THE JOURNAL OF THE INSTITUTE OF Metals

WITH THE BULLETIN
AND METALLURGICAL ABSTRACTS

Principal Contents:

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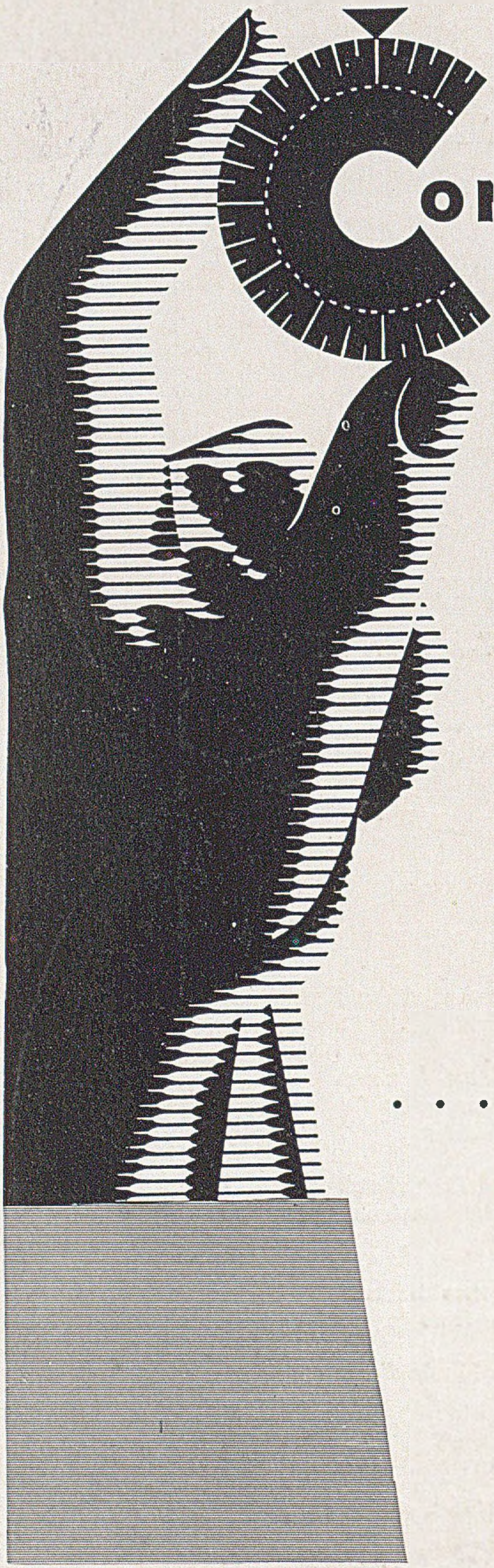
OCTOBER 1951



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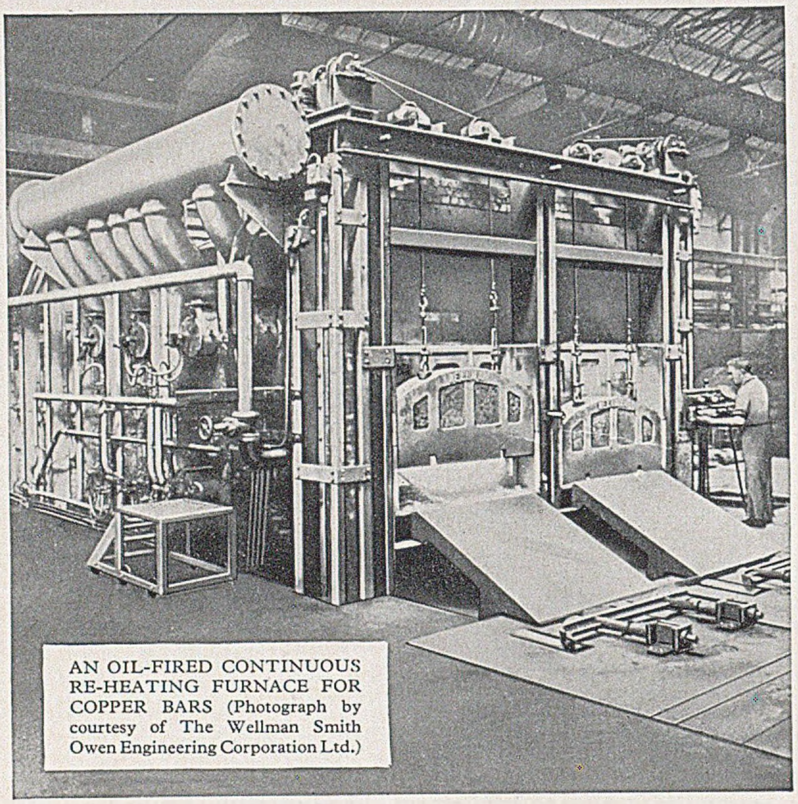

AN OIL-FIRED CONTINUOUS RE-HEATING FURNACE FOR COPPER BARS (Photograph by courtesy of The Wellman Smith Owen Engineering Corporation Ltd.)

. . . in the copper industry

Designed and constructed by The Wellman Smith Owen Engineering Corporation Limited for Enfield Rolling Mills Limited, this furnace has achieved an hourly output of 27 tons of copper bars heated from cold to 850° C. The air is preheated in a metallic recuperator to 300° C. Oil consumption over a period of several months' actual production time works out at about 5½ gallons per ton of output, which represents an efficiency of over 35 per cent.

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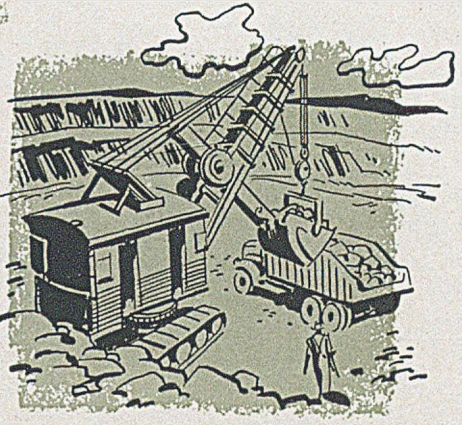
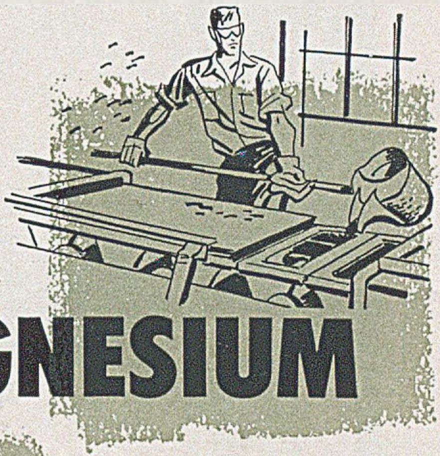
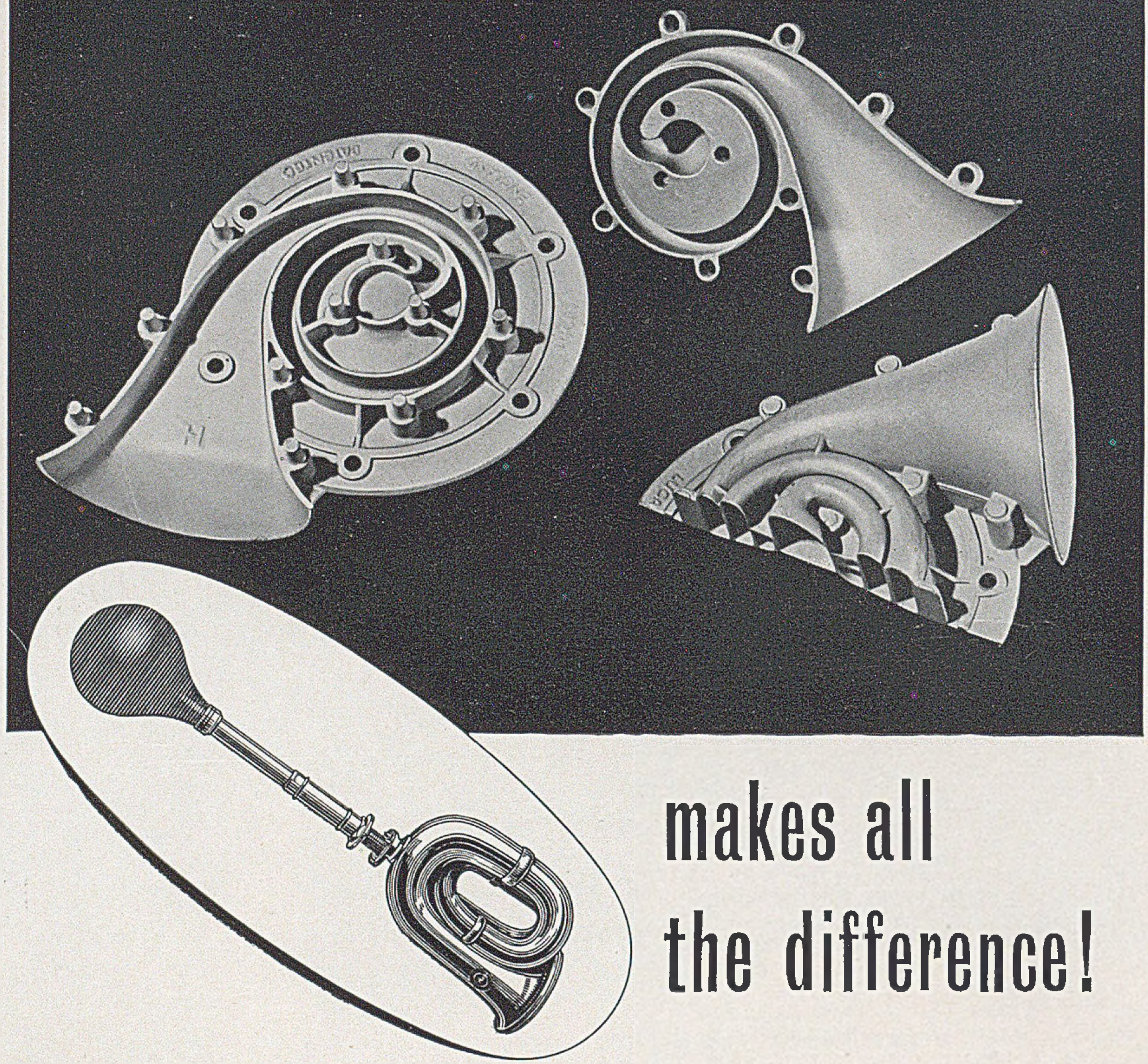




ZINC ALLOY DIE CASTING



makes all the difference!

The simple brass bulb horn was essential to early motorists, who gave it a proud position on their machines; and Africans valued them so much that they hoarded them instead of money! But ideas changed, and modern horns are so complex that they are only economical when they are zinc alloy die cast.

The windtone horn shown above is one of a pair that sound in harmony. Each is a good example of an intricate geometrical pattern which has been faithfully reproduced by die casting. The two parts of each horn are held tightly together by integral rivets on one engaging with cored holes on the other.

Some facts about zinc alloy die casting

Speed of production is an outstanding feature of the die casting process — the shortest distance between raw material and finished product. Zinc alloys are the most widely used of all metals for die casting because they yield castings with the following qualities:

ACCURACY: Castings can be made practically to finished dimensions and need little or no machining.

STRENGTH: Good mechanical properties for stressed components.

STABILITY: Close tolerances are maintained throughout the life of the casting.

British Standard 1004

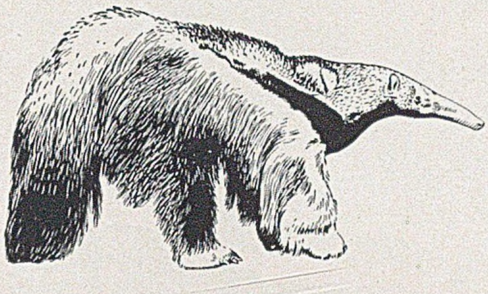
It is essential that alloys conforming to B.S.1004 should be specified for all applications.

The Association welcomes enquiries about the use of zinc alloy die castings. Publications and a list of Members are available on request.

ZADCA

ZINC ALLOY DIE CASTERS ASSOCIATION
LINCOLN HOUSE, TURL ST., OXFORD

Photograph reproduced by courtesy of Joseph Lucas Ltd.

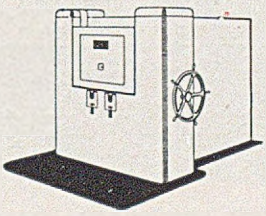


Deep in the forests of Central and South America roams the ant-eater — although he eats plenty of other insects besides. If ever an animal was perfectly designed to do the job, this is it. With his large bushy tail he shades himself when resting in the sun by curling it round him. With his strong front claws he tears away termites' nests, and with his long, thin head and sticky tongue he ferrets out the ants themselves for breakfast, lunch and tea. (They don't have dinner in the South American jungles.)

perfectly designed to do the job

The Wild-Barfield Heavy-Hairpin Furnace is designed to do the job too. With a temperature range of 700°C. to 1050°C. (or with "Tubular-Hairpin" elements up to 1150°C.) they are suitable for the majority of batch type heat-treatment processes. They are specially made for the job of hardening, carburising, normalising and annealing, nitriding, vitreous enamelling and decorating and glazing.

Wild-Barfield
Electric Furnaces



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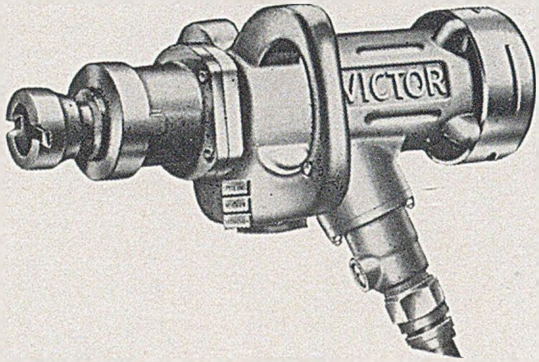
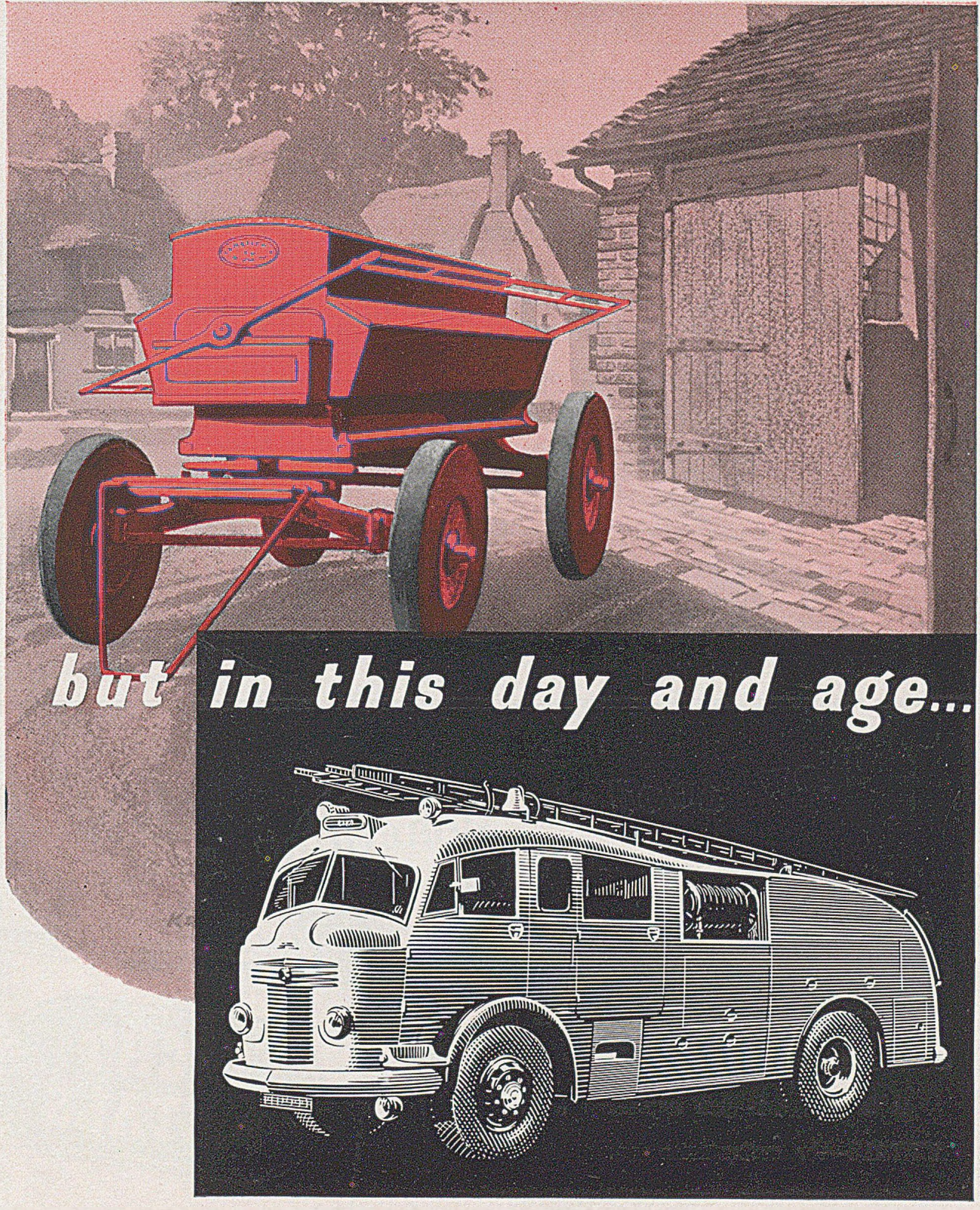
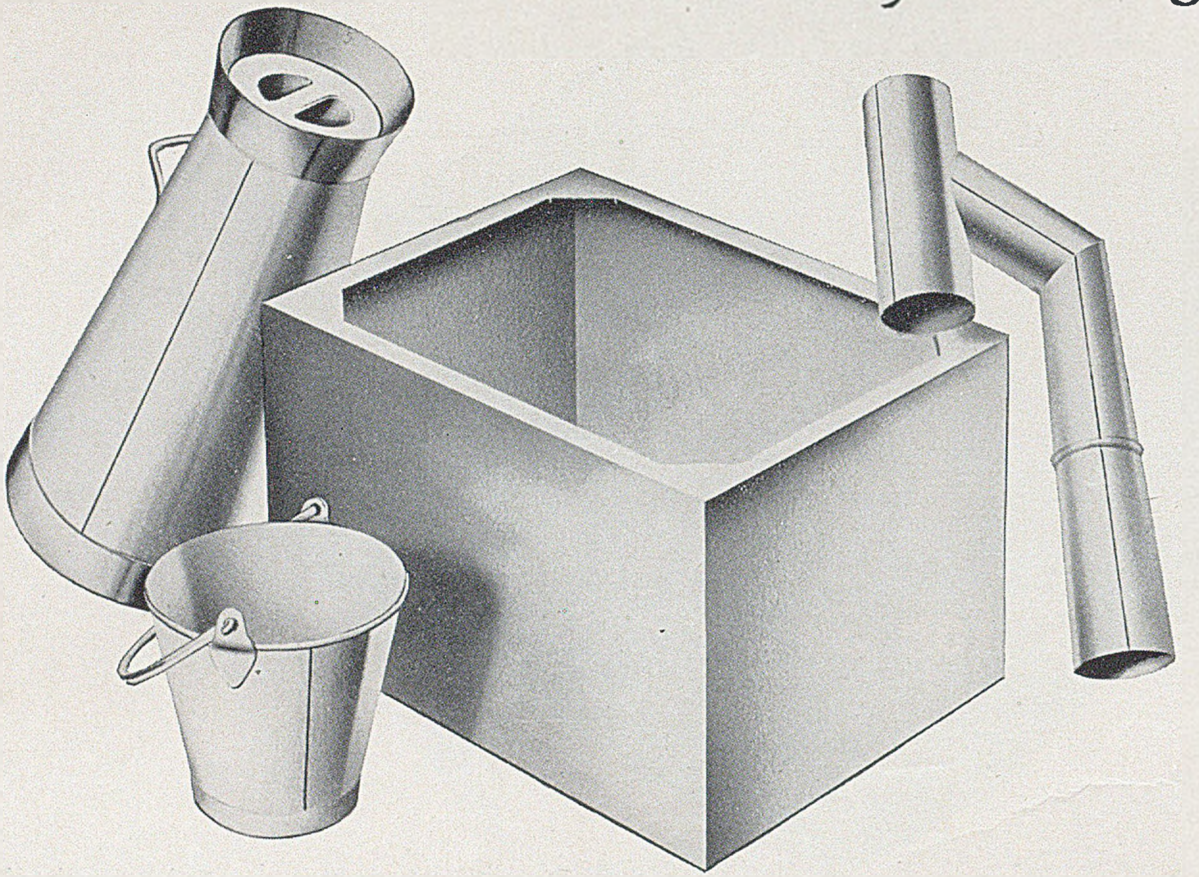

VICTOR

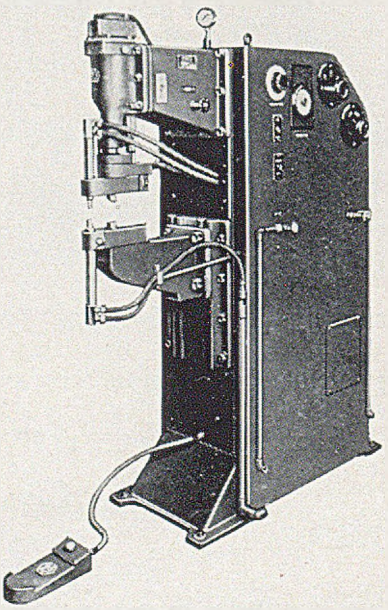

but in this day and age...

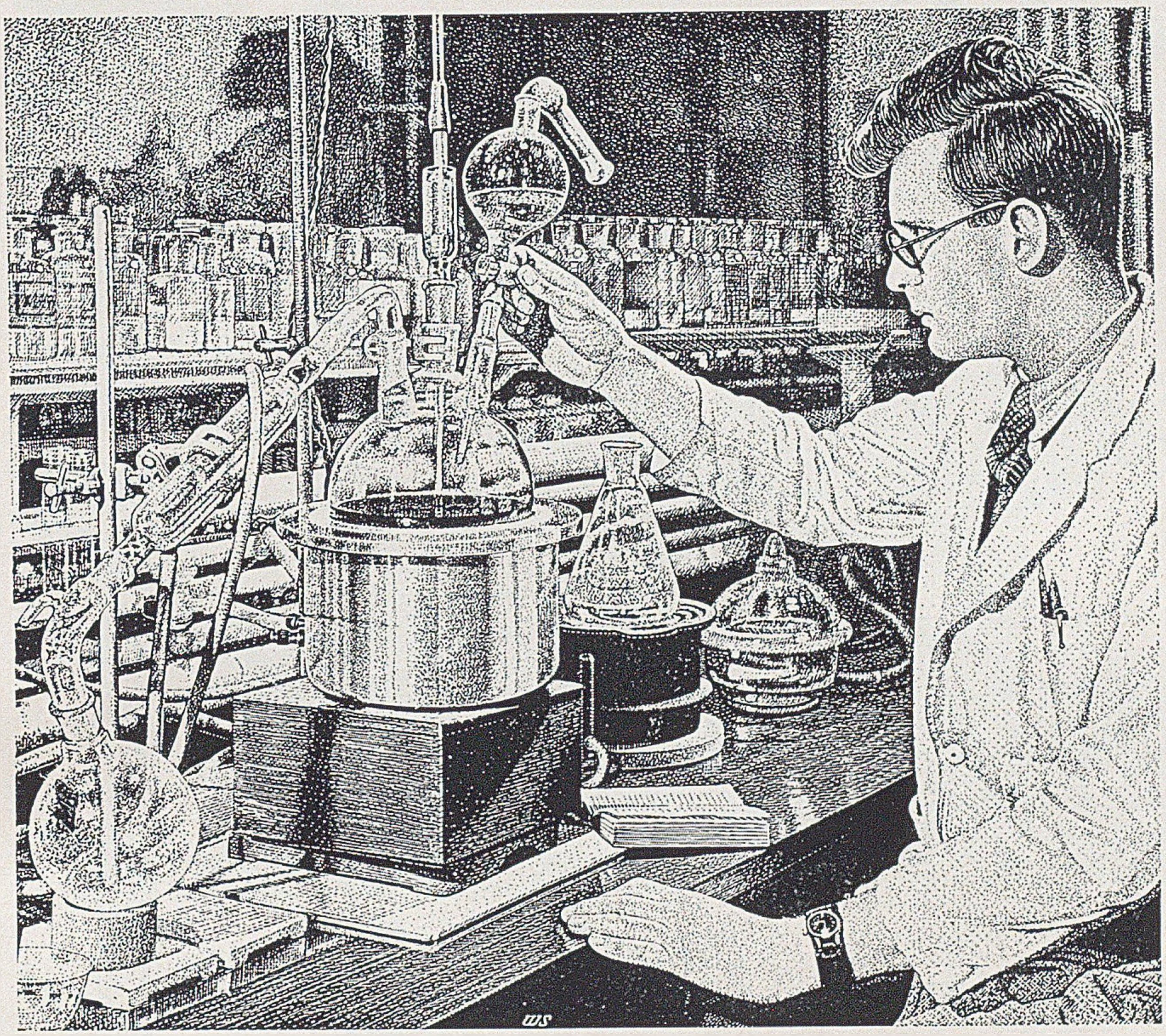

LIGHT
METALS


'C' for STANDARD



Metrovick Standard Type
" AS 60/24 " Spot
Welding Equipment

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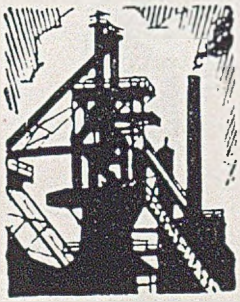

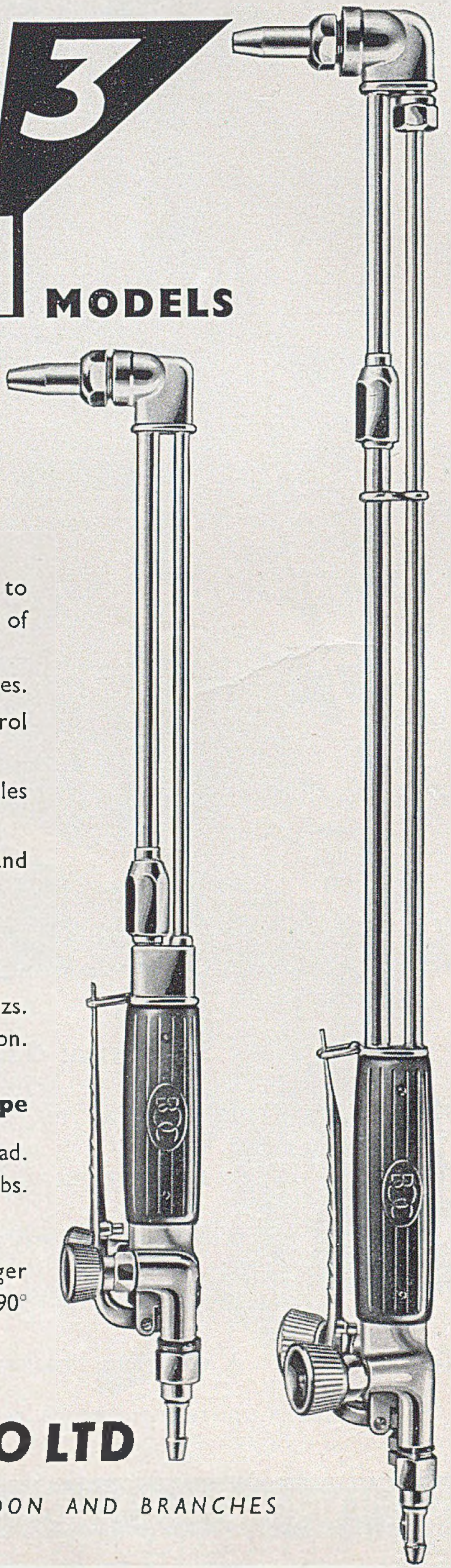

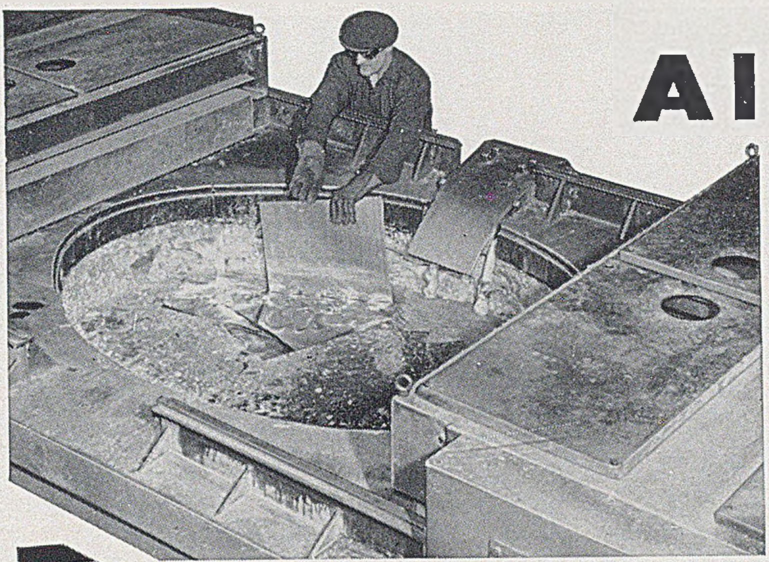

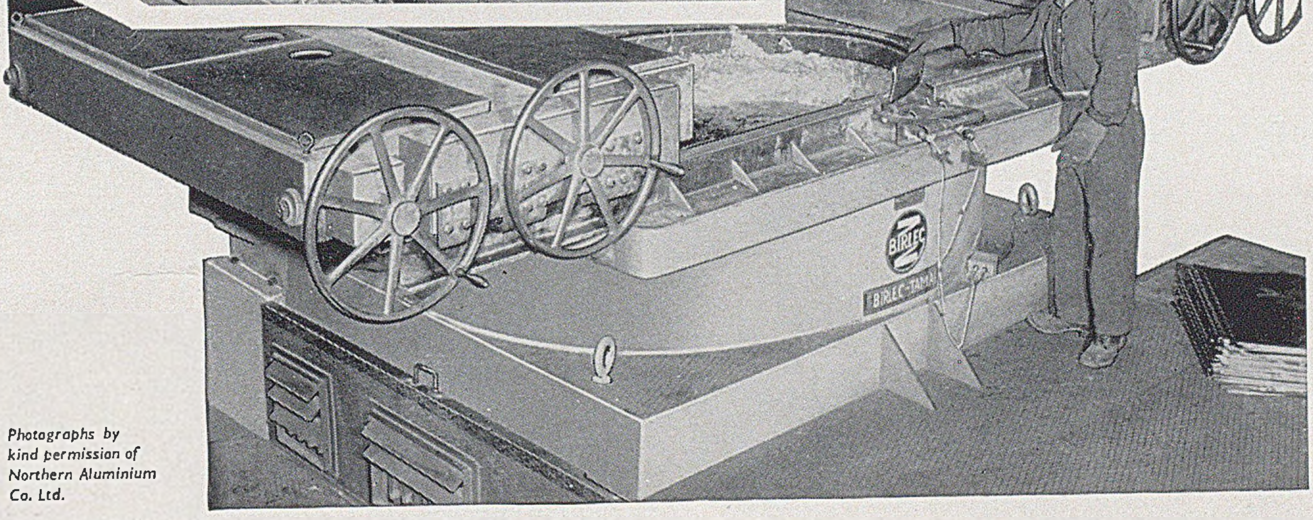

Photo Walter Nurnberg F.I.B.P., F.R.P.S.

Physico-chemical research

The production, control and service performance of the products of The British Aluminium Co. Ltd., require the extensive application of chemical and particularly physico-chemical science. This is true not only of the chemical products of the Company but also of the purely metallic products and specialities such as aluminium paste. Thus the physical and chemical characteristics of the alumina from which the virgin aluminium is made must be carefully controlled. These characteristics and the methods of controlling them at both the alumina and reduction factories are particular responsibilities of the Research Laboratories.

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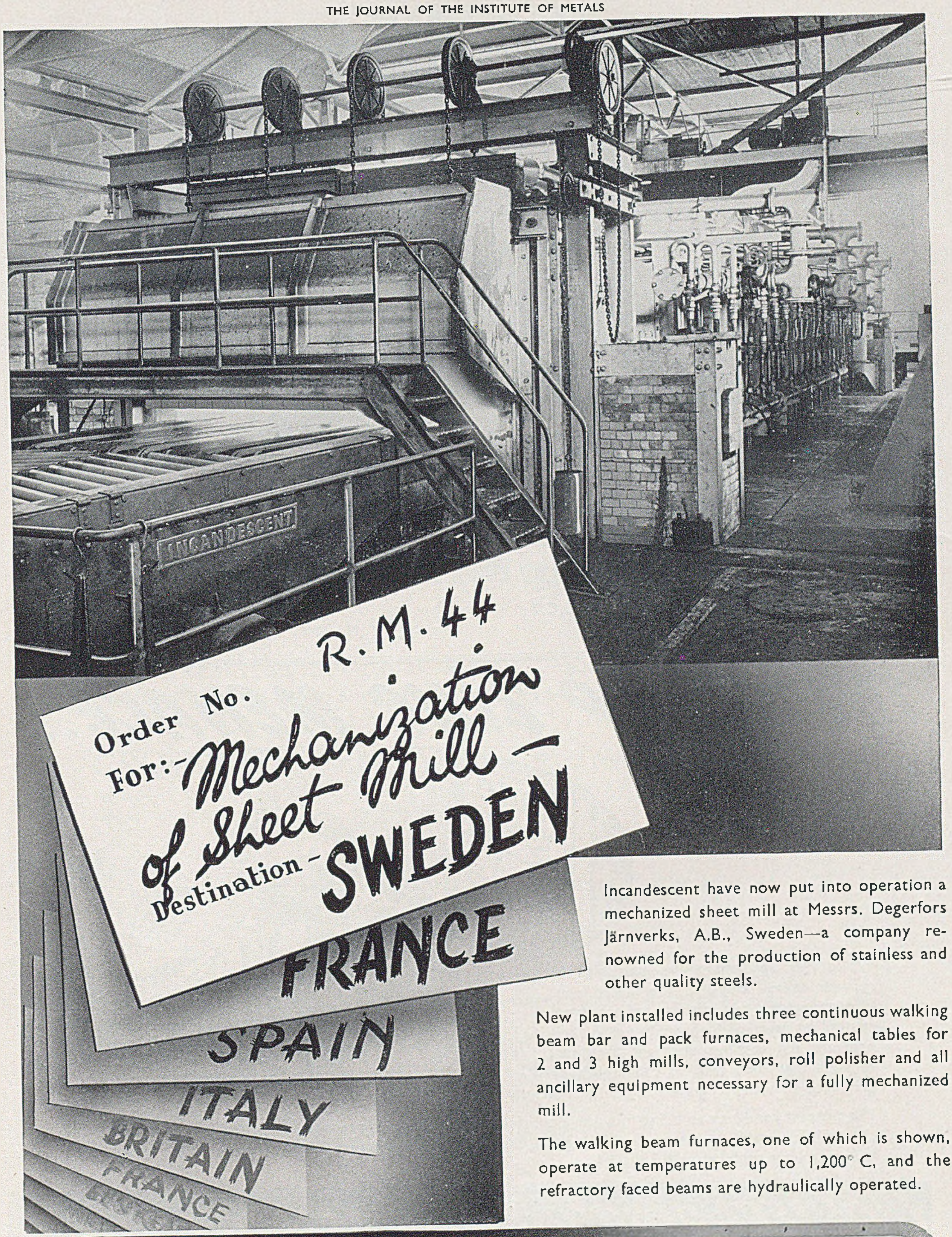
The B.S.I. Golden Jubilee—British Aluminium products are supplied to British Standards.



Incandescent have now put into operation a mechanized sheet mill at Messrs. Degerfors Järnverks, A.B., Sweden—a company renowned for the production of stainless and other quality steels.

New plant installed includes three continuous walking beam bar and pack furnaces, mechanical tables for 2 and 3 high mills, conveyors, roll polisher and all ancillary equipment necessary for a fully mechanized mill.

The walking beam furnaces, one of which is shown, operate at temperatures up to 1,200° C, and the refractory faced beams are hydraulically operated.

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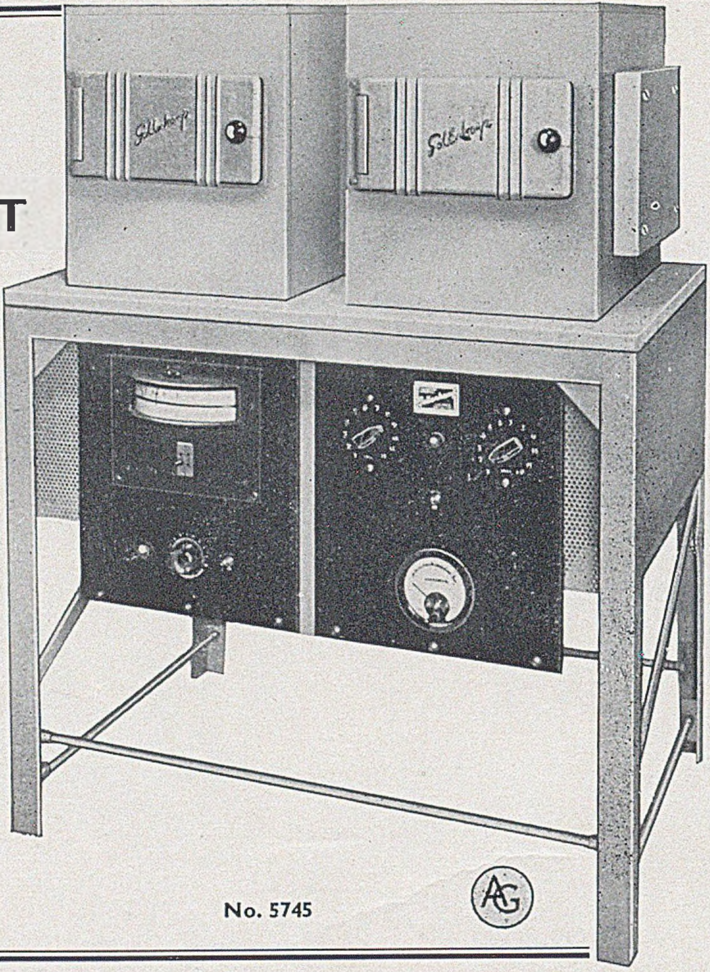
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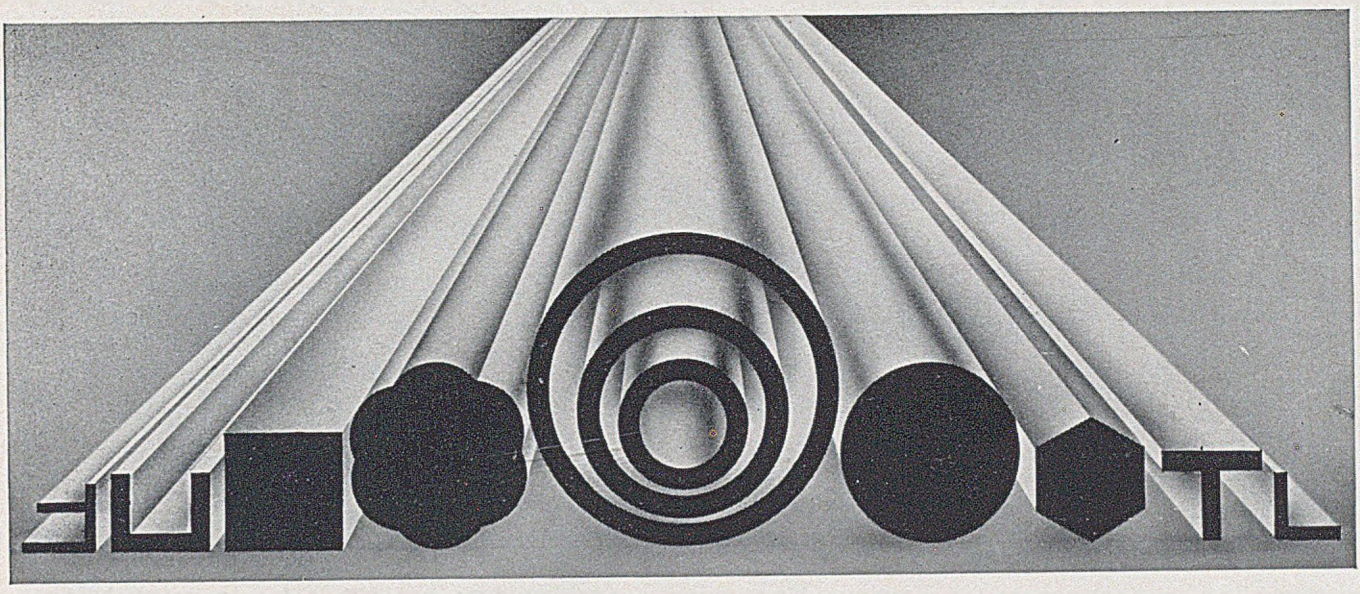
IRON
BRASS
B&T

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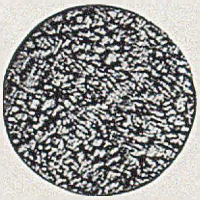

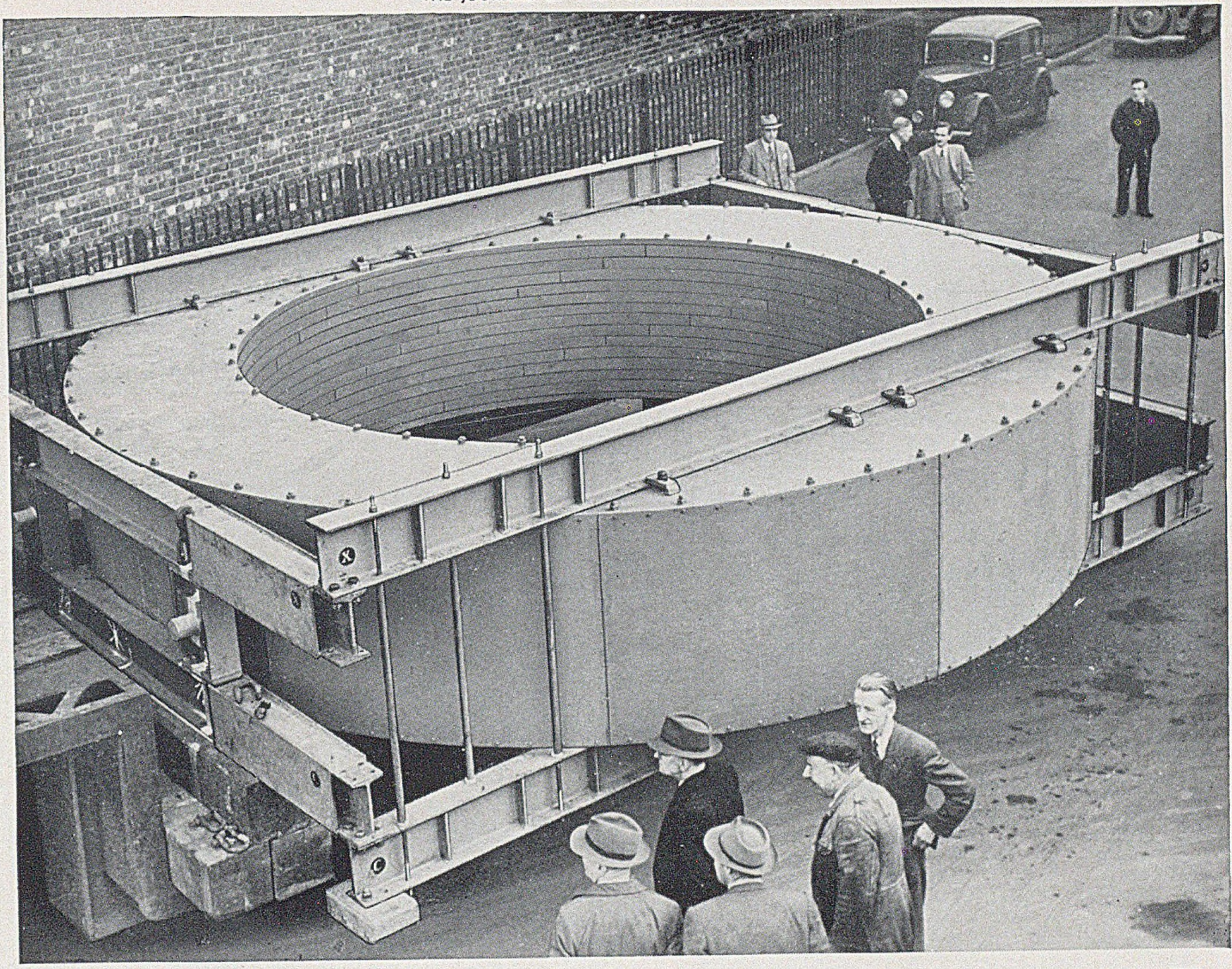

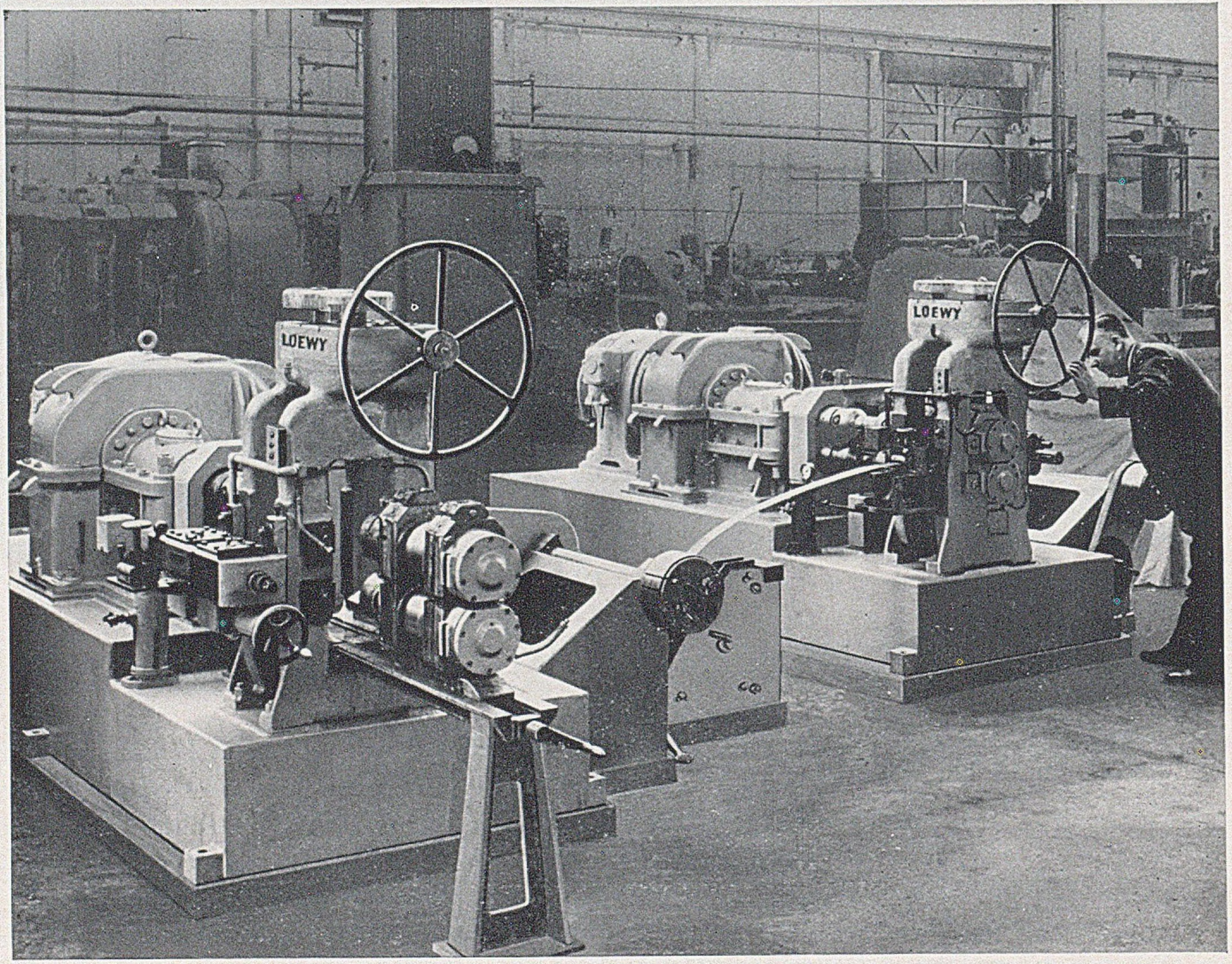
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EDITORIAL AND ADVERTISEMENT OFFICES
4 Grosvenor Gardens, London, S.W.1
Telephone and Telegrams: SLOane 5928 & 6233

Editor of Publications
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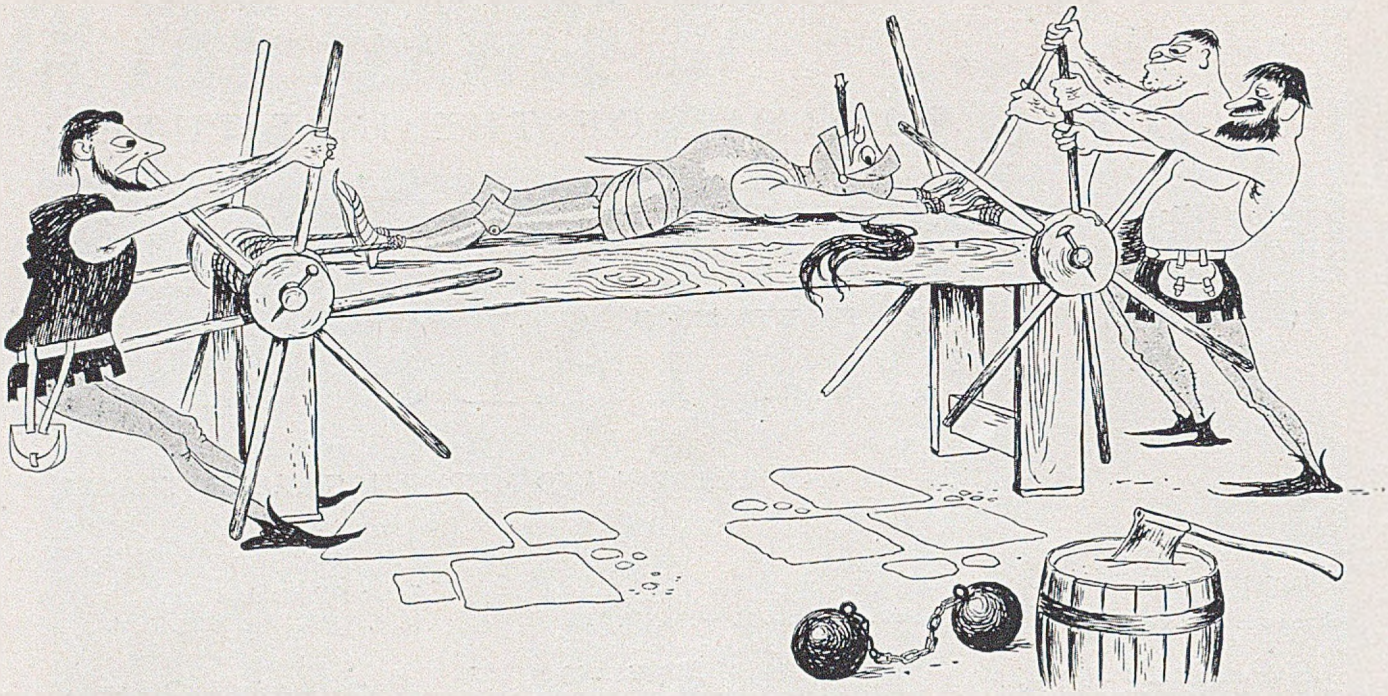

BULLETIN

OF THE INSTITUTE OF METALS

VOLUME I OCTOBER 1951 PART 2

GENERAL MEETINGS OF THE INSTITUTE

Abridged Report of a Special Committee Appointed by The Council

Under the Chairmanship of Dr. C. J. Smithells, Senior Vice-President, a special committee appointed by the Council has recently held a series of meetings to consider, in the light of changes of circumstances since the formation of the Institute, and having in mind the financial aspects of these matters, all questions relating to the organization of General Meetings. The committee of eight members was representative of the different interests within the Institute, including production metallurgists and metallurgical engineers, research workers, university and technical college staffs, specialists, and Junior and Student Members.

It is thought that Members may be interested to have some details of the committee's report and of its recommendations which have been accepted by the Council. They are therefore given below in much abbreviated form.

Needs of Members as Regards Meetings

The needs of the various classes of members as regards General Meetings of the Institute are:

(a) *Production Metallurgists and Metallurgical Engineers.*—(i) Symposia on production practice; (ii) review-type papers (at present provided to some extent by papers contributed to symposia on industrial subjects); (iii) more papers on plant and plant developments (which papers should be encouraged or—if not otherwise received in sufficient numbers to meet the needs of this class of members—invited); (iv) visits to works, &c.; and (v) social functions, to facilitate personal contacts.

(b) *Research Workers in General.*—(i) Papers describing fundamental work over wide fields of interest; (ii) visits to works and laboratories; and (iii) social functions, to facilitate personal contacts.

(c) *Specialists.*—(i) Facilities to discuss topics of common interest, even though the total numbers present may be very small (such discussions could take place concurrently with other meetings at which the subjects under discussion were sufficiently diverse); (ii) visits to works and laboratories; and (iii) social functions, to facilitate personal contacts.

(d) *Junior and Student Members.*—Undergraduates and post-graduate students in their first year really have no need for meetings, other than those of the University Metallurgical Societies. The Institute meets their needs with the Library, the *Journal*, *Metallurgical Abstracts*, and the Monograph Series. After obtaining their first appointments, however, and for post-graduate students after their first year, meetings could provide Junior and Student Members with useful, and even very valuable services. Though they may not be ripe enough in experience to present papers, Junior and Student Members could (and should be encouraged to) take part in discussions, and Local Sections can usefully cater for their needs by having one "Students' Night" in each session. As they gain experience, Junior and Student Members are in as good a position as others to present papers and take part in discussions. Students' Tours, and visits to works and laboratories (arranged by the parent body or by Local Sections) would be valuable services.

Committee's Recommendations

Place of Meeting.—As the amenities, and general atmosphere, of accommodation at an hotel are much to be preferred to those in the buildings of even the largest scientific societies, and the difference in cost is very small, General Meetings should be held in hotels.

Concurrent Sessions.—To ensure that as many as possible of the papers published in the *Journal* are discussed, concurrent sessions may often be desirable at General Meetings; there is nothing against them, provided that the subjects discussed are sufficiently diverse.

General Meetings.—Two main meetings should be held each year, viz. the Annual General Meeting and the Autumn Meeting. In addition, when invited to do so, the Institute should arrange one-day or half-day meetings in Local Section areas for the presentation and discussion of papers previously published in the *Journal*. These meetings (which would be meetings of the parent body), and any social functions in connection with them, should be organized by the Secretary and his staff.

May Lecture.—The present isolation of the May Lecture makes attendance difficult, on grounds of expense. It should be delivered, with title unchanged, in connection with the Annual General Meeting.

Annual General Meetings should be held in London, in March, on the following lines:

Day 1. May Lecture, in the evening.
Day 2. *Morning*: Business meeting; Presidential Address; Luncheon (at which medals would be presented). *Afternoon*: Discussion of scientific papers. *Evening*: Dinner-Dance at an hotel.
Day 3. All-Day Symposium on a subject of interest to production metallurgists and metallurgical engineers. *Evening*: Conversazione, with music and possibly an exhibition.
Day 4. *Morning*: Discussion of scientific papers. *Afternoon*: Visits to works and laboratories.

Autumn Meetings should be held in September and, when practicable, after the beginning of the children's school term and before the beginning of the University term. The time at which these meetings can be held may, however, depend on the locality (e.g. when college or hostel accommodation can be made available) or the wishes of the Institute's hosts.

Of every three Autumn Meetings, one should be held abroad; one should be a normal General Meeting in the provinces; and one should be a meeting in London, with extensions to the provinces, in connection with which an invitation would be extended to a foreign metallurgical society to hold its meeting side by side with that of the Institute.

When an Autumn Meeting is held abroad, a General Meeting should also be held in London, about 3 or 4 weeks afterwards, for the discussion of scientific papers.

Autumn Meetings in Great Britain should normally last from Monday to Friday, inclusive; abroad, the duration of meetings will depend on varying circumstances. An Autumn Lecture should be invited, but no symposia should be arranged at Autumn Meetings, at which the social side and works visits should be emphasized. Visits to works, laboratories, and places of cultural interest should be arranged for members and ladies. A dinner-dance should be held, at which the Institute can return any local hospitality.

Symposia.—Papers contributed to symposia should be distributed 3 to 4 weeks before the meeting, to stimulate discussion. A rapporteur should summarize the papers, which should not be separately presented by the authors. Successful General Discussions can, however, be held without the preprinting of papers.

Visits to Works and Laboratories.—All classes of members need to have visits to works and laboratories arranged for them, and they should be organized in connection with Annual General Meetings as well as Autumn Meetings. Our hosts should be advised that activities which may not be considered to be of interest to experienced members may, in fact, be of great interest to Junior and Student Members (e.g. the planning and progress of work through the factory).

Social Functions.—A dinner-dance (evening dress), at an hotel, should be arranged in connection with each Annual General Meeting; there is a strong demand for such a function among certain classes of members. Allowing for overheads, the present cost of such functions will be 25*s.* to 35*s.* per head. A dinner-dance is more attractive to members than a dinner. The difference in cost between a buffet dance at an hotel and a dinner-dance does not make the former type of function worth while.

Lunches.—Buffet lunches are not popular. Luncheons should not be organized in connection with Autumn Meetings, but one should be arranged in connection with each Annual General Meeting, at which medals will be presented. The cost, including overheads, will be about 18*s.* 6*d.* per head at present prices. The advantages of presenting medals at a luncheon greatly outweigh the disadvantages.

The Secretary should provide members with a simple guide (with prices) to restaurants and inns at which members may obtain lunch near to the place of meeting.

Conversazione.—A conversazione is the only type of function that can be arranged to meet the needs of those with slender means. In connection with each Annual General Meeting there should be a conversazione, with music and possibly an exhibition. Members would be required to bear only the cost (about 3*s.* 6*d.* per head, at present prices) of the very simple light refreshments provided.

Cost of Social Functions: General.—The cost of entertaining official guests should be borne by the Institute and not by the members taking part in the function to which they are invited. The price charged for tickets for social functions (as, also, for visits to works and laboratories) should otherwise cover all expenditure by the Institute on such functions, including overheads.

After having examined information obtained from a wide range of hotels, restaurants, and caterers in London regarding the accommodation that can be made available and the charges they would make for various types of function, the Committee is satisfied that, though some restaurant and hotel proprietors can—for obvious reasons—make low or modest charges for lunches and dinners served daily in their rooms open to the public (where several meals in succession may be served at the same table by their permanent staff of waiters), the overheads associated with the provision of the necessary accommodation for exclusive use and the costs of engaging temporary staff, make it impossible in present times to arrange dinner-dances, dinners, and luncheons for large numbers at small cost.

INSTITUTE NEWS

General Meeting, London, 17 and 18 October 1951

Tuesday, 17 October

GENERAL DISCUSSION ON "METAL ECONOMICS"

As previously announced, a General Discussion on "Metal Economics" will be held at the Park Lane Hotel, Piccadilly, London, W.1, on Tuesday, 17 October 1951. The President, Professor A. J. MURPHY, M.Sc., will occupy the Chair.

The meeting will be divided into two half-day sessions. At each session several short papers will be presented, after which there will be a General Discussion on the subject of the Session. A document giving synopses of the papers to be presented at the meeting has been dispatched to all Members resident in the British Isles. Other Members who can attend are requested to apply for copies. Visitors will be welcome, and in view of the importance of the subject, it is hoped that there will be a large attendance. The programme is as follows:

SESSION I.—PRIMARY RESOURCES OF FERROUS AND NON-FERROUS METALS (10 a.m.–12.45 p.m.)

(i) The PRESIDENT: Opening Address.

(ii) Mr. R. LEWIS STUBBS (*British Non-Ferrous Smelters' Association*): "The World Supply of Non-Ferrous Metals, Including the Light Metals".

(iii) Professor S. Zuckerman, C.B., F.R.S. (Birmingham University): "Metals as Natural Resources".

(iv) Dr. T. P. Colclough, C.B.E. (*British Iron and Steel Federation*): "Iron Ore: World Demand and Resources".

(v) General Discussion on Primary Resources.

Session II.—Scrap Reclamation, Secondary Metals, and Substitute Metals (2.30–5 p.m.).

(i) Mr. C. A. Bristow, Mr. A. J. Sidery, and Dr. H. Sutton (*Ministry of Supply*): "The Scope for Conservation of Metals, Ferrous and Non-Ferrous".

(ii) Mr. C. Dinsdale (*British Railways*): "Economy by Standardization of Alloys and of the Method of Reclamation of Scrap Metals".

(iii) Mr. F. Hudson (*The Mond Nickel Co., Ltd.*): "The Influence of Specifications on Productivity and the Economic Utilization of Ferrous and Non-Ferrous Metals".

(iv) Mr. E. H. Jones (*Capper Pass and Son, Ltd.*): "Secondary Heavy Metals".

(v) Colonel W. C. Devereux, C.B.E. (*Almin, Ltd.*): "Secondary Aluminium and Magnesium".

(vi) General Discussion on Scrap Reclamation, Secondary Metals, and Substitute Metals.

CONVERSAZIONE

In the evening (8–11 p.m.) there will be an informal (lounge suits) conversazione at the Institute's headquarters, 4 Grosvenor Gardens, London, S.W.1, at which there will be a small exhibition of apparatus, equipment, specimens, and recent books likely to be of interest to members.

A charge of 3*s.* 6*d.* will be made to cover the cost of simple light refreshments, and alcoholic drinks will be obtainable. Members wishing to attend the conversazione are requested to send the necessary remittance to the Secretary, from whom tickets are obtainable both for members and their guests.

Wednesday, 18 *October*

VISITS TO WORKS

By kind invitation of the Directors, visits to the following works (including lunch) have been arranged for members attending the meeting. A charge will be made to cover transport and administrative expenses, and tickets must be obtained from the Secretary in advance.

Members wishing to take part in one of these visits (the total number in each case is limited) are requested to advise the Secretary (on the form already circulated), stating their order of preference, and sending the appropriate remittance.

Transport will leave 4 Grosvenor Gardens, London, S.W.1, at the times stated.

Time of Departure		*Works*
9.30 a.m.	(1)	Enfield Rolling Mills, Ltd., and Enfield Cables, Ltd., Brimsdown, Middlesex. (Ticket price 5*s.*)
9.30 a.m.	(2)	High Duty Alloys, Ltd., Slough. (Ticket price 5*s.*)
9.30 a.m.	(3)	Telegraph Construction and Maintenance Co., Ltd., Greenwich. (Ticket price 5*s.*)

Annual General Meeting, London, 25, 26, and 27 March 1952.

The next Annual General Meeting of the Institute will be held in London on Tuesday, Wednesday, and Thursday, 25, 26, and 27 March 1952.

The provisional programme is as follows:

Monday, 24 *March*

May Lecture.

Tuesday, 25 *March*

Location: Park Lane Hotel, Piccadilly, London, W.1.

10.0 a.m.—General business of the Institute.
Report of Council.
Report of the Honorary Treasurer.
Induction of the new President.
Presidential Address, by Dr. C. J. Smithells, M.C.
12 noon.—Adjournment; drinks will be served in an anteroom.
1.0 p.m.—Luncheon at the Park Lane Hotel, followed by Presentation of the Institute of Metals (Platinum) Medal, the W. H. A. Robertson Medal, and the Rosenhain Medal.
2.45 p.m.—Scientific session; discussion of papers.
5.0 p.m.—Adjournment.
Evg.—Dinner-Dance at the Park Lane Hotel.

Wednesday, 26 *March*

Location: Park Lane Hotel, Piccadilly, London, W.1.

9.30 a.m.–12.45 p.m.; 2.30–5.0 p.m.—Symposium on "Equipment for the Thermal Treatment of Non-Ferrous Metals and Alloys" (invited papers will previously have been published in the *Journal*).
Evg.—Conversazione and exhibition at 4 Grosvenor Gardens, S.W.1.

Thursday, 27 *March*

Location: Park Lane Hotel, Piccadilly, London, W.1.

10.0 a.m.–12.30 p.m.—Scientific session; discussion of papers.
Aft.—Visits to works and laboratories.

Symbols and Abbreviations in "Metallurgical Abstracts"

The Council has decided, in the interests of economy, on an extension of the use of symbols and abbreviations in *Metallurgical Abstracts*. Beginning last month, the chemical symbols will replace the names of the elements throughout the abstracts, and chemical formulæ will be used instead of the names of common inorganic compounds. In addition, a number of words that appear repeatedly in the abstracts will in future be abbreviated. A full list of symbols and abbreviations will be found facing the first page of *Metallurgical Abstracts*.

Journal Binding Costs

Mr. W. A. Newark, of 2 Clerkenwell Green, London, E.C.1., to whom many Members send their *Journals* for binding, announces that, owing to increases in costs, the charge for binding each volume in the old 8vo size will in future be 6*s.* 3*d.*, inclusive of postage to Members' addresses.

The price per volume in the new 4to size will be 10*s.* 3*d.*, inclusive of postage.

Binding cases for the volumes for 1945, 1946, 1947, 1948, and 1948–49 are still available (*free*) to Members on request to the Secretary.

Election of Members

The following 21 Ordinary Members, 4 Junior Members, and 3 Student Members were elected on 9 August 1951:

As Ordinary Members

Améen, Einar Louis, Managing Director, Surahammars Bruks Aktiebolag, Surahammar, Sweden.

Andresen, Erling, Stavanger Electro-Staalverk A/S, Jörpeland, Norway.

Broadbent, Brian Lynn, Thomas Broadbent and Sons, Ltd., Central Ironworks, Huddersfield, Yorks.

Chandler, Henry, Vice-President, Vanadium Corporation of America, 420 Lexington Avenue, New York 17, N.Y., U.S.A.

Clayton, William Wikeley Ward, M.I.Mech.E., Chairman and Managing Director, Hudswell, Clarke and Company, Ltd., Railway Foundry, Jack Lane, Leeds 10.

Corfield, Reginald Holbeche, B.A., Managing Director, Corfield-Sigg, Ltd., and Corfield and Buckel, Ltd., Trafalgar Works, Station Road, Merton Abbey, London, S.W.19.

Davies, Ivor Bowen, In Charge of Light Alloy Test House, Richard Thomas and Baldwins, Ltd., Byass Works, Port Talbot, Glam.

Dewhirst, Eric Victor, A.I.M., Chief Metallurgist, Specialloid, Ltd., Black Bull Street, Leeds 10.

Duran Rigol, Enrique, Ingeniero Industrial, Productos AGMA, S.A., Refineria de Aluminio, Ronda San Pedro, 32, 1, A, Barcelona, Spain.

Gifkins, Robert Cecil, B.Sc., A.I.M., Research Officer, Commonwealth Scientific and Industrial Research Organization, Baillieu Laboratory, University of Melbourne, Carlton N.3, Vic., Australia.

Hancock, Peter Francis, B.A., F.I.M., Chief Metallurgist, Birlec, Ltd., Tyburn Road, Erdington, Birmingham 24.

Hogan, Leonard McNamara, B.Sc., Student, Metallurgy Department, University of Melbourne, Melbourne, Vic., Australia.

Kirkup, William Brookes, T.D., B.A., G.I.Mech.E., Brass Rolling and Extrusion Mill Engineer, Vickers-Armstrongs, Ltd., Elswick, Newcastle-on-Tyne 4.

Leroy, André Georges Paul, Directeur, Institut de Soudure, 32 Boulevard de la Chapelle, Paris 18e, France.

Malcor, Henri, 12 rue de la Rochfoucauld, Paris 9e, France.

Matuschka, Bernhard, Dr.Mont.Ing., Consulting Engineer, Vereinigte Österreichische Eisen und Stahlwerke, Linz, Austria.

Muir, Neil Baird, B.Sc., A.I.M., Technical Officer (Metallurgical), Research Department, Imperial Chemical Industries, Ltd., Metals Division, Kynoch Works, Witton, Birmingham 6.

Plantema, Frederik J., Ing., Acting Chief, Materials Section, Nationaal Luchtvaartlaboratorium, Sloterweg 145, Amsterdam-W., Holland.

Ridge, Charles William, O.B.E., Assoc.Met., F.I.M., General Manager, Scaw Metals, Ltd., P.O. Box 6334, Johannesburg, Transvaal, South Africa.

Staples, Ronald Thomas, Production Metallurgist, T.I. Aluminium, Ltd., Redfern Road, Tyseley, Birmingham 11.

Vargas, Fernando Gonzalez, Professor of Metallurgy at the Universidad Nacional Autonoma de México, and at the Instituto Politecnico Nacional, Mexico City; mail: 2a Priv. C. Beistegui 14 Col. Valle, Mexico 12, D.F.

As Junior Members

Auld, John Hugh, B.Met.E., Metallurgist, Metallurgy Department, Royal Aircraft Establishment, Farnborough, Hants.

McKinlay, D. F. A., Electrometallurgist, Standard Telephones and Cables, Ltd., New Southgate, London, N.11.

Pitts, Gordon Roy, B.Sc., G.I.Mech.E., Metallurgist, Bristol Aeroplane Co., Ltd., Aircraft Division, Filton, Bristol.

Vaughan, Thomas Bernard, Metallurgist, Walter Somers and Co., Ltd., Halesowen, Birmingham.

As Student Members

Evans, Ronald Ernest, Trainee, Research Department, Imperial Chemical Industries, Ltd., Metals Division, Witton, Birmingham 6.

Hines, John Grahame, B.A., Research Student, Department of Metallurgy, The University, Pembroke Street, Cambridge.

Taylor, Ian, Laboratory Assistant, Fulmer Research Institute, Ltd., Stoke Poges, Bucks.

The following 24 Ordinary Members, 4 Junior Members, and 4 Student Members were elected on 4 September 1951:

As Ordinary Members

Ardern, Lawrence L., Librarian, College of Technology, Manchester 1.

Binder, William Oakley, B.S., Research Metallurgist, Union Carbide and Carbon Research Laboratory, Inc., P.O. Box 580, Niagara Falls, N.Y., U.S.A.

Colmant, Raymond, Works Director, Sollac, Seremange, (Moselle), France.

Crooks, Laurence Edward, Chief Control Officer, Control Commission for Germany, Metallurgical Industries Department, Land Commissioner's Office, Düsseldorf, B.A.O.R., 4.

Dawihl, Walther, Dr.Ing.habil., Société Franco-Sarroise de Métaux Durs, Ursulinenstrasse 43, Sarrebruch/Sarre.

Devereux, Wallace Deane, Structural and Mechanical Development Engineers, Ltd., Slough, Bucks.

Elstub, St. John, B.Sc., Production Director (Wrought Metals), Imperial Chemical Industries, Ltd., Metals Division, Witton, Birmingham 6.

Ferrall, Lorin L., Director of Metallurgy, Crucible Steel Company of America, P.O. Box 88, Pittsburgh 30, Pa., U.S.A.

Fletcher, Frank, B.Sc., Research Engineer, Hopkinson's, Ltd., Huddersfield.

Goicoechea, Manuel, Director and General Manager, Hierro Maleable de Mexico, Viena no. 16-Department 12, Mexico City, Mexico.

Graham, Robert, Ph.D., B.Sc., A.R.T.C., Metallurgist, Shell Refining and Marketing Co., Ltd., Thornton Research Centre, P.O. Box 1, Chester.

Houghton, Frank, Director, Pickford, Holland and Co., Ltd., Sheffield.

Jolivet, Henri, Service Général des Recherches, Société d'Electro-Chimie, 10 rue du Général Foy, Paris 8e, France.

Martinez, Giorgio, M. A., Director, Barron and Crowther, Ltd., Eastleigh, Hants.

Nourse, Louis M., A.B., M.S., Librarian, St. Louis Public Library, St. Louis 3, Mo., U.S.A.

Owe, Aage W., Managing Director, A/S Ardal og Sunndal Verk, Nedre Vollgate 4, P.O. Box 180, Oslo, Norway.

Price, Bartlett R., Westinghouse Electric Corporation, East Pittsburgh, Pa., U.S.A.

Sandström, Karl Eric Viktor, Chairman, S. and M. Engineering Pty., Ltd., 88 Fox Street, Johannesburg, Transvaal, South Africa.

Sherwood, Charles Noel, B.Comm., Public Relations Officer, Metals Division, Imperial Chemical Industries Ltd., Witton, Birmingham 6.

Tagliaferri, Leone, *Ing*., General Manager, Leone Tagliaferri & C.S.p.A., via Principe Eugenio 11, Milan, Italy.

Tenland, Waldemar, *Eng*., Hedåsvägen, 47d, Sandviken, Sweden.

Vanick, James Sebold, B.S., International Nickel Company, Inc., 67 Wall Street, New York 5, N.Y., U.S.A.

White, Charles M., President, Republic Steel Corporation, Room 1707, Republic Building, Cleveland 1, O., U.S.A.

Willners, Sven Harry, A.B. Ferrolegeringar, P.O. Box 16150, Stockholm 16, Sweden.

As Junior Members

Brown, Derek James, B.Sc., Metallurgist, Magnesium Elektron, Ltd., Clifton Junction, near Manchester.

Devereux, Robert Wayne, Assistant to Works Superintendent, Southern Forge, Ltd., Langley, Bucks.

Hagel, William P., B.Met.Eng., Junior Metallurgist, Oak Ridge National Laboratory, Oak Ridge, Tenn., U.S.A.

Harris, Ian Robert, Research Metallurgist, The British Aluminium Company, Ltd., Chalfont Park, Gerrards Cross, Bucks.

As Student Members

Cina, Bernard, Student of Metallurgy, Sheffield University.

Holder, Sydney George, Jr., M.Sc., Student of Metallurgy, University of Tennessee, Knoxville, Tenn., U.S.A.

Liu, T. S., M.S., Graduate Student, University of Notre Dame, Ind., U.S.A.

Thomas, W. Roy, M.A.Sc., Graduate Student, University of Toronto, Canada.

PERSONAL NOTES

Dr. F. Adcock is now Chief Research Officer to the Broken Hill Proprietary Co., Ltd., Australia.

Mr. F. E. Asbury has been awarded the B.Met. degree of Sheffield University.

Mr. U. G. Bhat has received a Master's degree in Metallurgical Engineering Practice at Lehigh University, Bethlehem, Pa., and is now working for his Ph.D.

Mr. S. Binner has left Stewarts and Lloyds, Ltd., Corby, and taken up a position with the Ministry of Supply, Atomic Energy Production, Sellafield, Cumberland.

Mr. A. J. Bourne, who has been connected with the Company since 1908 and for the past 27 years has been Works' Manager, has been elected a Director of Earle, Bourne and Co., Ltd., of Birmingham.

Mr. R. R. Brooks, Metallurgical Superintendent, Commonwealth Aircraft Corporation Pty., Ltd., Melbourne, is on a business trip to England.

Mr. J. M. Capus has been awarded the degree of B.Sc. (2nd Class Honours) in Industrial Metallurgy at Birmingham University.

Miss S. Constantine is now Chief Chemist to M. L. Alkans, Ltd., Greenford, Middlesex.

Dr. Maurice Cook has been appointed Joint Managing Director of the Metals Division, Imperial Chemical Industries, Ltd. He has also been appointed a Vice-Chairman of the British Non-Ferrous Metals Research Association.

Mr. P. Cutler has been appointed Superintendent, Ordnance Factory, Muradnagar, Meerut, U.P., India.

Mr. A. Dando has been appointed a Vice-Chairman of the British Non-Ferrous Metals Research Association.

Mr. R. C. Davidson, who recently graduated B.Sc. in the University of Melbourne, has been appointed Metallurgist to David Duncan and Co., ironfounders, Melbourne.

Mr. T. J. Dennis has been appointed Production Superintendent at the Byass Light Alloy Works of Richard Thomas and Baldwins, Ltd., Port Talbot. He obtained the B.Sc. degree with first-class Honours in Industrial Metallurgy at Birmingham University last July.

Mr. J. P. Dennison has been appointed Lecturer in Metallurgy at the University College, Swansea.

Mr. H. B. Dunthorne has left England for Canada on his appointment as metallurgist in the Gas Turbine Engineering Division Laboratory of A. V. Roe (Canada) Ltd., Malton, Ont.

Mr. A. M. Edge has been awarded the B.Met. degree of Sheffield University.

Mr. R. D. Hamer has been appointed to fill a casual vacancy on the Council of the British Non-Ferrous Metals Research Association.

Mr. V. C. Hanson has become Managing Director of Kent Alloys, Ltd., Rochester, Kent.

Mr. G. T. Harris has been appointed Research Manager of Messrs. William Jessop and Sons, Ltd., Sheffield.

Mr. T. Hewitt has left British Copper Refiners, Ltd., Prescot, and joined the Smelting Company (Egypt), S.A.E., Cairo.

Dr. W. R. Hibbard, Jr., formerly Assistant Professor of Metallurgy at Yale University, is now a Research Associate in the laboratories of the General Electric Co., Schenectady, N.Y.

Mr. G. W. Horsley has joined the Metallurgy Division, Atomic Energy Research Establishment, Harwell.

Dr. B. T. Houlden has left the British Non-Ferrous Metals Research Association and joined John Dale, Ltd.

Mr. B. P. James has been awarded the B.Met. degree of Sheffield University and has taken up a Short-Service Commission in the Education Branch of the Royal Air Force.

Mr. V. B. John has graduated B.Sc. in Metallurgical Chemistry at University College, Cardiff.

Mr. J. R. Lee has been awarded the degree of B.Sc. in Industrial Metallurgy at Birmingham University.

Mr. H. R. Mason, a recent B.Sc. graduate of Melbourne University, has joined the staff of the Broken Hill Proprietary Co., Ltd., Newcastle, N.S.W.

Mr. A. D. Michael has been appointed an Investigator in the Mechanical Testing Section of the British Non-Ferrous Metals Research Association. He was previously with J. Stone and Co., Ltd.

Dr. R. H. Myers, who has recently been working at the Atomic Energy Research Establishment, Harwell, has been appointed the first Professor of Metallurgy in the University of Sydney.

Mr. N. P. Pinto has joined Rem-Cru Titanium, Inc., Midland, Pa.

Mr. P. Potter has taken up an appointment with H. J. Enthoven and Sons, Ltd., Rotherhithe.

Dr. J. N. Pratt has resigned his post as University Research Fellow in the Department of Metallurgy of the University of Birmingham, to take up an appointment as a Research Fellow in the Department of Metallurgical Engineering in the University of Toronto. His new address is Department of Metallurgical Engineering, University of Toronto, Toronto S, Ont., Canada.

Mr. Eric Raybould has relinquished his position as Chief Metallurgist, Foundry Division, High Duty Alloys, Ltd., Slough, and has been appointed Foundry Manager with Messrs. H. M. Hobson, Ltd., Wolverhampton.

Professor G. V. Raynor has been appointed a Visiting Professor of Metallurgy for the session 1951–52 at the University of Chicago, and will be working at the Institute for the Study of Metals, Chicago 37, Ill. In connection with this appointment, Professor Raynor has been selected by the Board of Foreign Scholarships for a Fulbright Award and by the Department of State for a Smith–Mundt Award.

Mr. S. F. Reiter has received the degree of D.Eng. (Metallurgy) of Yale University and is now on the staff of the Research Laboratory of the General Electric Co., Schenectady, N.Y.

Mr. Lynn G. Richards is now at the U.S. Naval Engineering Experiment Station, Annapolis, Md.

Mr. N. B. Rutherford has been transferred from the Melting and Casting Section to the Liaison Department of the British Non-Ferrous Metals Research Association.

Dr. Richard Seligman (Past President and a Fellow of the Institute), founder and chairman of The A.P.V. Co., Ltd., has been presented by his workpeople and staff with a bronze plaque bearing a representation of his head in profile and the words "Presented by those who work for him as a token of their high regard." It is proposed to mount the plaque in the new works now being constructed for the firm at Crawley, Sussex.

On 22 October, Dr. Seligman is to receive the gold medal of the Society of Dairy Technology. This medal, which is awarded not more than three times in ten years, is for distinguished service to the dairy industry in any part of the world.

Dr. Dana W. Smith has been appointed Associate Director of the Division of Metallurgical Research, Kaiser Aluminum and Chemical Corpn., Spokane, Wash.

Mr. P. W Smith has resigned his post with Murex (Australasia) Pty., Ltd., on appointment as an Examiner of Patents, with the Department of Patents, Canberra.

Mr. L. J. Stone, who for the last few months has been on a business visit to England, has returned to Australia. Mr. Stone is Process Superintendent at Commonwealth Aircraft Corporation Pty., Ltd., Melbourne.

Dr. Roy E. Swift has joined the faculty of the Department of Mining and Metallurgical Engineering of the University of Kentucky, Lexington, Ky. He was formerly on the staff of the Mackay School of Mines of the University of Nevada, Reno, Nev.

Mr. Julian S. Tritton, a partner in Messrs. Rendel, Palmer, and Tritton, consulting engineers, has been elected President of the Institution of Locomotive Engineers for 1951–52.

Mr. H. Vickers has left King's College, Newcastle, and taken up an appointment with the Ministry of Supply, Division of Atomic Energy, Risley, near Warrington.

Professor H. K. Worner has been appointed by the Victorian Governor-in-Council as a member of the Council of the Swinburne Technical College, Melbourne.

Note: Will members (in addition to informing the Institute's administrative department of changes of address, occupation, &c.) kindly notify the Editor, *separately*, of all changes of occupation, appointments, awards of honours and degrees, &c., as these matters interest their fellow members.

JOINT ACTIVITIES

Capper Pass Awards

The Capper Pass Awards, which are made annually from a sum of £200 placed at the disposal of the Councils of the Institution of Mining and Metallurgy and of the Institute of Metals by Capper Pass and Son, Ltd., Bristol, are as follows:

(*a*) £100 per annum for one or more awards to authors of papers on some aspect of non-ferrous extraction metallurgy.

(*b*) £100 per annum for one or more awards to authors of papers relating to some process or plant used in the fabrication of non-ferrous metals.

The Adjudicating Committee will meet early in each year to consider all papers published by both societies during the previous year. Papers on extraction metallurgy should be offered to the Institution of Mining and Metallurgy and papers on fabrication to the Institute of Metals. Authors need not be members of either society, and may be resident in any country. MSS. should preferably be submitted in duplicate to either the Secretary, Institution of Mining and Metallurgy, Salisbury House, Finsbury Circus, London, E.C.2, or the Secretary, The Institute of Metals, 4 Grosvenor Gardens, London, S.W.1, from whom further particulars may be obtained.

NEWS OF KINDRED SOCIETIES

Symposium on the Corrosion of Buried Metals

A Symposium on the Corrosion of Buried Metals, organized by the Iron and Steel Institute in conjunction with the British Iron and Steel Research Association and the Corrosion Group of the Society of Chemical Industry, will be held at the offices of the Iron and Steel Institute, 4 Grosvenor Gardens, London, S.W.1, on Wednesday, 12 December 1951. Sir Charles Goodeve, O.B.E., D.Sc., F.R.S., Director of the British Iron and Steel Research Association, will be in the Chair. The sessions will begin at 10.0 a.m. and 2.30 p.m. and will be open to all interested in the subject under discussion.

The papers will be presented in brief individually and discussed in groups, as indicated in the programme below. A buffet luncheon will be available in the Library of the Institute.

Members of the Institute of Metals are invited to participate in this meeting on the same basis as members of the Iron and Steel Institute.

Detailed Programme

Morning Session

10.0 a.m. Opening remarks by the Chairman.

10.05 a.m. (i) Joint discussion on:

"Tests on the Corrosion of Buried Iron and Steel Pipes", by J. C. Hudson and G. P. Acock.

"Investigations on Underground Corrosion", by K. R. Butlin, W. H. J. Vernon, and L. C. Whiskin,

11.45 a.m. (ii) Joint discussion on:
"Cathodic Protection", by K. A. Spencer.
"Cathodic Protection of Buried Metal Structures", by M. R. de Brouwer.
1.30 p.m. Buffet Luncheon in the Institute Library.

Afternoon Session

2.30 p.m. (iii) Joint discussion on:
"Corrosion of Buried Copper and Ferrous Strip in Natural and Salted Soils", by G. Mole.
"Tests on the Corrosion of Buried Aluminium, Copper, and Lead", by P. T. Gilbert and F. C. Porter.
4.30 p.m. Conclusion of the Meeting.

Charges

Admission: There will be no charge for admission to the Meeting, but intending participants are requested to complete Part A of a Reply Form to be obtained from the Secretary of the Iron and Steel Institute.

Papers: The papers presented and the discussion at the Meeting will be issued as a single bound volume (No. 45 in the Special Report Series of The Iron and Steel Institute) the published price of which will be 15*s.* 0*d.* (post free).

Orders received before the Meeting, if accompanied by a remittance, will be supplied at the reduced rate of 10*s.* 0*d.* (post free), and one set of advance copies of the papers will be provided in respect of each volume so ordered, without extra charge. Advance copies will not be supplied separately, nor will more than one set be provided in respect of each bound volume ordered before the Meeting.

Requests for bound volumes (and preprints) should be made not later than 13 October 1951.

Buffet Luncheon: The charge for the Buffet Luncheon will be 6*s.* 0*d.* per person.

Remittances: The appropriate remittances should accompany the Reply Forms, which may be obtained from The Secretary, The Iron and Steel Institute, 4 Grosvenor Gardens, London, S.W.1.

Société Française de Métallurgie

The Annual Autumn meeting of the Société Française de Métallurgie will be held from 22 to 27 October at the Maison de la Chimie, 28 rue St. Dominique, Paris 7e. During the course of the meeting the Osmond Medal will be presented to Professor E. N. da C. Andrade, F.R.S., Director of the Royal Institution.

The Chemical Society Research Fund

The Research Fund of the Chemical Society provides grants for the assistance of research in all branches of Chemistry. About seven hundred pounds per annum is available for this purpose, the income being derived from a donation of the Worshipful Company of Goldsmiths, from the Perkin Memorial Fund, and from other sources.

Applications for grants will be considered in November next, and should be submitted on the appropriate form not later than Thursday, 1 November 1951. Applications from Fellows will receive prior consideration.

Forms of application, together with the regulations governing the award of grants, may be obtained from the General Secretary, The Chemical Society, Burlington House, Piccadilly, London, W.1.

OTHER NEWS

Metals Economy Advisory Committee

The Minister of Supply, in conjunction with the Lord Privy Seal as Minister of Materials, has appointed an advisory committee on metals economy to help him to ensure the most economical use of metals in the engineering industry and in the defence programme.

Mr. D. A. Oliver, Director of Research to the B.S.A. Group of Companies, who recently became metals economy adviser to the Minister, is chairman of the committee.

Members are drawn from industry and Government departments. The industrial members are: Mr. G. L. Bailey, British Non-Ferrous Metals Research Association; Mr. H. A. R. Binney, British Standards Institution; Mr. H. H. Burton, English Steel Corporation, Ltd.; Mr. W. C. F. Hessenberg, British Iron and Steel Research Association; Mr. E. W. Grinham, Standard Motor Co., Ltd.; Dr. Ivor Jenkins, General Electric Co., Ltd.; Dr. L. B. Pfeil, The Mond Nickel Co., Ltd.; and Dr. C. J. Smithells, The British Aluminium Co., Ltd. The secretary is Mr. K. M. McLeod, of the engineering industries division of the Ministry of Supply.

The terms of reference of the committee are: "To bring under review, in consultation with the various Government and industrial authorities concerned, ways of economizing in the use of scarce metals in the design, specification, and the manufacturing process of metal goods for both rearmament and civil purposes, and to advise the Government how it can best assist in promoting such developments."

As measures of economy in non-ferrous metals have an effect on, and are affected by, questions of supply of these metals, the committee will report to the Minister of Materials as well as to the Minister of Supply.

Courses in Applied X-Ray Diffraction and Industrial Radiography

Two courses of 30 lectures each are being given during the winter months at the Northampton Polytechnic, St. John Street, London, E.C.1.

The first course, to be given by Mr. A. E. De Barr, is intended to give graduate or Higher National Certificate students an introduction to the fundamental principles and practical technique of X-ray diffraction, with particular reference to industrial applications. The course, the fee for which is 30*s.*, will be held from 7.0 to 9.30 p.m. on Mondays, beginning on 24 September. Additional laboratory work can be taken by arrangement.

The second course, to be given by Dr. R. J. Barnes, will provide a survey of principles and practice for those engaged in industrial radiography. The fee is 30*s.*, and the lectures will be from 7.0 to 9.30 p.m. on Tuesdays, beginning on 25 September.

Advanced Course in Applied Physics

A course of twelve post-graduate lectures is to be given at the Harrow Technical College, Station Road, Harrow, on Fridays at 7.30 p.m.

The subjects to be dealt with are: (1) "Industrial Radiography", by Dr. L. Mullins (5, 12, 19 October); (2) "Photography Applied to Nuclear Physics", by Dr. R. H. Herz (26 October, 2, 9 November); (3) "The Measurement of Colour", by Mr. R. W. G. Hunt (16, 23, 30 November); and (4) "Electron Optics", by Mr. B. J. Mayo (7, 14, 21 December).

The fee for the full course is 30*s.*; for three lectures, 10*s.*

Course on Quantum Theory of Metals

Professor C. A. Coulson, F.R.S., is to give a course of six lectures on "Quantum Theory of Metals" at the Sir John Cass College, Jewry Street, Aldgate, London, E.C.3. The lectures will be from 6 to 7 p.m. on successive Tuesdays, beginning on 30 October. The fee for the course is 15*s*. Application for enrolment should be made to the Principal.

Course on Crystal Physics

The Sir John Cass College, Jewry Street, Aldgate, London, E.C.3, is holding a special course of ten lectures on Crystal Physics. The lecturer is Mr. L. A. Thomas of the Research Laboratories of the General Electric Co., Ltd., who will treat both theoretical and experimental properties of the crystalline state. The course, the fee for which will be 25*s*, will be given at 6 p.m. on Thursdays, beginning on 4 October.

DIARY

The Institute

17 and 18 October. General Meeting, London. For details see pp. 10–11.

Local Sections

4 October. London. "Pressure Welding", by R. F. Tylecote. (4 Grosvenor Gardens, London, S.W.1, at 7 p.m.)

8 October. Scottish. Visit to the works of Henry Wiggin and Co., Ltd., Thornliebank.

9 October. Oxford. "What is a Dislocation?" by Dr. B. A. Bilby. (Physical Chemistry Laboratory, South Parks Road, Oxford.)

9 October. South Wales. "Metallurgical Problems of Atomic Energy", by Dr. H. M. Finniston. (Metallurgy Department, University College, Swansea, at 6.30 p.m.)

15 October. Sheffield. "Radioactive Tracers in Metallurgy", by Dr. H. M. Finniston. Joint meeting with the Sheffield Society of Engineers and Metallurgists. (The University, St. George's Square, Sheffield 1, at 7.30 p.m.)

23 October. Sheffield. Lecture by Dr. P. A. Taylor. Joint meeting with the Sheffield Metallurgical Association. (Grand Hotel, Sheffield, at 7 p.m.)

25 October. Birmingham. Debate: "That in the opinion of this House further encroachment of Metal Physics into the sources of instruction would be detrimental to training in Metallurgy". Opening speakers: Dr. J. W. Jenkin and Mr. H. W. G. Hignett. (James Watt Memorial Institute, Great Charles Street, Birmingham 3, at 7 p.m.)

Other Societies

10 October. Manchester Metallurgical Society. Presidential Address, by A. B. Ashton. (Engineers' Club, Albert Square, Manchester, at 6.30 p.m.)

12 October. Liverpool Metallurgical Society. "The Problem of the High-Temperature Oxidation of Metals", by Professor A. Preece. (Lecture Theatre, Electricity Service Centre, Whitechapel, Liverpool, at 7 p.m.)

24 October. Manchester Metallurgical Society. "Recent Developments in Electrically Welded Chains", by Dr. H. O. Smerd. (Engineers' Club, Albert Square, Manchester, at 6.30 p.m.)

26 October. Chemical Society. "Some Aspects of Oxidation and Corrosion", by Dr. U. R. Evans. Joint Meeting with the Birmingham University Chemical Society. (Chemistry Lecture Theatre, The University, Birmingham 15, at 4.30 p.m.)

26 October. Institution of Mechanical Engineers. "The Principles of Continuous Gauge Control in Sheet and Strip Rolling", by W. C. F. Hessenberg and R. B. Sims. (The Institution, Storey's Gate, London, S.W.1, at 5.30 p.m.)

APPOINTMENTS VACANT

CHEMISTS AND METALLURGISTS are invited by the Ministry of Supply to apply for the following appointments in the EXPERIMENTAL OFFICER class at a Research Establishment in Berkshire.

Post 1. (Ref. F.591/51/A) Metallurgist with research experience in electro-deposition or in the investigation of the properties of metallic films and coatings. Experience in the preparation of microscopical specimens would be an advantage. Candidates should possess a Higher National Certificate or equivalent qualification in metallurgy.

Post 2. (Ref. F.592/51/A) Physical chemist or physicist with interest and preferably experience in work on ceramic materials. Candidates should have a minimum qualification of Higher School Certificate with chemistry as a principal subject, but higher qualifications and relevant experience would be an advantage.

Salary will be determined on age and on an assessment of the successful candidate's qualifications and experience within the ranges: Senior Experimental Officer (minimum age 35) £742 to £960; Experimental Officer (minimum age 28) £545 to £695. Rates for women somewhat lower. Posts are unestablished.

Selected candidates may be required to work elsewhere for a short time before taking up duty in Berkshire. Housing accommodation may be available later. Application forms obtainable from Ministry of Labour and National Service, Technical and Scientific Register (K), York House, Kingsway, London, W.C.2, quoting appropriate reference number. Closing date 15 October 1951.

CHEMISTS, METALLURGISTS, AND PHYSICISTS are invited by the Ministry of Supply to apply for the following appointments in the SCIENTIFIC OFFICER CLASS at a Research Establishment near London and (for one of the posts under 1) the Royal Aircraft Establishment, South Farnborough, Hants.

1. (Ref. F.582/51/A) Physical Metallurgists (3) for the determination of thermal and physical properties of metals and alloys. Research experience of metallography, electron microscopy, or X-ray or electron-diffraction techniques would be an advantage.

2. (Ref. F.583/51/A) Physical chemist or physicist with an interest in metallurgy or ceramics, for research investigations connected with the preparation of novel ceramic materials.

3. (Ref. F.584/51/A) Physical Chemist or physicist for work on surface phenomena and for research investigations into the properties of metallic films and coatings. Experience of electron-diffraction techniques or of electrochemistry would be an advantage.

Candidates must have a 1st or 2nd class honours degree in appropriate subject or equivalent qualification. For the senior posts they must be at least 26 years of age and have had at least 3 years' post-graduate research experience. Salary will be determined on age and on an assessment of the successful candidates qualifications and experience within the ranges: Senior Scientific Officer £720–£910; Scientific Officer £380–£620. Rates for women somewhat lower. Posts are unestablished but carry benefits of F.S.S.U. Application forms obtainable from Ministry of Labour and National Service, Technical and Scientific Register (K), York House, Kingsway, London, W.C.2, quoting appropriate reference number. Closing date 15 October 1951.

GRADUATE, preferably with a good Honours degree in Metallurgy, is required for research and development work on magnetic materials. Preference will be given to men with 2–3 years' research experience. Apply to the Staff Manager (Ref. GBLC/814) Research Laboratories of The General Electric Co., Ltd., East Lane, North Wembley, Middlesex, stating age, experience, and qualifications.

INHOMOGENEITIES IN THE PLASTIC DEFORMATION OF METAL CRYSTALS—I.–II.*

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I.—Occurrence of X-Ray Asterisms

By R. W. K. HONEYCOMBE,† M.Sc., Ph.D., Member

SYNOPSIS

A study has been made of the occurrence of X-ray asterisms from deformed single crystals of a typical hexagonal metal (cadmium) and a typical cubic metal (aluminium). Cadmium can be extended by more than 100% elongation in tension without the appearance of asterisms in X-ray Laue photographs; asterisms occur only when the crystals are macroscopically bent or kinked. On the other hand, they are present in photographs from aluminium crystals after less than 4% elongation in tension. These asterisms contain intensity maxima which are shown to be a direct consequence of the deformation and not of a secondary phenomenon such as polygonization.

On annealing, extended cadmium crystals give sharp Laue spots, and the bent crystals polygonize very uniformly at 300° C. The appearance of asterisms from lightly extended aluminium crystals is unaltered by annealing at 250° C., at which temperature partial recovery of the yield stress occurs; but when the temperature is raised to 475° C. the asterisms become further fragmented as a result of polygonization.

These experiments suggest that tensile deformation causes aluminium, but not cadmium, crystals to break down into a series of slightly disoriented blocks connected by regions of distortion or lattice curvature.

I.—INTRODUCTION

It has been realized for a long time that plastic deformation of metal crystals does not consist merely of simple translations along crystallographically defined slip planes. This is clearly shown in the shape of the stress/strain curves, which indicate that strain-hardening occurs to a greater or lesser degree. There is also metallographic evidence that even in deformed single crystals, deformation does not result in uniform shearing along glide lamellæ. The simple conception of plastic deformation is further belied by the occurrence of certain X-ray phenomena, such as asterisms in Laue photographs and line broadening in Debye-Scherrer photographs. X-ray asterisms show that local disorientations occur in deformed crystals, but these are related to the original orientation and do not not amount to a random fragmentation of the crystal. In this and the second part of the paper, investigations of the nature of the phenomena in the deformed crystals giving rise to some of the above deviations from pure deformation by slip are described. These phenomena will be called inhomogeneities until at a later stage they can be referred to in more specific terms. A good starting point for the investigation is provided by a study of the origin and occurrence of X-ray asterisms.

II.—THE ORIGIN OF X-RAY ASTERISMS

During the last twenty years there have been three main theories advanced to explain X-ray asterisms. Their origin has been variously attributed (1) to local curvatures on the slip planes, (2) to macroscopic curvatures of the crystal, and (3) to fragmentation.

(1) Yamaguchi [1] considered that asterisms arose from the presence on the slip planes of local curvatures formed when glide processes became anchored in the lattice. This hypothesis was later supported by Taylor [2] and by J. M. Burgers,[3] who assumed that local curvatures occurred when dislocations of opposite sign approached each other. W. G. Burgers and Lebbink [4] did not consider macroscopic curvatures to be the basic cause of asterism, because they found that X-ray photographs of aluminium crystals showed asterisms even when the crystals were sheared along the slip plane in the slip direction by a special apparatus.

(2) Other workers hold the view that asterisms are caused by macroscopically bent regions of the crystals, and there are in fact cases where asterism clearly arises from this cause, e.g. the elastic bending of mica or thin metal sheet where asterism disappears on return to the unbent condition; this also occurs with some plastically bent crystals when they are re-straightened. Komar and Mochalov [5] have shown that with deformed magnesium crystals, the lengths of the Laue striæ diminish as the thickness of the crystals is reduced by etching, a result which suggests that the asterisms are due to macroscopic curvatures. Orowan and Pascoe [6] obtained further evidence for this view by the examination of X-ray rotation photographs from slightly deformed cadmium crystals. They found that certain reflections were very sharp, whereas other reflections from the same planes were

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† Formerly Armourers and Brasiers' Company Research Fellow, Cavendish Laboratory, Cambridge; now at Metallurgy Department, University of Sheffield.

blurred, a result they explained by assuming a macroscopic curvature of the plane, the sharp reflection coming from the concave side, the blurred one from the convex. Kochendörfer[7] found that asterisms were absent in X-ray photographs from naphthalene crystals which had been sheared along the glide plane and in the glide direction, while tensile and compressive tests imposing similar strains resulted in strong asterisms. He concluded that the asterism was not due to local curvatures, as its occurrence depended clearly on the nature of the deformation process.

(3) A third explanation attributes X-ray asterism to the formation of lattice fragments with very small differences in orientation between neighbouring fragments, which if sufficiently small would make the asterism continuous. Manteuffel[8] and Komar[9] supported this view because they observed discontinuities in the asterisms from deformed metals. Wood,[10] on the basis of extensive experiments on X-ray line broadening, also considers that fragmentation occurs as a direct result of deformation. There is, in fact, much evidence that under certain conditions, e.g. deformation at high temperatures, X-ray asterisms contain a fine structure. This has been studied in recent years by Cahn[11] and by Guinier and Tennevin,[12] and from their experiments it seems clear that the breaking up of the asterisms results from the recovery of a bent lattice to a series of closely oriented, unbent blocks. This takes place by diffusion of dislocations to form the walls between the "fragments". It appears that this process, which Orowan has called polygonization, is a secondary one which removes lattice distortions caused by plastic deformation.

Apart from X-ray phenomena, there is direct metallographic evidence that inhomogeneities occur when metals deform. Band-like structures have been observed within deformed grains in many different metals in a variety of circumstances. In 1939 Barrett and Levenson[13] published an account of a detailed investigation into the occurrence of what they called *deformation bands* in iron and aluminium, using both polycrystalline and single-crystal specimens. In the early stages of deformation the bands occurred on planes of low indices, and the lattice within each band rotated away from the parent orientation to a gradually increasing degree. After heavy deformations the crystallographic features were obscured and the bands became curved and distorted. Barrett suggested that in view of the fact that the ranges of orientation in the bands correspond approximately with those determined from X-ray asterisms, the bands are a major source of these asterisms. These experiments demonstrated the important role of such inhomogeneities in the deformation of metals, but many points remain to be elucidated.

It is difficult to decide the real causes of asterism on the available evidence, because the experiments have been carried out on a variety of metals—and even non-metals such as mica, naphthalene, and rock salt—under widely differing conditions of deformation. It is perhaps unwise to expect non-metals to behave similarly to metals, or indeed for metals as a class to behave uniformly. There is, in fact, ample evidence that metal crystals of cubic crystal structure, when plastically deformed, behave very differently from metals of hexagonal structure; e.g. the resolved shear stress/strain curves fall into two distinct groups,[4] the hexagonal metal crystals showing only a small degree of strain-hardening. It therefore seemed necessary to compare the behaviour of a typical hexagonal metal with that of a typical cubic metal. Experiments to this end on cadmium and aluminium are described below, in which the occurrence of asterisms is investigated together with the behaviour of the deformed crystals on annealing.

III.—STUDY OF ASTERISMS FROM DEFORMED CRYSTALS

1. Cadmium

The single crystals used in these experiments were in the form of 1-mm.-dia. wires produced from the melt by the Andrade method. These were made from both spectroscopically pure (99·99%) and 99·9% pure metal. Crystals were selected in which the angle between the glide plane and the wire axis was 40°–60° (soft orientations), so that a large extension could be expected before twinning occurred. Specimens with gauge-lengths between 2 and 5 cm. long were carefully extended on a glass plate using small tweezers; in this way handling of the extremely fragile crystals was reduced to a minimum, and accidental distortion eliminated. Crystals were extended by this method to 100, 200, and even 300% elongation. Short lengths were then cut from the resulting flat ribbons by means of a sharp razor blade, and X-ray Laue photographs were taken using gold radiation. It was found that crystals extended by 100% produced practically no asterism in the Laue photographs and only a slight fine structure could be detected in the reflections (Fig. 4, Plate X). Prolonged annealing of the extended crystals in no case resulted in recrystallization and X-ray photographs revealed that the Laue spots were again extremely sharp and typical of an undeformed crystal.

Specimens from the extended crystals were then bent by controlled amounts around cylindrical mandrels (with radii of curvature from 5 to 1 mm.), the basal plane being almost parallel to the axis of bending. X-ray Laue photographs showed marked asterisms which were homogeneous, i.e. there were no pronounced intensity maxima (Fig. 5, Plate X). On annealing the bent crystals at 290° C. for 24 hr., a striking uniform fragmentation of the asterisms occurred, indicating that the bent lattice had polygonized (Fig. 6, Plate X). Prolonged annealing resulted in a coarsening of the polygonized structure, the range of orientation remaining constant. Recrystallization only occurred in the most severely bent crystals (radius of curvature 1·75 mm.), in which the new grain grew at the expense of a polygonized matrix (Fig. 7, Plate X).

Certain crystals were chosen with their basal plane parallel to the wire axis, so that on compression kinking took place. The structure of a kinked crystal, as given by Orowan[15] is shown in Fig. 1. This configuration occurs when the crystal suddenly collapses under load; there are two kink planes, *k* and *k'*, separated by a region in which the crystal is bent severely. X-ray photographs of kinked crystals did, in fact, show marked asterism in this region, but not elsewhere in the crystal. That kinking had led to a macroscopic bending of the crystal planes was shown directly by polishing flat faces on crystals such that the displacement would occur parallel to the face. The crystals were then kinked and examined microscopically. Fig. 8 (Plate X) shows a typical micrograph of a portion of a kink; the sharp change in direction of the slip lines at the kink plane is shown, and the slip lines in the region between the kink planes are macroscopically bent.

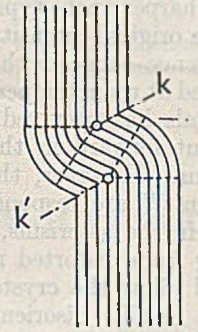

Fig. 1.—Schematic Diagram of a Kinked Crystal (*Orowan*[15]).

The bending caused by kinking was so severe that spectroscopically pure cadmium crystals after kinking either polygonized or recrystallized at room temperature if left for several weeks. These changes were restricted to the bent regions of the crystal. If the kinked crystals were annealed at high temperatures (300° C.), the kinked region was almost invariably replaced by two new grains, each of which occupied one curved region of the kinked crystal, their boundaries with the old crystal being in the vicinity of the two kink planes.

2. Aluminium

The aluminium crystals were prepared in a similar way to the cadmium crystals. They were in the form of wires 0·5 mm. in dia. and were of spectroscopic purity (99·99%). Specimens 1 in. long were cut from the crystals, and small copper U's were soldered to the ends so that the specimens could be suspended and deformed in a single-crystal extensometer.

Preliminary experiments showed that asterisms appeared in X-ray Laue photographs when the crystals had been elongated in tension by only small amounts. After 1% extension the Laue reflections were blurred and after 4–5% marked asterisms had developed. In all the cases examined the asterisms possessed a pronounced fine structure which became apparent in the very early stages of the deformation (Fig. 9, Plate X). It was considered that a more valid comparison could be made with the experiments on cadmium if the aluminium crystals were deformed at elevated temperatures. However, asterisms were still obtained from crystals deformed at 400° C., and even after deformation at 600° C. Fig. 10 (Plate X) shows asterisms from a crystal deformed 20% at 600° C. Under such circumstances the asterisms were always built up of small discrete spots, indicating that fragmentation had occurred during deformation despite the fact that the deformed crystal was at 600° C. for only about 2 min. Microscopic examination confirmed that discrete crystallites with straight boundaries had been formed, in other words that the crystal had polygonized.

The fine structure in asterisms from crystals deformed at room temperature might be attributed to the partial occurrence of polygonization, which is pronounced in crystals deformed at much higher temperatures. However, such a recovery process would hardly be expected in aluminium crystals only slightly deformed at room temperature. For example, Guinier and Tennevin,[12] using a very sensitive X-ray focusing method, were unable to detect polygonization in aluminium at temperatures less than 300° C. and in most cases a considerably higher temperature was required. If polygonization does occur in aluminium crystals deformed slightly at room temperature, then changes in the mechanical properties, e.g. the yield point, might be expected to take place. To determine whether this was so, crystals were strained by small amounts in a single-crystal extensometer and the stress/strain curve obtained. The yield stress was subsequently measured after various periods at room temperature. Deformed crystals were also held at elevated temperatures (250°–450° C.) and changes in the yield stress were plotted against time to give recovery curves. The crystals thus investigated were mounted in small jigs that enabled them to be removed from the extensometer without distortion and to be fixed to the spindle of an X-ray camera. X-ray back-reflection Laue photographs were taken at various stages of the treatment and correlated with the changes in yield stress.

Several crystals were deformed small amounts (between 2 and 6% elongation) and allowed to remain at room temperature. It was found that the X-ray patterns showed intensity maxima when the exposure was made immediately after deformation. The pattern did not alter after two days at room temperature, and in the same time no change in the yield stress of the deformed crystals could be detected.

Certain of the deformed crystals were subsequently annealed at 250°–300° C., and the yield stress measured at intervals during the process. For example, Fig. 2 shows both the stress/strain curve and the recovery curve for crystal E Al 5; marked recovery has taken place on annealing at 250° C. X-ray Laue photographs taken with a fine collimator (0·3 mm.) revealed no change from those taken immediately after deformation. If a crystal annealed at 300° C. was subsequently annealed for 30 min. at 450° C. then the X-ray asterisms broke down further, giving the spots characteristic of polygonization. These new spots occurred in the originally diffuse portions of the asterisms, while the original intensity maxima remained (Fig. 11, Plate X).

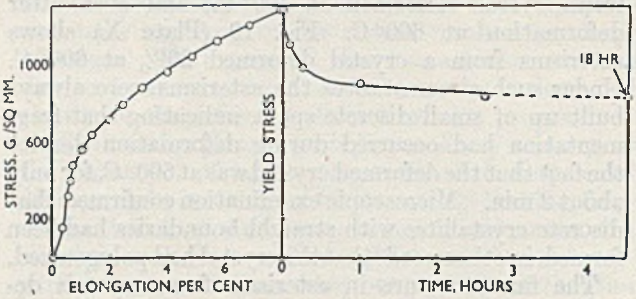

FIG. 2.—Stress/Strain and Recovery Curves for Aluminium Crystal E Al 5.

Other crystals after similar deformations were annealed immediately at 475° C. The yield stress fell markedly in the first 5 min. of the anneal (Fig. 3), during which time no perceptible change could be seen in the X-ray Laue patterns. However, at a later stage, when the recovery curve was almost horizontal, marked changes characteristic of polygonization were observed. It is interesting to note that after 5 minutes' annealing, the yield stress dropped almost to its final value. Further prolonged annealing did not result in a closer approach to the yield stress of the original crystal. It seems that changes resulting in the major part of the recovery are beyond the resolution of the X-ray method, which reveals only the final stage of polygonization when growth is taking place with little influence on the mechanical properties.

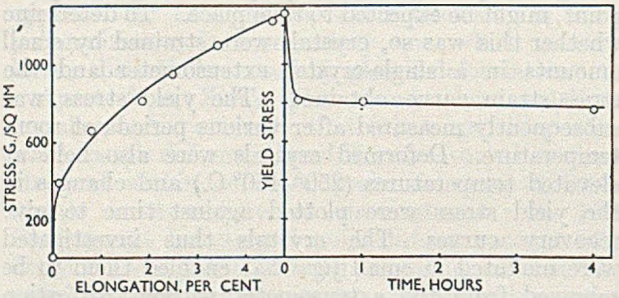

FIG. 3.—Stress/Strain and Recovery Curves for Aluminium Crystal E Al 1.

IV.—DISCUSSION AND CONCLUSIONS

The experiments with cadmium deformed in tension show that X-ray asterisms are not necessarily associated with the plastic deformation of this metal. That asterisms are obtained from cadmium crystals deformed inhomogeneously by bending or by kinking is entirely in accord with the view that they result from macroscopic curvatures of the crystal, the occurrence of which in turn depends on the mode of deformation. On the other hand, asterisms appear in the photographs of aluminium crystals even after small elongations in tension, when marked macroscopic curvatures would not be expected. These asterisms differ from those obtained from bent cadmium crystals in so far as they contain marked intensity maxima after small deformations. It seems unlikely that these maxima are a result of polygonization, because they occur concurrently with the deformation at relatively low temperatures. They do not appear to result from spontaneous recovery at room temperature, because the Laue streaks show no further breakdown after several days at room temperature; furthermore, no change in yield stress occurs during this time. In fact, the asterisms remain unaltered even after annealing at 300° C., at which stage there is a definite fall in the yield stress which indicates that recovery is taking place. If the temperature is then raised to 450° C. for a short time, the Laue streaks alter, the diffuse arc breaking up into sharper spots typical of polygonization; however, the original intensity maxima remain.

The X-ray evidence suggests that in the case of aluminium deformed at room temperature, a fragmentation of the crystals has occurred. This view has been frequently put forward in the past to explain similar observations. However, the author believes that the "fragments" are responsible only for the intensity maxima in the asterisms, the diffuse background originating from distorted regions which join the fragments and allow the crystal to remain continuous although locally disoriented. In support of this hypothesis there is the observation referred to earlier that polygonization occurs primarily in the diffuse regions of the asterism, as would be expected if these regions were more severely bent than the rest of the crystal. It is clear that the tensile deformation of cubic metal crystals results not only in a greater degree of strain-hardening than is the case in hexagonal metals, but that the deformation is accompanied by much more distortion of the crystal. The obvious deduction is that the increased strain-hardening is a result of the greater amount of distortion in the crystals of cubic metals. Before this hypothesis can be further explored, the exact nature of the distortions should be known. This is dealt with in Part II.

A comparison of the behaviour of deformed crystals of cadmium and aluminium on annealing is significant. On the one hand cadmium, after extensions of 200–300% in tension, would not recrystallize or polygonize even on prolonged annealing at temperatures close to the melting point. The Laue spots originally only slightly affected by the severe deformation became extremely sharp after annealing. It thus appears that complete recovery occurred in these cases. In fact, measurements of the change in yield point of deformed zinc crystals made by Haase and Schmid[16] showed that even after 100% elongation complete recovery to the original yield stress occurred, and Davies[17] has recently confirmed this behaviour for cadmium. On the other hand aluminium crystals deformed in tension do not recover to their original yield stress when annealed at high temperatures after relatively small deformation. Moreover, the X-ray reflections remain complex. It seems clear that once disorientations occur within a crystal they are not removed by recovery; nucleation and grain growth are the only way in which a uniform orientation can be re-created.

The experiments do not show conclusively whether polygonization is the sole mechanism by which recovery occurs, for the resolution obtained in the X-ray Laue technique is insufficient to decide this. It is, however, significant that extended cadmium crystals are capable of complete recovery without the occurrence of polygonization. In this case it could be assumed that dislocations in the slip planes migrate through to the surface of the crystal, whereas if the crystal is bent this freedom of movement is restricted and the excess dislocations of one sign form polygon boundaries, leading to the observed X-ray effects. On the other hand, with aluminium crystals the movement of dislocations is hindered by the regions of distortion, which give rise to the X-ray asterisms.

REFERENCES TO PART I.

1. K. Yamaguchi, *Sci. Papers Inst. Phys. Chem. Research (Tokyo)*, 1929, **11**, 151.
2. G. I. Taylor, *Proc. Roy. Soc.*, 1934, [A], **145**, 362.
3. J. M. Burgers, *Proc. Phys. Soc.*, 1940, **52**, 23.
4. W. G. Burgers and F. J. Lebbink, *Rec. Trav. Chim.*, 1945. **64**, 321.
5. A. P. Komar and M. D. Mochalov, *Physikal. Z. Sowjetunion*, 1936, **9**, 613.
6. E. Orowan and H. J. Pascoe, *Nature*, 1941, **148**, 467.
7. A. Kochendörfer, "Plastische Eigenschaften von Kristallen und metallischen Werkstoffen." Berlin: **1941** (Julius Springer).
8. I. Manteuffel, *Z. Physik*, 1931, **70**, 109.
9. A. P. Komar, *Physikal. Z. Sowjetunion*, 1936, **9**, 413.
10. W. A. Wood, *Proc. Phys. Soc.*, 1940, **52**, 110, &c.
11. R. W. Cahn, *J. Inst. Metals*, 1949–50, **76**, 121.
12. A. Guinier and J. Tennevin, *Compt. rend.*, 1948, **226**, 1530.
13. C. S. Barrett and L. H. Levenson, *Trans. Amer. Inst. Min. Met. Eng.*, 1939, **135**, 327; 1940, **137**, 112.
14. E. Schmid and W. Boas, "Plasticity of Crystals with Special Reference to Metals", p. 125. London: **1950** (F. A. Hughes and Co.).
15. E. Orowan, *Nature*, 1942, **149**, 643.
16. O. Haase and E. Schmid, *Z. Physik*, 1925, **33**, 413.
17. P. T. Davies, private communication.

II.—X-Ray and Optical Micrography of Aluminium

SYNOPSIS

A technique of X-ray microscopy is used in conjunction with optical micrography to investigate the structure of large aluminium crystals deformed by small amounts in tension. Two types of microscopic inhomogeneity which cause local variations in orientation are described. First, there are narrow regions of curvature or *kink bands* separating slightly disoriented lamellæ of the crystal; these bands occur initially on (110) planes, the normal to which coincides with the active slip direction. The second type of inhomogeneity comprises *bands of secondary slip*, which are regions nearly devoid of primary slip traces, in which slip on another system occurs preferentially. These bands are almost parallel to the primary slip planes in the early stages of deformation.

The effects of crystal orientation, temperature and speed of deformation, and purity of metal on the occurrence of kink bands are investigated, and it is found that these are usually present after deformation, except when this occurs from the beginning by conjugate slip. Bands of secondary slip also generally occur, and in crystals deforming by conjugate slip, they are often of macroscopic size.

The inhomogeneities play an important role in the deformation of aluminium and account for many of the differences between this metal and cadmium, such as the occurrence of X-ray asterisms, the marked differences in strain-hardening, and the extent to which recovery can occur on annealing.

I.—INTRODUCTION

Much work has been done on the occurrence of so-called *deformation bands* in deformed metals, in particular by Barrett and Levenson,[1] which indicates that these bands may be the seat of the distortions in the crystals. However, the wealth of information relating to the bands is rather confusing and their exact nature is far from clear. If inquiry is restricted to aluminium single crystals deformed in tension, it is found that there is some evidence for the existence of regions of distortion on a microscopic scale. As long ago as 1929, Yamaguchi[2] found microscopic regions in which the slip lines were bent, and Collins and Mathewson[3] observed narrow bands of crystallographic habit which they considered to be deformation bands. More recently Crussard[4] found regions in deformed aluminium crystals which he likened to the kinking that occurs in hexagonal metal crystals. Finally, Cahn,[5] during the course of the present work, has investigated by microscopic means bands of local flexural glide in deformed aluminium crystals of special orientation. There seems little doubt that all these observed phenomena are related and that they are in need of further systematic study. Bearing in mind the limitations of optical micrography for detecting small local changes in orientation within crystals, a method of X-ray microscopy similar to that described by Berg[6] and Barrett,[7] which is suitable for the examination of lightly deformed crystals, has been developed. The main results obtained with deformed aluminium crystals are described below, but for convenience, the method employed and the detailed interpretation of the X-ray micrographs have been discussed in a separate paper.[8]

II.—EXPERIMENTAL WORK

1. Preparation and Examination of Crystals

The small crystals used in the previous experiments (reported in Part I) were unsuitable for micrographic examination, so large flat crystals 20 cm. long, 12·5 mm. wide, and 1·5–3 mm. thick, with a gauge-length of 7 cm., were produced by the Carpenter–Elam critical-strain method. Two grades of aluminium were used one 99·5% pure and the other 99·99% pure. Before deformation the crystals were electropolished, without previous mechanical polishing.

The crystals were deformed by various amounts between 0·5 and 20% elongation in a simple straining jig which, if necessary, could be heated in a furnace. The deformed crystals were examined in the optical microscope, often in oblique illumination, and by X-ray microscopy. Whereas the optical microscope revealed very small displacements in the crystal surface, e.g. slip lines, X-ray micrography showed local distortions and changes in orientation. As these disorientations did not occur on the slip planes, the latter were not usually an important feature of the X-ray photographs.

2. Summary of Results

In aluminium crystals deformed between 0·5% and 20% elongation, two distinct types of inhomogeneity which had caused local disorientations were observed. First, narrow bent regions were found which separated relatively unbent regions of the crystals. These, which will be referred to as *kink bands*, were detected at a very early stage of the deformation on (110) planes. The essential feature of this type of inhomogeneity is the bending which can give rise to X-ray asterisms.

The other type of inhomogeneity comprised bands which were initially deficient in primary slip traces, i.e. traces of the slip system which would normally be expected to operate (applying the criterion of maximum resolved shear stress). In the very early stages of the deformation these bands were roughly parallel to the active slip planes. As the deformation proceeded, the bands became the preferential sites for secondary slip on planes not predicted by the criterion of maximum shear stress. For clarity, this type of inhomogeneity will be referred to as *bands of secondary slip*. These bands also lead to disorientations in the crystal, but are primarily free from severe curvatures.

3. Kink Bands

(a) *Detection by X-Ray Micrography*

X-ray micrographs of aluminium crystals of 99·5% purity after 4% elongation in tension were shown to possess a fine structure absent from images of undeformed crystals. When the X-ray images recorded on Kodak Maximum Resolution plates were enlarged to 50 diameters, two sets of dark bands were revealed (Fig. 12, Plate XI). One set, which predominated, had a spacing of about 0·05 mm.; furthermore, most of the bands ran completely across the crystal face. The fact that the reflections from the deformed crystal comprised alternate dark and light bands indicated that more or less regular disorientations had occurred. The crystal had broken up into long thin blocks, many of which were not in a position to reflect the incident X-rays. Optical examination at this stage of the deformation did not reveal the bands, but confirmed that they could not be due to the slip traces (Fig. 14, Plate XI). However, at heavier deformations (over 10% elongation) bands became visible and proved to be narrow regions within which the crystal was bent (Fig. 15, Plate XI). Optical examination at higher magnification showed clearly that the bands possessed a double curvature similar to that observed in kinked cadmium crystals. However, the planes bounding the bent regions were not as well defined as those in cadmium. Occasionally the presence of a small included grain caused the bands in its vicinity to widen and become more pronounced (Fig. 16, Plate XI).

The X-ray reflections from the slightly deformed crystals occurred over a greater angular range than those from the annealed crystals. It was not possible to relate directly the angular range of reflection to a given part of the crystal, because, as the crystal was rotated, the area contributing to the reflection changed gradually. However, by taking a series of photographs 6′ apart, and by comparing the same region of the specimen represented in each, it was possible to determine the angular displacement over which a particular region contributed to the reflection. After 4% elongation, the angle was usually between 1° and 2° and increased with increasing deformation. While a small area, say 1 mm.^2, might contribute to the reflection for 1° of rotation, it was obvious that different microscopic parts within the area contributed to the reflection at different stages of this rotation.

At certain settings of the crystal it was possible to obtain reflections from the slip planes rather than from the kink bands. Fig. 13 (Plate XI) shows the change which occurred in the image of Fig. 12 (Plate XI) after a rotation of only 20′. The dark bands representing the kink bands have been entirely replaced by a series of fine dark striations which are broken by horizontal white bands of similar orientation to the dark bands of Fig. 12. The kink bands are no longer in a reflecting position. In general, it was found that the kink bands caused striations which predominated in the image over most of the angular range of reflection, whereas the reflections from the slip planes usually appeared over a much more restricted range (between 10′ and 20′). This predominance of the kink-band reflections is a consequence of the range of orientation created by these bent regions of the crystal, while the high intensity of the reflection is probably a result of the distortion in the bands.[8]

(b) *Crystallography of the Kink Bands*

A number of crystals of different orientations were examined by X-ray micrography to determine whether the occurrence of the kink bands was dependent on orientation. The tension axes of 11 crystals which were systematically investigated are represented in the stereographic triangle of Fig. 26. Kink bands were detected in the crystals represented by the open circles, while the black circles represent crystals in which kink bands were not observed. In these two cases, conjugate slip on two or more systems occurred from the beginning of plastic deformation. While the kink bands occurred to the same extent in crystals whose orientations lay near the centre of the stereographic triangle, they were much finer and more difficult to detect in crystals whose orientations were approaching the (111) pole. In these crystals the bands were not visible optically even after relatively heavy deformations. It is possible that the bands would be absent in orientations very close to (111), but crystals of this orientation have not yet been examined. Fig. 26 shows that several crystals have very similar orientations; these were chosen so that the investigation of such variables as speed and temperature of deformation could be made independent of orientation.

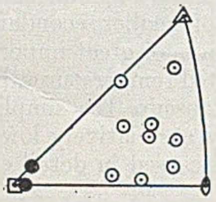

Fig. 26.—Stereographic Projection of Crystal Tension Axes.
○ Kink band present. ● Kink band absent.

In all crystals which developed kink bands, the X-ray micrographs showed clearly that the bands possessed a marked crystallographic habit, so in every case the plane of the bands was determined in the manner described in a previous paper.[8] In addition to the plane of the kink bands, the active slip plane, as determined by measurement of angles of slip traces on two surfaces of the crystal, was plotted. The operative slip direction was obtained by selecting the [110] direction in the slip plane which gave the maximum resolved shear stress. In all cases it was found that in the early stages of plastic deformation, the kink bands were on (110) planes, the normal to which coincided with the active slip direction.

(c) *The Formation of the Bands*

It was found that the kink bands could occur after less than 1% elongation in tension. Fig. 21 (Plate XIII) is an X-ray micrograph of an aluminium crystal after 0·75% elongation. At this stage the local differences in orientation were very small and most of the crystal was contributing to the X-ray reflection; however, two sets of bands were visible because of differences in intensity within the reflection. On further deformation, one set of bands became much more pronounced in the X-ray images, and analysis showed that they were kink bands on (110) planes. The other set of bands originated from the second type of inhomogeneity, i.e. bands of secondary slip, which will be discussed later. In other crystals deformed by similar amounts the kink bands were not visible and appeared only after 1–2% elongation, but bands of secondary slip were always observed.

Optical examination revealed that many of the slip traces stopped in the kink bands (Figs. 15 and 16, Plate XI). This could mean that kink bands originated from barriers initially present in the crystal, but microscopic examination of crystals at different stages during deformation showed that the arrested slip lines were those which formed in the later stages. Initially all the slip lines were continuous, apart from occasional breaks due to cross-slip; moreover, no bending could be detected. The behaviour of the slip lines that appeared later was shown by a crystal deformed to 17·5% elongation, electropolished to remove all slip traces, and then re-strained slightly. All the new slip lines stopped abruptly in the kink bands (Fig. 17, Plate XII), which were also clearly shown to be regions of curvature.

Fig. 17 also shows that after moderate deformation, slip on a different set of planes occurs in the kink band. This suggests that rotation within the bands is more pronounced than in the bulk of the crystal. The amount of rotation necessary before secondary slip occurs is obviously dependent on the initial orientation of the crystal and on the direction of rotation. This occurrence of secondary slip may cause confusion between the kink bands and bands of secondary slip, particularly if the specimen is examined after heavy deformations. However, in the early stages of deformation, the difference can be easily seen and confirmed by determination of the crystallographic habit.

(d) *Purity of the Aluminium*

Kink bands also occurred in deformed high-purity aluminium crystals (99·99%), but although they approximated to the same crystallographic habit as those in the less pure crystals, the bands were less regular and much further apart. Fig. 19 (Plate XII) is an X-ray micrograph of such a crystal after 2·8% elongation, which should be compared with Fig. 12 (Plate XI). There was also a more gradual development of the bands, the earlier ones being between 0·2 and 1 mm. apart, but as the deformation became more severe, new bands appeared between them. This resulted in a more complex breakdown of the crystal, as shown in Fig. 20 (Plate XII) which represents the same area as Fig. 19 after 6% elongation. While the density of the bands was much less than in the less pure crystals, the rotations which occurred in the initial bands were greater. This usually resulted in a rumpling of the surface which was visible to the naked eye after small extensions (4%). Examination of such surfaces with a "Talysurf" profilometer revealed directly the tilting of the lamellæ between the kink bands. Optical examination at heavier deformation showed that these disoriented regions between the kink bands had frequently rotated to positions where duplex slip had begun (Fig. 18, Plate XII).

(e) *Temperature of Deformation*

Kink bands occurred in crystals deformed at widely differing temperatures. For example, crystals deformed rapidly at 500° C. developed kink bands to just as marked a degree as crystals deformed at room temperature. To investigate more precisely the effect of temperature, two crystals (99·5% pure) of similar orientation were compared; one was deformed rapidly at room temperature to an elongation of 4%, while the other was strained rapidly at 450° C. and immediately air-cooled. X-ray examination of the latter crystal showed that a widely spaced set of bands had formed.* This should be compared with a typical result obtained at room temperature, as shown in Fig. 12 (Plate XI), from which it is clear there has been a two- or three-fold increase in spacing of the bands for an increase of 430° in the temperature of deformation. Analysis of the bands obtained in the crystals deformed at high temperatures showed that they had the same crystallographic habit as those formed at room temperature.

A crystal deformed at 450° C. was subsequently annealed at 630°–640° C., and X-ray micrographs were taken at various stages. It was found that the banded structure produced as a result of plastic deformation was still visible even after 89 hours' annealing, but the dark lines representing the kink bands were no longer intense and in many cases the incident X-rays were not reflected. This would be expected because of the tendency of the most severely bent regions, i.e. the kink bands, to polygonize during the long anneal and so eliminate the curvatures. However, as the kink bands are very narrow, this local polygonization is only a secondary source of disorientation, the primary source being the larger lamellæ formed between the kink bands during deformation.

Several high-purity crystals were deformed at the temperature of liquid nitrogen and again kink bands were detected in X-ray micrographs. These were much more closely spaced (0·05 mm. after 4·5% elongation) than those formed at room temperature (Fig. 19, Plate XII) and could not be observed optically.

(f) *The Speed of Deformation*

The speed of deformation appeared to have little effect on the formation and habit of the kink bands. As in the investigation of the effect of temperature, crystals of similar orientation were compared after small deformations at widely differing rates of strain. A crystal of 99·5% aluminium deformed to 3·3% elongation at room temperature at a rate of 0·018% per hr. developed kink bands to much the same degree as a crystal deformed the same amount in several seconds. Again the bands occurred on (110) planes with the usual relation to the slip direction. Another crystal (99·5% purity) deformed at the same slow rate at 300° C., likewise developed kink bands. On the other hand the results obtained with high-purity (99·99%) crystals deformed slowly at 300° C. were very different from those at room temperature. Again, widely spaced, pronounced kink bands could be detected by X-ray microscopy. However, while the primary disorientations in the crystal were a result of the kink bands, the long lamellæ between the bands became the sites of smaller secondary disorientations.† This phenomenon is of great importance in the study of creep at elevated temperatures, but as discussion at this stage may obscure the simpler case of crystals deformed rapidly at relatively low temperatures, the matter will be discussed in detail elsewhere.

(g) *Crystals with Conjugate Slip*

During the investigation several crystals were obtained, the orientations of which showed that conjugate or double slip could be expected at a very early stage of the deformation. The orientations of two crystals which were investigated in detail are represented in Fig. 26 as black circles. These crystals were of high-purity aluminium and were deformed at room temperature. They were examined at stages during the deformation both by X-ray and optical micrography and in neither method were kink bands detected. A third crystal started to deform by conjugate slip after only a small amount of deformation, but in this case fine kink bands had already occurred in the early stages of the deformation. The bands, however, did not develop to the same degree as those found in crystals deforming on one primary set of slip planes.

4. BANDS OF SECONDARY SLIP

In X-ray micrographs of lightly deformed aluminium crystals two sets of bands were usually observed (Fig. 21, Plate XIII), particularly at small extensions (about 1% elongation). At a later stage the kink bands predominated in the X-ray micrographs, but optical examination showed that the other bands were still present. Microscopic examination revealed that in the early stages of deformation this second type of

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* See Fig. 8(*a*) (Plate IX), *J. Inst. Metals*, this vol.

† See Fig. 6(*a*) and (*b*), Plate VIII, *J. Inst. Metals*, this vol.

PLATE X.

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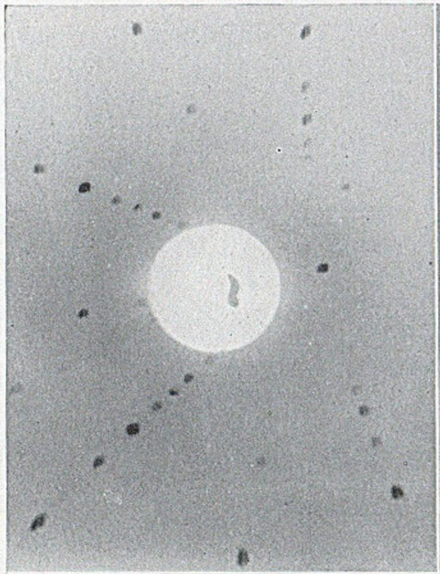

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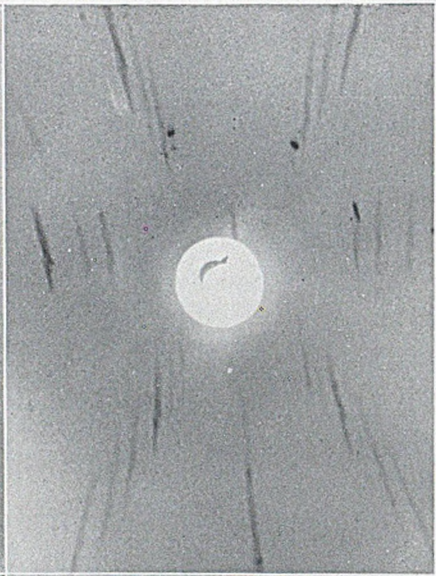

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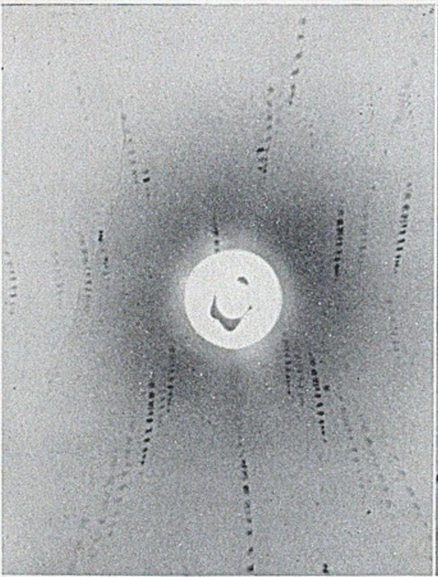

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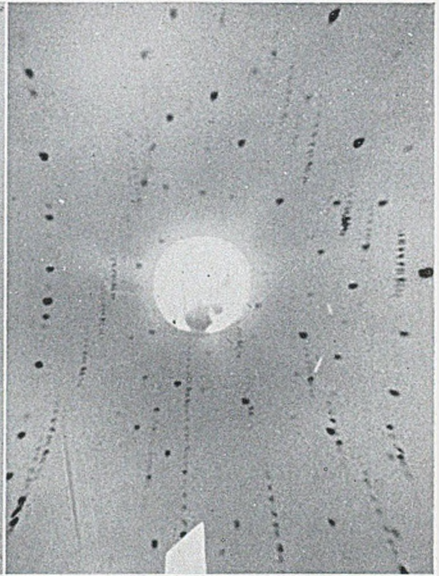

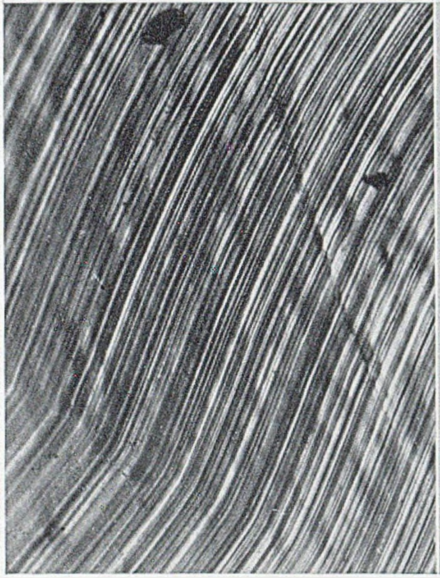

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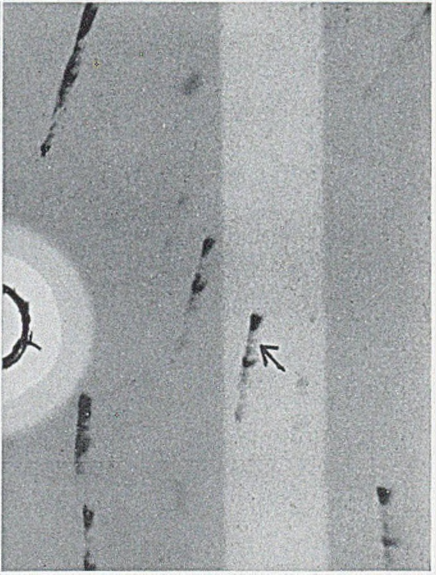

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Figs. 4–7.—X-Ray Laue Photographs of Cadmium Crystal After 100% Elongation. Au Radiation (Back Reflection).

Fig. 4.—As Extended.

Fig. 5.—Bent to 3·5 mm. Radius.

Fig. 6.—Bent to 3·5 mm. Radius and Annealed 24 Hr. at 290 °C.

Fig. 7.—Bent to 1·75 mm. Radius and Annealed 24 Hr. at 290° C.

Figs. 9–11.—X-Ray Asterisms from 99·99% Aluminium Crystals. × 2.

Fig. 8.—Micrograph of Kink Region of Cd Crystal. × 250.

Fig. 9.—4·6% Elongation at Room Temp. (Back Reflection).

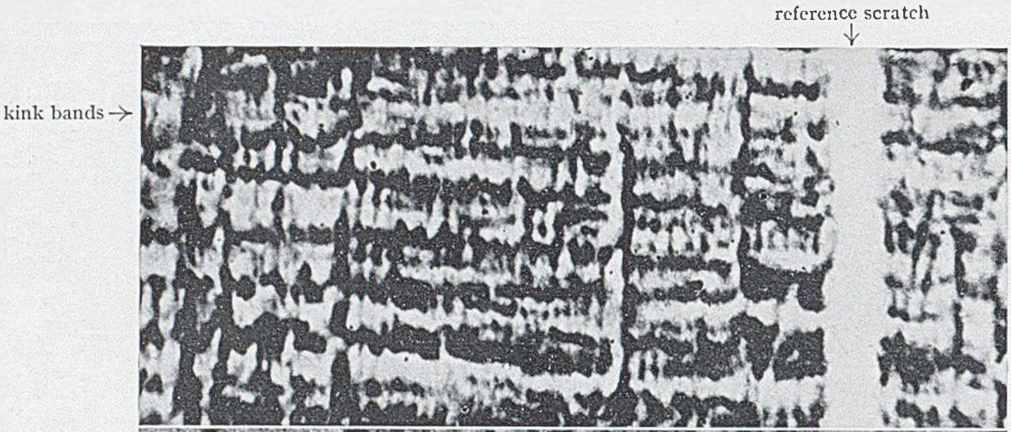
Fig. 10.—20% Elongation at 600° C. (Transmission).

Fig. 11.—5·4% Elongation followed by Annealing for 4 Hr. at 300° C. + 30 Min. at 450° C.

[*To face p.* 52.

PLATE XI.

MICROGRAPHS OF 99·5% ALUMINIUM CRYSTAL AFTER 4% ELONGATION. × 50.



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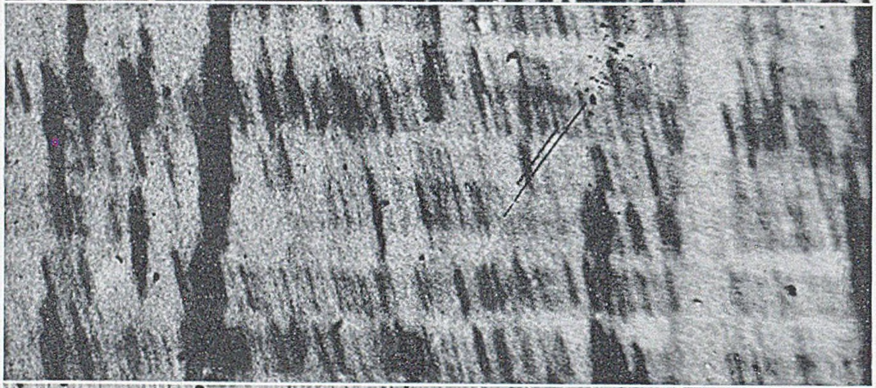

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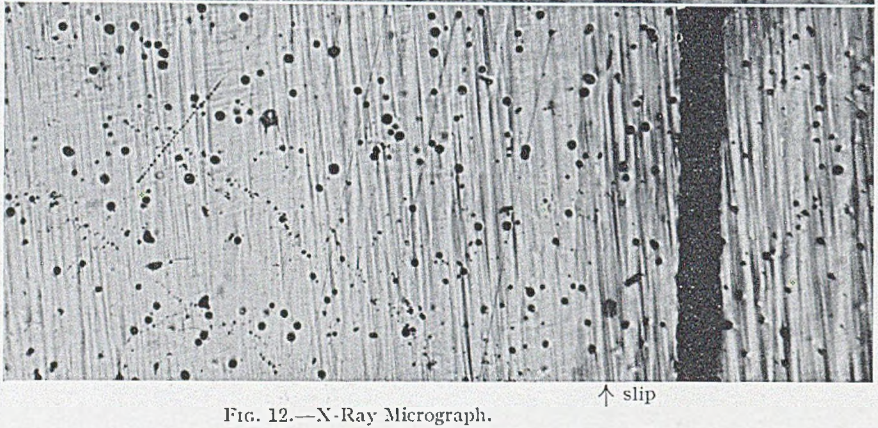

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Fig. 12.—X-Ray Micrograph.
Fig. 13.—Same as Fig. 12 After Rotation of 20′.
Fig. 14.—Optical Micrograph.

OPTICAL MICROGRAPHS OF 99·5% ALUMINIUM CRYSTAL AFTER 20% ELONGATION.

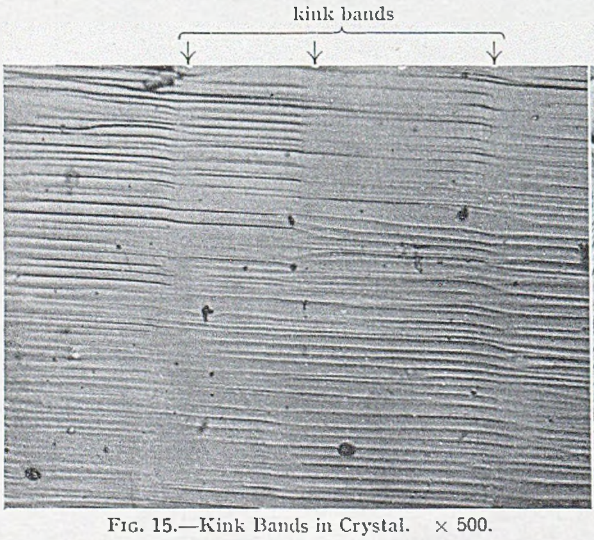

Fig. 15.—Kink Bands in Crystal. × 500.

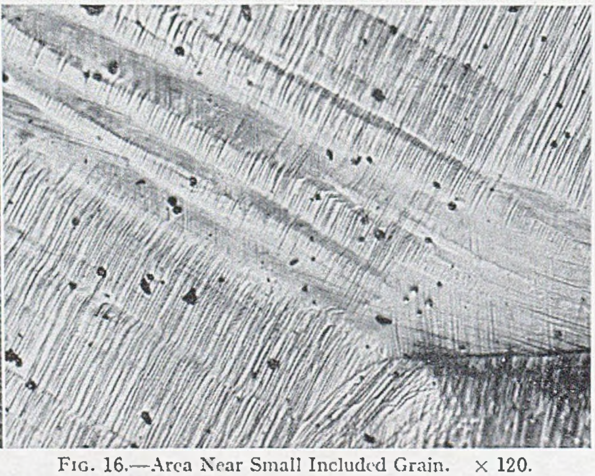

Fig. 16.—Area Near Small Included Grain. × 120.

Plate XII.

Optical Micrographs of 99·99% Aluminium Crystal.

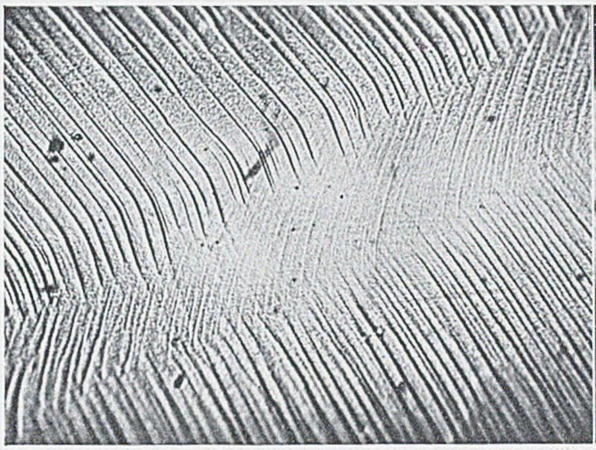

Fig. 17.—After 17·5% Elongation, Repolished, and Slightly Re-strained. × 250.

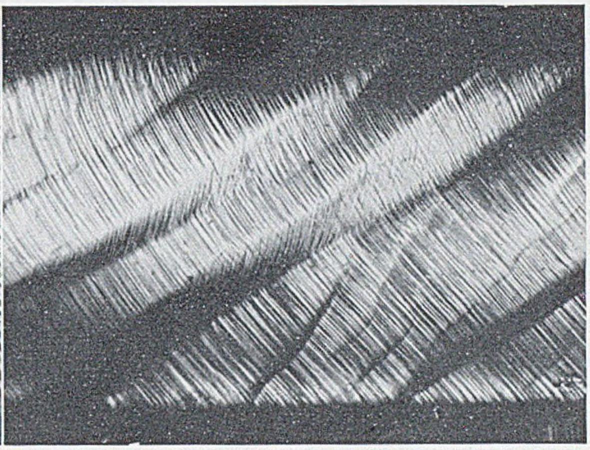

Fig. 18.—After 17·5% Elongation. × 100.

X-Ray Micrographs of 99·99% Aluminium Crystal. × 20.

Fig. 19.—After 2·8% Elongation.

Fig. 20.—After 6% Elongation. Same area as Fig. 19.

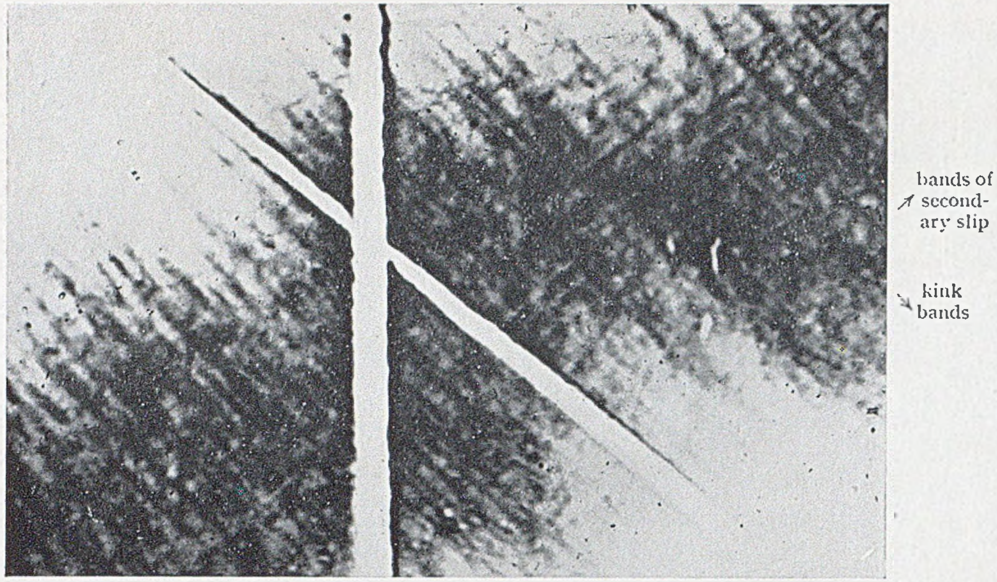

Fig. 21.—X-Ray Micrograph of 99·5% Aluminium Crystal After 0·75% Elongation. × 25.

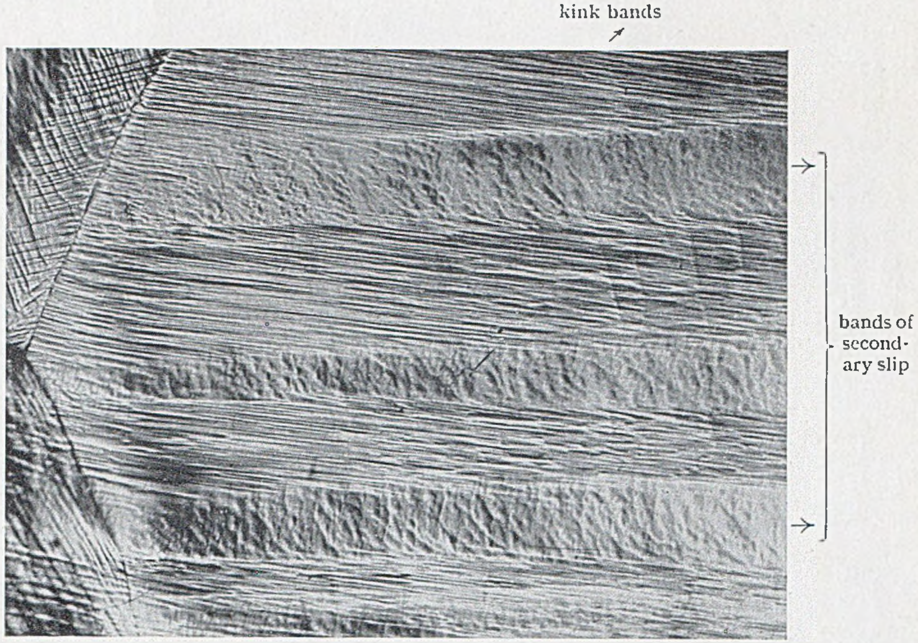

Fig. 22.—Optical Micrograph of Bands of Secondary Slip in a Grain of Coarse-Grained 99·9% Aluminium After 17% Elongation. × 100.

Optical Micrographs of a Band of Secondary Slip in Aluminium Crystals.

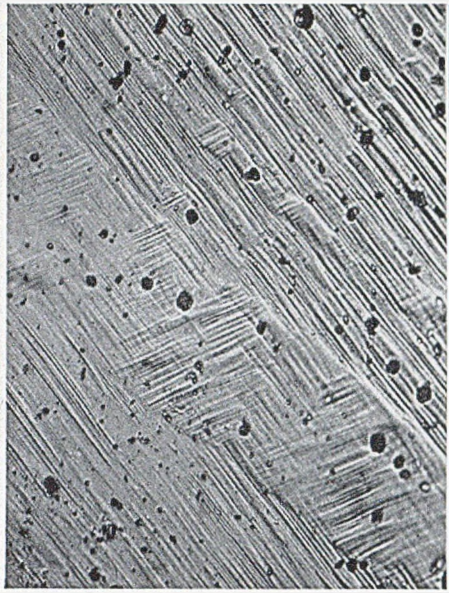

Fig. 23.—99·5% Al After 10% Elongation. × 150.

Fig. 24.—99·99% Al After 5·6% Elongation. × 100.

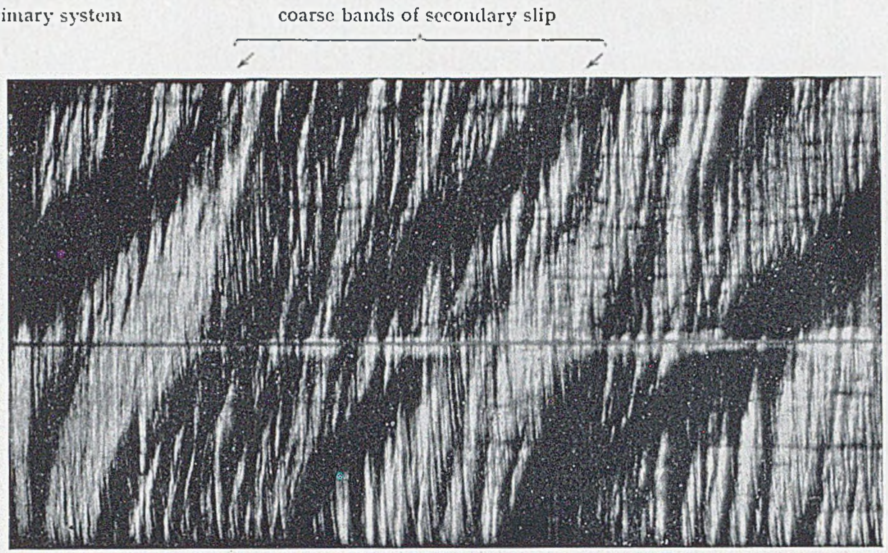

Fig. 25.—Optical Macrograph of 99·99% Aluminium Crystal of Symmetrical Orientation After 5·6% Elongation. × 5.

Plate XIII.

inhomogeneity occurred almost parallel to the octahedral slip planes, and comprised regions in which the primary slip traces were very weak or even absent. As the deformation became more severe, these bands were the preferential sites for slip on a system of planes different from those operating in adjacent parts of the crystal. For this reason the term " band of secondary slip " has been used to describe this type of inhomogeneity. A good example in an aluminium crystal (99·5% pure) after 10% elongation is shown in Fig. 23 (Plate XIII); in this case the secondary slip has developed to a considerable extent.

In many crystals the bands did not show sharp slip lines, but a rumpled appearance which at low magnifications revealed little crystallographic regularity. However, examination at higher magnifications showed that the rumpling was caused by the slip continually changing from the primary to the secondary slip system and back, thus following an irregular zig-zag path. The individual components of each slip line, as described by Heidenreich and Shockley [9] and Brown,[10] crossed over to the new glide plane at different points, thus somewhat obscuring crystallographic features. Fig. 22 (Plate XIII) shows some bands of secondary slip in which this has occurred; the photograph includes the major part of one grain of a coarse-grained aggregate which had been extended by 17% in tension after electrolytic polishing.

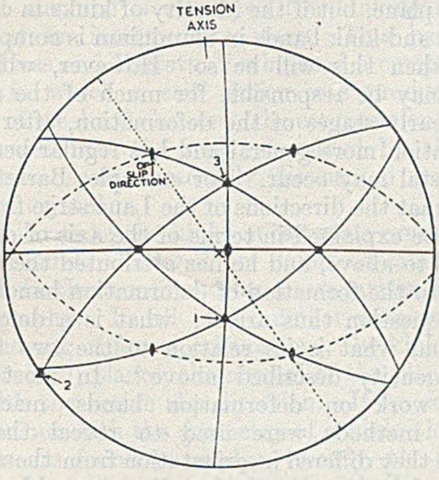

FIG. 27.—Stereographic Projection of Crystal PAl2 Which Deformed by Conjugate Slip.

KEY.

— · — · — · — Primary plane.

- - - - - - - - - - - First conjugate.

— — — — — Second conjugate.

Crystals in which more than one slip system operated from the beginning of deformation allowed a detailed study of this type of inhomogeneity, for in these circumstances bands of macroscopic size were often obtained. Fig. 27 shows the stereographic projection of the orientation of one of these crystals, and it is clear that no less than four slip systems could take part in the deformation at an early stage. The data obtained by measurement on the crystal and its projection are tabulated in Table I.

TABLE I.—*Primary and Secondary Slip in Crystal PAl2 (99·99% Aluminium).*

Angles	Slip Planes		
	Primary	Secondary	
	1	2	3
λ-angle between axis and slip direction . . .	42°	41°	42°
ϕ-angle between axis and slip plane normal . . .	55°	55°	59°
$\cos\lambda . \cos\phi$ . . .	0·42	0·43	0·39

After slight tensile deformation, slip plane 1 operated, but there were large regions in which no primary slip lines were visible. After 5·6% elongation slip on at least one of the alternative systems occurred in these bands (Fig. 24, Plate XIII), in many cases to the complete exclusion of the primary system. Fig. 25 (Plate XIII) is a photomacrograph taken at this stage of the deformation, in which the bands of secondary slip show up as dark areas. When the specimen was deformed further to 16·3% extension, the bands became more pronounced as they rotated in a different direction from adjacent parts of the crystal, thus resulting in macroscopic disorientations within the crystal. By this stage the other parts of the crystal were deforming by conjugate slip on the primary plane and the first of the secondary planes. The smaller, more regularly spaced bands of Fig. 25 are approximately parallel to the primary slip plane and correspond to the bands observed in the many other aluminium crystals examined. The larger bands comprising about five large inhomogeneities were more or less regularly spaced along the crystal, and although they are similar in origin to the smaller bands, their unusual size and orientation must be attributed to the special orientation of the crystal.

5. POLYCRYSTALLINE HIGH-PURITY ALUMINIUM

While the majority of the experiments have been done with single crystals, some observations have also been made on polycrystalline high-purity aluminium deformed in tension. For convenience in observation the grain-size chosen was very large—between 2 and 10 mm.-dia. Under these conditions the deformation is much less homogeneous than in the case of single crystals, the effect of the crystal boundaries being to cause different parts of the one grain to deform to different extents (Boas and Hargreaves [11]). However, neglecting the variations in the vicinity of the boundaries, it can be said that the same inhomogeneities develop as in the single crystals. In fact, the two types of band are quite pronounced and can often be distinguished by macroscopic examination if the grain-size is large enough. Fig. 22 (Plate XIII) illustrates an example of bands of secondary slip typical of coarse-grained, high-purity aluminium; fine kink bands can also be seen. The grains which showed the most pronounced kink bands were those in which slip had predominantly occurred on one system; these grains also usually developed bands of secondary slip. Grains in which conjugate slip occurred at a very early stage of the deformation did not usually contain kink bands but the bands of secondary slip were quite pronounced. Again in some grains conjugate slip occurred as an almost uniform network.

6. SOME OBSERVATIONS ON SINGLE CRYSTALS OF CADMIUM

Large cadmium crystals (10 × 0·5 × 0·3 cm.) were prepared by solidification from the melt in a graphite-coated steel boat, the resultant undersurface of the crystal after electropolishing being tolerably flat for X-ray and optical micrography. The crystals were deformed in tension, and it was found that after 6% elongation X-ray micrographs showed no fine structure at all, whereas optical micrography revealed many straight slip lines. After 20% elongation some X-ray images showed some fine lines corresponding with optically observed slip bands; however, in no case were markings seen which could be attributed to kink bands or bands of secondary slip.

III.—DISCUSSION AND CONCLUSIONS

In Part I it was stated that the structure of X-ray asterisms obtained from slightly deformed aluminium crystals could be interpreted in terms of microscopic disorientations within the crystals, the disoriented blocks being connected by bent regions. The results of X-ray and optical micrographic examinations in the present part have provided experimental evidence for this view, namely the occurrence of narrow kink bands separating disoriented lamellæ of the crystal. It is suggested that the bands give rise to the diffuse background of the asterism, while the lamellæ are responsible for the observed intensity maxima. The bands of secondary slip which are also present in aluminium crystals will result too in fragmentation of the asterisms, but because they are not primarily regions of curvature, would not be expected to contribute to the continuous background. Thus asterisms from slightly deformed aluminium crystals can be explained without recourse to the "local curvature" hypothesis of Burgers, which envisaged curvatures more on an atomic scale.

Burgers and Ploos van Amstel[12] have shown that the angular range of asterisms from deformed aluminium crystals is less for a given strain when the crystals deform from the start on two slip systems, than when slip occurs on a single system of planes. They contended that the asterism was less in the former case because the local curvatures were spread over twice as many glide planes. However, the above experiments indicate that the decrease in asterisms in crystals deforming by conjugate slip from the beginning of deformation is really due to the absence of kink bands. This view of the origin of asterisms is further supported by the experiments with cadmium crystals which showed no asterism after 100–200% elongation in tension; X-ray micrographic examination of cadmium crystals did not reveal kink bands or other inhomogeneities after considerable plastic extension.

The axes of bending corresponding to the observed asterisms have been determined by numerous workers for various metal crystals. For example, Burgers and Louwerse[13] have shown that in aluminium crystals the curvatures are about an axis which lies in the glide plane perpendicular to the glide direction. Andrade and Tsien[14] have shown that a similar type of distortion occurs also in body-centred cubic metals such as sodium. Now kink bands occur on (110) planes, the normal to which coincides with the slip direction, so that the axis of rotation determined by Burgers and Louwerse is clearly compatible with the type of rotation occurring in the kink bands. It is not immediately evident that the axis of bending lies in the slip plane, but if the identity of kinks in cadmium crystals and kink bands in aluminium is complete (see later), then this will be so. However, while kink bands may be responsible for much of the asterism in the early stages of the deformation, after heavier deformation more general and less regular bending of the crystal may occur. For example, Barrett[15] has shown that the directions of the Laue striæ frequently cannot be explained in terms of the axis of curvature referred to above, and he has attributed these irregularities to the formation of deformation bands.

The question thus arises: what is a deformation band and what is its relation to the two types of inhomogeneity described above? In most of the earlier work on deformation bands, macroscopic etching methods were used to reveal the bands because they differed in orientation from the adjacent regions of the crystal. In fact, Barrett and Levenson[1] defined a deformation band as a region in which the orientation progressively rotated away from that in the neighbouring parts of the crystal, so it is likely that their deformation bands included the two types of inhomogeneity described in the present paper. Barrett and Levenson state in one of their papers: "we believe that the [deformation] bands form as a result of the operation of different sets of slip systems in different band-shaped regions". This would account for the bands of secondary slip, but not for the kink bands which appear to play an equally important part in the deformation. As the two types of inhomogeneity occur together in deformed crystals and as they possess different crystallographic habits, it is perhaps not surprising that the crystallographic features of deformation bands observed by earlier workers were rather variable. Furthermore, as the deformation proceeds, the bands rotate, and at heavy deformations there is no reason why they should occur any longer on planes of lower Miller indices.

The term *deformation band* has been used in the past to cover all disorientations encountered in deformed metals, and its continued use without qualification may lead to considerable confusion. In this paper the terms *kink band* and *band of secondary slip* have been deliberately used instead of the term *deformation band,* because the two types of inhomogeneity arise through different initial mechanisms whatever their final result may be.

There is a close relationship between the kinking in compressed cadmium crystals described by Orowan [16] and the narrow kink bands found in aluminium crystals. It is clear from optical examination that both processes result in the formation of sharply bounded regions of the crystal within which the orientation is continuously changing because of lattice curvature. The kinks which occur when certain cadmium crystals are compressed are a more severe form of distortion than that in kink bands of aluminium crystals, but this is due to the different stress conditions. The characteristic kinking of cadmium crystals occurs suddenly on compression in a soft machine as a result of the inherent instability, but if a hard machine is employed the kinking can be stopped at an intermediate stage. This has been done by Hess and Barrett, [17] who used a compression machine in which a stiff action was produced by insertion of steel rods between the compression plates. They showed that the region bounded by the two kink planes became gradually disoriented from the rest of the crystal.

The similarity of the kinks in cadmium crystals and the kink bands observed in deformed aluminium crystals is further confirmed by a comparison of their crystallography. In all of a number of aluminium crystals having different orientations, the kink bands formed on (110) planes, the normal of the plane coinciding with the active slip direction. If we compare this behaviour with the geometry of a kink, the same conditions are found to apply; namely, the normal to the plane of kinking is identical with the original slip direction.

Kink bands are not observed in cadmium crystals elongated considerable amounts in tension; however, flexural glide, i.e. slip associated with macroscopic bending of the crystal, is found near the grips. This arises directly from the fact that although the crystal is being pulled in tension, it is attempting to deform by shear along slip planes. To do this the grips should be able to rotate relative to each other, but as is usual, they remain rigid and hinder rotation of the crystal, a circumstance which leads to macroscopic bending in their vicinity. It seems that in aluminium, the restraint of the grips is accommodated by the formation of kink bands which allow small units of the crystal to undergo rotation relative to neighbouring parts. It is significant that kink bands do not occur in crystals which deform from the beginning by conjugate slip on two or more slip planes. If another slip plane is available on which the resolved shear stress is similar to that on the primary plane, then the formation of kink bands is unnecessary, because the stresses set up as a result of the restraints imposed on the crystal, are reduced by slip on the alternative system.

The view that kink bands arise as a result of the mode of deformation is fully borne out by the recently published experiments of Röhm and Kochendörfer, [18] who deformed long aluminium crystals in a special shear apparatus. Unlike results of earlier experiments carried out with the Bausch shear apparatus, they found that by arranging for the shear to take place along a (111) plane in the slip direction, they could obtain " pure gliding ". This pure gliding was characterized by an absence of asterism in X-ray photographs, a very low value for the critical shear stress, and only a small degree of strain-hardening; in other words, aluminium crystals behaved rather like cadmium crystals in simple tension. It seems very likely that in these experiments kink bands were absent and that the grips had little influence on the deformation in the middle of the crystal.

The bands of secondary slip are in the first instance not regions in which the crystal is bent, but as the deformation becomes more severe, considerable distortions may arise within them. Evidence for this is provided by Barrett and Levenson,[1] who observed that deformation bands (into which general category bands of secondary slip certainly fall) become curved and distorted at high deformations. The present author has confirmed this observation in aluminium single crystals deformed in compression. This type of inhomogeneity may be related to the phenomenon of unpredicted cross-slip as described by Maddin, Mathewson, and Hibbard [19] in α-brass crystals and Cahn [5] in aluminium crystals. However, this phenomenon in its various manifestations is usually very localized in character and the authors mentioned have not described the somewhat coarser band structures observed in the present work. The bands appear to be initially free from slip, but the presence of unpredicted cross-slip at the beginning of deformation could be the reason why primary slip traces are almost absent from the bands. It is well known that in deformed single crystals the latent slip systems are hardened by the primary slip, so that they do not operate until well past the position at which the resolved shear stresses on the two slip systems are the same. Thus the occurrence of cross-slip in a region before the primary slip would tend to inhibit slip on the primary system. The work of Maddin, Mathewson, and Hibbard [19] has established that cross-slip frequently occurs before primary slip. They explain it as an alternative to flexural glide: " [stress] relief could take place by action of the conjugate slip planes just prior to slip on the primary glide plane carrying the highest conventionally resolved shear stress ".

The secondary slip observed in the bands was frequently straight, but if the bands were observed on a different face of the same specimen they then appeared to contain a zig-zag type of slip as a result of repeated alternate slip on two different planes. Recent work by Cahn [5] has shown that this is a general characteristic of slip processes in aluminium, which when observed in a plane containing the slip direction are straight but which frequently exhibit cross-slip when viewed in other planes. The apparent rumpling in the bands of secondary slip can be satisfactorily explained on this basis.

The occurrence of inhomogeneities in the tensile deformation of aluminium, which are absent in cadmium crystals deformed in a similar manner, may help to account for the great difference in strain-hardening in the two metals. The experiments with aluminium crystals indicate that the two types of inhomogeneity act as inhibitors of slip. On the one hand slip processes, particularly those occurring after the early stages of deformation, are halted at kink bands. Similarly the bands of secondary slip resist the penetration of primary slip traces and if examined at a late stage of the deformation, they clearly interrupt the progress of slip processes which have begun elsewhere (Fig. 22, Plate XIII). Thus, if we define the softness of a crystal as the ease with which a dislocation can proceed through it, then it is evident that both types of inhomogeneity are inhibitors of moving dislocations and hence should cause strain-hardening. Perhaps the most convincing support for this view is found in the experiments of Röhm and Kochendörfer already referred to. They found that the hardening was directly proportional to the amount of glide that occurred during deformation, i.e. the strain-hardening curve was linear and corresponded closely with those determined many years ago for hexagonal metals.[20] It therefore appears that inhomogeneities are responsible for a large part of the strain-hardening in aluminium crystals. The residual hardening as found in both cadmium and aluminium is considered by Kochendörfer to be due to the interaction of dislocations at their formation.

In Part I it has been shown that aluminium crystals deformed in tension do not recover completely and that the range of orientation produced in the crystals remains unaltered. However, Röhm and Kochendörfer [18] found that their sheared crystals recovered almost completely on annealing for 1 hr. at 350° C. after strains of 30–40%, i.e. they behaved like the cadmium crystals deformed in tension. On the other hand, they found that aluminium crystals deformed similar amounts in tension recovered by only 20–30% after the same annealing treatment. Thus the inhomogeneities which lead to X-ray asterisms and increase strain-hardening also influence the course of recovery. It is, in fact, difficult to see how disorientations of the type described above can be removed except by nucleation and grain growth.

In conclusion, it has been shown that in the tensile deformation of aluminium single crystals, inhomogeneities develop at an early stage and play an important part in determining the behaviour of the deformed crystal. In the first place, the microscopic inhomogeneities that have been described are the cause of the disorientations occurring in the crystal and are thus responsible for the asterisms in X-ray diffraction patterns. Further, it seems likely that the occurrence of inhomogeneities increases strain-hardening, and thus alters the shape of the stress/strain curve during deformation. Lastly, the inhomogeneities influence to a large degree the behaviour of a crystal during recovery. It appears that the inhomogeneities develop as a result of restrictions imposed by the mode of deformation, although the exact mechanisms involved are not yet understood. Quasi-homogeneous deformation, i.e. deformation by slip without the formation of kink bands, &c., can be achieved in special circumstances, and it has been shown that this condition is more readily attained in hexagonal metals, such as cadmium, than in aluminium. At the other extreme, the deformation of polycrystalline metals provides ample opportunity for the general development of the two types of inhomogeneity described in this paper, and which must undoubtedly be taken into account when the detailed plastic behaviour of metal aggregates is studied.

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REFERENCES TO PART II.

1. C. S. Barrett and L. H. Levenson, *Trans. Amer. Inst. Min. Met. Eng.*, 1939, **135**, 327; 1940, **137**, 112.
2. K. Yamaguchi, *Sci. Papers Inst. Phys. Chem. Research (Tokyo)*, 1929, **11**, 151.
3. J. A. Collins and C. H. Mathewson, *Trans. Amer. Inst. Min. Met. Eng.*, 1940, **137**, 150.
4. C. Crussard, *Bull. Soc. Franç. Minéral.*, 1945, **68**, 174.
5. R. W. Cahn, *Dissertation, Univ. Cambridge*, **1949**.
6. W. F. Berg, *Naturwiss.*, 1931, **19**, 391; *Z. Krist.*, 1934, **89**, 286.
7. C. S. Barrett, *Trans. Amer. Inst. Min. Met. Eng.*, 1945, **161**, 15.
8. R. W. K. Honeycombe, *J. Inst. Metals*, 1951–52, **80**, 39.
9. R. D. Heidenreich and W. Shockley, *Phys. Soc.: Rep. Conf. on Strength of Solids*, **1948**, 57.
10. A. F. Brown, *Metallurgical Applications of the Electron Microscope* (*Inst. Metals Monograph and Rep. Series* No. 8), **1950**, 103.
11. W. Boas and M. E. Hargreaves, *Proc. Roy. Soc.*, 1948, [A], **193**, 89.
12. W. G. Burgers and J. J. A. Ploos van Amstel, *Z. Physik*, 1933, 81, 43.
13. W. G. Burgers and P. C. Louwerse, *Z. Physik*, 1931, **67**, 605.
14. E. N. da C. Andrade and L. C. Tsien, *Proc. Roy. Soc.*, 1937, [A], **163**, 1.
15. C. S. Barrett, *Trans. Amer. Inst. Min. Met. Eng.*, 1939, **135**, 296.
16. E. Orowan, *Nature*, 1942, **149**, 643.
17. J. B. Hess and C. S. Barrett, *Trans. Amer. Inst. Min. Met. Eng.*, 1949, **185**, 599.
18. F. Röhm and A. Kochendörfer, *Z. Metallkunde*, 1950, **41**, 265.
19. R. Maddin, C. H. Mathewson, and W. R. Hibbard, Jr., *Trans. Amer. Inst. Min. Met. Eng.*, 1948, **175**, 86; 1950, **185**, 527.
20. E. Schmid and W. Boas, "Plasticity of Crystals with Special Reference to Metals." London: **1950** (F. A. Hughes and Co.).

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THE FLOW OF LIQUID METALS ON SOLID METAL SURFACES AND ITS RELATION TO SOLDERING, BRAZING, AND HOT-DIP COATING*

By G. L. J. BAILEY,† Ph.D., A.R.C.S., D.I.C., F.Inst.P., MEMBER,
and H. C. WATKINS,‡ L.I.M., MEMBER

(Communication from The British Non-Ferrous Metals Research Association.)

SYNOPSIS

Earlier investigations of soldering performance are reviewed and, after a theoretical discussion of factors expected to control the flow of liquids on solid surfaces, experimental investigations of the behaviour of several liquid metals and alloys in contact with various solid metals, are described. Most of the work was carried out using hydrogen as flux, but a few experiments with liquid fluxes are also described.

It is found that the contact angle between solid and liquid surfaces of the metals examined is not, in general, zero. Its true value in given circumstances is obscured by the effects of roughness of the solid surface, which cannot be allowed for. Roughness effects could also account for the fact that the contact angle against a surface over which liquid has receded is generally lower than that against a surface over which liquid has advanced. The development of the particularly low contact angles which are formed against copper by tin–lead alloys with compositions in the range preferred for practical soldering, appears to be preceded by the formation of an alloy layer in the surface of the copper over which the liquid metal spreads. The alloy layer may be formed relatively slowly by diffusion through the surface layers of the copper from the bulk of the liquid metal, or it may be formed relatively quickly as a result of transfer of metal ions through suitable liquid fluxes. The production of particularly low contact angles between lead and iron or copper and between tin and copper, through very small additions to the lead or tin, is described.

I.—INTRODUCTION

THE use of soft soldering has for many centuries afforded a ready means of joining common metals such as steel, brass, and copper, without the use of equipment other than means of heating the joint to a comparatively low temperature. The essential characteristic of soldering is the ease and rapidity of making joints which are adequate for their purpose, and the most natural ways of assessing a soldering operation are in terms of ease of application of the solder and suitability for service of the resulting product.

The mechanical properties of joints can be measured, and the effects of variations of joint design, of method of soldering, and of materials used thereby usefully compared. In making joints, however, the liquid metal is required to flow, to an extent depending on the design of the joint, and it is found that, whatever methods of soldering are used, some combinations of solder, flux, and solid metal surfaces have better flow properties than others.

Although readily recognized in practice, the flowing power or "solderability" of a solder/flux/stock system cannot be measured quantitatively. The causes of differences in "solderability" are not known, for the basic factors controlling flow are not well understood.

The British Non-Ferrous Metals Research Association first began to examine the properties of solders and soldered joints in 1922, and, since that time, has completed many investigations in this field. The most important conclusions of these researches are summarized in a monograph first issued in 1932 and subsequently, in a revised form, in 1942.[1] The imperative need for tin economy in the 1939–45 war stimulated much further work on low-tin or tin-free soft solders of which a description was recently published in another monograph.[2]

Much of the experimental data contained in these monographs is in the form of measurements of the mechanical properties of joints. In the earlier work, solders to British Standard No. 219 having tin contents in the range 30–65% were used. The war-time research was concerned largely with solders based on lead and containing small percentages of tin, antimony, and silver, either alone or in combination. Throughout, the effects on mechanical properties of

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† Deputy Research Manager, British Non-Ferrous Metals Research Association, London.

‡ Formerly Research Assistant, British Non-Ferrous Metals Research Association; now with Messrs. H. J. Enthoven and Sons, Ltd.

such variables as soldering temperature and time and joint design were of primary interest. In the work with conventional solders, sound joints were readily produced and the need for an investigation of the reasons for poor performance was therefore not felt, but war-time experience in the use of lead-rich solders containing less than 20% tin brought sharply into prominence the fact, already referred to, that marked variations existed between differing molten solder compositions in their readiness to flow when applied to solid metal surfaces, even when due allowance was made for differences in liquidus temperature. Difficulties were particularly evident in bit-soldering work and, since the reasons were not apparent, it was hard to foresee means of improving the performance.

At the end of the war, a study was started, on fundamental lines, of the factors of importance in making sound soldered and brazed joints. The initial stages of this research are described in the present paper.

As soon as the difficulties of using solders consisting substantially of lead became apparent, efforts were made to devise tests in which the power of a solder to fill a joint could be measured and its likely performance in practice readily assessed. Three such tests are described in the recent monograph,[2] in which, however, the significance of the results obtained is not discussed in detail. These tests are briefly described in the next section to illustrate the difficulties of explaining the results and assessing their practical value.

II.—TESTS OF SOLDERING POWER

1. Bit-Soldering Tests

Simple lap joints, 6 in. long, were made between strips cut from sheet material and clamped in contact. A gas-heated soldering bit, held at any desired temperature, was drawn at controlled rates along the lap which had been fluxed with zinc–ammonium chloride solution. These tests were intended to yield quantitative data on the penetration of solder into the lap joint, but "reproducibility was not generally adequate for this purpose, and the results had to be assessed on a purely qualitative basis". Special note was therefore taken of the external appearance of the joint, the penetration of the solder into and the soundness of the joint, and the ease with which the soldering bit could be tinned and maintained in a well-tinned condition.

Tests were made with joint members of blackplate and high-conductivity tough-pitch, and arsenical coppers, using a wide range of conventional and "tin-economy" solders. It was found that, even when allowance was made for the higher liquidus temperatures of the lead-rich solders by the use of higher bit temperatures, they did not adhere well to the bit and failed to form sound joints. Essentially, solders containing more than 30% tin gave good performance and those with less behaved poorly in application and gave badly filled joints.

2. Area-of-Spread Tests

The markedly poorer flow characteristics of the lead-rich solders stimulated a series of experiments in which the area to which a pellet of solder spread when melted under flux on a heated copper or steel plate was measured. It was felt that, while the factors promoting spreading were not understood, the direct measurement of area of spread produced under standardized conditions approximating to bit soldering might form a rapid and simple means of classifying solders according to their expected performance in practical bit-soldering applications. In carrying out these tests, small square plates of copper or steel were fluxed and carried in the centre a pellet of solder in the form of a disc 8 mm. in dia. and 0·1 ml. in volume. They were placed on a hot plate held at a controlled temperature. The solder began to melt in about 10 sec. and spreading had ceased within a further 50 sec. After not longer than $1\frac{1}{2}$ min., the plate was removed and cooled. No attempt was made to determine differences in the rate of spreading.

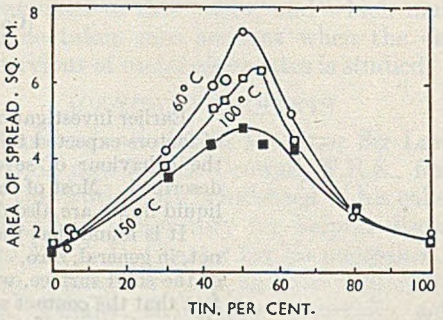

Fig. 1.—Spreading of Tin–Lead Solders on Copper With Zinc/Ammonium Chloride Flux at Various Degrees of Superheat.

The results obtained on bright, electropolished copper surfaces, with zinc/ammonium chloride flux, may be summarized as follows. The area of spread for tin–lead solders varied with composition as shown in Fig. 1, in which the curves correspond to different superheats. Silver additions up to 3% of the tin content did not influence the amount of spread obtained. Antimony additions up to 6% of the tin content gave a marked reduction in area of spread, proportional to the antimony content and amounting to about 50% in the 50% tin solder containing 3% antimony. This effect of antimony was reduced somewhat by the presence of silver.

When a 20% solution of resin in industrial spirit was used as flux in place of zinc/ammonium chloride, the areas of spread remained in the same relative order, but the larger areas of spread were very much reduced.

When, instead of clean electropolished copper, oxidized specimens were used, the spreads obtained using zinc/ammonium chloride were rather greater than on clean surfaces; with the resin flux, very small areas of spread were obtained.

Similar experiments on mild-steel sheet, with zinc/ammonium chloride flux, showed that tin–lead solders having tin contents of 20–50%, melted with 60° C. superheat, all spread to the same extent initially, the area of spread being about 3 cm.^{2}. With solders containing 20% and 30% tin it was noted that spreading occurred in two stages. In the first stage, spreading took place rapidly once the solder became fully molten and then appeared to cease altogether. Subsequently, after a period depending on the temperature of the test, spreading began again, but only where molten flux remained upon the surface. This spreading was slower and generally very irregular, but amounted in some cases to an increase of 100% in area.

As with copper, silver additions to the solder were without effect, whereas antimony additions reduced the spread considerably. Both with copper and, especially, with steel, the addition of up to 5% tin to a lead–silver solder containing 1·5% silver, gave notably large areas of spread; the 5% alloy on steel spreading to an area of 6 cm.^{2} at 360° C.

The applicability of the area-of-spread test to practice is not clear. Broadly, it may be stated that the solders most used industrially all show areas of spread greater than 3 cm.^{2} in tests made as described by McKeown, and that those preferred for critical bit-soldering applications show areas of spread, on copper, of more than twice this area under suitable conditions of superheat. The differentiation shown between fluxes is in agreement with practical experience.

3. Capillary-Penetration Tests

Observation of the performance of solders in practice suggests that the impression of ease of flow which they give may be connected with the rate at which they penetrate into joints. From measurements of the time taken for solder to traverse a horizontal capillary space 4½ in. long × 1 in. wide and with a depth of either 0·0035 in. or 0·006 in., Latin[2, 3] was able to calculate an " effective penetration coefficient ", characteristic of the solder/stock/flux combination at a chosen temperature. He compared this with the " ideal penetration coefficient " which he thought would be obtained if the solder wetted the solid metal perfectly.

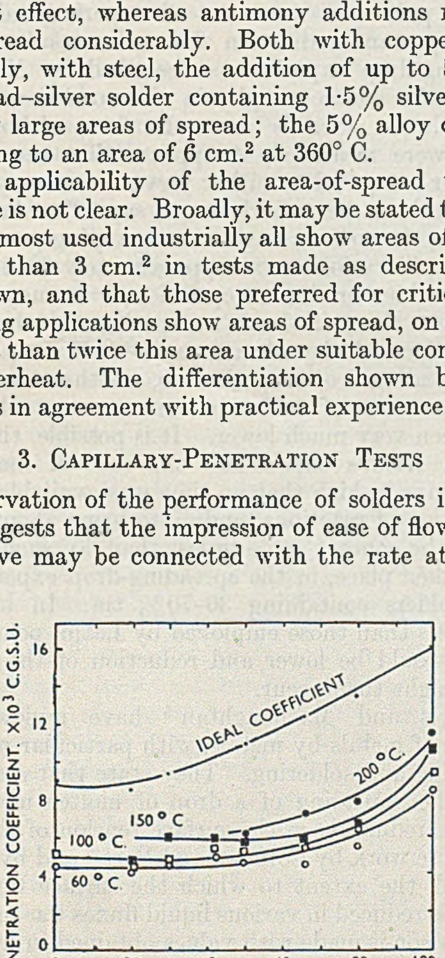

Fig. 2.—Penetration of Tin–Lead Solders in Copper Joints at Various Degrees of Superheat.

Latin's tests were made using joint members of copper and tinplate, with zinc–ammonium chloride and resin fluxes and with a wide range of solders. He found that, between copper surfaces, the highest effective penetration coefficient was obtained when the liquid metal was pure tin and that its value for any solder composition was always approximately half that of the corresponding ideal penetration coefficient. The effective penetration coefficient increased with increasing superheat, remaining at about half the ideal value, but was insensitive to the presence in the solder of additions of antimony, silver, and copper, and was the same whether zinc–ammonium chloride or resin flux was used. These results are shown in Fig. 2.

With tinplate surfaces, the penetration coefficient reached the ideal value when solders with tin contents in the range of 40–60% tin were used, but it fell to half the ideal value with solders containing less tin. No experiments on tinplate were reported with solders containing more than 63% tin.

The results of these tests are at variance with practical experience, which is that solder containing about 50% tin have the most useful flow characteristics, and that flow depends markedly on the flux used.

III.—FACTORS CONTROLLING THE FLOW OF LIQUIDS UPON SOLID SURFACES

1. Surface Tension and Contact Angle

In attempting to reconcile the results of the area and penetration tests with each other and with the results obtained in practice, it is necessary to consider the factors which control the flow of liquids in contact with solid surfaces. A liquid will flow under the action of forces to which it is subjected in an attempt to reach a position of equilibrium; the forces of particular importance in soldering, brazing, and hot-dip coating are those due to gravity and to surface tension. The surface-tension forces acting on the liquid metal are characterized by the value of the surface tension of the liquid metal and by the angle of contact between the liquid and solid surfaces at the line in which they meet.

The general features of the rate of flow and the equilibrium conditions under the combined influence of gravitational and surface-tension forces are most easily appreciated by considering that both the surface tension of a liquid and its contact angle against a solid surface remain constant. In these circumstances it is possible to examine flow and equilibrium theoretically in a number of examples of interest. The results of such examination are set out briefly in Appendix I. It will be noted that not every example can be given a completely satisfactory treatment. For instance, the relationship between the area of a drop of liquid on a solid surface, the surface tension of the liquid, and the contact angle is not simple unless the drop is small enough for gravitational distortion to be negligible. When gravity can be neglected, however, the drop has the shape of a spherical cap; the area of spread is independent of the surface tension of the liquid, except in so far as this is one factor in determining the contact angle, and is connected in a relatively simple way with the value of the contact angle. Again, neither the rate at which a drop spreads or retracts to gain its equilibrium state, nor the rate at which a meniscus is formed when a solid surface is dipped into a liquid are at present calculable. For capillary spaces, however, both the equilibrium state and, with a good approximation, the rate of penetration, are calculable in all circumstances.

2. SIGNIFICANCE OF THE CONTACT ANGLE

Even the most casual observation of the flow of a liquid metal on a solid metal surface suggests that the contact angle is not constant and characteristic of the system. A discussion of the factors which determine its value is given in Appendices II and III. Briefly, Appendix II shows that if the solid surface is perfectly smooth, the contact angle which will be observed depends upon the value of the surface tension of the liquid (γ_{23}) and on the values of two other surface tensions, attributable respectively to the solid surface where it is not covered by the liquid metal, (γ_{13}), and to the solid metal/liquid metal interface (γ_{12}).

In studies in fields other than those concerned with liquid metals,[4] it is almost always found that the contact angle of a liquid which has spread outwards over a smooth solid surface exceeds the contact angle observed after the liquid has retracted from the surface. This difference between the values of "advancing" and "receding" contact angles is generally attributed to the adsorption of molecules of liquid upon the solid surface and is known as "hysteresis of the contact angle". To distinguish such absorption hysteresis from hysteresis due to surface roughness, which is discussed below, it is proposed to employ the term "molecular hysteresis".

Unless the solid surface is perfectly smooth, the contact angle which is observed differs from the "true" contact angle, discussed in Appendix II, in a way which depends upon the surface roughness. The influence of roughness is discussed in Appendix III, in which it is shown that the contact angle observed after a liquid has spread outwards over a rough surface can exceed the angle observed after the liquid has receded over the surface. This effect may be described as "capillary hysteresis" to indicate the essential difference in its origin from "molecular hysteresis".

3. EFFECTS OF DIFFUSION BETWEEN SOLID AND LIQUID METALS

The discussion of the previous section indicates that, in a given system comprising liquid metal, flux, and solid metal, provided the three surface tensions referred to and the roughness of the solid surface remain constant, values of the advancing and receding contact angle observed immediately the liquid has come to rest will be characteristic of the system and of the roughness of the solid surface. In fluxed metallic systems no such characteristic contact angles will necessarily be obtained, however, since intermetallic diffusion normally occurs, and in consequence both the roughness and the composition of the solid surface changes. Chemical and electrochemical action of the flux, and its evaporation and decomposition may also contribute to these changes.

4. REVIEW OF EARLIER EXPERIMENTAL WORK

This discussion throws some light on the results of several published accounts of experimental work. One important indication from the results of the spreading-drop experiments described by McKeown is that the contact angle, in the conditions of the tests, always exceeds zero. In fact, although no values were measured, it appears that the contact angle for compositions either very poor or very rich in tin must have been fairly large, say 60°. According to Latin, the contact angle accompanying penetration in his capillary joints was approximately 60° irrespective of solder or flux compositions. It may readily be shown that, in Latin's experiments, the lowest penetration velocity always exceeded 1 cm./sec. In the spreading-drop experiments, on the other hand, the mean rate of solder movement must obviously have been very much lower. It is possible, therefore, that in Latin's experiment the rate of movement was always so high that no time was available for the completion of reactions tending to bring about reduction of the contact angle to less than 60° such as may have taken place, in the spreading-drop experiments, with solders containing 30–70% tin. In narrower capillaries than those employed by Latin, penetration speeds would be lower and reduction of the contact angle might then occur.

Daniels and Macnaughtan[5] have reviewed the wetting of metals by metals, with particular reference to tinning and soldering. They state that a flux will assist the spreading of a drop of molten metal if it causes a reduction in the surface tension of the drop, and quote work by Coffman and Parr[6] and by Latin[7] in which the extent to which the surface tension of solders is reduced in various liquid fluxes was measured (comparison is made with values obtained in hydrogen or *in vacuo*). Daniels and Macnaughtan later point out that, with a given contact angle, joint penetration is greater, the higher the surface tension of the liquid, and they find some difficulty in reconciling the facts that an effective flux promotes both spreading and joint penetration. However, the area of spread of a drop is determined by the contact angle alone, i.e. indirectly by the values of three surface tensions, whereas joint penetration is determined by the product $\gamma_{23} \cos \theta$. Since $\gamma_{23} \cos \theta = \gamma_{13} - \gamma_{12}$, it is evident that joint penetration is determined directly by the difference in surface tension of two solid surfaces.

Chalmers and Wadie,[8] in investigating the dewetting of tin and some tin alloys from hot-dipped O.F.H.C. copper, used the receding contact angle as a measure of the tendency to dewet and discussed the influence of the surface tension of the liquid and their experimental variables upon the measured receding contact angles. They derived Wenzel's expression for the effect of surface roughness upon contact angle (see Appendix III), but were unable to take roughness into quantitative account. In consequence, while they showed that the contact angle cannot depend only upon the value of the liquid surface tension, they were unable to decide whether the other factors causing contact angle variation were either roughness variations or variations in the two solid surface tensions, or a combination of these three variables. They added that the use of a flux which reduces the liquid surface tension will reduce the contact angle, but this involves the assumption that the other two surface tensions remain unaltered.

Daniels and Macnaughtan,[5] also discussing the dewetting of tin from oxygen-bearing copper, state that small additions of copper, nickel, or cobalt to the tin prevent collapse of the tin coatings. They account for this effect by supposing the viscosity of the coating to be increased by the small additions. It is improbable that any important change of viscosity is here involved, and it seems more likely that changes in roughness or true contact angle may provide an explanation.

In the course of his experiments designed to examine whether fluxes, besides acting as cleaning agents, also considerably lower the surface tension of the molten solder, Latin [7] noted that, around tin and solder drops heated in contact with copper in zinc–ammonium chloride flux, blue-coloured areas were obtained. He concluded that this blue coloration was due to tin which had been deposited from the flux and had subsequently alloyed with the copper. This action, he considered, might account for the greater spreading observed in zinc–ammonium chloride flux in comparison with that in resin flux. From a comparison of areas of spread of tin and eutectic tin–lead solder in either flux he deduced that the greater spreading of the eutectic solder was probably a consequence of its lower surface tension. If this were the only reason for greater spreading of the eutectic composition, it might be expected from measurements of surface tension in resin made by Chalmers and Wadie,[8] that the best spreading of all would be obtained with pure lead. Latin gives no evidence that this is so, and the spreading-drop results quoted by McKeown indicate that in fact lead does not spread as well as solders containing about 50% tin. The formation of "haloes" around drops of solder when a chloride flux is used, as observed by Latin, was also noted by Fine and Dowdell,[9] who suggested the addition of stannous chloride or lead chloride to zinc chloride fluxes used in the tinning and soldering of steel.

Daniels and Macnaughtan [5] point out that the rate of spreading of drops of solder upon solid surfaces has not been studied. However, Tammann and Arntz [10] and Alty and Clark [11] measured the rate of spreading of mercury on a number of solid metals at room temperature, in air and under several liquids. In similar experiments using mercury, copper, and stannic bromide solution at temperatures above atmospheric, Daniels and Macnaughtan [5] found the rate of spreading suddenly to increase greatly above a critical temperature. They refer to the discovery by Latin of such a critical temperature for eutectic tin–lead solder on copper under molten zinc–ammonium chloride. It must be that at the "critical temperature" some reaction occurs which rapidly changes one or more of the factors determining the value of the contact angle.

Even when no liquid flux phase was present, and electrochemical reaction was therefore impossible, Tammann and Arntz [10] and Alty and Clark [11] noted the appearance of a band surrounding the drop of mercury in the surface of the solid metal. This band could be formed only as a result of the diffusion of liquid metal in the surface layers of the solid.

Daniels and Macnaughtan comment that it is unknown whether the formation of a "halo" (which it seems clear may take place by diffusion from the liquid metal or by deposition from some liquid fluxes) always accompanies the spreading of a drop of molten metal on a solid metal, or if it is an indispensable preliminary to the spreading of the main drop.

It will be clear that there is room for further experimental study of contact angles in soldering systems and of the factors which influence them. Such information may well have a bearing on the assessment of "solderability" in practice, even though the significance of this property cannot yet be appreciated.

IV.—EXPERIMENTAL INVESTIGATIONS ON THE TIN–LEAD/COPPER SYSTEM

1. Observations of the Spreading of Drops of Liquid Metal

The possibility of changes of contact angle taking place as a consequence of intermetallic diffusion [10, 11] has been pointed out. When a liquid flux is used, there is the further possibility of changes due to chemical and electrochemical reactions. Flux effects of this kind can be eliminated by studying the behaviour of liquid metals in contact with solid metal surfaces freed from oxide in a reducing atmosphere. It was considered that spreading-drop experiments, carried out using hydrogen as a flux, should be made to ascertain if changes in contact angle occurring only as a result of diffusion can in fact occur in the tin–lead/copper system and if so, in what circumstances.

In view of their exploratory nature, the precision of measurement aimed at in these tests was not high; instead, a procedure was adopted which would allow a large number of observations to be made quickly.

(a) *Apparatus*

The furnace used is shown in Fig. 3. Specimens of copper sheet rested on the furnace baseplate.

Just above the baseplate was a loose-fitting silica rod, with a spoon-shaped cavity ground at its end to hold a bead of liquid metal which could be gently dropped on to the copper specimen by rotating the rod. Spreading was observed through a silica window having a water-cooled wax seal, which was broken for insertion and removal of specimens. To aid in the estimation of drop diameters at any instant, a square of wire gauze was usually attached to the silica spoon in such a way that it could be rotated to lie over the drop. The drop diameter was estimated by counting the meshes of the gauze. The furnace was levelled by means of adjustable feet. In the experiments to be described, the furnace atmosphere was cylinder hydrogen which had been purified by passage over heated platinized asbestos and through a series of drying tubes.

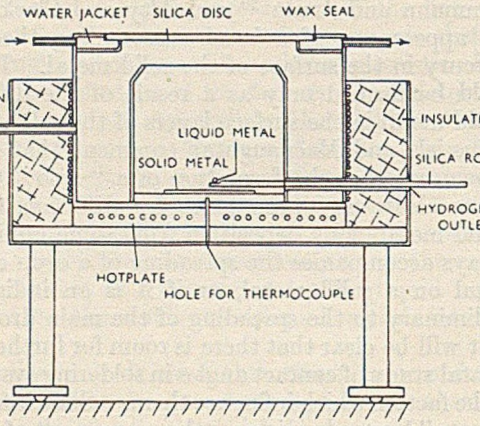

Fig. 3.—Sectional Diagram of Furnace for Spreading-Drop Experiments.

(b) *Materials and Procedure*

The copper used was oxygen-free, high-conductivity strip, 0·015 in. thick. Specimens $1\frac{1}{2}$ in. square, were cathodically degreased, annealed in hydrogen for 1 hr. at 600° C., flattened, and again degreased before insertion in the furnace. Pellets of high-purity tin and lead and of a range of alloys made up from them were cut and weighed to have a volume of 0·05 ml. A pellet of the required composition was loaded into the spoon. After the furnace had been sealed, hydrogen was passed for 15 min. to sweep out air and the temperature raised to 600° C. for a further 15 min. to complete the cleaning of the materials. The temperature was then adjusted to the required value and the drop of molten metal applied to the copper surface. Estimates of the area of spread were made at intervals over a period of 30 min.

(c) *Results*

It was found that, with all the liquid alloys examined, a well-defined drop was formed immediately the liquid came into contact with the copper. In every case, the diameter of the drop was in the range 10–12 mm. With alloys of tin content less than 30% or exceeding 70%, the shape of the drop did not change at all, or changed only slightly, during the 30 min. after its formation at any temperature between the liquidus of the alloy and 500° C. Within the composition range 30–70% tin, however, an appreciable and, in some cases, considerable increase of drop diameter occurred during this time. The extent of this secondary spreading was found to depend strongly on the composition of the alloy and on the temperature of the test.

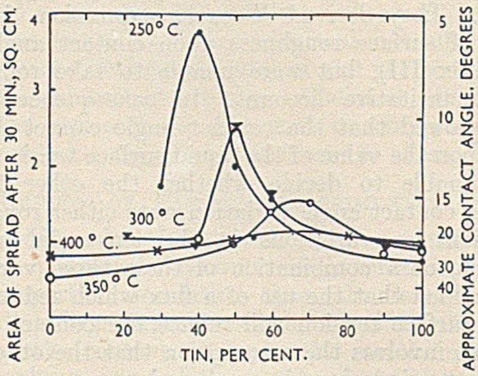

Fig. 4.—Effect of Tin Concentration on Spreading of Tin–Lead Alloys on Copper in Hydrogen Atmosphere at Different Temperatures.

The results are shown in Figs. 4 and 5. From Fig. 4 it is seen that a drop of 40% tin alloy spread in 30 min. to an area of about 4 cm.^{2} when the temperature was 250° C. The initial area of all drops at all temperatures was roughly 1 cm.^{2}. Fig. 5 shows that a drop of 40% tin solder, at 250° C., was still increasing in diameter at an approximately constant rate after

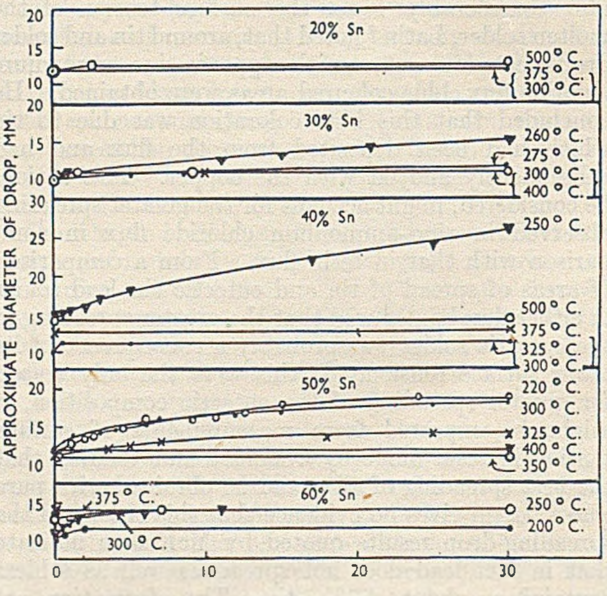

Fig. 5.—Effect of Temperature on Rate of Spreading of Tin–Lead Alloys on Copper in Hydrogen Atmosphere.

30 min., the average rate of movement of the liquid being 0·2 mm./min. At higher temperatures, a lower maximum was reached in 30 min. with alloys containing a greater percentage of tin, as shown in Fig. 4. The 30% tin alloy was still spreading at a comparatively low rate, after 30 min. at 260° C. (Fig. 5), but in the other cases a steady state had apparently been reached. Fig. 5 also illustrates the generally observed fact that when no marked secondary spreading took place, the area of drops was slightly greater at higher temperatures. This effect was examined over the temperature range 350°–950° C. for the lead/copper system. The contact angle of a drop of lead was found to decrease as the temperature was raised and to increase again as it was lowered, approximately as shown in Fig. 6, in which the contact angle is plotted against temperature. The fuller investigation of this effect in which the surface tension of the solid surface and that of the solid/liquid interface were measured is described elsewhere.[12]

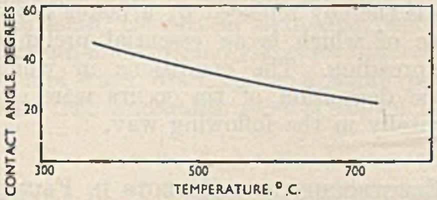

Fig. 6.—Change of Contact Angle with Temperature in the Lead/Copper System.

According to the composition of the liquid alloy and the temperature of experiment, it was possible to classify the spreading characteristics into three different categories :

(A) Marked secondary spreading, to areas greater than are obtained with the same composition at higher temperatures.

(B) Slight secondary spreading, to areas less than or equal to those obtained with the same composition at higher temperatures.

(C) No secondary spreading.

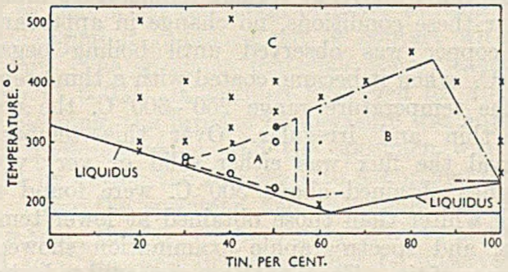

Fig. 7.—Spreading of Tin–Lead Alloys on Copper in Hydrogen Atmosphere.

The temperature/composition fields corresponding to these three classifications are drawn in Fig. 7. It will be noted that the region of marked secondary spreading includes a large part of the composition range within which the solders preferred in practice are found.

Two further observations of interest were made during the experiments. It was noted that when a drop of, for example, 40% tin alloy, applied at a temperature above 300° C. and therefore about 10 mm. in dia., was cooled slowly, it began to spread rapidly at the lower temperatures. A further feature frequently noted, particularly with alloys containing 30% tin or more, was the formation of a bright band in the copper surface immediately surrounding a drop. With compositions and at temperatures where slow spreading occurred, this band did not appear until the spreading became slower. Some observations on the character of these bands are made later in the paper (Section VI, 1 and 2).

2. Area of Spread and Contact Angle

As is pointed out in Appendix I, only if a drop is sufficiently small in volume is the area of spread related in a comparatively simple way to the contact angle. When the drop is so small in volume that gravitational effects are negligible it has the form of a spherical cap. The full curve of Fig. 8 shows the relationship between the diameter of the drop and the contact angle for a spherical cap of the volume used in these experiments. The points plotted in this figure show the measured diameter and contact angle of a number of drops of this volume. Contact angles were measured by an optical arrangement similar to that described by Chalmers and Wadie.[8] Various liquid and solid metals were used in obtaining these results, but the details are not of importance for the present purpose. It will be seen that the plotted points lie fairly close to the full curve of Fig. 8, and it is permissible therefore to derive an approximate value of the contact angle from the drop diameter or area of spread. Accordingly a scale for contact angle has been added to Fig. 4. It should be added that the measured height of the drops was in general appreciably less than it would have been if they were in fact spherical caps.

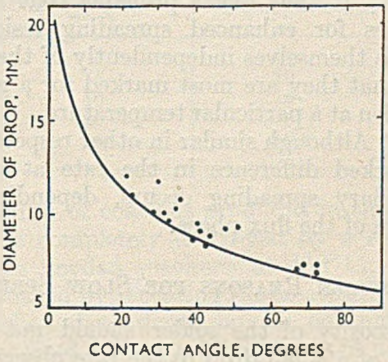

Fig. 8.—Relation of Drop Diameter and Contact Angle.

It will be noted from Fig. 4 that the secondary spreading undergone by certain of the alloys at appropriate temperatures corresponds to a reduction of the contact angle from the range 20°–30° down to 10° or less.

3. CONCLUSIONS FROM SPREADING-DROP EXPERIMENTS

The following conclusions may be drawn from the results of the simple experiments described above:

(i) The contact angle of tin, lead, and tin–lead alloys against a hydrogen-cleaned copper surface is not zero over the temperature range explored.

(ii) The enhanced spreading and consequent changes of contact angle obtained with certain compositions and temperatures are similar in extent to those found in spreading-drop tests using zinc–ammonium chloride flux, as comparison of Figs. 1 and 4 shows. In both sets of experiments, enhanced spreading is less marked at the higher temperatures. It may be noted that when, in the earlier tests, resin was used as flux, the same tendency to enhanced spreading was observed, though in this case its extent was slight. It thus seems probable that the potentialities for enhanced spreading reside in the metals themselves independently of the flux used and that they are most marked for a given composition at a particular temperature.

(iii) Although similar in other respects, there is a marked difference in the rate at which this secondary spreading occurs, depending on the nature of the flux phase.

4. POSSIBLE REASONS FOR SLOW SPREADING

The viscosity of the solder should not limit the spreading of a small drop to the rate observed during enhanced spreading in hydrogen unless the liquid became loaded with solid particles of intermetallic compounds. No evidence was found (e.g. by the exposure of particles in drops which had spread to large areas and become very thin) that this occurred, and it was concluded that such spreading is therefore due to a change in the conditions determining the contact angle, the rate of change controlling the rate of spreading. As has been pointed out earlier, a change of roughness or of composition, or a combination of these effects, may be expected to affect the contact angle. Whatever the detailed reason for the change of contact angle, it must certainly be associated with the occurrence of intermetallic diffusion between the liquid and solid metals.

If the surfaces are not perfectly clean in these experiments, the effect of diffusion may be merely to disperse contamination from the solid surface, allowing the drop to spread. If this is the only reason, it is difficult to see why pure tin should spread less well than some of the tin–lead alloys.

If, however, oxide films and gas-absorption effects are absent in the experiments in hydrogen, it can be concluded that, in the experiments using zinc–ammonium chloride flux, apart from any action in removing oxide film and absorbed gas from the solid surface, the flux must also have some other action which leads to rapid secondary spreading. It has been suggested by Latin [7] that tin ions from the solder are taken into solution and re-deposited electrolytically on to the copper surface. The significance which may now be attached to this observation is that the slow diffusion from the edge of the drop is thereby replaced by a faster process, the occurrence of which is an essential preliminary to further spreading. The conditions in which such electrolytic deposition of tin occurs were examined experimentally in the following way.

5. ELECTROCHEMICAL EFFECTS IN FLUXES

Experiments were carried out with the object of determining at what stage of heating zinc–ammonium chloride flux the deposition of metallic ions could occur as a result of chemical or electrolytic reaction. The flux was contained in a small crucible furnace. At the bottom of the crucible was a thin layer of tin, lead, or tin–lead alloy into which dipped the exposed tip of an insulated connection. The procedure adopted was to insert a strip of copper into the flux at a chosen temperature, taking care to avoid contact with the metal at the bottom of the crucible. The copper strip was then connected externally directly to this metal layer and withdrawn without breaking contact after 10 sec. A fresh strip of copper was used for each immersion. The copper was cathodically degreased and pickled in a mixture of acids before use.

A plain zinc chloride flux, consisting of zinc chloride 50 g. and water 50 ml., was slowly heated to 400° C. over a period of 2 hr., a copper electrode being inserted as described above at regular temperature intervals. Under these conditions, no change in appearance of the copper was observed until boiling began at 110° C., when it became coated with a thin grey film. In the temperature range 250°–300° C. the coating was thin and irregular. Over this temperature interval the flux was either solid or very viscous. Coatings obtained above 300° C. were found to be much whiter than those obtained at lower temperatures, and spectrographic examination showed the presence of tin. These coatings appeared to be brittle, and it was thought probable that the tin was alloyed with the copper. A similar result was obtained when an addition of 5 g. ammonium chloride was made to this flux, and it was noted that the flux retained its fluidity throughout the experiment. When the tin was replaced by a 50% tin–lead alloy similar deposition was observed. Deposition was observed only when the external circuit between the copper and the liquid metal was completed. These experiments provide evidence that tin from a solder may be deposited on to a copper surface by electrolysis when a zinc or zinc–ammonium chloride flux is employed. Such deposition might well account for the far higher rate of spreading which must occur when such a flux is used in comparison with the rate in hydrogen.

6. FLOODING EXPERIMENTS

Although no detailed explanation of the way in which diffusion controls the rate of spreading of a drop in a hydrogen atmosphere is yet possible, the fact that it does so makes it important to obtain further information on diffusion characteristics. Useful results in this connection were obtained in experiments in which copper surfaces, cleaned in hydrogen, were covered with tin, lead, and tin–lead alloys for different lengths of time and then drained for a short period and observed. The apparatus used is shown in Fig. 9.

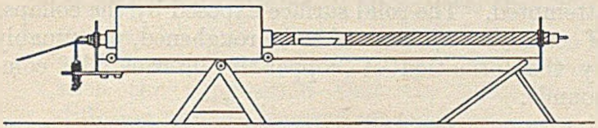

FIG. 9.—Apparatus for Flooding Tests.

It consisted essentially of a horizontal tube furnace which could be tilted through about 15°, and which was so arranged that the specimen could be examined visually during an experiment. A silica tube with a short central transparent insert was mounted concentrically within a horizontal tube furnace capable of operation at temperatures up to 1000° C. The furnace was carried on rails so that it could be withdrawn from the transparent section of the central tube, within which the specimen could thus be observed at will. The specimen was a narrow strip of metal foil, pressed on to the bottom of a silica boat which contained, in addition, a small piece of the more fusible metal, placed at one end, out of contact with the foil. When making an experiment, the furnace was tilted so that when the metal of lower melting point became liquid it remained out of contact with the solid foil. The specimen materials were cleaned by heating to 600° C. in purified hydrogen for 15 min. After cleaning, the furnace temperature was adjusted to the required value, and the specimen was then flooded by tilting the furnace into a horizontal position. After the specimen had been immersed for a chosen time, the furnace was again tilted to allow the liquid to drain, and draining could be observed by displacing the furnace from the window of the central tube. This was normally done 15 sec. after the beginning of draining. The draining angle was small, and the specimen cooled rapidly after removal of the furnace, and, in consequence, the apparatus was not operated in such a way as to provide a severe test of coating stability, the need for which was not appreciated at that time.

The solid surface was classified by observation after draining as follows:

(*a*) Wetted surface: covered by a smooth, continuous liquid coating.

(*b*) Dewetted surface: drops of liquid, formed by collapse of coating, adhering to the surface.

(*c*) Unwetted surface: no liquid adhering as a film or as drops to the solid surface.

It was found that copper was unwetted by lead after immersion for 2 min. at 400° C. and also after 24 hr., at the same temperature. Experiments with tin–lead alloys containing tin in the range 2–50% were made at temperatures of 380° C. and 450° C. (below and above the decomposition temperature of the copper–tin η-phase, Cu_6Sn_5). It was found that, whilst a coating was in all cases obtained after flooding for the shortest practicable time (about 10 sec.), it was unstable and broke up rapidly on draining, exposing a matt alloy layer on the surface of the copper foil. The stability of the coating was not improved by increasing the flooding time at 450° C., but, at 380° C., stability, within the limitations of the apparatus as mentioned above, was obtained by sufficiently prolonged immersion. The results of an assessment of the time required to develop a stable coating are shown in Table I.

TABLE I.—*Time Required to Produce Stable Coating on Copper at* 380° *C.*

Tin Concentration, wt.-%	Time Required, min.
2	60
5	20
10	6
20	5
50	<0·3

The stability of coatings produced in this way was found to be completely destroyed by a short treatment of the flooded specimen at 450° C., but could be re-established by repeating the treatment at 380° C.

The marked dependence on tin concentration of the time required for the establishment of a relatively stable coating is noteworthy, and it may be suggested that, in the spreading tests in hydrogen, the rate of diffusion outwards from the drop might be extremely slow with solders containing less than 30% tin and thus not be observed in the 30 min. period allowed. Correspondingly, in the spreading tests using zinc–ammonium chloride flux, the deposition of tin might well fail to occur when low-tin solders are used. It may be suggested that if tin ions could be incorporated in the flux and be deposited electrolytically, enhanced spreading should be obtained even with low-tin or tin-free solders.

7. IMMERSION AND DRAINING EXPERIMENTS

Use of the tilting furnace just described did not conveniently allow the examination of effects produced during immersion periods of less than about 10 sec. In addition, the method failed to give as much information as was required on the approximate values of contact angle. An alternative method was therefore followed.

The object of these experiments was to observe:

(*a*) The shape of the liquid meniscus formed against a vertically dipped, smooth, bright copper surface during slow immersion and while held immersed in a fixed position for a few seconds.

(*b*) The shape of the meniscus during withdrawal from the liquid and the permanence of any liquid coating adhering to the withdrawn surface over a period of 30 sec., during which it was held vertically and maintained at the immersion temperature.

Tests were made with tin, lead, and a range of tin-lead alloys at temperatures up to 500° C., in hydrogen at atmospheric pressure, using the furnace shown in Fig. 3, in which a silica crucible was stood upon the furnace baseplate and a suitably bent strip of copper foil was attached to the rotatable silica rod.

It was found that the meniscus shape could be characterized by four contact angles as follows:

(i) ϕ_I: the immersion contact angle, accompanying slow immersion of the solid surface.

(ii) ϕ_A: the advancing contact angle, lower than ϕ_I, assumed while the specimen was held immersed at a fixed depth.

(iii) ϕ_W: the withdrawal contact angle, usually lower than ϕ_A, accompanying slow withdrawal of the surface.

In most cases, the withdrawal contact angle was zero, i.e. a coating was withdrawn. This coating usually broke up into drops, and observations were made of a fourth angle.

(iv) ϕ_R: the receding contact angle, less than ϕ_A, typical of the "dewetted" drops formed on the collapse of a uniform coating.

It will be noted that two of the angles, ϕ_A and ϕ_R are "equilibrium" angles, whereas the other two, ϕ_I and ϕ_W, are unstable and change either when the immersion rate becomes zero or after the surface is withdrawn completely.

The magnitude of the angles was estimated by eye. To supplement these observations, the withdrawn surface could be described, at any stage after withdrawal, as wetted, dewetted, or unwetted. It was found that, with all liquid compositions and at all temperatures, the immersion angle, ϕ_I, was approximately 90° and that this angle fell, very rapidly at the higher temperature and tin contents, to an advancing angle, ϕ_A, of 40°–60°, as soon as the movement of the specimen stopped.

With tin and the tin-rich alloys the end of the fall from ϕ_I to ϕ_A was accompanied by the appearance of a band in the copper surface immediately above the meniscus, indicative of diffusion of the tin through the surface layers of the copper. With pure lead as liquid, the withdrawal angle ϕ_W was estimated at 30° and no coating was formed. The copper surface remained smooth during immersion. When liquids containing 20% tin or more were used, the withdrawal angle was invariably zero, i.e. a coating was obtained, but the permanence of the coating depended on the composition of the liquid alloy and on the temperature. Thus, coatings formed at 250° C., using a 45% tin alloy were classified as stable. A coating formed at 250° C. using a 60% tin alloy broke up slowly into separate drops. Coatings formed with a 45% tin alloy at 350° and 500° C., with a 60% tin alloy at 350° C., and with a 70% tin alloy or pure tin at temperatures in the range 250°–500° C. collapsed within two or three seconds of their withdrawal from the melt. The receding contact angle, ϕ_R, was always in the range 5°–10°. With liquid alloys containing 20 and 30% tin a more stable coating could be obtained by repeated immersion. With alloys of high tin content, however, the copper sheet was completely dissolved if this was attempted. The solid surface exposed by the collapse of coatings was observed to be roughened, presumably by the formation of copper-tin intermetallic compounds.

8. CONCLUSIONS FROM COATING EXPERIMENTS

The changes occurring in these coating experiments are evidently of great complexity, and it is difficult to describe them with precision on the basis of the simple observations which were made. These observations do, however, enable the following suggestions to be put forward:

(*a*) Unless the receding contact angle, ϕ_R, is less than about 20°, no coating can be withdrawn. When the receding contact angle is less than 20° a coating can be obtained, but such coatings exhibit varying degrees of stability. The degree of stability is indicated by the time which elapses after withdrawal before the coating is seen to break from a continuous film and coalesce into separate drops. It is also indicated by the area of uncoated surface relative to that covered by drops after coalescence. The lower the value of the receding contact angle, the higher is the stability of the coating.

(*b*) Coating stability increases with time of immersion only when some tin is present. This is clear for alloys containing tin in the range 1–50%. With more than 50% tin present in the lead, it may be presumed that the time required to develop the lowest value of receding contact angle is too short to be measurable. These observations indicate that formation of an alloy layer is required to enable the lowest value of receding contact angle to be developed. The reversible "wetting–dewetting," produced by heating at 380° and 450° C. supports this view and furthermore, indicates that the solid surface with which the lowest receding contact angles are associated must be the η phase, Cu_6Sn_5, rather than the ϵ phase Cu_3Sn.

(*c*) It appears that coatings having the highest stability of any are obtained only with those solder compositions and in those temperature ranges for which marked secondary spreading is found in the spreading-drop experiments. It is possible that similar highly stable coatings may be produced with solders of lower tin content than about 30% if sufficient time of immersion is allowed.

(*d*) There is evidence of marked contact-angle hysteresis. Except for lead, the receding contact angle, after a sufficient immersion time, is never more than about 10°, even with the least stable coatings. On the other hand, the advancing contact angle, determined in spreading-drop experiments, is not less than 30°, except in the cases of marked secondary spreading, for which the receding contact angle must be close to zero.

(*e*) The observation that the contact angle accompanying immersion, ϕ_I, exceeds the advancing contact angle, ϕ_A, to which ϕ_I falls when the specimen becomes stationary, is in agreement with the supposition that the occurrence of diffusion reduces the contact angle.

9. Summary of Main Conclusions on Contact Angles in the Copper/Tin–Lead/Hydrogen System

The main conclusions to be drawn as a result of the qualitative survey of contact angles in the copper/tin–lead/hydrogen system are as follows:

(*a*) There is no single value of contact angle characteristic of the liquid metal composition and temperature.

(*b*) The contact angle which is observed depends on the extent to which diffusion has occurred. This is evident in different ways according to whether the advancing or receding angle is examined.

(*c*) The experiments provide no evidence on the relative contributions of change of roughness and change of surface structure to the changes of contact angle consequent upon diffusion.

(*d*) Very low values of both advancing and receding contact angles may be obtained with certain liquid alloy compositions within limited temperature ranges.

V.—EXPERIMENTAL OBSERVATIONS ON OTHER METAL SYSTEMS

It was of interest to compare the observations made upon the tin–lead/copper system with similar observations on other combinations of liquid and solid metals. Qualitative experiments, on the lines already described, are briefly reported below for several other combinations of solid and liquid metals.

1. Flooding Experiments

Flooding experiments, as described in Section IV, 6, were carried out with 38 pairs of pure metals selected from the following:

Liquid metal: Ag, Sb, Te, Zn, Pb, Cd, Bi, Sn, Al.
Solid metal: Fe, Ni, Cu, Au, Ag.

In 25 of the 38 pairs, it was expected that, at the temperature of the test, an intermetallic compound would be formed at the interface between solid and liquid metal and in all these cases wetting was observed. Of the remaining pairs, seven were examples in which the solid could take the liquid metal into solution. Here again, the result was in all cases classified as "wetting." In the six remaining pairs,

Table II.—*Summarized Results of Flooding Tests.*

System	Temp., ° C.	Result
(a) *Compounds Formed Between the Two Metals.*		
Fe–Al	700	Wetting
Fe–Sb	700	Wetting
Ni–Sb	700	Wetting
Cu–Sb	600	Wetting †
Ag–Sb	550	Wetting †
Fe–Te	500	Wetting
Ni–Te	500	Wetting
Cu–Te	400	Wetting †
Ag–Te	500	Wetting
Fe–Zn	500	Wetting
Ni–Zn	500	Wetting
Cu–Zn	500	Wetting
Au–Zn	450	Wetting
Ag–Zn	500	Wetting
Au–Pb	400	Wetting
Ni–Cd	400	Wetting
Cu–Cd	350	Wetting
Au–Cd	350	Wetting
Ag–Cd	400	Wetting
Ni–Bi	400	Wetting
Fe–Sn	400	Wetting
Ni–Sn	400	Wetting
Cu–Sn	400	Wetting
Au–Sn	275	Wetting
Ag–Sn	300	Wetting
(b) *No Compounds Formed: the Solid Metal Can Take the Other into Solution.*		
Ni–Ag	1000	Wetting
Cu–Ag	850	Wetting †
Au–Ag	1000	Wetting
Ni–Pb	(Fig. 10)	Wetting *
Ag–Pb	400	Wetting
Au–Bi	300	Wetting *
Ag–Bi	360	Wetting
(c) *No Compounds or Solid Solutions Formed.*		
Fe–Ag	1000	Dewetting
Fe–Pb	400	Non-wetting
Cu–Pb	400	Dewetting
Fe–Cd	400	Non-wetting
Fe–Bi	400	Non-wetting
Cu–Bi	400	Dewetting

* A time factor was detected. † Using eutectic liquid.

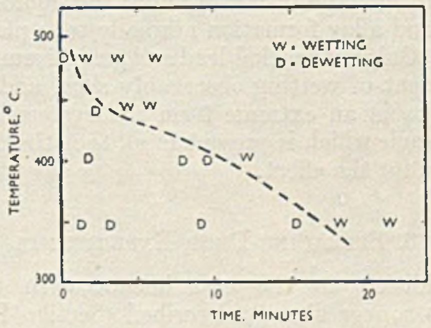

Fig. 10.—Effect of Time and Temperature of Immersion on Wetting of Nickel by Lead.

no intermetallic compounds or solid solutions were expected to form at the temperature of experiments, and the result was either "dewetting" or "non-wetting." The results are set out briefly in Table II.

The combinations nickel/lead and gold/bismuth, both in the solid-solution-forming group, were of particular interest in that the development of a stable coating required a considerable time interval. An estimate of the time required to produce a stable lead coating on nickel is given in Fig. 10.

It was noted that although lead failed to wet copper and iron, the adhesion was strong when it was allowed to solidify in contact with either.

2. IMMERSION AND DRAINING EXPERIMENTS

Immersion and draining experiments were made with the following metal pairs:

(*a*) Ag–Sn; Ni–Sn; Fe–Sn.
(*b*) Ag–Pb; Ni–Pb.
(*c*) Cu–Bi; Fe–Pb.

The pairs (*a*) above form intermetallic compounds at the solid liquid interface, pairs (*b*) are solid-solution types, and pairs (*c*) do not form solid solutions.

It was found that the liquid metals in group (*a*) formed coatings which dewetted rapidly, while those of group (*b*) gave comparatively stable coatings. In the nickel/lead system, immersion of at least 1 min. was needed at 500° C. to produce coating stability. The pairs of group (*c*) did not give a coating.

The nickel/lead pair was of interest in that the immersion contact angle exceeded 90° and did not change during the immersion period. The withdrawal angle was nevertheless zero.

It is clear from the results of these flooding and immersion experiments that there is a general correlation between the occurrence of alloy formation at the immersed solid surface and the occurrence of wetting. Coatings on "solid-solution" surfaces appear to be more stable than those on "intermetallic compound" surfaces. Coatings are not obtained on surfaces on which no alloy formation occurs, even though the solid metal may go into solution in the liquid. Both wetting and alloy formation normally take place very rapidly. Only with nickel/lead and gold/bismuth is the development of wetting observably slow, and nickel/lead shows in an extreme form the hysteresis of the contact angle which is present in all the other systems examined for the effect.

3. SPREADING-DROP EXPERIMENTS

In addition to the experiments with tin–lead alloys on copper already described (Section IV, 1, 2, and 3), similar observations were made with tin, lead, tin–lead alloys, and bismuth on iron, nickel, and silver.

(a) *Spreading on Iron* (*Cold-Reduced, White-to-Edge Blackplate*)

Both lead and bismuth showed contact angles exceeding 90° against iron at temperatures up to 1000° C., but it was noted that the angle decreased with increase in temperature, especially near 1000° C., when it was probably just less than 90°. The contact angle of tin–lead solders on iron at 500° C. was 60°–70° irrespective of the solder composition. No slow secondary spreading was observed at this temperature, and no experiments were made at any other temperature.

(b) *Spreading on Nickel*

The contact angle of bismuth on nickel was about 40° at 400° and 500° C., and no secondary spreading was observed. Using tin–lead alloys at 500° C., contact angles of about 30° were observed. Secondary spreading to a very low contact angle occurred fairly rapidly with a 1% tin alloy, but was not obtained with lead, tin, or tin–lead alloys containing 20% tin or more.

(c) *Spreading on Silver*

Marked secondary spreading was observed when drops of tin, lead, and tin–lead alloys, and of bismuth, were applied to silver at temperatures above 400° C. Bismuth and lead behaved almost identically in this respect.

4. PRODUCTION OF STABLE LEAD COATINGS ON COPPER AND IRON

As has been pointed out, the failure of lead to form a hot-dipped coating on copper or iron is a consequence of the relatively high receding contact angles which characterize these systems. The immersion experiments just described show that comparatively stable lead coatings are readily produced upon silver and nickel surfaces. It was considered that the addition to lead of a small proportion of nickel should enable coatings of substantially pure lead to be formed on copper or iron surfaces, since the nickel by reason of its solubility in the solid metal, would tend to become adsorbed from the liquid solution at the solid/liquid surface. Dipping tests were carried out in hydrogen, using lead containing 0·1% nickel, on strips of copper and iron either as-rolled or after light etching. After immersion for 1 min. at 400° C., a coating was obtained on copper which showed little tendency to dewet. A similar coating was obtained on steel after immersion for 5 min., at 500° C. It was evident that the addition of a small proportion of nickel exerted a strongly marked effect in reducing the receding contact angle of lead against copper and iron.*

5. PRODUCTION OF STABLE TIN COATINGS ON COPPER

The fact that hot-dipped coatings of tin upon surfaces of O.F.H.C. copper and iron appear to be inherently unstable has been noted earlier. As a matter

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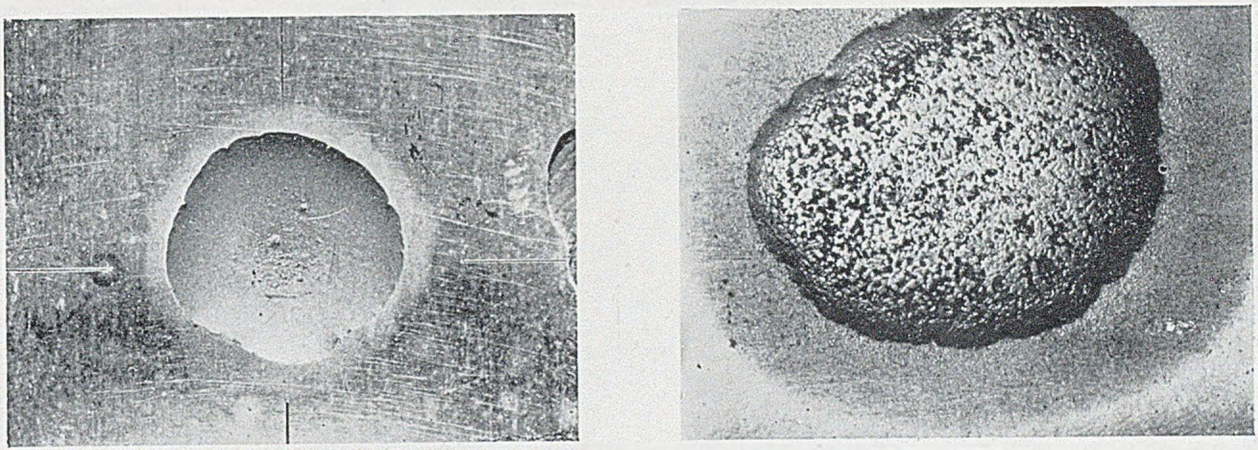
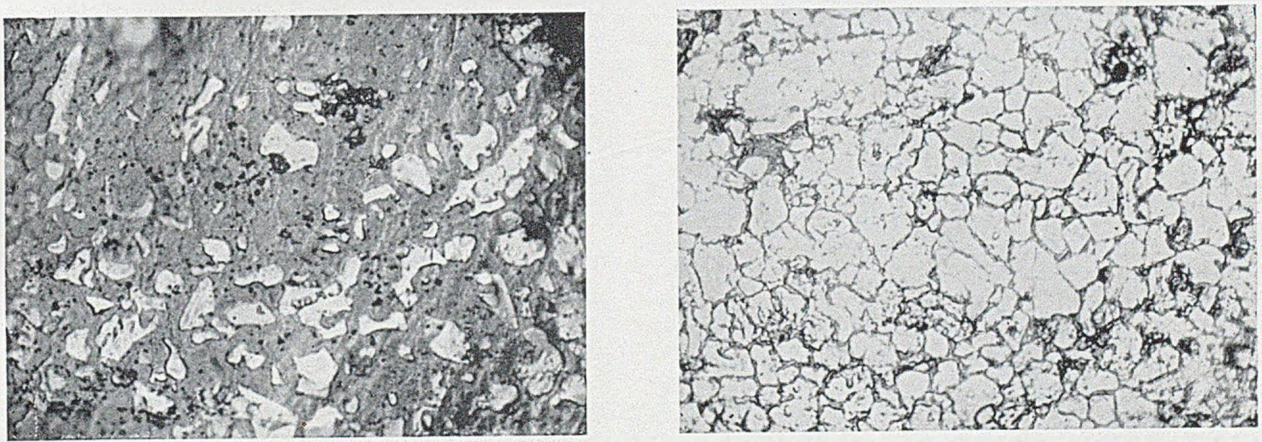
* Patent protection is being sought, in the interests of members of the Association, for processes for the production of lead coatings based upon these observations.



(*a*) 400° C.

(*b*) 500° C.

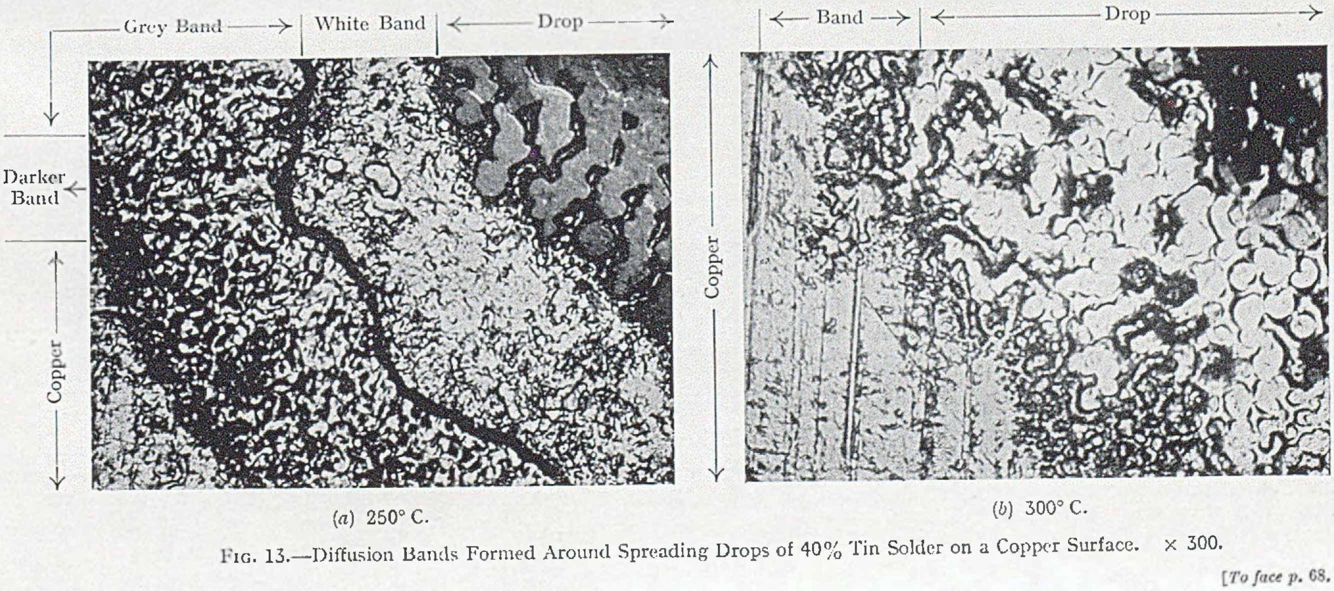
FIG. 11.—Diffusion Bands Formed Around Drops of Lead Spreading upon Silver. × 3.



(*a*) Inner zone. Diffusion from grain boundaries into grains is evident.

(*b*) Outer zone. Grain-boundary penetration is evident.

FIG. 12.—Diffusion Band Formed at 500° C. Around a Drop of Lead Spreading on Silver. × 750.



(*a*) 250° C.

(*b*) 300° C.

FIG. 13.—Diffusion Bands Formed Around Spreading Drops of 40% Tin Solder on a Copper Surface. × 300.

[*To face p.* 68.

PLATE XV.

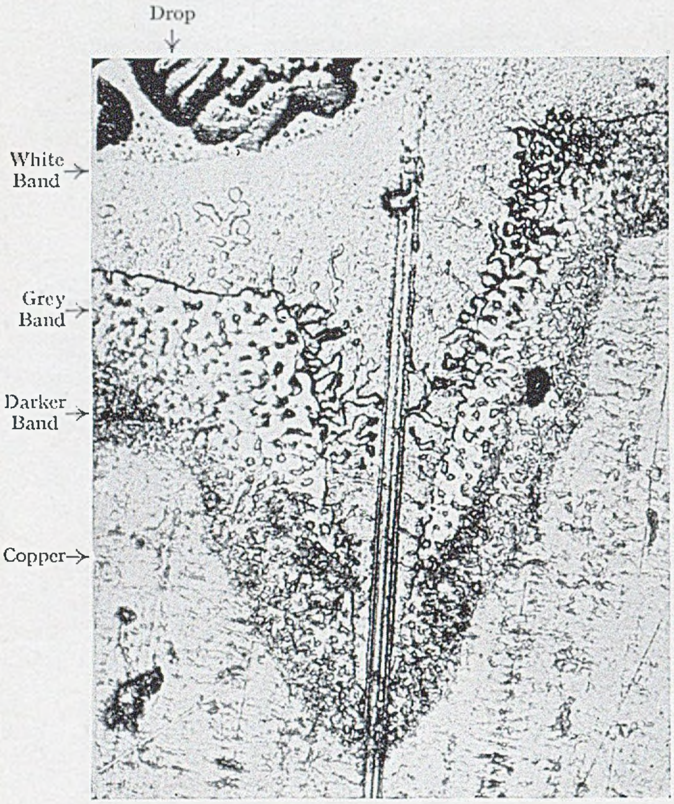


FIG. 14.—45% Tin Solder Spreading at 240° C. Along a Scratch in a Copper Surface. × 150.

FIG. 16.—Dewetted Copper Surface Showing Attack at Grain Boundaries. Copper immersed in lead at 600° C. for 2 hr. × 400.

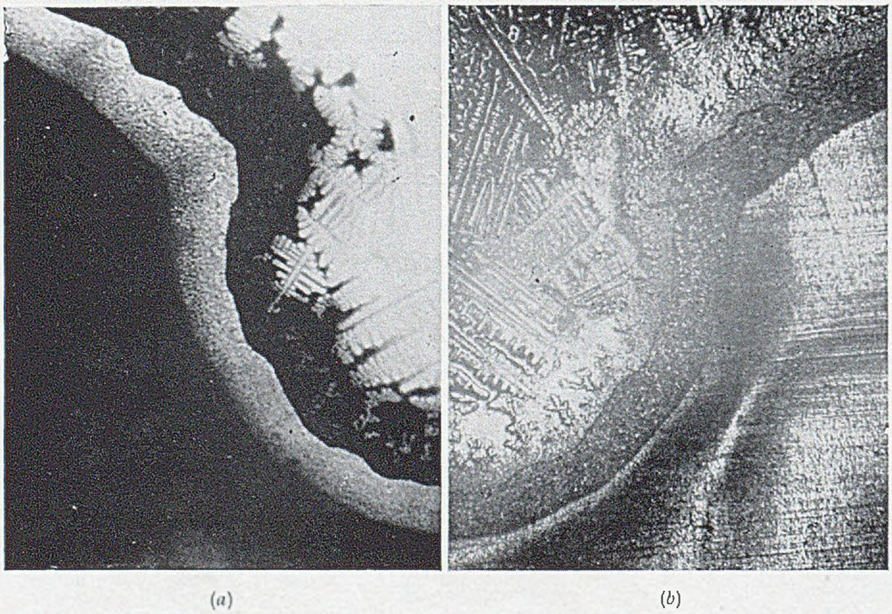

(*a*) (*b*)

FIG. 15.—Diffusion Band Formed Around Spreading Drop of 45% Tin Solder on Copper at 240° C. (*a*) Radiograph (the light areas correspond to strong X-ray absorption). (*b*) Micrograph of same field. × 40.

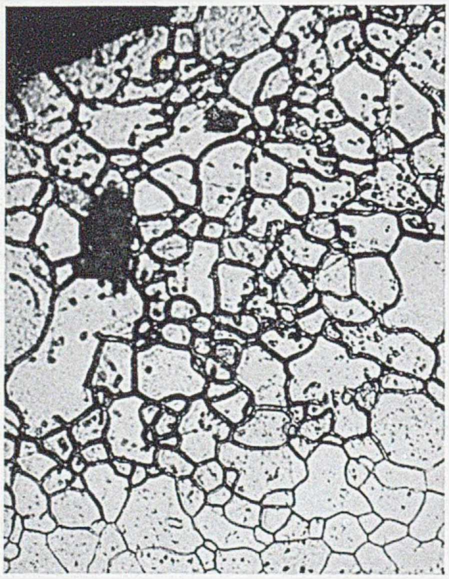


FIG. 17.—Dewetted Copper Surface Showing Lead Retained in Grain Boundaries. Copper immersed in lead at 600° C. for 2 min. × 150.

FIG. 18.—Appearance of Copper Grain Face after Immersion in Lead Saturated with Copper and Very Slow Cooling to Precipitate Primary Copper. Showing development of preferred planes and "liquid" retained in grooves of structure. × 400.

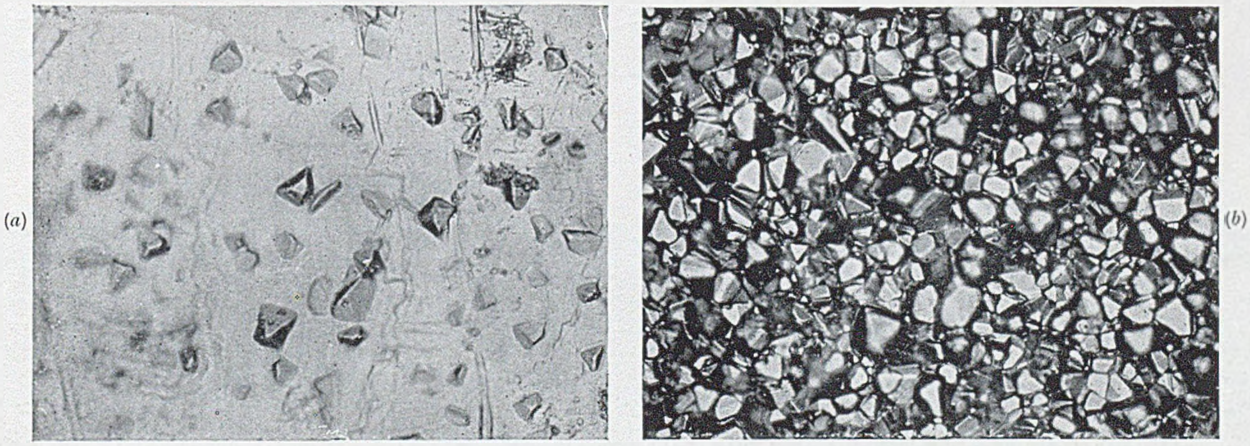


FIG. 19.—Copper Foil, Having Initially Smooth Surface, Flooded with 98 : 2 Lead–Tin Alloy for 60 min. and Drained at 450° C. Same area of surface photographed: (*a*) As dewetted, (*b*) after etching for 1 min. in 3 : 1 by volume glacial acetic acid and 20 vol. hydrogen peroxide. × 500.

FIG. 20.—Copper Surface Immersed for 10 sec. at 400° C. in Lead Containing 0·05% Tin, Showing Cubic Etch-pits on the Least Attacked Grains. × 750.

FIG. 21.—Copper Surface Immersed for 15 min. at 500° C. in Lead Containing 2·5% Silver, Showing Form of Etching. × 500.

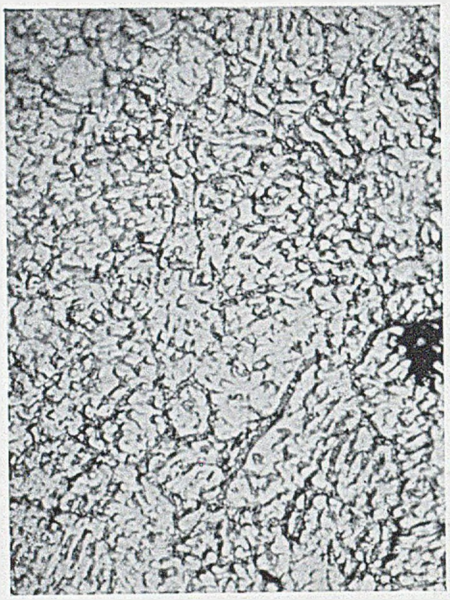


Fig. 22 (*a*).—Copper Surface after Immersion in Lead Containing 0·1% Nickel for 15 sec. at 400° C. × 750.

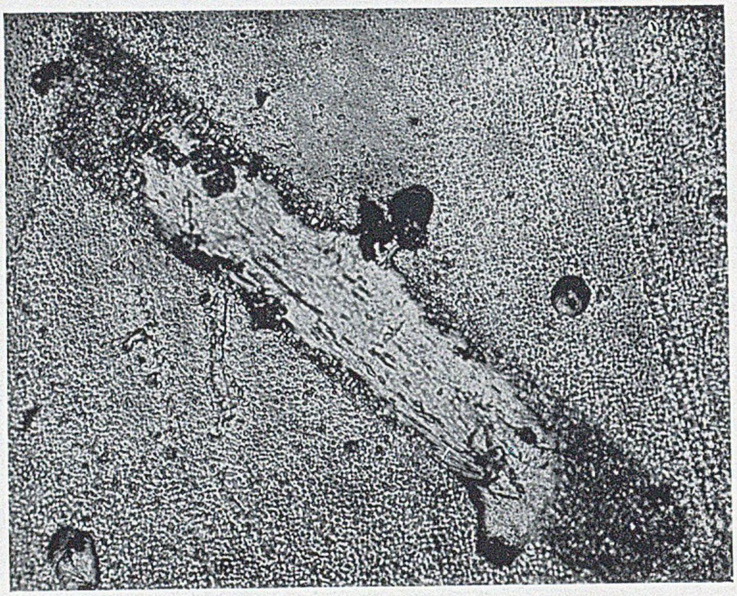


Fig. 22 (*b*).—Copper Surface (larger grain-size produced by annealing 1 hr. at 1050° C.) after Immersion as in (*a*) Showing Enhanced Attack on Twin which has an Adherent Drop of Liquid. × 400.

Fig. 23 (*a*).—Section of Copper Immersed for 2 min. at 300° C. in Pure Tin. × 500.

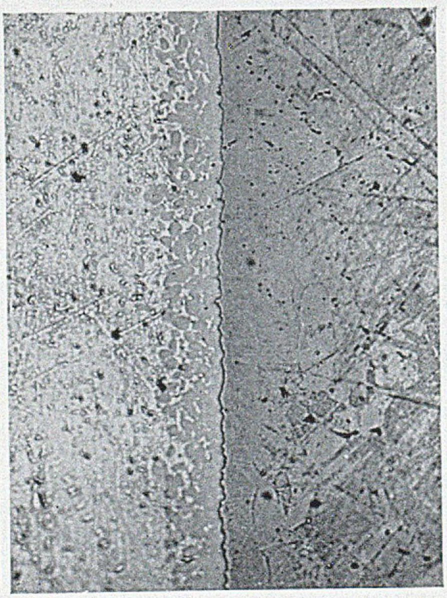


Fig. 23 (*b*).—As (*a*), but Immersed in Tin Containing 0·1% Nickel. × 500.

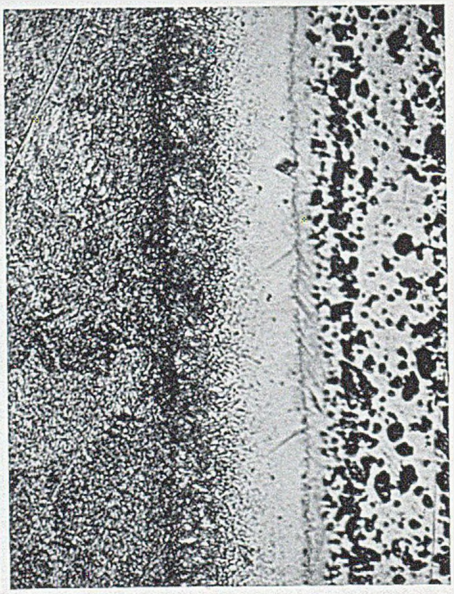
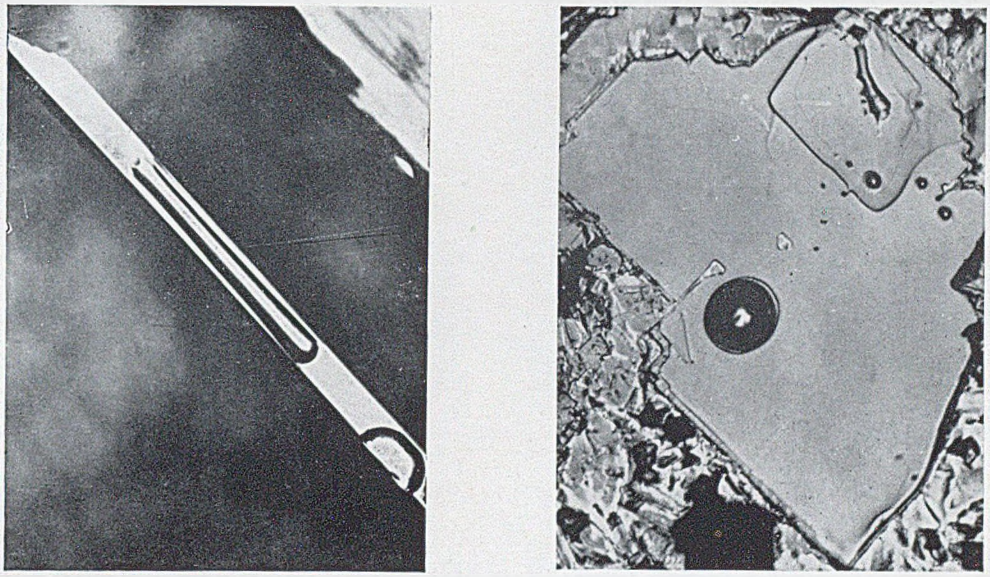


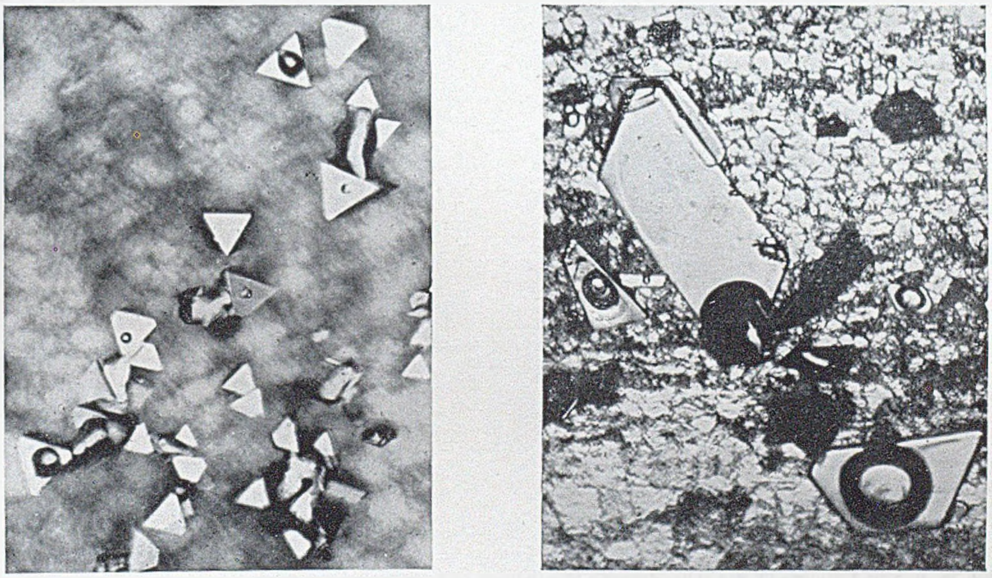
Fig. 24.—Oblique Section of 50% Tin Solder Coating Formed on Copper at 350° C. × 500.

PLATE XVIII.



(*a*) Cu–Sn.

(*b*) Ag–Sn.



(*c*) Cu–Bi

(*d*) Cu–Pb

FIG. 25 (*a*)–(*d*).—Extracted Primary Crystals, Showing Adhesion of Drops of the Liquid. × 200.

PLATE XIX.

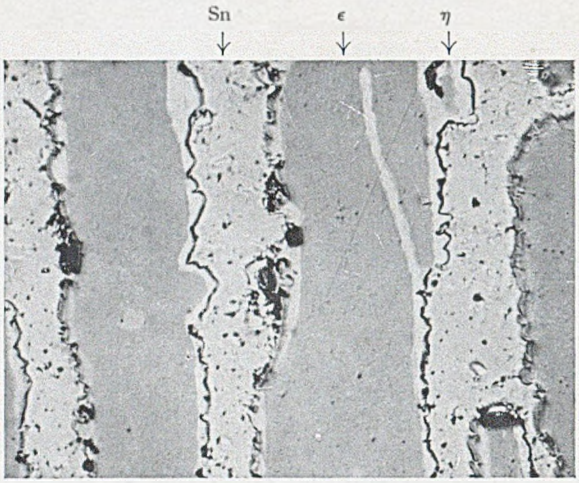


FIG. 26 (*a*).—Copper–Tin Alloy. As cast. × 150.

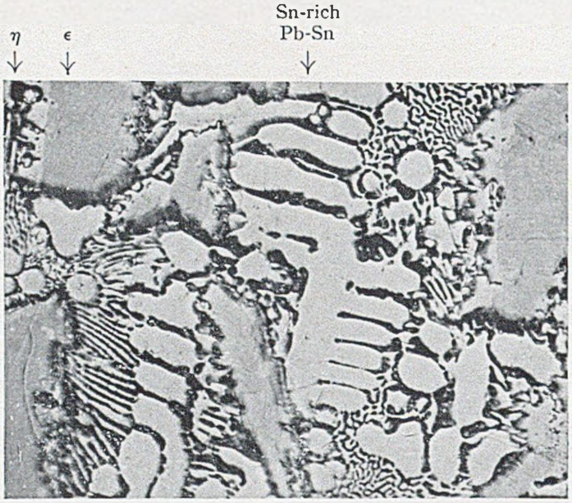


FIG. 26 (*b*).—Copper–Tin–Lead Alloy. As cast. × 150.

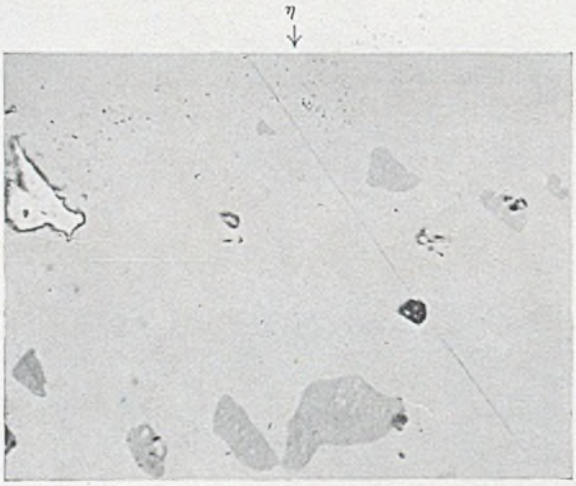


FIG. 27 (*a*).—Copper–Tin Alloy. Annealed 7 days at 400° C. × 150.

FIG. 27 (*b*).—Copper–Tin–Lead Alloy. Annealed 7 days at 400° C. × 150.

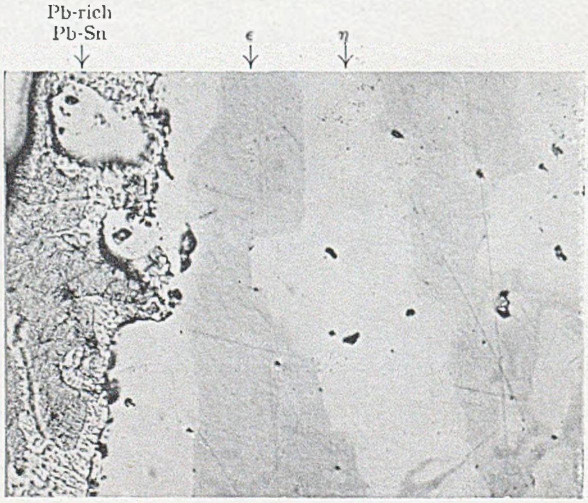


FIG. 28.—Copper–Tin–Lead Alloy. Annealed 21 days at 300° C. × 150.

of interest arising from the improvement obtained by the addition of nickel to lead for coating copper and iron, similar experiments were made to observe the stability of a coating of tin, to which 0·1% of nickel had been added, upon these two metals. Strips of copper or iron were dipped simultaneously, at 300° C. for 2 min., one into pure tin and the other into tin containing 0·1% nickel. On withdrawal, both coatings on the iron specimens dewetted, but the nickel-bearing tin coating on the copper surfaces appeared to be very much more stable than that of pure tin.

6. COATINGS OF LEAD–SILVER ALLOY ON COPPER

In similar experiments with a lead–silver alloy containing 2·5% silver upon copper surfaces with periods of immersion up to 15 min. at temperatures up to 500° C., it was found that, unlike lead, a coating could be obtained, but that this coating was comparatively unstable and dewetted fairly rapidly.

7. ELECTROCHEMICAL DEPOSITION FROM FLUXES

The deposition of tin on to a copper surface by electrochemical action in a short-circuited cell consisting of tin/zinc–ammonium chloride/copper has been described (Section IV, 5), and its possible importance in promoting rapid spreading of solders in practice referred to. It was of interest to examine in a preliminary way the possibility of similar deposition on copper and iron from other metallic chlorides which might be incorporated into fluxes. Since the majority of the chlorides which were considered have high melting points, they were mixed with other chlorides with which eutectic systems are formed. The following compositions were examined:

(*a*) Stannous chloride 80 g., potassium chloride 20 g.

(*b*) Lead chloride 80 g., potassium chloride 20 g.

(*c*) Cadmium chloride 60 g., potassium chloride 40 g.

In each case, a layer of the flux was heated in a silica crucible above a little of the appropriate metal (tin, lead, or cadmium) until both were molten and strips of copper and iron were dipped through the flux to make contact with the molten metal. After a few seconds' immersion, the specimen was removed and the part which had been in contact with the flux was examined. It was found that a coating had been deposited from the flux in every case except with steel immersed in the lead chloride flux. The coating of lead on the copper specimen had dewetted.

To repeat and extend the investigation, including other chlorides of interest, a standard flux base was adopted in which they could be incorporated. This consisted of equal parts of lithium, potassium, and ammonium chlorides, 10 g. of each being dissolved in 100 ml. of water. To this basic solution was added 10 g. of one of the following: nickel chloride, manganese chloride, cobalt chloride, stannous chloride, cadmium chloride.

These fluxes were tested by comparing their effectiveness in the hot-dip coating of copper and iron with lead at 400° C., zinc–ammonium chloride flux being used as a standard.

Before fluxing, the specimens were cathodically degreased, the copper was lightly etched in a chromate pickle and the steel in dilute nitric acid. The results showed that, while wetting of copper by lead could be produced with all the fluxes, only the stannous chloride, cadmium chloride, and zinc chloride fluxes were effective with iron. It was found that dewetting of copper was much less likely to occur with the flux containing nickel than with the standard flux.

Open-circuit voltage measurements at room temperature with lead as one electrode and copper or iron as the other, using the aqueous fluxes as electrolytes, gave the results shown in Table III.

TABLE III.—*E.M.F. Measurements.*

Flux	Open-Circuit Voltage at Room Temperature, V.	
	Steel	Copper
$NiCl_2$	0·008 *	0·315
$CoCl_2$	0·010 *	0·300
$MnCl_2$	−0·015 *	0·252
$SnCl_2$	0·095	0·215
$CdCl_2$	0·100	0·235

* No coating of lead obtained.

It will be seen that coatings were obtained only where an open-circuit voltage exceeding about +0·1V. existed between the electrodes. The polarity of the cells was in all but one case such as would result in the deposition of metallic ions on to the copper or steel.

VI.—METALLOGRAPHIC OBSERVATIONS

Throughout the experimental work described, metallographic studies were made of features of special interest. An account is given below of some of these observations.

1. DIFFUSION BANDS IN SOLID SURFACES

The formation of bands in the exposed solid surfaces adjacent to the advancing edge of the liquid metal has been mentioned. These bands were most evident where secondary spreading occurred and were due to diffusion of the liquid in the surface layers of the solid metal. The appearance, after solidification, of drops of lead on a silver surface is shown in Figs. 11 (*a*) and (*b*) (Plate XIV). In both cases, the time of contact at temperature was 30 min., but the drop in Fig. 11 (*a*) was applied at 400° C. and showed no secondary spreading, whereas that of Fig. 11 (*b*) was applied at 500° C. and showed marked secondary spreading. In both cases a diffusion band is evident in the solid surface surrounding the drop. Closer examination of this band showed that in its outer zone grain-boundary penetration had occurred

(Fig. 12 (*b*) Plate XIV) while, nearer to the liquid, diffusion from the grain boundaries into the grains was evident (Fig. 12 (*a*) Plate XIV).

2. DIFFUSION BANDS IN THE COPPER/TIN–LEAD SYSTEM

The width and appearance of the diffusion bands formed in a copper surface at the periphery of a drop of molten tin–lead alloy was observed to depend on the tin content of the alloy, on the temperature, and on the duration of the experiment. Detailed examination could be made only after cooling, which may have been accompanied by an increase of contact angle, leading, with differential contraction, to shrinkage of the drop before solidification.

With pure tin, the diffusion band was always narrow, though wider at higher temperatures. In the range 250°–400° C. it consisted of two well-defined zones, a narrow, bright inner band, presumably of η phase (Cu_6Sn_5) and a broader outer band, dark in colour, presumably of ϵ phase (Cu_3Sn). The diffusion zone round drops of tin applied at 500° C. showed only one band, presumably of ϵ phase, since the η phase is unstable at that temperature.

No diffusion bands were observed surrounding drops of lead. With tin–lead alloys, the bands were broad only with compositions in the range 30–50% tin, and within limited ranges of temperature, i.e. in the circumstances in which secondary spreading was observed.

Reference to Fig. 4 shows that marked secondary spreading occurs with an alloy containing 40% tin applied at 250° C., the effect being entirely absent at 300° C. The corresponding diffusion zones formed at the periphery of the drop are compared in Figs. 13 (*a*) and (*b*) (Plate XIV). A similar difference was also obtained using a 50% tin alloy at 300° and 350° C. respectively. The width of the bands is sensitive to the texture of the surface. The effect of a scratch is shown in Fig. 14 (Plate XV).

An attempt was made, using the technique of micro-radiography, to obtain information on the materials composing the bands shown in Fig 13 (*a*) (Plate XIV). A drop of 45% tin alloy was allowed to spread at 240° C. on a copper foil 0·0006 in. thick and a radiograph was made of part of its periphery using cobalt radiation. The result is shown in Fig. 15 (*a*) (Plate XV). Fig. 15 (*b*) (Plate XV) is a normal micrograph of the same field. A marked difference in the X-ray absorption of the two principal diffusion bands is evident, suggesting that if, as is likely, the outer band is no thicker than the inner, it is lead-rich in comparison with the inner band. That this is very probably the case was confirmed by attacking the specimen with an etchant selective for lead. A micro-radiograph, taken after etching in acid ferric chloride solution, showed that the dense material was being removed. Removal by this etchant of the supposed lead-rich material exposed a light-coloured substrate similar to that seen in the inner band, Fig. 15 (*a*). Prolonged etching removed the light constituent from both inner and outer bands, exposing a darker surface. It must be remembered, however, that the X-ray examination could be made only on solidified drops and any interpretation of the result must take this fact into account.

3. ROUGHENING OF SURFACES ON IMMERSION

The examination of the solid/liquid interface of a coated surface is not easy, though some information is, of course, obtainable from sections. Dewetted surfaces may, however, be readily examined, and the changes which occur as a result of immersion are of interest. Thus, Fig. 16 (Plate XV) shows the dewetted surface of an initially polished copper specimen which has been covered by a small volume of lead for 2 hr. at 600° C. The resultant etching of the grain boundaries is clear. Fig. 17 (Plate XV) shows a similar specimen which has been immersed for 2 min. in a larger volume of lead at 600° C. The retention of lead in the grain boundaries near the lead drop at top left of the micrograph is evident, as is also the fact that the grain faces themselves are attacked. This attack results in the development of etch pits. The effect on a copper surface of slowly cooling a copper-saturated melt of lead in which it is immersed is shown in Fig. 18 (Plate XVI), in which the presence of residual " liquid " in the grooves of the surface is of interest.

Such retention of liquid from a coating drained off a roughened surface is clearly shown in Figs. 19 (*a*) and (*b*) (Plate XVI). A specimen of copper foil, having initially a smooth surface, was immersed for 60 min. in lead containing 2% tin, at 450° C. On withdrawal, the coating immediately dewetted, and the dewetted surface appeared as in Fig. 19 (*a*). This surface was treated in a lead-selective etch consisting of 3 : 1 by volume glacial acetic acid and 20 vol. hydrogen peroxide. Fig. 19 (*b*) shows the same field as Fig. 19 (*a*) after etching for 1 min. and the exposure of crystals, probably of ϵ-copper–tin, Cu_3Sn, as " liquid " is removed is evident.

The marked effect in roughening a copper surface of the presence of as little as 0·05% tin in lead is shown in Fig. 20 (Plate XVI) which shows a polished copper surface after immersion for only 10 sec. at 400° C. It may be noted that those grains having a cubic plane in the surface appear to be least attacked, showing only a few cubic etch-pits. Similar selective roughening is shown when a copper surface is immersed in lead containing 2·5% silver, as is clear from Fig. 21 (Plate XVI), and when it is immersed in lead containing 0·1% nickel (Fig. 22, Plate XVII). The selective wetting of a twin is of interest in this last example, Fig. 22 (*b*). When intermetallic compounds are formed on immersion the solid surface is grossly roughened by the growth of crystals of the intermediate phase, as is clear from the sections shown in Fig. 23 (Plate XVII). The mode of growth of crystals of copper–tin compound appears to be greatly modified by the addition to the tin of 0·1% nickel, as shown by Fig. 23 (*b*), which should be compared with Fig. 23 (*a*).

The appearance of a section through a coating of 50% tin solder applied to copper at 350° C. shown in Fig. 24 (Plate XVII) resembles that of the tin–nickel alloy of Fig. 23 (*b*), and the solder coating had a similarly high stability.

4. Extraction of Primary Crystals From Molten Alloys

Some indication of the magnitude of the contact angle which is formed on comparatively smooth surfaces was obtained by the examination, after solidification, of the liquid phase adhering to the surface of primary crystals thrown out when saturated alloys were slowly cooled. These primary crystals were removed from the molten alloy on a copper or steel spatula, and allowed to cool in the furnace atmosphere of purified hydrogen. They were seen, by microscopic observation, in general to have comparatively smooth surfaces, to which there often adhered solidified drops of the liquid alloy. It was noted that the contact angle exhibited by these drops appeared to be comparatively high. Fig. 25 (*a*) (Plate XVIII) shows drops of tin-rich liquid adhering to the smooth face of a needle of η phase (Cu_6Sn_5), extracted from a copper–tin liquid alloy at 250° C., Fig. 25 (*d*) (Plate XVIII) shows drops of lead-rich liquid on primary copper crystals extracted at 475° C., and Figs. 25 (*b*) and (*c*) (Plate XVIII) illustrate respectively the systems silver–tin and copper–bismuth. Similar behaviour was observed with molten copper–tin–lead alloys. Thus, with crystals extracted at 325° C. from a liquid containing 20% tin, drops with high contact angle were observed on the large primary crystals, blue-grey in colour, which had been formed by cooling the melt. Similarly, crystals extracted at 250° C. from a liquid containing 60% tin, showed adherent drops and were similar in appearance to those of Fig. 25 (*a*). However, crystals extracted at 300° C. from a 45% tin alloy appeared in general to be coated with liquid; where the coating had dewetted the contact angles were much lower than in the examples in which only adherent drops were observed.

5. Comparative Structures of Tin–Copper and Tin–Copper–Lead Alloys

The spreading and coating experiments with tin-lead alloys on copper surfaces in a hydrogen atmosphere show that the lowest contact angles are obtained upon an alloy layer, and that lower contact angles are observed when the layer is of the η phase (Cu_6Sn_5) than when it is of the ϵ phase (Cu_3Sn). A possible reason for the failure of alloys containing less than about 30% tin to exhibit slow secondary spreading is that the rate of diffusion outwards from the drop is very slow because the reactivity of tin towards copper decreases as its dilution by lead increases. Relatively stable hot-dipped coatings are formed with alloys of lower tin content than 30% if sufficient time of immersion is allowed.

A micrographic study of alloy layers with the object of obtaining supporting evidence for these conclusions might prove to be difficult, since undetectably thin alloy layers could influence the contact angle. An effort was therefore made to obtain information of the kind required by comparing the structures of cast and heat-treated tin–copper and tin–copper–lead alloys.

Small ingots were cast to the following compositions :

	Sn, wt.-%	Cu, wt.-%	Pb, wt.-%
Alloy A . . .	59·0	41·0	...
Alloy B . . .	53·1	36·9	10

In both alloys, the proportion of copper to tin was that of the η phase (Cu_6Sn_5).

The as-cast structures of the two alloys are compared in Figs. 26 (*a*) and (*b*) (Plate XIX). It will be seen that these are similar in consisting of an open banded structure of Cu_3Sn (grey) fringed with Cu_6Sn_5 (white), the intervening areas showing either tin (alloy *A*) or tin–lead eutectic with tin-rich primaries (alloy *B*). Figs. 27 (*a*) and (*b*) (Plate XIX) show the two alloys after annealing for 7 days at 400° C. It will be noted that while, in alloy *A*, the reaction $\epsilon + Sn \rightarrow \eta$ has proceeded almost to completion, alloy *B* is apparently little changed. Annealing alloy *B* for a further period of 11 days at the lower temperature of 350° C. results in the appearance of substantial quantities of the white η phase and the depletion of the residual liquid approximately to the tin–lead eutectic composition, while annealing for 21 days at 300° C. gives a structure containing more of the white η phase and a lead-rich liquid phase Fig. 28 (Plate XIX).

Qualitatively, it appears that the lead present competes with the copper for the tin required to transform the ϵ phase to the η phase, and that it competes more successfully at the higher than at the lower temperatures. At a temperature of 400° C., therefore, Cu_6Sn_5 might be comparatively slow to form as an interfacial layer even in a tin-rich solder, and secondary spreading would accordingly not be marked with any solder composition at this temperature. At 350° C. there is a better chance of Cu_6Sn_5 forming with solders containing less tin, while at 300° C. even less tin is necessary for the ready formation of Cu_6Sn_5. The rising liquidus temperature of lead-rich solders sets a limit to the range of solder compositions with which ready formation of Cu_6Sn_5 is possible at appropriate temperatures. This tentative deduction from the examination of alloys *A* and *B* may in part explain the fact, shown in Fig. 4, that the greatest spreading occurs at lower tin contents as the temperature is decreased, and also the curious features shown in Figs. 13 (Plate XIV), 14, and 15 (Plate XV). It is evident, however, that a more extensive investigation would be needed to strengthen these views.

VII.—GENERAL CONCLUSION

The object of the investigation reported in this paper is to contribute to an understanding of the factors controlling the flow of liquid metals on solid metal surfaces, with particular reference to soldering, brazing, and hot-dip coating. The experimental work described is of a preliminary kind, and is in several directions incomplete. It would be fruitless to attempt to summarize here the many experimental observations which have been made: conclusions are drawn at appropriate points in the paper. A more useful purpose is served by surveying briefly the main indications arising from the work.

Starting from the probable assumption that the forces controlling the flow of liquid are those due to surface tension aided or opposed by gravity, it is pointed out that the surface-tension forces depend on the value both of the surface tension of the liquid and of the contact angle. In capillary penetration both the rate and ultimate extent of penetration depend on the product of liquid surface tension and the cosine of the contact angle, whereas in spreading on a plane surface, particularly of small drops, the area covered depends solely on the value of the contact angle. This dependence is no longer as the cosine of the contact angle, but is more nearly inversely as the value of the contact angle itself.

It is not necessarily surprising, therefore, that solders are placed in differing orders of merit by spreading-drop and capillary-penetration tests, which, as hitherto employed, measure different surface-tension properties. The apparent discrepancies between the results of the two types of test may be explicable solely on these grounds. However, these discrepancies suggest alternatively, that with all tin-lead solder compositions the contact angle first formed against a solid surface is high (*c.* 60°), but that, if the solder moves sufficiently slowly over the surface, the contact angle of solders containing about 50% of tin falls to a low value. The experimental work on spreading over a copper surface in a hydrogen atmosphere shows that such slow spreading can occur. How quickly it occurs in the presence of liquid fluxes has not yet been determined. It evidently takes place to a greater extent under zinc–ammonium chloride flux than under plain resin flux. It appears that electrolytic deposition of metal from the flux on to the solid surface ahead of the spreading solder produces a modified solid surface against which the liquid develops a lower contact angle and as a result both the rate and the extent of the spreading are increased.

Theory anticipates and experiment readily shows that substantial hysteresis of the contact angle is usual. It is possible that the discrepancies between the results of spreading-drop experiments and practical assessments of solder performance may be explained by taking hysteresis into account. In spreading-drop experiments an advancing contact angle is established, whereas in most soldering practice, a coating of solder is formed, e.g. by tinning, dipping, or working with the bit and, if the contact angle is to be considered, the receding value should be more appropriate. It remains to be explained, however, why an area measurement should even approximately represent practical solder performance, in which the ability to penetrate joints appears to be the main requirement.

At present, theory does not allow the prediction of true contact angles, nor can they in general be measured directly because of the effects of surface roughness, which cannot be given quantitative expression. The nature of roughness effects can be foreseen to some extent, however. In particular, the promotion of spreading by previously roughening the solid surface by interlacing scratches is substantiated by theory.

Although true contact angles may be neither calculated nor measured, an indication of their value is obtainable by experiment, and some use may be made of such observations. For example, the observation that the receding contact angle of lead upon nickel is very low led to the successful attempt to produce lead coatings upon copper and iron by the addition of a small proportion of nickel to the lead.

ACKNOWLEDGEMENT

The authors are indebted to the Director and Council of the British Non-Ferrous Metals Research Association for permission to publish this paper.

APPENDIX I

Spreading and Penetration on Smooth Surfaces: Constant Surface Tension and Contact Angle

The purpose of this appendix is to state briefly and in simple form a number of expressions which are almost all derived in classical surface-tension theory[4] and relate to cases of particular interest in jointing and hot-dip coating.

(a) *Shape of a Drop of Liquid on a Horizontal Plane Surface* (Fig. 29)

The fundamental equation for the shape of the surface of a drop resting on a horizontal plane may be written:

$$\gamma\left(\frac{1}{r} + \frac{\sin\psi}{x}\right) = \frac{2\gamma}{b} + g\rho y \quad . \quad . \quad (1)$$

where γ = surface tension of liquid
ρ = density of liquid
g = acceleration due to gravity
x, y = the co-ordinates of any point, P, on the surface (Fig. 29 (*a*))
ψ = angle between the tangent to the drop at P and the x-axis
r = that radius of curvature, PQ, at P which lies in the plane of the figure
$b = OM$, the radius of curvature at the apex of the drop

The terms $\sin\psi$, r, and b in equation (1) may be written as differential functions of x and y, so that equation (1) is in fact a differential equation in x, y, and the constants γ, ρ, and g. No general solution of this differential equation is possible, however. For a drop which is so large that its upper surface is flat, and with contact angles exceeding 90° (Fig. 29 (*b*)), it is found by approximation that :

$$\gamma = \frac{g\rho h^2}{2} \quad . \quad . \quad . \quad . \quad (2)$$

$$\cos\theta = 1 - \frac{H^2}{h^2} \quad . \quad . \quad . \quad . \quad (3)$$

When the drop is so small that the gravitational term in equation (1) can be neglected, its surface forms part of a sphere. In these circumstances, the area of contact between the drop and the plane is independent of its surface tension and depends only on the volume of liquid composing the drop and the contact angle. This relationship may be written, Fig. 29 (*c*) :

$$V = \frac{\pi h}{b}(3R^2 + h^2) \quad . \quad . \quad . \quad . \quad . \quad . \quad (4)$$

or

$$V = \frac{\pi R^3}{3}\tan^2\frac{\theta}{2}\left(3\operatorname{cosec}\theta - \tan\frac{\theta}{2}\right) \quad . \quad (5)$$

where V is the volume of the drop.

No useful solution of equation (1) is available when the drop is so large that the gravitational term cannot be neglected and, at the same time, the contact angle is less than 90°.

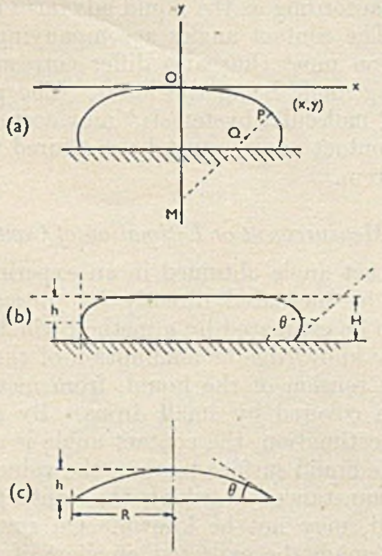


Fig. 29.—Shape of Liquid Drop on Horizontal Plane Surface.

(b) *Rate at which a Drop of Liquid Attains its Equilibrium Shape*

Because the fundamental equation for equilibrium drop shape, equation (1), is incapable of general solution, it is not possible to deduce either the rate at which a spherical drop applied to the surface will expand or the rate at which a given volume of liquid applied as a film of specified uniform thickness will contract to take up the equilibrium drop shape.

(c) *Downward Pull Exerted by a Meniscus*

When a plane surface is dipped vertically into a liquid which exhibits against it a contact angle θ, the liquid surface assumes a shape shown in section in Fig. 30. The curve ABC is the " capillary curve " and its equation may be derived. Of greater interest, however, are the height (OA, Fig. 30) of the top of the liquid meniscus and the downward pull, W, exerted by the meniscus on the solid surface.

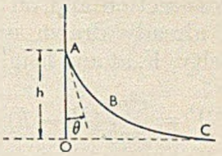


Fig. 30.—Shape of Liquid Surface at Vertical Plane Surface.

(i) The height of the meniscus is found from :

$$h = \sqrt{\frac{2\gamma}{g\rho}(1 - \sin\theta)} \quad . \quad . \quad . \quad (6)$$

(ii) The downward pull, W, is simply, per unit length of meniscus,

$$W = \gamma\cos\theta \quad . \quad . \quad . \quad . \quad (7)$$

(d) *Rate of Formation of a Meniscus*

The rate at which a meniscus achieves the equilibrium shape has not hitherto been calculated.

(e) *Draining of Dipped Coatings*

Jeffreys [13] derived a relationship for the thickness x of the liquid coating at a distance y below its top edge at any time t after withdrawal from immersion. This expression is :

$$x = \sqrt{\frac{\eta y}{gt}} \quad . \quad . \quad . \quad . \quad (8)$$

in which η is the kinematic viscosity of the liquid. It was derived under the assumption that the draining surface is vertical and that the contact angle is zero. No treatment has hitherto been given of the case when the contact angle is greater than zero, or of the collapse of coatings to form separate drops.

(f) *Penetration of Capillary Spaces*: *Equilibrium Penetration Height*

The height, H, to which a liquid rises in a vertical tube of circular cross-section is given by the expression :

$$H = \frac{2\gamma\cos\theta}{g\rho r} \quad . \quad . \quad . \quad . \quad (9)$$

where r is the radius of the tube. An identical expression in which r is now the separation of the surfaces, may be obtained for penetration between parallel plates. These expressions are also true for penetration in capillaries inclined to the vertical, H being the vertical component of the penetration distance ; in the limiting case of a horizontal capillary it is clear that equilibrium can never be reached in a tube of finite length.

(g) *Penetration of Capillary Spaces: Rate of Penetration*

The rate of flow into a capillary at any moment depends on its cross-section and on the length of the column of liquid already drawn up. In a vertical capillary, the velocity, V, at any height, h, is given by :

$$V = \frac{r^2\rho g}{8\eta h}(H - h) \quad . \quad . \quad . \quad (10)$$

In this expression, η is the coefficient of viscosity of the liquid and H the equilibrium height. From equation (10), the time, t, to reach height h is found to be :

$$t = \frac{8\eta}{r^2\rho g}\left(H \log_e \frac{H}{H - h} - h\right) \quad . \quad (11)$$

In a horizontal capillary, the corresponding expressions may be written :

$$V = \frac{r^2\rho g H}{8\eta h} \quad . \quad . \quad . \quad . \quad (12)$$

$$t = \frac{4\eta h^2}{r^2\rho g H} \quad . \quad . \quad . \quad . \quad (13)$$

These expressions form useful approximations for the early stages of penetration into a capillary space of any inclination. They may conveniently be rewritten

$$V = \frac{r}{4\eta h} \cdot \gamma \cos\theta \quad . \quad . \quad . \quad (14)$$

$$t = \frac{2\eta h^2}{r} \cdot \frac{1}{\gamma \cos\theta} \quad . \quad . \quad . \quad (15)$$

APPENDIX II

FACTORS DETERMINING THE VALUE OF THE LIQUID SURFACE TENSION AND OF THE CONTACT ANGLE ON SMOOTH SURFACES

(a) *Surface Tension and Contact Angle*

For a general discussion of surface tension and contact angle reference should be made, for example, to Adam.[4] Here it is stated only that every surface separating different kinds of matter possesses, by virtue of its existence, surface energy, which resides in a surface layer only an atom or two thick, the value of which is determined by the nature and arrangement of the atoms in this layer. When a surface is mobile, as with a liquid, it will tend to adjust its shape until a condition of minimum free energy is achieved locally. The possession by a surface of a specific free energy per unit area may be regarded as equivalent to the presence of a numerically equal tension force per unit length, the surface tension, acting in the surface.

When a liquid is in contact with a smooth solid surface, under equilibrium conditions, their surfaces meet at a characteristic angle, the contact angle, the value of which is determined by the surface tensions of three surfaces, as shown in Fig. 31, according to the expression : *

$$\gamma_{13} = \gamma_{12} + \gamma_{23} \cos\theta \quad . \quad . \quad . \quad (16)$$

Of these three surface tensions, only that of the liquid, γ_{23}, is normally measurable. Clearly if γ_{13} equals or exceeds the sum $\gamma_{12} + \gamma_{23}$ the contact angle is zero and the liquid spreads indefinitely over the solid surface.

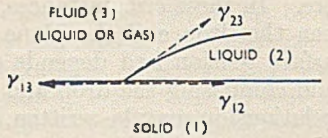


FIG. 31.—Determination of Contact Angle by Surface Tension of Three Surfaces.

It is known that adsorption may occur from both the liquid and vapour phases on to a solid surface. The form of adsorption may well differ in the two cases, and thus the surface tension of a smooth solid surface may differ according as the liquid advances or recedes upon it. The contact angles accompanying advance and recession must thus also differ correspondingly, the receding angle being the lower. The possibility that such " molecular hysteresis " may occur indicates that two contact angles must be measured to specify a given system.

(b) *Direct Measurement or Estimation of Contact Angle*

The contact angle obtained in an experiment may frequently be measured directly by optical means. It may also be estimated by a method which does not involve any knowledge or assumption of the value of the surface tension of the liquid, from measurement of the area covered by small drops. By any other method of estimation, the contact angle is derived in terms of the liquid surface tension, the value of which in the circumstances in which the contact angle is investigated, may not be known. On smooth solid surfaces, it might be expected, in view of the possibility of molecular hysteresis, that different advancing and receding angles would be obtained, but these should be characteristic of the system. In Appendix III it is shown that, in general, when the surface is rough, it is impracticable to correct the observations for the effects of roughness and so derive these characteristic values. It is, however, the characteristic values which are of fundamental importance for spreading, capillary penetration, and coating stability. Since they cannot readily be measured directly, it is desirable to discuss individually the three surface tensions which determine them.

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* Surface tension at the surface between the solid (1) and liquid (2) is indicated by γ_{12}. Similarly the surface tension at solid (1)/fluid (3) interface is γ_{13}, and at liquid (2)/fluid (3) interface γ_{23}.

(c) *Direct Determination of Surface Tensions*

While it is clear that the surface tension of a surface is determined by its atomic arrangement, the value cannot at present be calculated with accuracy for given arrangements of atoms. In any case, in view of the possible concentration at surfaces of impurities which are below detectable limits when a bulk sample is taken, such an approach would have little practical value. The surface tension of the liquid surface in contact with a fluid phase, either liquid or gaseous, may be measured directly, but those of the liquid metal/solid metal surface and of the solid metal/flux surface are not normally measurable.

(d) *Surface Tension of Liquid Metal/Flux Phase*

Direct measurement of the surface tension of a liquid metal in contact with a flux phase is possible by several methods which are independent of the contact angle. Bircumshaw[14] gives values for a number of tin–lead solders in contact with hydrogen, Chalmers and Wadie[8] obtained similar data employing a resin flux and Latin[7] determined the surface tension of tin and tin–lead solder of eutectic composition in contact with resin, zinc–ammonium chloride, and stannous chloride fluxes. Some of these results may be compared in Table IV.

TABLE IV.—*Surface Tensions of Tin, Lead, and Tin–Lead Eutectic (dynes/cm.) at Temperatures Just Above the Liquidus.*

Metal	Flux				
	Hydrogen[14]	Resin[8]	Resin[7]	Zinc/Ammonium Chloride[7]	Stannous Chloride[7]
Tin . . .	550	420	456	422	342
Tin–lead eutectic .	490	380	390	331	...
Lead . . .	440	316	...	...	...

The complete series of results from which the first two columns of Table IV are selected show that, in both hydrogen and resin, surface tension decreases continuously with tin content. These results do not therefore at once suggest an explanation for the marked spreading of alloys in the centre of the composition range. If the two other surface tensions which determine the contact angle do not change, the largest spreads would be expected with the lead-rich solder compositions. It is, however, possible that the surface tension of the liquid metal may be influenced by the presence of the solid metal which is in contact with both solder and flux in soldering. This possibility has not hitherto been investigated, though the investigation might be attempted. One method would be to measure the downward pull exerted by a meniscus upon a vertical surface of the solid metal and at the same time to observe the contact angle directly.

(e) *Surface Tensions of Solid Surfaces*

No general methods are available for measurement of the surface tension of solid surfaces. An experimental estimate for solid copper just below its melting point by Udin, Shaler, and Wulff[15] gives a value of 1370 dynes/cm., and another by Bailey and Watkins[12] gives a figure of 1800 dynes/cm. at temperatures of 800°–900° C. Bailey and Watkins were also able to measure the solid metal/liquid metal surface tension for the copper/lead/hydrogen system and obtained a value of 340 dynes/cm. at 800°–900° C. These values indicate that lead should spread completely on a copper surface at these temperatures. In fact, however, the contact angle is finite, though low. This is accounted for by supposing the surface tension of copper to be reduced as a result of adsorption of lead from the vapour phase. The surface tension of copper with an adsorbed lead layer was estimated to be 780 dynes/cm., less than half the " clean " value. A high value of the solid metal/flux phase surface tension, in the absence of adsorption, is not always to be expected, however. Shuttleworth[16] has calculated the surface tensions of some ionic crystals and found them to be of a lower order than those of metallic crystals. It might thus be expected that the surface tension of intermetallic compounds would in general be lower than that of metallic solid solutions.

C. S. Smith[17] considers that the solid metal/liquid metal surface tension is normally much lower than that of the solid metal surface in contact with its own vapour. He also describes experiments from which it is clear that the solid metal/liquid metal surface tension of lead–bismuth alloys in contact with copper varies considerably with the composition of the liquid alloy.

APPENDIX III

INFLUENCE OF SURFACE ROUGHNESS ON THE VALUE OF THE CONTACT ANGLE

(a) " *True* " *and* " *Observed* " *Contact Angles*

The influence of roughness of the solid surface on the contact angle obtained in practice was first discussed by Wenzel.[18] He considered that, if the true area of a rough surface is r times greater than its nominal area, r being called the " roughness ratio," the contact angle observed, ϕ, is related to the true

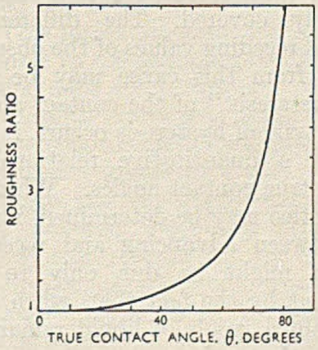

FIG. 32.—Relations Between True Contact Angle and Roughness Ratio.

contact angle, θ, which is the angle observed on a smooth surface, by the expression:

$$\cos \phi = r \cos \theta . \quad . \quad . \quad . \quad (17)$$

Thus, when θ is less than 90°, the observed contact angle is smaller than the true angle. The relationship between true contact angle and roughness ratio which gives an observed angle just equal to zero is plotted in Fig. 32 from which it will be seen, for example, that the observed angle is zero when $r>2$ and $\theta<60°$.

(b) *Influence of the Form of the Surface Roughness*

That Wenzel's solution of the problem is incomplete can be seen qualitatively from a consideration of Fig. 33. This represents an idealized rough surface in which the roughening has the form of a network of interlacing grooves. The spreading of a liquid on such a surface will be enhanced by the capillary effect of the grooves. Now consider a surface which is the "negative" of that of Fig. 33. This evidently consists of an array of pits. The roughness ratio of the "positive" and "negative" surfaces is the same, but spreading of a liquid on the negative will not be enhanced, as on the positive, by a network of capillary channels.

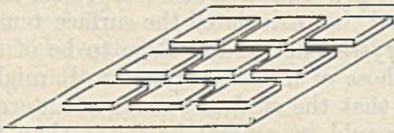

Fig. 33.—Idealized Rough Surface.

Using a similar model, Shuttleworth and Bailey [19] showed that Wenzel's expression, equation (17), is valid only when the roughness has the character of interlacing grooves. When this is not so the contact angle which is observed depends on whether the liquid has come to rest after spreading outwards over the surface or after receding over a surface which it has previously covered. The difference between advancing and receding values of the observed contact angle arising from this cause may be described as "capillary hysteresis" of the contact angle.

When this type of hysteresis occurs, it is impracticable to derive a quantitative relationship between observed and true contact angles. Whether or not it occurs in practice may be determined, not solely by a difference between advancing and receding contact angles which might be due only to "molecular hysteresis" but by the fact that, when an immersed surface is drained, pools of liquid remain trapped in the pits upon the surface.

By combining equation (16) (Appendix II) with equation (17) it is found that:

$$r\gamma_{13} = r\gamma_{12} + \gamma_{23} \cos \phi \quad . \quad . \quad (18)$$

This expression has the same form as equation (16), (Appendix II) and shows that, if Wenzel's equation is obeyed, the effect of the roughness is to increase the effective value of the surface tensions γ_{13} and γ_{12} by the roughness ratio, r.

The suggestion may be added that, with any real rough surface on which hysteresis is observed, the lower the true contact angle the more nearly does the surface behave as if it consisted of interlacing grooves. This suggestion arises from the view that a real surface consists of interlacing grooves of varying width and depth. When the true contact angle is high, grooves adjacent to the edge of a liquid drop will be incompletely filled, so that the variations in their depth will prevent capillary penetration. If the true contact angle is lower, this limitation on capillary penetration is less severe and the more nearly is an ideal system of interlacing grooves approached.

REFERENCES

1. S. J. Nightingale and O. F. Hudson, "Tin Solders: A Modern Study of the Properties of Tin Solders and Soldered Joints", (*Brit. N.-F. Metals Research Assoc. Research Monograph* No. 1), 1942.
2. J. McKeown, "The Properties of Soft Solders and Soldered Joints", (*Brit. N.-F. Metals Research Assoc. Research Monograph* No. 5), 1948.
3. A. Latin, *J. Inst. Metals*, 1946, **72**, 265.
4. N. K. Adam, "The Physics and Chemistry of Surfaces", 3rd Edition. London: **1941** (Oxford University Press).
5. E. J. Daniels and D. J. Macnaughtan, *Tech. Publ. Internat. Tin Research Develop. Council, Series B*, 1937, (**6**).
6. A. W. Coffmann and S. W. Parr, *Indust. and Eng. Chem.*, 1927, **19**, 1308.
7. A. Latin, *Trans. Faraday Soc.*, 1938, **34**, 1384.
8. B. Chalmers and R. H. Wadie, *J. Inst. Metals*, 1940, **66**, 241.
9. M. E. Fine and R. L. Dowdell, *Trans. Amer. Soc. Metals*, 1946, **37**, 245.
10. G. Tammann and F. Arntz, *Z. anorg. Chem.*, 1930, **192**, 45.
11. T. Alty and A. R. Clark, *Trans. Faraday Soc.*, 1935, **31**, 648.
12. G. L. J. Bailey and H. C. Watkins, *Proc. Phys. Soc.*, 1950, [B], **63**, 350.
13. H. Jeffreys, *Proc. Camb. Phil. Soc.*, 1930, **26**, 204.
14. L. L. Bircumshaw, *Phil. Mag.*, 1934, [vii], **17**, 181.
15. H. Udin, A. J. Shaler, and J. Wulff, *Trans. Amer. Inst. Min. Met. Eng.*, 1949, **185**, 186.
16. R. Shuttleworth, *Proc. Phys. Soc.*, 1949, [A], **62**, 167.
17. C. S. Smith, *Trans. Amer. Inst. Min. Met. Eng.*, 1948, **175**, 15.
18. R. N. Wenzel, *Indust. and Eng. Chem.*, 1936, **28**, 988.
19. R. Shuttleworth and G. L. J. Bailey, *Discussions Faraday Soc.*, 1948, (**3**), 16.

THE SPECTROCHEMICAL DETERMINATION OF ZINC, LEAD, AND IRON IN COPPER AND COPPER ALLOYS* 1326

By FREDERICK V. SCHATZ,† M.A.

SYNOPSIS

A spectrochemical method suitable for the routine works control of copper and copper alloys has been developed. The procedure outlined is applicable to high-speed analysis, has a precision capable of determining zinc in brass with an error (expressed as a standard deviation) of 1·18% of the content or less, and has a sensitivity capable of detecting common impurities in copper alloys down to 0·005% or less. The determination of zinc, lead, and iron in brasses and leaded brasses is considered in detail. Factors important to the accuracy of the analysis are also considered; these include discharge characteristics, sample polarities, effects due to the nature of the lead dispersion, and curve shifts due to varying quantity of the matrix element.

I.—INTRODUCTION

THE analysis of copper and copper alloys was among the earliest applications of the spectrograph in the field of practical analysis, and a great many papers have been published which describe spectrochemical methods designed to solve the special problems of individual investigators. Papers outlining procedures for practical works' control in the copper and brass industry, however, have been conspicuously small in number, and the development of suitable process control for copper alloys by means of the spectrograph has tended to lag behind the parallel development in the steel and aluminium works. This lag is not surprising when it is realized that a proper spectrographic procedure for the analysis of a simple high-purity brass should combine the extremes in spectrographic analysis, namely high-precision analysis of a constituent ranging from 5 to 40%, with a sensitive analysis of impurities ranging down to 0·005% or less. Only in recent years have the invention of high-precision sources, the development of photographic emulsions of great uniformity, and the progress in direct-reading equipment enabled the analyst to achieve control compatible with the specification limits of brasses.

The author has been engaged for several years in studying equipment and developing spectrochemical methods suitable for the complete analytical control of high-quality copper and brass alloys. This effort has been attended with considerable success, since at the present time up to 90% of all analyses at the Divisional Laboratories of Revere Copper and Brass Inc. are carried out on the spectrograph. The work described is concerned in detail only with the determination of zinc, lead, and iron in copper and the brasses. These elements are the most important encountered in routine quality control, and analyses for them represent the greatest majority of those performed. Moreover, the procedures outlined are capable of extension to other elements. Work is in progress at the moment on a detailed consideration of the more complex copper alloys.

II.—SOURCE CONSIDERATIONS

When the determination of high zinc contents in brass first engaged the author's attention, a new source, known as the Multisource, was being described in the literature.[1, 2] Since a high-voltage Feussner interrupted spark had been used for high-zinc determinations with only fair results, a Multisource was obtained for detailed investigation.

The principle underlying the operation of the Multisource is very simple (Fig. 1). A half-wave rectifier charges a condenser to 940 V. during one half cycle. In the succeeding half cycle, the analytical gap is ionized by a low-power, high-tension spark discharge. This ionization allows the energy stored in the condenser to discharge across the gap through a selected inductance and resistance. Since the input rectifier and suitable filters isolate the discharge from all parts of the circuit except the analytical gap and a selected condenser, inductance, and resistance, the type of discharge and the energy associated with it can be very precisely controlled and maintained. Settings are combinations of 20, 20, 10, 5, 2, 2, and 1 μF.; 200, 100, 50, 25, 10, 5, 5, 2, 2, and 1 ohm, and 200, 100, 50, and 25 μH. There is also a distributed inductance of 25 μH. and 0·4 ohm distributed resistance associated with the source, plus the distributed inductance, capacitance, and resistance of the leads used to connect the source and analytical gap.

The inventors of the Multisource claim that close to

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* Manuscript received 16 December 1950.

† Research Department, Revere Copper and Brass Inc., Rome, N.Y., U.S.A.

300,000 separate sources are possible. Of this multiplicity of sources, three have been found to be useful in the analysis of brasses. The three discharges are all unidirectional and pulse-like in nature. Oscillatory discharges from this type of circuit were found to be unsatisfactory owing not only to polarity considerations, but also to the nature of the copper spectrum, which in general was composed of broad, diffuse lines against a high background.

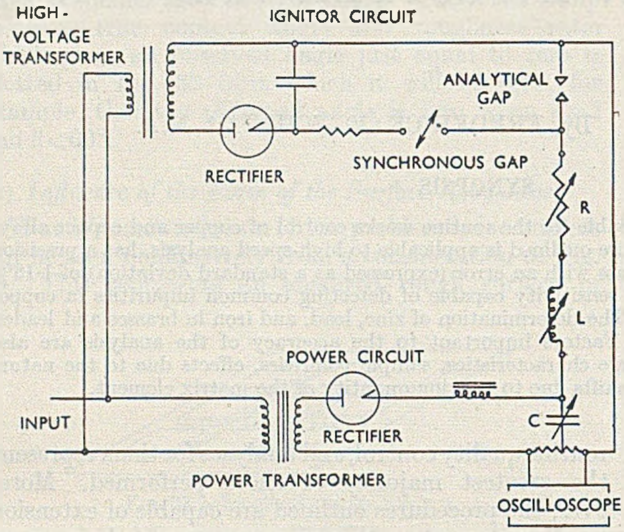


Fig. 1.—Schematic Diagram of Basic Multisource Circuit.

The discharges used are summarized in Table I.

Table I.—*Types of Discharge.*

Type	Sample Polarity	Precision	Sensitivity	Typical Setting
1. Slightly over-damped	+	< 2·0% Error	Poor	10 μF., 200 μH.,[*] 10 Ω
2. Heavily over-damped	–	<10% ,,	Excellent	60 μF., 400 μH.,[*] 20 Ω
3. Slightly under-damped	+	< 2·0% ,,	Good	10 μF., 200 μH.,[*] 3 Ω

[*] Settings include 25 μH. distributed inductance.

Oscilloscope patterns of each type of discharge are shown in Fig. 2 and form a convenient way of correlating settings between different source models. The Type 1 discharge, as applied to copper alloys, was first investigated by Hasler and Kemp,[2] who used it for aluminium bronze. They claimed a precision of better than 2% and a reduction in the effects of the specimen's metallurgical history. However, the Type 1 discharge is of only limited value in its applications to high-purity brasses, since its use makes necessary the application of either Type 2 or Type 3 discharges for the determination of impurities. The use of two discharges and the doubling of the analysis time may be unimportant in some laboratories, but not in a production laboratory where every minute counts. Consequently, for most work, the Type 3 or Type 2 discharge has been used.

Examination of the oscilloscope pattern of the Type 3 discharge indicates that electrically it is slightly oscillatory. The contribution of the lower pip to the gap discharge, however, is so small that the pulse is unidirectional and has characteristics associated with high peak current and short duration.

If the Type 1 discharge is compared with Type 2, it is obvious that precision is associated with a short pulse and a low average current, while sensitivity is associated with a long pulse and a high average current. Consequently, there is a fundamental incompatibility between precision and sensitivity. Fortunately, the Type 3 discharge, which combines a pulse of short duration with a very high peak current, offers a compromise that enables the analyst to combine precision and sensitivity within reasonable limits.

Though the author has had no experience with the B.N.F.M.R.A. general-purpose source unit, descriptions [3, 4] in the literature indicate that it is basically similar to the Multisource. The outstanding difference is that the triggered discharge voltage is 310 instead of 940 V. Consequently, equivalent settings on both units can be determined by the use of the relation:

$$R_c = 2\sqrt{L/C}$$

where R_c = critical damping resistance, L the inductance, and C the capacitance.

Distributed circuit constants may complicate the picture, especially in arriving at a proper resistance equivalent to the Type 3 discharge. This resistance is quite critical if a spectrum with optimum line-to-background intensity is desired. Walsh [4] in carrying out the analysis of uranium with a B.N.F.M.R.A. general-purpose source unit, discovered a setting comparable in sensitivity to the over-damped arc-like discharge at resistance values between 0 and 1 when inductance

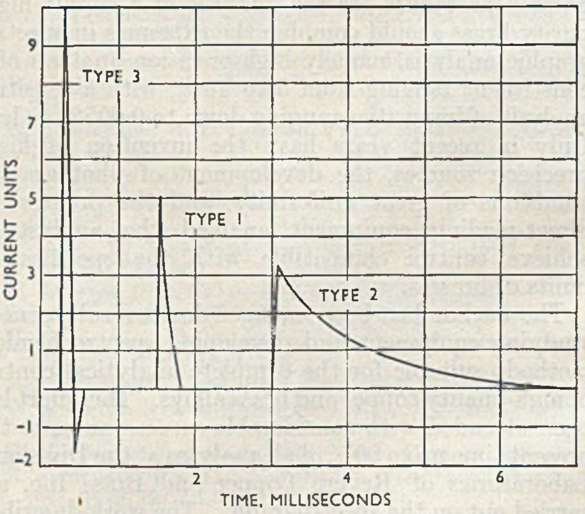


Fig. 2.—Discharge-Pulse Shapes.

and capacitance settings were equal to 500 μH. and 240 μF. respectively. Calculation shows that if $R = 0{\cdot}97$ ohm Walsh setting is equivalent to the Type 3 discharge.

To summarize, the following working rules can be applied in deciding which discharge is most suitable for a specific copper alloy:

(1) Use Type 1 discharge on complex copper alloys where the metallurgical history is important and maximum impurity content is not too low, e.g. nickel silver, aluminium brass, and copper–silicon–manganese alloys.

(2) Use Type 2 discharge on materials where a very low maximum impurity content is important, e.g. copper, phosphor bronzes, and high-purity red brasses.

(3) Use Type 3 discharge where precision as well as low impurity content is important, e.g. copper, high-purity yellow brasses, leaded brasses, tin brasses, &c.

III.—POLARITY CONSIDERATIONS

The sparking technique employed for these analyses is the point-to-plane spark, using a specimen with a machined flat opposed by a pointed graphite counter-electrode. Since the discharge is operating between dissimilar electrodes, polarity effects are important. For short-duration pulses of high peak current, positive polarity of the sample has been found to produce more desirable analysis conditions than negative polarity: the line-to-background ratios for impurity lines are greater, the curve for zinc has a greater slope, and random curve shifts are substantially less. A study of the zinc curve over a period of two years, under a variety of conditions, showed very little shift for positive sample polarity. At the same time curves for negative sample polarity showed shifts as high as 3% for 30% zinc.

For arc-like pulses of long duration, different conditions exist. For this type of discharge, sensitivity is the important factor. Investigation revealed that for the analysis of copper and brasses a five-fold increase in sensitivity resulted when the sample was negative instead of positive. Since a Type 2 discharge is over-damped as much as possible and the pulse maintained within the confines of one half cycle, it borders on a D.C. arc and as such is subject to some of the conditions controlling the D.C. copper arc.

Milbourn,[5, 6] in an investigation of the physical nature of the D.C. copper arc, demonstrated conclusively that in the presence of oxygen copper at the cathode is readily attacked and volatilizes as CuO. He also demonstrated that when no melting occurs at the anode, very little of the copper volatilizes from the positive pole. If the above conditions are observed, a copper arc of great uniformity results, having excellent analytical properties.

Early in 1946, the author had the opportunity to discuss personally with Mr. Milbourn the application of his D.C. copper-arc theories to a unidirectional pulse-like discharge, and there was agreement on the idea that this type of discharge would give good precision and high sensitivity if the sample were negative instead of positive. As investigation proved, the over-damped discharge displayed these properties to a marked degree. In addition, the precision of the Type 2 discharge, though not as good as that of Types 1 and 3, is greatly superior to that of the D.C. arc.

IV.—APPARATUS

Samples for analysis are in the form of small castings: discs 2½ in. in dia. and ½ in. thick; rectangular blocks 2½ × 2½ × 1 in. thick; or small squares 1 × 1 × 1 in. (see Fig. 4, Plate XX). Suitable flats for sparking are machined in a lathe or milling machine. In most cases, the analysis curves developed were independent of the shape or origin of the casting.

The spectrograph used is a large Bausch and Lomb prism spectrograph with quartz and glass optics, having a reciprocal dispersion of 4Å/mm. in the 2900Å. wave-length region. A cylindrical quartz lens of 150 mm. focal length is placed directly in front of the slit. Its curvature is in the vertical plane and serves to focus the discharge vertically on the collimator opening. The distance between the discharge and the slit (23 cm.) keeps the wandering spark image within the horizontal aperture of the collimator. The discharge gap used (2·5 mm. or 0·1 in.) serves to keep the image of the electrodes just outside the vertical opening of the collimator. The slit width is 0·04 mm. With the above adjustments, the spectrum produced consists of easily measured lines against a satisfactory background.

The plates used are Eastman Kodak SA No. 1, which are developed at 70° F. (21° C.) in Kodak developer D19 for 5 min. The plates have high contrast, fine grain, and low background.

A Hilger non-recording microphotometer is used to read line densities. The scanning slit setting is 0·10 × 10 mm., equivalent to a scanning area of 10,000 square microns at the plate. All line densities are read in terms of the percentage transmission on a scale 50 cm. long. Readings are recorded to within 0·5 mm. or 0·1% transmission. The percentage transmission readings are converted to arbitrary intensities by means of an emulsion characteristic curve.

The spectrum of a D.C. iron arc, photographed with a rotating stepped sector in front of the slit, is used to set up the emulsion characteristic curve. The spectrum consists of eight steps with an intensity relationship of the form $\log \frac{I_1}{I_2} = 0{\cdot}2$ between adjacent steps. The percentage transmissions of a sequence of steps of a number of iron lines are plotted against the corresponding intensity intervals on logarithmic co-ordinate paper. Since the curves obtained by this method are not sufficient to give the whole density range desired for an emulsion characteristic curve, the various partial curves are shifted parallel to the intensity axis until they all overlap. Satisfactory overlapping is a good indication of self-consistency for all lines in the wave-length region used. Since logarithmic intensity ratios are used exclusively, there is no need to resort to absolute intensity determinations. Consequently, the emulsion characteristic curve may be very conveniently used with a negative logarithmic percentage transmission scale as the ordinate and a sliding logarithmic intensity scale as the abscissa.

Fundamental formulæ are :

$$D = \gamma \log E$$

and $$D = \log 100/T$$

$\therefore$ for lines L_1 and L_2

$$D_1 - D_2 = \gamma \log \frac{E_1}{E_2} = \gamma \log \frac{I_1}{I_2}$$

or $$-\log \frac{T_1}{T_2} = \gamma \log \frac{I_1}{I_2}$$

where $D =$ film density, $E =$ exposure, $I =$ intensity, $T =$ percentage transmission, and $\gamma =$ development factor or film gamma.

Since the highest sensitivity as well as precision is required, background considerations are relevant, and in many cases background corrections are made. If the line intensity read is considered to be the sum of actual line intensity plus background intensity, the following derivation applies :

$$I\,(\text{line}) = I\,(\text{read}) - I\,(\text{background})$$

$$\therefore \quad \frac{I_L'}{I_L''} = \frac{I_R' - I_B'}{I_R'' - I_B''} \quad \text{or}$$

$$\frac{I_L'}{I_L''} = \frac{I_R'/I_B' - 1}{I_R''/I_B'' - 1} \times \frac{I_B'}{I_B''}$$

Thus the relative intensity of any two lines can be determined by manipulating two sliding logarithmic intensity scales which function as the scale of abscissæ for the emulsion characteristic curve.

V.—DETERMINATION OF ZINC

As mentioned earlier, the Type 3 discharge has proved to be the most useful for the brasses, and the paper is confined to a detailed consideration of its application to the determination of zinc, lead, and iron.

The curve for the estimation of zinc in the range 20·0–40·0% appears in Fig. 3. Examination of curve *A* (relative intensity Zn 3018/Cu 3022 against zinc concentration, corrected for background) shows that its slope is greater than 45°. The slope of curve *B* (relative intensity Zn 3018/Cu 3022 against Zn/Cu relative concentration, corrected for background) is also greater than 45°. Therefore, the intensities of the zinc and copper lines are not linear functions of their respective concentrations. The increased slope of the zinc curve is probably due to the reduction in the boiling point of the alloy with higher zinc content and a resulting enhancement of the intensity of the zinc line. (For the determination of nickel in cupro-nickel, the curves are quite flat probably owing to the opposite effect). This steep slope greatly contributes to the accuracy of the zinc determination by reducing the relative effect of emulsion and microphotometer errors.

For the determination of the precision of the spectrographic estimation of zinc in 70 : 30 brass, the author is indebted to the methods used by Vincent and Sawyer [7–9]. The total error associated with spectrochemical analysis can be considered as composed of errors due to : (1) sample inhomogeneity, (2) source, (3) microphotometer, and (4) emulsion. Inhomogeneity error was ignored, since copper and zinc form a homogeneous solid solution at 30% zinc. Emulsion and microphotometer error, though incidental to the analysis, were computed in order to arrive at a proper evaluation of error associated with the Type 3 discharge.

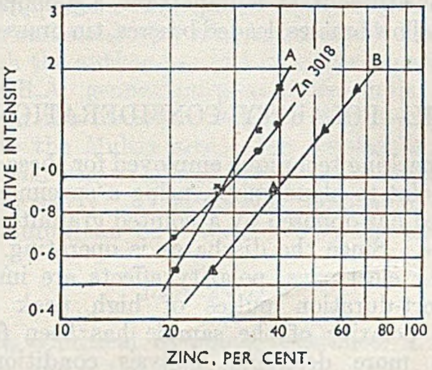


Fig. 3.—Curves for Determination of Zinc in Brass, with Type 3 Discharge, Using Cu 3022 as Reference.

Curve A : Relative intensity corrected for background vs. zinc concentration.

Curve B : Relative intensity corrected for background vs. relative zinc concentration.

To arrive at a total error, three samples, poured from the same heat, were each sparked 32 times, making a total of 96 analyses. Six plates were used, 16 analyses per plate, and the samples were paired on the plates in the following manner :

Plate 1	Sample 1—eight analyses	
	Sample 2— ,, ,,	
Plate 2	Sample 3— ,, ,,	
	Sample 1— ,, ,,	
Plate 3	Sample 2— ,, ,,	
	Sample 3— ,, ,,	&c.

The plates were sparked over a period of a week in order to include variables associated with the passage of time, such as random curve shift, variation in plate-development conditions, optical misalignment, &c.

The standard deviation, σ_1, from an overall average was computed and found to be $\pm 1{\cdot}18\%$ of content or 30% Zn $\pm$ 0·355%. In order to break this figure down into a standard deviation per plate, the sixteen results on each plate were averaged and the difference between the individual results and the plate average was determined. The standard deviation, σ_2, of these differences was computed for the 96 analyses and found to be ±0·87% of content or 30% Zn ± 0·261%.

Microphotometer error, σ_m, was computed by taking three readings of percentage transmission R_1, R_2, and R_3 on 40 lines. Differences D_1, D_2, and D_3 for successive lines R' and R'' were determined as $D_1 = |R_1' - R_1''|$, $D_2 = |R_2' - R_2''|$, and $D_3 = |R_3' - R_3''|$. Deviations were computed as follows $d_1 = |D_1 - D_2|$, $d_2 = |D_1 - D_3|$, and $d_3 = |D_2 - D_1|$. The standard deviation per pair of lines, computed from these deviations, was ±0·004 intensity units.

Examination of the Zn 3018/Cu 3022 curve shows that a change of 0·01 in intensity unit at 30% zinc is equivalent to a change of 0·222% zinc or 0·74% of the content. Consequently, the microphotometer error is equivalent to ±0·30 of the content or 30% Zn ± 0·09%. This result is comparable with that of Grossman, Sawyer, and Vincent,[9] who obtained ±0·40% of the content, and Vincent and Sawyer,[8] who obtained ±0·18% of the content under the most favourable conditions.

Emulsion error σ_e, was determined by reading the relative intensity of Zn 3018/Cu 3022 at each end of the line pair. The paired readings, after adjustment for a slight constant difference due to the optics of the system, were subtracted to obtain the deviation due to emulsion error plus microphotometer error. The standard deviation per line pair for 47 deviations was computed and was found to be ± 0·0086 intensity unit. To compute σ_e, the following relation is used:

$$\begin{aligned}\sigma_e &= \pm\sqrt{0{\cdot}0086^2 - \sigma_m^2}\\ &= \pm\sqrt{0{\cdot}0086^2 - 0{\cdot}004^2}\\ &= \pm\ 0{\cdot}0076 \text{ intensity unit.}\end{aligned}$$

This result is equivalent to an error of ±0·57% of content or 30% Zn ± 0·17%.

To compute the source error due to the Type 3 discharge, the following relation is used:

$$\begin{aligned}\sigma_s &= \pm\sqrt{\sigma_2^2 - \sigma_m^2 - \sigma_e^2}\\ &= \pm\sqrt{0{\cdot}886^2 - 0{\cdot}30^2 - 0{\cdot}57^2}\\ &= \pm\ 0{\cdot}61\%\end{aligned}$$

Consequently, the source error is equivalent to ±0·61% of the content or 30% zinc ± 0·18%. Owing to the indirect nature of its computation, the source error can be properly viewed with some scepticism. However, a check on this figure was furnished by Mr. M. F. Hasler who, at the request of the author, performed the analysis on the Quantometer, a direct-reading instrument. He reported a result of 30% zinc ± 0·18%, using Type 3 excitation.

Note Added in Proof

For two under-damped discharges to be completely equivalent, both damping factor and period should be equal. For a Type 3 discharge, the period of pulse length is closely approximated by:

$$\text{Period} = 2\pi\sqrt{LC} \times 10^{-3} \text{ millisec.}$$

Thus L and C are uniquely defined by LC and L/C.

The author has recently been testing Type 3 discharges with pulse lengths ranging from 0·1 to 0·8 millisec., the maximum obtainable on the Multisource. As was to be expected, there is a deterioration in precision with increase in pulse length. For a discharge with constants 40μF., 400μH., and 2 ohms, and of 0·8 millisec. duration, the standard deviation in the zinc determination computed from 2 plates and 32 analyses is ±1·55% of content or 30% ± 0·47% zinc. Source error is 1·39% of content or 30% ± 0·42% zinc. Consequently source error, though still low, has more than doubled over that of the discharge ordinarily used, which has a pulse duration of approximately 0·3 millisec. Any discharge of 1·0 millisec. and over will probably have a precision equivalent to the over-damped case.

VI.—DETERMINATION OF LEAD IN LEADED BRASSES

Repeat determinations of 3% lead in selected leaded brass standards gave essentially the same accuracy as that associated with zinc. This accuracy is valid, only for samples with a uniform, very fine dispersion of the lead particles such as that shown in Figs. 5 (*a*) and (*b*) (Plate XX). In general, this very desirable fine particle dispersion can be obtained by pouring small samples into iron or steel moulds and causing a quick chill. Unfortunately, a structure where large agglomerates exist renders the spectrochemical determination of lead subject to large errors, and this structure sometimes occurs in the analysis sample in routine works' sampling procedures.

An investigation of the effect of lead dispersion on the lead curves shows a correlation between spectrographic relative intensities and the type of dispersion. In general, for samples of the same lead concentration, relative intensities for selected lead/copper lines increase with increased size of the lead particles. Moreover, this increase is complicated by greater scatter in individual results. Consequently, if lead dispersion is not controlled, it is impossible to obtain either a standardized curve or lead results with the required precision.

The photomicrographs of the six samples in Figs. 5 (*a–f*) (Plate XX) illustrate the forms of lead dispersion encountered in spectrographic samples of 3–4% leaded brass cast by routine procedures in the works. The relative intensities of the line pair Pb 2823/Cu 3022 were plotted against pre-spark time for the above six samples (Fig. 6). Samples 16 and 18, in which the lead is uniformly dispersed and has fine particle size, show the same decrease in relative intensity until 60 sec. is reached, when the relative intensities level off. The samples with large particle size all show much higher initial relative intensities and steeper curves before the relative intensities level off. Border-line cases, such as sample 81, will eventually fall into line if the pre-sparking time is long enough. However, in most cases, relative intensities will level off at values much higher than those compatible with the curve. For routine procedures the author has found a 30-sec. pre-spark period adequate to align most samples with a normal range in lead dispersion. However, even with the greatest care, curve shifts due to differences in lead dispersion are all too common and contribute greatly to the analysis error. Changing the source excitation from Type 3 to Type 1 offers no improvement in a condition which originates in casting practice.

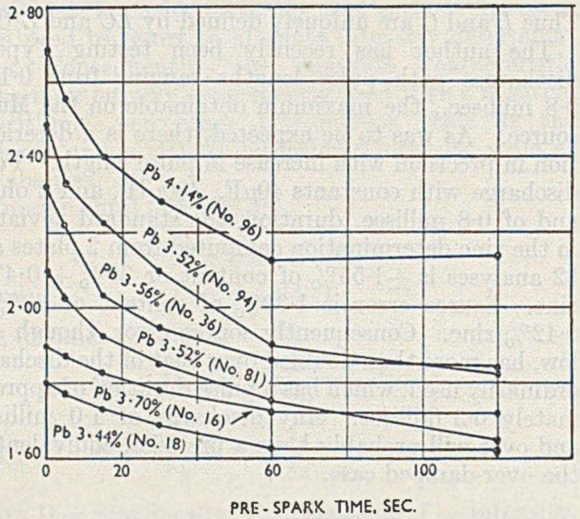


Fig. 6.—Relative Intensity of Pb 2823 to Cu 3022 Plotted Against Pre-Spark Time for Leaded Brass, Using Type 3 Discharge.

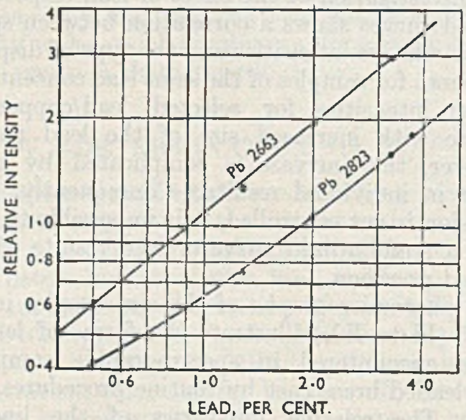


Fig. 7.—Curves for Determination of Lead in Leaded Brasses, with Type 3 Discharge, Using Cu 3022 as Reference.

Control of lead dispersion is a factor that enters into the analysis in the range 1–2% lead and becomes increasingly critical as lead content increases. The curves for the determination of lead in Fig. 7 are all based on samples that have been examined under the microscope and found to have a suitable fine particle dispersion.

VII.—DETERMINATION OF IMPURITY ELEMENTS IN BRASS

Lead and iron in the range 0·01–0·2% are the two impurities most commonly encountered in high-purity brasses. Figs. 8 and 9 show the curves for these two impurities at various copper concentrations, using Type 3 discharge. Since background densities are an appreciable part of the measured densities, the curves are corrected for background in order to determine the curve shifts due to a variable matrix element.

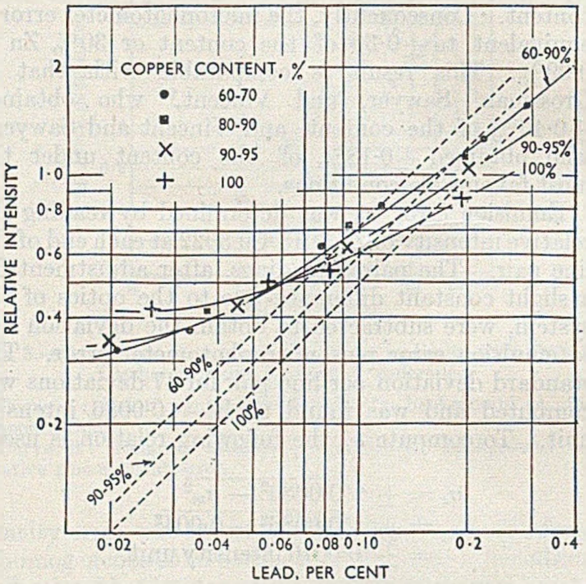


Fig. 8.—Relative Intensity of Pb 2833 to Cu 3022 Plotted Against Lead Concentration, Using Type 3 Discharge.
Broken curves corrected for background.

With the exception of the curve for the determination of iron in copper, all curves, corrected for background, have slopes of 45°. Therefore, at a given copper concentration, the relative intensities of these lines of iron and lead are directly proportional to their respective concentrations, and no self-reversal is present. In the case of iron in copper, if the iron concentration is plotted against arbitrary intensity units for the iron line instead of relative intensities, to eliminate the effect of variation in the intensity of the copper line, the slope of this line also is 45°. Therefore, the relatively shallow slope of the line for the determination of iron in copper is due to the progressive enhancement of the copper spectrum by increasing amounts of iron. This enhancement extends to the lead line as well, since the curve for lead in copper maintains its 45° slope irrespective of the amount of iron present.

A single curve is adequate for lead in all brasses in the range 60–90% copper. Between 90 and 100% copper, however, close tolerances should be maintained on the copper content in order to preserve accuracy in the lead determination. With iron the situation is slightly different. Close tolerances should be maintained in the ranges 70–80% and 95–100% copper to preserve accuracy in the iron determination.

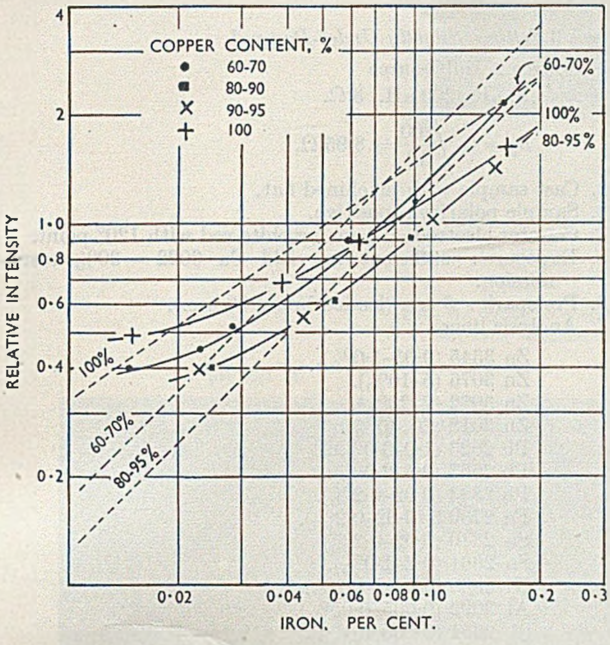


Fig. 9.—Relative Intensity of Fe 2739·6 to Cu 3022 Plotted Against Iron Concentration, Using Type 3 Discharge.

Broken curves corrected for background.

ACKNOWLEDGEMENTS

The author wishes to express his appreciation to Dr. R. A. Wilkins for permission to publish the results obtained in this work, and to thank Messrs. F. F. Poland and L. H. Decker, whose co-operation enabled him to devote extensive time to this investigation. Thanks are also due to Mr. Robert Hahn for his many chemical analyses and to Mr. Donald Destito for his careful preparation of the photomicrographs.

REFERENCES

1. M. F. Hasler and H. W. Dietert, *J. Opt. Soc. Amer.*, 1943, **33**, 218.
2. M. F. Hasler and J. W. Kemp, *J. Opt. Soc. Amer.*, 1944, **34**, 31.
3. A. Walsh, *Bull. B.N.F.M.R.A.*, 1946, (**201**), 60.
4. A. Walsh, *Spectrochim. Acta*, 1950, **4**, 47.
5. M. Milbourn, *J. Inst. Metals*, 1943, **69**, 441.
6. M. Milbourn, *Proc. Phys. Soc.*, 1947, **59**, 273.
7. H. B. Vincent and R. A. Sawyer, *J. Opt. Soc. Amer.*, 1941, **31**, 639.
8. H. B. Vincent and R. A. Sawyer, *J. Opt. Soc. Amer.*, 1943, **32**, 686.
9. H. H. Grossman, R. A. Sawyer, and H. B. Vincent, *J. Opt. Soc. Amer.* 1943, **33**, 185.

APPENDIX

SUMMARY OF PROCEDURE

Type 1 Setting, Slightly Over-Damped

1. Source : Multisource
   10 μF., 200 μH., 10 Ω.
   $$R_c = 2\sqrt{\frac{200}{10}} = 8{\cdot}95\ \Omega.$$
2. Cast sample with machined flat.
3. Sample polarity : positive.
4. Counter-electrode : ¼ in. graphite rod with 120° point.
5. Exposure; sufficient to yield Cu 2997 = 30% transmission.
6. Pre-spark : 5 sec.
7. Analysis line : Zn 3072 (20%–40% Zn).
8. Reference line : Cu 2997.
9. No background correction required.
10. Accuracy : <2·0% error.

Type 2 Setting, Heavily Over-Damped

1. Source : Multisource
   60 μF., 400 μH., 20 Ω.
   $$R_c = 2\sqrt{\frac{400}{60}} = 5{\cdot}2\ \Omega.$$
2. Cast sample with machined flat.
3. Sample polarity : negative.
4. Counter-electrode : ¼ in. graphite rod with 120° point.
5. Exposure : sufficient to bring out Cu 2858·2 (background will vary according to amount of zinc present).
6. Pre-spark : 5 sec.
7. Analysis lines :
   Pb 2833 (0·005%–0·05%).
   Fe 2739·6 (0·005%–0·05%).
   P 2535·7 (0·02%–0·2%) (phosphor bronze).
   Te 2385·7 (0·05%–0·3%) (90 : 10 tellurium brass).
   As 2349·8 (0·02–0·1%).
8. Reference line : Cu 2858·2.
9. Background correction required for low impurity concentration in high-copper alloys.
10. Accuracy : <10% error.

Type 3 Setting, Slightly Under-Damped

1. Source : Multisource
   10 μF., 200 μH., 3 Ω.
   $$R_c = 2\sqrt{\frac{200}{10}} = 8{\cdot}95\ \Omega.$$
2. Cast sample with machined flat.
3. Sample polarity : positive.
4. Counter-electrode : ¼ in. graphite rod with 120° point.
5. Exposure : sufficient to yield Cu 3022 = 30% transmission.
6. Pre-spark : 5 sec. (leaded brass, 30 sec.).
7. Analysis lines :
   Zn 3345 (0·05–1·0%).
   Zn 3076 (5–10%).
   Zn 3072 (7–20%).
   Zn 3018 (20–40%).
   Pb 2823 (1·0–3·0%).
   Pb 2663 (0·5–2·0%).
   Pb 2833 (0·02–0·2%).
   Fe 2739·6 (0·02–0·2%).
   Sn 2840 (0·02–0·2%)
   Sn 2661 (0·2–1·0%).
   Ni 3050·6 (0·02–0·2%).
   Al 3092 (0·005–0·05%).
   Si 2881 (0·005–0·1%).
   Ag 3382·9 (0·003–0·05%).
8. Reference line : Cu 3022.
9. No background correction required except to maintain accuracy in very low concentration ranges.
10. Accuracy : <2·0% error.

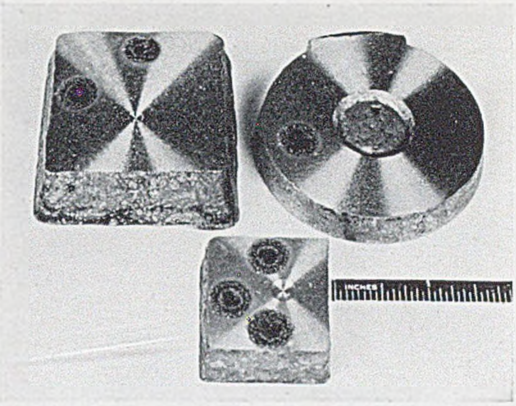
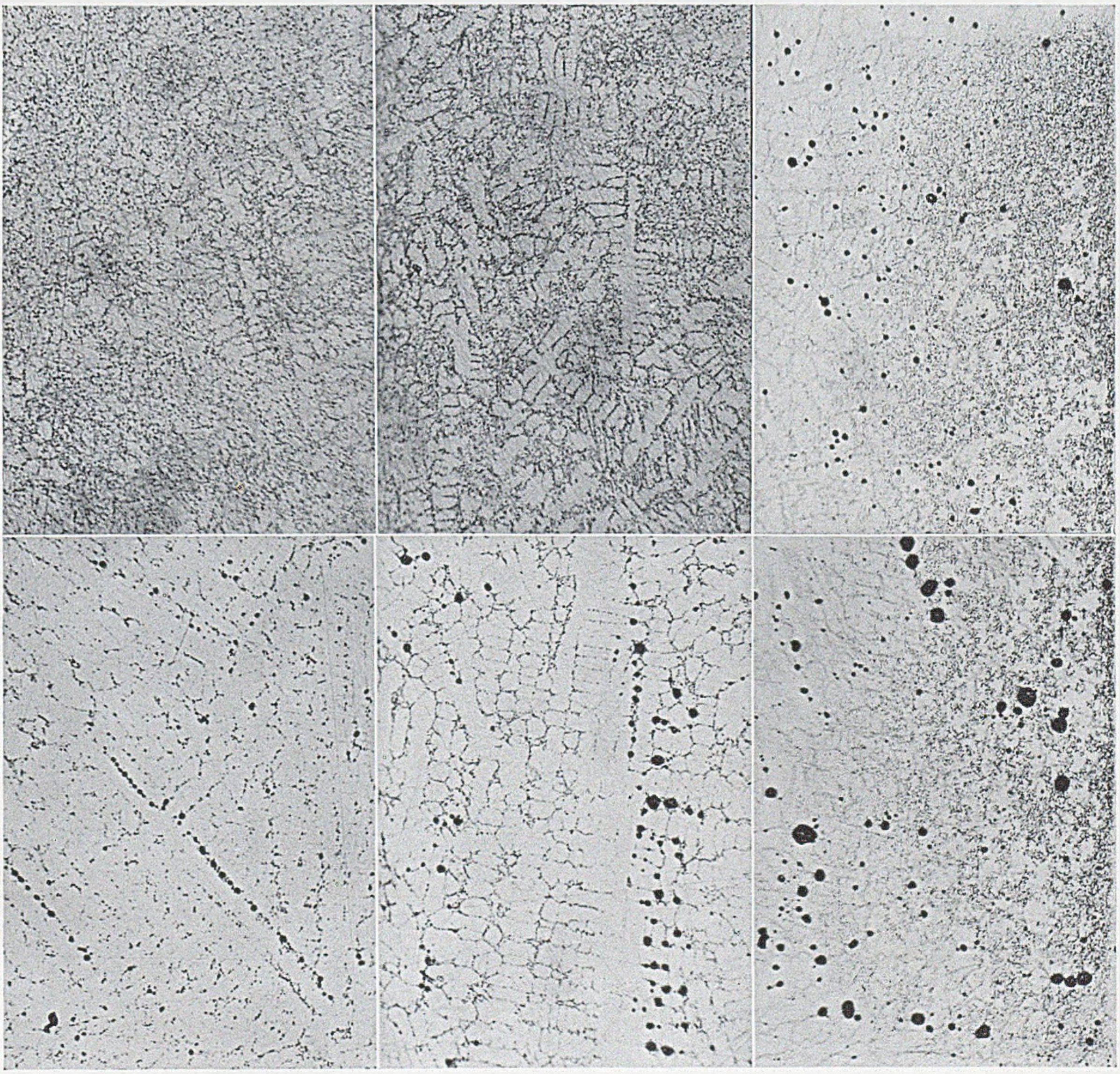


FIG. 4.—Sample Castings for Spectrographic Analysis, Showing Machined Surface and Discharge Spots.



(*a*) No. 16 (*b*) No. 18 (*c*) No. 81

(*d*) No. 36 (*e*) No. 94 (*f*) No. 96

FIG. 5 (*a*)–(*f*).—Photomicrographs Showing Range of Lead Particle Size Encountered in Leaded Brass Samples for Spectrographic Analysis. × 100.

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THE EQUILIBRIUM DIAGRAM OF THE SYSTEM NICKEL–MANGANESE *

By B. R. COLES,† B.Sc., Junior Member, and
W. HUME-ROTHERY,‡ M.A., D.Sc., F.R.S., Member

SYNOPSIS

The equilibrium diagram of the nickel–manganese system above 800° C. has been determined by a combination of thermal, microscopical, and X-ray methods; some information about the diagram at lower temperatures was also obtained. The liquidus and solidus pass through a smooth minimum at 62 at.-% manganese and 1018° C. Nickel is freely soluble in γ-manganese and increases the axial ratio of the face-centred tetragonal structure until it becomes cubic and unites with the solid solution of manganese in nickel. Immediately below the solidus there is thus a complete range of solid solution extending across the diagram. At lower temperatures (c. 910° C.) this solid solution in the equi-atomic region is transformed into a β phase with a body-centred cubic structure which cannot be retained by quenching. At a still lower temperature the β phase is transformed into a phase with a face-centred tetragonal superlattice of the CuAu type. The temperature of the $\delta \rightleftharpoons \gamma$ transformation of manganese is raised by the addition of nickel, whilst those of the $\gamma \rightleftharpoons \beta$ and $\beta \rightleftharpoons \alpha$ transformations of manganese are lowered. The diagram in some respects resembles that of the system nickel–zinc, and this suggests that manganese acts as a divalent element in some of the alloys.

I.—INTRODUCTION AND PREVIOUS WORK

The present investigation is concerned mainly with the equilibrium diagram of the system manganese–nickel at temperatures above 800° C., although some information has also been gained of the structures at lower temperatures. In describing the previous work it is not therefore proposed to discuss the earlier investigations of nickel-rich alloys at the relatively low temperatures where interesting magnetic properties are associated with ordered structures. When the present work was started the only general investigation of the system was that of Dourdine,[1, 2] who used impure alumino-thermic manganese. Shortly after the present work was begun, the results of a complete investigation of the system were published by Köster and Rauscher,[3] who used electrolytic manganese for alloys containing more than 75% of this metal, and less pure manganese for the remaining alloys. Their diagram is reproduced in Fig. 1 and is of interest in many ways. It shows a minimum in the liquidus at 62 at.-% manganese (61 wt.-% manganese). For convenience all alloys will be referred to by their atomic percentages of manganese. The minimum in the liquidus is smooth, and at high temperatures a continuous solid solution extends across the diagram from nickel to γ-manganese, the axial ratio of which is increased by the addition of nickel until, between 25 and 30 at.-% manganese, it becomes equal to 1·00, and the resulting face-centred cubic phase is continuous with the face-centred cubic solid solution in nickel. This solid solution is, however, broken into by a phase denoted δ in the equi-atomic region which, according to Köster and Rauscher, rises almost, but not quite, to the solidus curve, in contrast with the results of Dourdine, which suggested that a peritectic reaction was involved. There is general agreement that at low temperatures the MnNi phase has a face-centred tetragonal structure. According to the thermal analysis of Köster and Rauscher MnNi transforms between 600° and 700° C. to an ordered face-centred cubic structure, but this has not been confirmed by the present work, which shows that at high temperatures the structure is that of a body-centred cube. The work of Dourdine (*loc. cit.*) had suggested phases based on the compositions Mn_3Ni_2 and Mn_3Ni_4, and Kaya and Kussmann[4] claimed to have discovered a face-centred cubic phase corresponding to the latter, although this was not confirmed by Valentiner and Becker,[5] and Köster and Rauscher (*loc. cit.*) considered that the only intermediate phases were those based on MnNi and $MnNi_3$, the latter being a superlattice structure. The work of Köster and Rauscher was not very thorough in the equi-atomic region, and their reliance on the results of X-ray examination of quenched specimens did not appear justified. For the manganese-rich alloys their results suggested that the temperature of the $\alpha \rightleftharpoons \beta$ transformation was lowered markedly by the addition of nickel, which produced a similar but less-marked effect on the $\beta \rightleftharpoons \gamma$ transformation, for which a broad $(\beta + \gamma)$ region was shown. The lines for the $\gamma \rightleftharpoons \delta$ transformation in Fig. 1 were inserted on the basis of the microscopical

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† Lecturer, Imperial College of Science and Technology, London. Formerly Inorganic Chemistry Laboratory, Oxford.

‡ Royal Society Warren Research Fellow, Oxford.

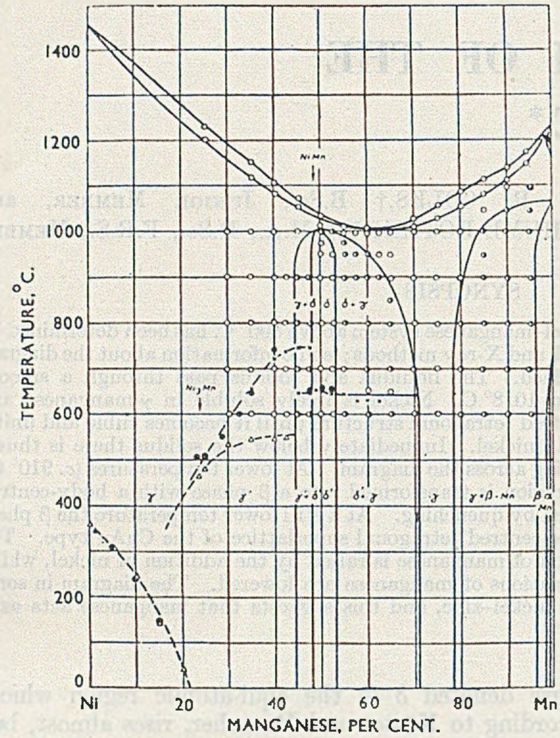

Fig. 1.—Equilibrium Diagram of the System Nickel–Manganese (according to Köster and Rauscher [3]).

examination of cooled alloys and were not detected by thermal analysis. In these circumstances complete re-investigation of the system has been made at the higher temperatures.

II.—EXPERIMENTAL PROCEDURE

1. Materials Used

The nickel used in the present work was specially selected Mond carbonyl nickel in the form of medium-sized shot. After the shot had been cleaned with ether to remove surface grease and dirt, the analysis obtained was 99·95% nickel, 0·02% carbon, and 0·02% iron. We have to thank The Mond Nickel Co., Ltd., for kindly presenting this metal. The manganese used was electrolytic metal of 99·98% purity, containing traces of iron and chromium. This was treated in purified hydrogen at 800° C., in order to remove superficial oxidation.

2. Preparation of Alloys

The alloys, both for thermal analysis and for annealing, or X-ray experiments, were prepared in a valve-operated induction furnace without the use of a heater sleeve. The first alloys were melted in ΔRR alumina crucibles (Morgan Crucible Company), using an atmosphere of purified hydrogen, but these contained large gas holes owing to the great difference between the solubilities of hydrogen in the solid and liquid alloys. Later alloys were therefore prepared under an atmosphere of purified argon and sound ingots were obtained. The vacuum system, furnace arrangements, and methods for the purification of hydrogen and argon have been described by Carlile, Christian, and Hume-Rothery [6] in their work on chromium–manganese alloys. The lower temperatures and reactivities involved in the present work reduced the risk of contamination by nitrogen, and as will be seen from what follows a satisfactory standard of purity was obtained. The loss of manganese by volatilization was also much smaller than in the work on chromium–manganese alloys, and even in manganese-rich alloys the loss rarely exceeded 1–2 g. in a 50-g. ingot.

3. Thermal Analysis of the Alloys

Thermal analysis was carried out by thermocouple methods, using the platinum-wound furnace shown in Fig. 1 of the paper by Carlile, Christian, and Hume-Rothery (*loc. cit.*). For this purpose the alloys were previously melted in the induction furnace, and were then remelted in the platinum furnace using a crucible with a re-entrant thermocouple sheath; to accommodate this the ingots were cut in half and grooved in order to allow the sheath to be inserted when the crucible was packed. The brittle manganese-rich alloys were broken up and packed into the crucible. The thermal analysis was carried out in an atmosphere of purified argon, and owing to the volatility of the manganese, whose vapour penetrates alumina thermocouple sheaths, it was necessary to calibrate the thermocouple during the actual experiment by the immersion-couple technique of Carlile, Christian, and Hume-Rothery. In this method a cooling curve is taken, the temperatures being read by means of the re-entrant thermocouple which may become contaminated during the process. As soon as the arrest is established the wires of a second or immersion thermocouple are pushed down a sheath which has also been in the melt and serves as a stirrer. Alternate readings are then taken on the two thermocouples until their temperature/time curves are parallel, after which the wires of the immersion thermocouple are withdrawn into a cold part of the sheath. In this way the re-entrant thermocouple is calibrated immediately after the arrest point against the immersion thermocouple which is exposed to high temperatures only for short periods. The immersion thermocouple itself was frequently recalibrated, and for high-temperature calibration it was found convenient to calibrate against the freezing point of pure nickel (1453° C.); the results obtained in this way agreed well with those obtained by the wire method for the gold and palladium points.

A complete experiment was usually carried out in the following way. After packing the crucible with material previously melted in the induction furnace, the apparatus was assembled and evacuated and the temperature raised to about 700° C. until the whole assembly was thoroughly outgassed. Argon was introduced and a heating curve was taken in which the solidus was approached at a rate of about 5° C./min. The beginning and the end of the arrests on the heating curve gave a rough indication of the solidus and liquidus points, and the latter was then determined accurately by a cooling curve taken, with stirring, at a rate of 1°–3° C./min. When the alloy was completely solid, it was maintained at about 30° C. below the solidus for about half an hour, after which a slow heating curve was taken in order to determine the solidus point, and a second slow cooling curve to confirm that no appreciable change in composition had occurred. In general, the two liquidus determinations were in very good agreement, and if any slight difference occurred the second value was preferred because the composition was determined by the analysis of the complete ingot after the experiment. In some of the later experiments it was possible to prepare the initial ingots in the induction furnace in crucibles of the same size as those used for the subsequent thermal analysis. Under these conditions a close fit between the thermocouple sheath and alloy could be ensured, and after a preliminary anneal 30° C. below the solidus it was thought justifiable to take a single slow heating curve to determine the solidus, followed by a slow cooling curve for the liquidus. Experiments showed that it was essential to give a homogenizing treatment just below the solidus if the heating-curve points were to be relied upon; in work where this precaution was omitted the apparent solidus point might be as much as 20°–30° C. too low owing to coring of the ingot.

In general, the experiments for the determination of the liquidus and solidus were run by themselves, but in some cases cooling curves were followed to low temperatures, and the arrests due to low-temperature transformations were observed, and are discussed later.

The analysis of the cooling-curve ingots was carried out by Messrs. Johnson, Matthey & Co., Ltd., and the authors must express their thanks to Mr. A. R. Powell for his interest in the work. Both nickel and manganese were determined, and for the ingots included in Table I the analytical totals for the two metals lie between 99·92 and 100·06.

III.—EXPERIMENTAL RESULTS

1. The Liquidus

The results obtained from the cooling curves are shown in Table I. From the freezing point of nickel the liquidus falls smoothly to a flat minimum at 1018·3° C. and 62 at.-% manganese. The liquidus curve in this region is so flat that the exact composition of the minimum point could not be determined, although the curvature of the line through the adjacent points shows that it cannot be far from 62 at.-% manganese. In the experiment with alloy 62·5, the cooling curve was taken successfully, but an accident then occurred as a result of which the specimen was oxidized and could not be analysed. The composition of this alloy was therefore estimated from the weights of metal used, and in view of the close agreement between the intended and analysed compositions in this region, the value is not likely to be in error by more than a few tenths per cent. The temperature of the minimum is appreciably higher than that obtained by previous workers, probably owing to the greater purity of the metals used. This temperature was checked not only by the calibration methods described above, but also by a calibration against the freezing point of high-purity copper. In view of the previous suggestions of a peritectic horizontal connected with the formation of the NiMn phase, and of a eutectic in place of a smooth minimum, the cooling curves in the region 45–65 at.-% manganese were taken with great care and were examined in detail, but no sign of a second arrest could be detected. Microscopical examination of the cooling-curve ingots in the region of minimum freezing point gave no indication of a eutectic structure, and the evidence is therefore that the liquidus passes through a smooth minimum. With increasing manganese content the liquidus rises to 1162° C., where a change in direction occurs owing to the peritectic reaction : $\gamma \rightleftharpoons \delta$ + liquid.

Table I.—*Liquidus and Solidus Temperatures of Nickel–Manganese Alloys.*

Alloy	Mn, wt.-%	Mn, at.-%	Liquidus, °C.	Solidus, °C.
5	94·86	95·19	1198·2	1172·1
7·5	92·59	93·04	1181·0	1156·4
10	89·63	90·21	1161·6	1135·8
17	83·17	84·07	1124·0	1092·7
20 *	78·91	79·99	1094·8	1048·0
25	72·15	73·46	1057·6	1038·7
30	68·85	70·22	1038·7	1023·4
35	63·58	65·07	1018·1	
39 †	61	62·5	1018·5	
40	58·74	60·32	1018·3	1018·3
45	54·96	56·59	1027·4	1020·7
48·5	51·28	52·93	1039·3	1031·8
50	48·44	50·09	1053·0	1036·0
55	44·91	46·55	1072·8	1051·3
60	39·69	41·29	1110·3	1084·0
65	34·97	36·49	1145·5	1117·0
70	29·78	31·19	1188·2	1155·2
75	24·90	26·16	1228·6	1197·4
80	21·03	22·11	1265·4	1233·4
85	15·78	16·68	1315·9	1288·3
90	10·59	11·20	1363·2	1340·0
95	6·21	6·59	1407·8	1386·5

* Showed effect of insufficient homogenization.
† Oxidized after liquidus arrest and not analysed.

2. The Solidus

The results of the solidus points taken from the final heating curves are shown in Column 5 of Table I. As in the case of the liquidus, a special search was made for the existence of a peritectic or eutectic horizontal in the middle of the diagram, but no evidence for this could be found and the conclusion is again that the liquidus and solidus fall to a smooth minimum at 62 at.-% manganese, and that a continuous solid solution exists at the higher temperatures. The results of the heating and cooling curves are shown in Fig. 2. In the course of the work some annealing and quenching experiments were carried out which extended into the semi-liquid region, and the results of these are included in Fig. 2 and confirm the results obtained by the heating-curve method. The details of this work are described in later sections, and unless otherwise stated the microscopical points for the solidus in Fig. 2 were obtained by annealing previously homogenized specimens for at least 30 min. at the temperatures concerned; the temperature was kept constant to within ± ½° C. by hand control of a Variac transformer. In this figure and in Fig. 3 the full points refer to alloys whose compositions were determined by the chemical analysis of the actual specimen examined microscopically, whereas the open points refer to alloys whose compositions were inferred from the analysis of another portion of the same ingot.

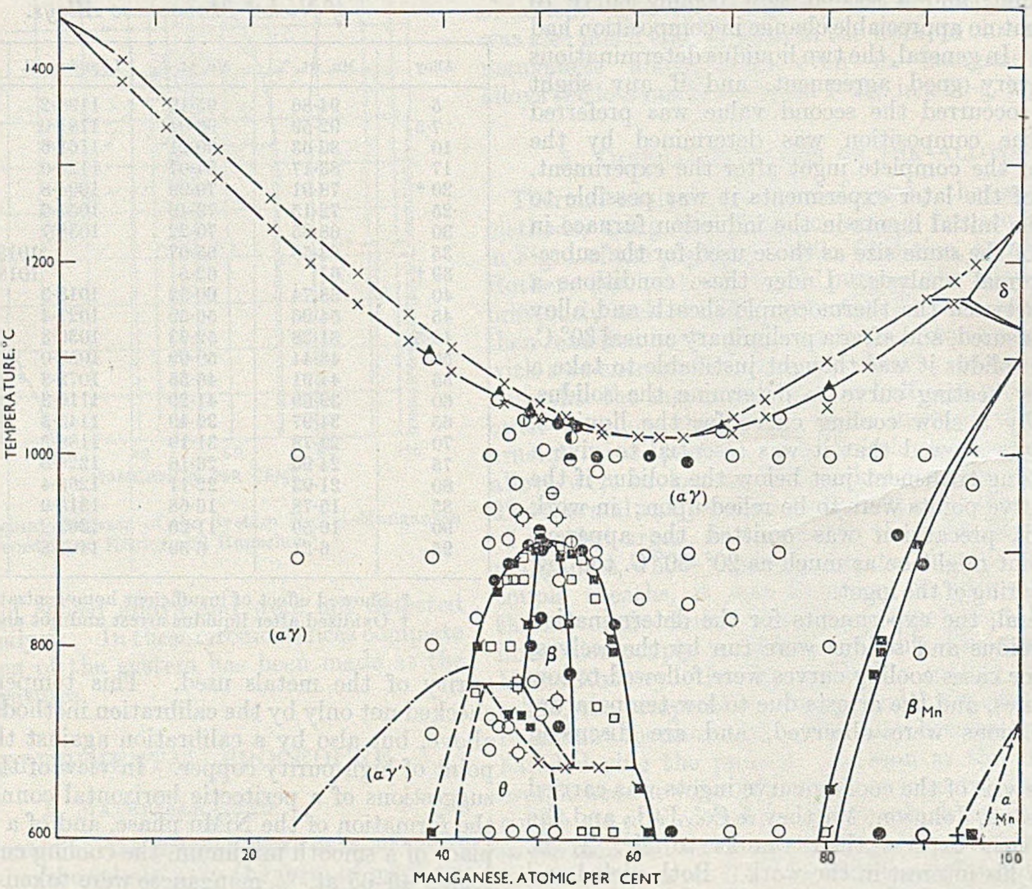

Fig. 2.—The Nickel–Manganese System.

Key.

○ ● Single-phase alloy.

⊖ ◓ Single-phase alloys completely decomposed.

⦶ ◐ Single-phase alloys partly decomposed.

▪ Trace of second phase.

□ ■ Two-phase alloy.

▲ Solid + liquid alloy.

⌖ High-temperature X-ray single-phase.

⌖ High-temperature X-ray two-phase.

× Thermal arrests.

\+ X-ray boundary.

The full or half-full symbols refer to alloys analysed after the heat-treatment shown. Open symbols refer to alloys whose compositions are based on the analysis of another portion of the same ingot, and sometimes to the synthetic compositions.

3. The Continuity of the Ni $\rightarrow$ γ Mn Solid Solution at High Temperatures

Microscopical examination showed that all specimens in the range 0–49 at.-% manganese, when annealed and quenched from temperatures above 910° C., gave single-phase undecomposed structures, and single-phase structures were also obtained in alloys containing more than 54 at.-% manganese until the (γ-Mn + β-Mn) region was reached. In the range 49–54 at.-% manganese, alloys quenched from above 910° C. showed decomposed microstructures; in some of these the whole section was of uniformly decomposed appearance, whilst in others structures were obtained which simulated those of two-phase alloys, although careful examination suggested that these effects were really due to incomplete decomposition of the specimen. Thus Figs. 4 and 5 (Plate XXI) illustrate the structures of alloys 49·1 and 53·5 after quenching from 1035° and 995° C., respectively. They show smooth, light-etching areas in a background of decomposed material. The boundaries between the two are irregular and different in shape from those to be expected from an alloy consisting of two phases in equilibrium at the quenching temperature, and as these structures were not reproducible it was concluded that in Figs. 4 and 5 the light areas were portions which had not undergone decomposition during quenching. These regions were further examined by high-temperature Debye–Scherrer X-ray photography, using the camera of Hume-Rothery and Reynolds.[7] The filings were contained in evacuated silica capillaries, and chemical analyses were carried out on the actual specimens after the X-ray exposure. The first specimen examined contained 48·1 at.-% manganese and was prepared from a lump of alloy previously homogenized at 920° C. and quenched. The X-ray specimen was heated to 935° C. and kept at this temperature for 20 min., after which an exposure of $1\frac{1}{2}$ hr. was made with unfiltered iron radiation. The film showed the diffraction lines of a face-centred cubic structure with $a = 3{\cdot}73$ kX, but an accidental fogging of one end of the film prevented an accurate determination of the lattice spacing. This result confirms the microscopical evidence. A similar experiment with an alloy containing 52·3 at.-% manganese also showed only the lines of a face-centred cubic structure with $a = 3{\cdot}741(0)$ kX. This experiment indicates that the intermediate MnNi phase which, as described below, has a body-centred cubic structure, does not exist at the higher temperature, but is replaced by a face-centred cubic solid solution. The conclusion of Köster and Rauscher is thus confirmed, although the temperature of the transformation is clearly much lower than was previously imagined. For convenience we shall denote this phase by the symbol $\alpha\gamma$.

4. The Intermediate Phase in the Equi-Atomic Region

The work of previous investigators had shown that the low-temperature modification of the phase in the equi-atomic region possesses a face-centred tetragonal structure. This has been confirmed in the present work and a superlattice of the CuAu type has been identified; this phase will be denoted by θ. In the region 670°–760° C. this phase is transformed into another which may be denoted β. Experiments with filings quenched from above the transformation temperature gave sharp lines of a face-centred cubic structure with no indication of the decomposition suggested by the microstructures of the quenched alloys. These X-ray films agree with those of Köster and Rauscher, but work with the high-temperature camera has shown conclusively that the high-temperature phase has a body-centred cubic structure. The following two experiments are of interest:

(1) Alloy 52·3 when photographed at 745° C. showed only the lines of a body-centred cubic structure of $a = 2{\cdot}968(3)$ kX.

(2) Alloy 48·1 was carried through the sequence of treatments indicated in Table II in the high-temperature camera with the results given.

Table II.—*Effect of Heat-Treatment on Structure of Alloy Containing* 48·1 *at.-% Manganese.*

Heat-Treatment	Structure
Exposure at 763° C.	b.c.c. + f.c.c.
Cooled to room temp.	f.c. tetragonal.
Exposure at 783° C.	b.c.c. + f.c.c.
Cooled to room temp.	f.c. tetragonal.
Exposure at 700° C.	f.c. tetragonal.
Cooled to room temp. and then exposed at 713° C.	mixture of blurred lines.
Cooled from 713° to 687° C. and then exposed	f.c. tetragonal.
Heated from 687° to 800° C.	b.c.c. + f.c.c.
Cooled to room temp.	f.c. tetragonal.

The specimen was analysed after the above series of exposures, and the total of the percentages of nickel and manganese was 99·2. Mr. A. R. Powell gave it as his opinion that this low total was probably due to the extreme smallness of the sample and not to contamination. Another specimen made from the same batch of filings showed a face-centred tetragonal structure at room temperature, and a mixture of body-centred cubic and face-centred tetragonal structures at 759° C. These results show that the peritectoid decomposition of the θ phase takes place at 761° ± 2° C. The way in which filings quenched from the homogeneous β region gave sharp lines of a face-centred cubic solid solution is very interesting, and emphasizes the desirability of combining X-ray and microscopical methods, because no suggestion of decomposition would have been obtained from the X-ray films.

5. The ($\alpha\gamma + \beta$), ($\theta + \beta$), and ($\beta + \alpha\gamma$) Regions

The boundaries of the phase fields in the region 40–60 at.-% manganese were determined by microscopical methods. For this purpose the furnace-cooled ingots were first homogenized by heating for 3 days at a temperature about 50° C. below the solidus of the alloys concerned. This treatment was carried out in a platinum furnace which was outgassed and filled with argon. For subsequent anneals in the range 800°–950° C. each specimen was placed in a sealed, evacuated silica capsule. Analysis of the specimens showed that no contamination by silicon occurred in this temperature range, although for the higher temperatures near to the solidus it was desirable to place the specimens in alumina collars inside the silica capsules. The capsules were then heated in furnaces controlled by Foster temperature regulators, the accuracy of the control being of the order ± 2° C.

After quenching into iced water, the specimens were examined microscopically. When quenched from 950° C. all alloys except those in the range 45–55 at.-% manganese could be cut with a saw, but with specimens annealed at lower temperatures most of the alloys in the range 40–60 at.-% manganese were too hard for this and were therefore sectioned with a high-speed bonded carborundum disc, using a very light pressure and a continuous stream of cold water to prevent overheating. After the usual grinding on emery paper, the alloys were polished on a rotary wheel, using magnesia powder.

For alloys in the range 0–60 at.-% manganese the most suitable etching reagents were Merica's reagent,* and the acidified alcoholic solution of ferric chloride recommended by Carapella.† The latter reagent does not remove much of the deformed layer produced by grinding and polishing, and it was therefore only used after a preliminary etch with Merica's reagent.

In the nickel-rich alloys the face-centred cubic α phase is etched light by the above reagents, which also reveal the grain boundaries. On approaching the equi-atomic region decomposition readily takes place on quenching. Thus alloy 53·5 quenched from 1017° C. gave the structure shown in Fig. 6 (Plate XXI), with decomposition in the grain boundaries of the $\alpha\gamma$ solid solution. Alloy 52·3, quenched from 1036° C., gave the structure of Fig. 7 (Plate XXI), showing a little chilled liquid between crystals of the $\alpha\gamma$ phase which has decomposed to an acicular structure resembling that illustrated in Figs. 4 and 5 (Plate XXI).

Alloys in the $(\alpha\gamma + \beta)$ region etched so that the α phase appeared light, while the β phase always decomposed. The pure β phase decomposed on quenching to give the characteristic martensitic structure shown in Fig. 8 (Plate XXI), which is a photomicrograph of the alloy 50·5 quenched from 830° C. In the $(\beta + \gamma)$ region the γ phase appeared as light crystals in contrast to the decomposed β structure.

The present paper is concerned mainly with the diagram above 800° C., but some quenching experiments were made at slightly lower temperatures, which showed that in an $(\alpha + \theta)$ alloy, using Carapella's reagent, the θ phase etched as the darker of the two phases. In the $(\theta + \beta)$ alloys the θ phase appeared light against the characteristic structure of decomposed β. In the $(\theta + \gamma)$ region fine structures were obtained of the discontinuous precipitation type, and the γ phase was stained dark in comparison with the θ phase. Single-phase θ alloys at 608° C. were identified by X-ray powder photographs, because the microstructures were not easily distinguished from those of the α phase.

6. THE $\alpha\gamma/(\alpha\gamma + \beta)$, $(\alpha\gamma + \beta)/\beta$, $\beta/(\beta + \alpha\gamma)$, AND $(\beta + \alpha\gamma)/\alpha\gamma$ BOUNDARIES

The results of the annealing experiments for these phase boundaries are shown in Fig. 2 and in greater detail in Fig. 3. Annealing periods of from 1 week at the higher to 3 weeks at the lower temperatures were used in this part of the diagram, and further anneals up to 5 weeks at all these temperatures confirmed that equilibrium had been obtained.

The results show that the β phase field includes the equi-atomic composition. The phase field rises to a maximum which is drawn at 50·3 at.-% manganese, but it is doubtful whether this can really be distinguished from the exact equi-atomic composition. The temperature of this maximum was determined as 910° C. by microscopical methods.

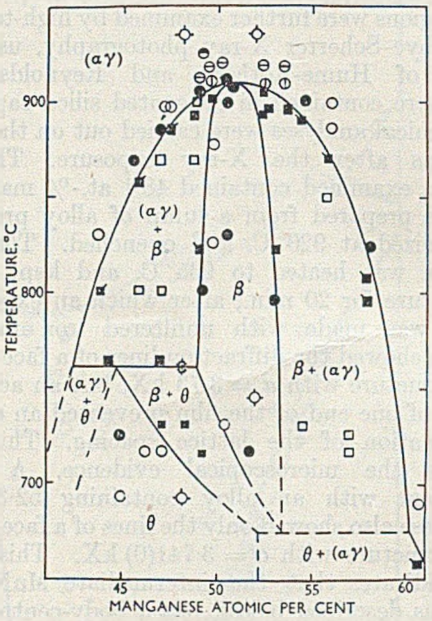

FIG. 3.—Constitution of Nickel–Manganese Alloys Below 1000° C. in the Range 40–60 At.-% Manganese.

On the nickel-rich side the $(\alpha\gamma + \beta)/\beta$ phase boundary with falling temperature slopes slightly in the direction of lower manganese content until 761° C., at which temperature the θ phase is formed by a peritectoid reaction which may be represented :

$$\alpha(43 \text{ at.-\% Mn}) + \beta(49 \text{ at.-\% Mn}) \rightleftharpoons \theta(45 \text{ at.-\% Mn})$$

Below this temperature the nickel-rich boundary of the β phase moves in the direction of higher manganese content until at about 675° C. it undergoes a eutectoid decomposition which may be represented :

$$\beta(53{\cdot}5 \text{ at.-\% Mn}) \rightleftharpoons \theta(52 \text{ at.-\% Mn}) + \gamma(60 \text{ at.-\% Mn})$$

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* 1 part conc. nitric acid, 1 part glacial acetic acid, and 2 parts acetone by volume.

† 5 g. ferric chloride, 2 c.c. conc. hydrochloric acid, and 100 c.c. ethyl alcohol.

It is to be noted that the composition of the eutectoid is distinctly to the manganese-rich side of the equi-atomic value.

It is thought that the phase boundaries shown in Fig. 2 have been established accurately above 800° C. The low-temperature portion of the diagram is to be made the subject of a separate study, and the dotted lines shown in Figs. 2 and 3 are to be regarded as preliminary only.

7. THE SOLID SOLUTIONS IN MANGANESE

In pure manganese the $\delta \rightleftharpoons \gamma$ transformation occurs at 1133° C., and in the present work the thermal analysis of alloy 95·2 showed arrests due to the transformation on both heating and cooling curves. The liquidus curve shows a slight change in direction at 1162° C., and in view of these facts the δ field has been included in Fig. 2. The maximum solubility of nickel in δ-manganese is about 6 at.-%.

In pure manganese the γ modification is stable between 1133° and 1100° C., below which the β-manganese allotrope is the stable phase. The temperature of the $\beta \rightleftharpoons \gamma$ transformation is lowered by the addition of nickel. On cooling curves of manganese-rich alloys, arrests due to this transformation were detected, but they always showed some undercooling and were not at the equilibrium values. The phase boundaries indicated in Fig. 2 were determined by microscopical methods, using specimens which had first been homogenized in the γ-manganese region. Anneals varying from 4 days at 1000° C., to 7 weeks at 600° C. were used in this part of the diagram, though it is probable that these changes are fairly rapid and equilibrium is soon established. The alloys were etched in 1½% alcoholic nitric acid, which darkens the β_{Mn} phase. When present in small amounts the β_{Mn} phase appears olive after light etching, and brownish-blue after further etching. Fig. 9 (Plate XXI) illustrates the structure of alloy 85·5, quenched from 796° C., in which the β_{Mn} phase appears dark.

TABLE III. *Lattice Spacing of β_{Mn} Phase with Varying Manganese Content.*

Mn, at.-%	Lattice Spacing of β_{Mn} Phase, kX
100	6·3030 *
94·63	6·3242
89·76	6·3434
85·36	6·3581

* This value was obtained by Dr. J. W. Christian.

The lattice spacings of some alloys in the β_{Mn} field were measured with the results shown in Table III. For this purpose lumps of the alloys were first homogenized in the β_{Mn} region and quenched. The brittle material was then ground to powder in an agate mortar, and was so fragile that sharp diffraction lines were obtained without further annealing.* The chemical analyses were carried out on the actual powder after the above treatment, and the totals of the analytical percentages of nickel and manganese lay between 99·94 and 99·97.

V.—DISCUSSION

The present work confirms the conclusion of Köster and Rauscher that at high temperatures the α solid solution in nickel is continuous with the solid solution in γ-manganese, the axial ratio of the latter being increased by the solution of nickel. The present work suggests that the extent of the γ-manganese phase is much greater than had previously been imagined, and that the equi-atomic region has a quite different form. There is an interesting resemblance between the present equilibrium diagram and that of the system nickel–zinc, which also shows a typical body-centred cubic β phase in the equi-atomic region. It is of course well known that the divalent Mn^{++} salts are very stable owing to the presence of five 3*d* electrons which exactly half-fill the $(3d)^{10}$ sub-group. The resemblance between zinc and manganese can thus be understood, and if the β phases in the nickel–manganese and nickel–zinc systems were simple 3/2 electron compounds it would suggest that nickel was acting as a univalent element. On the other hand, the ferromagnetic properties of solid solutions of both zinc and manganese in nickel suggest that the holes in the 3*d* band of nickel have been filled by about 30 at.-% zinc or manganese. In the β phase of the nickel–zinc system, the proportion of zinc atoms is greater than in the α (nickel-rich) solid solution, and there should be a greater tendency to fill up the holes in the *d* shell, so that the co-existence of univalent nickel and divalent zinc would seem improbable. The work of Schramm,[8] however, shows that the low-temperature β' modification of the nickel–zinc phase is paramagnetic and may involve holes in the *d* shell, so that the true state of affairs in this system is not yet understood. The resemblance between the systems nickel–zinc and nickel–manganese undoubtedly suggests that the manganese is acting as a divalent element in the equi-atomic region of the system manganese–nickel, but more extensive knowledge of nickel alloys is desirable before these phases can be understood.

Reference to Fig. 3 will show the very symmetrical nature of the $(\alpha\gamma + \beta)$ phase boundary about the composition 51 at.-% manganese. It is found that, except for a short length of the order of 1 at.-% at the extreme peak, this curve can be represented by the equation :

$$T = 2C^2,$$

where T is the temperature in °C. measured down-

---

* Experiments by Dr. J. W. Christian have shown that in other solid solutions in β-manganese, no change in lattice spacing is found if the "as-ground" powder is re-annealed and quenched from the β-region.

wards from 910° C., and C is the change in composition in either direction measured from the peak at 51 at.-% manganese.

ACKNOWLEDGEMENTS

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REFERENCES

1. A. Dourdine, *Zhur. Russ. Met. Obshch.*, 1912, **1**, 341 (see *Rev. Mét.*, 1915, **12**, (Extraits), 125).
2. A. Dourdine, *Rev. Mét.*, 1932, **29**, 507 and 565.
3. W. Köster and W. Rauscher, *Z. Metallkunde*, 1948, **39**, 178.
4. S. Kaya and A. Kussmann, *Z. Physik*, 1931, **72**, 293.
5. S. Valentiner and G. Becker, *Z. Physik*, 1935, **93**, 795.
6. S. J. Carlile, J. W. Christian, and W. Hume-Rothery, *J. Inst. Metals*, 1949–50, **76**, 169.
7. W. Hume-Rothery and P. W. Reynolds, *Proc. Roy. Soc.*, 1938, [A], **167**, 25.
8. J. Schramm, *Z. Metallkunde*, 1938, **30**, 122, 131, and 327.

NICKEL–MANGANESE ALLOYS.


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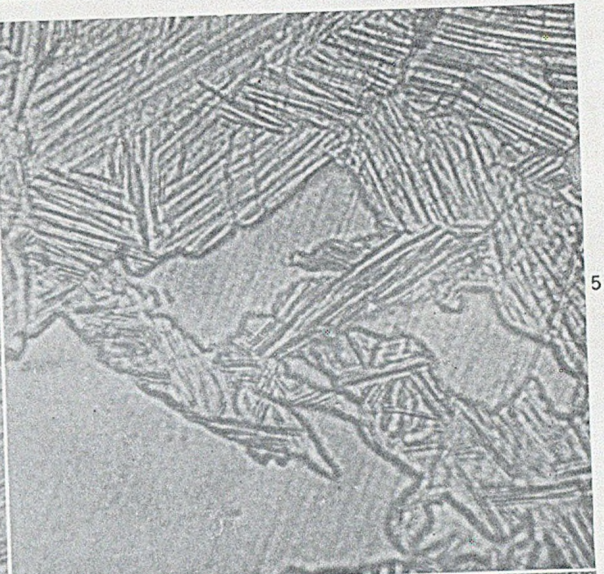
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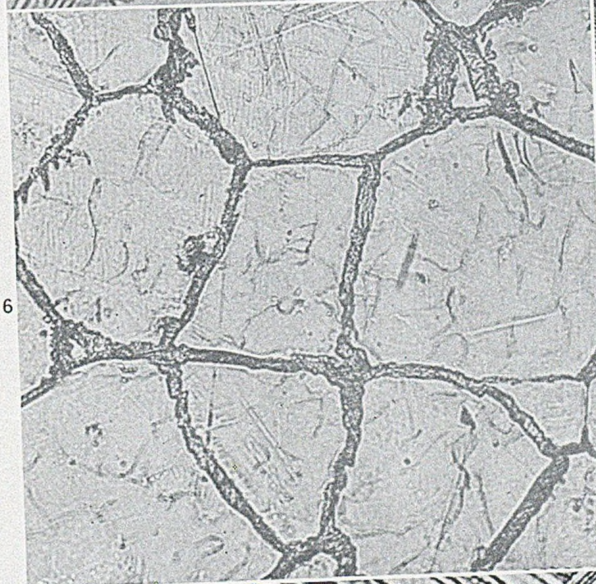
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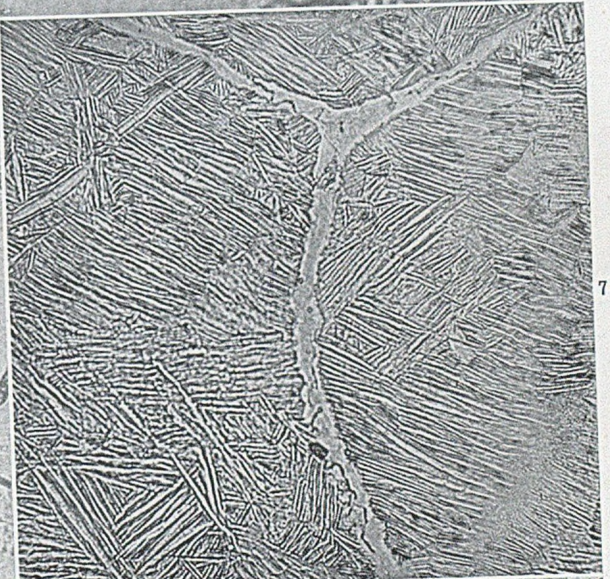
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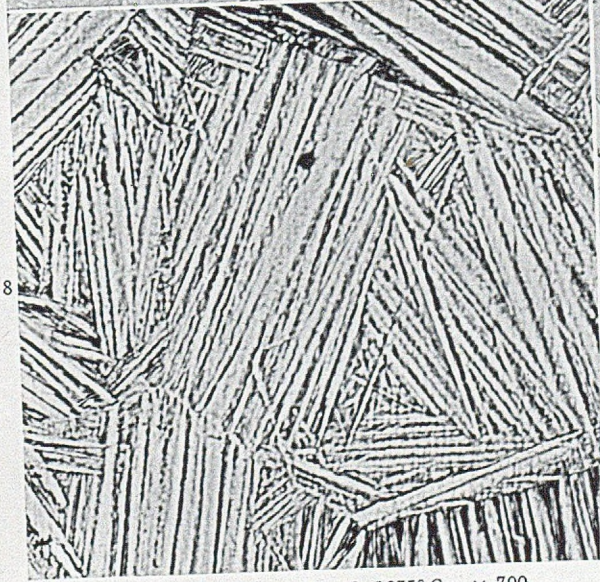
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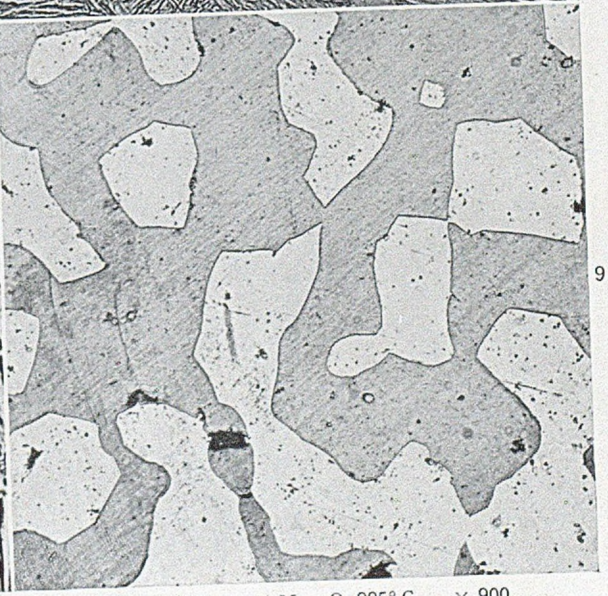
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FIG. 4.—49·1 at.-% Mn Q. 1035° C. × 700.
FIG. 6.—53·5 at.-% Mn Q. 1017° C. × 420.
FIG. 8.—50·5 at.-% Mn Q. 830° C. × 200.

FIG. 5.—53·5 at.-% Mn Q. 995° C. × 900.
FIG. 7.—52·3 at.-% Mn Q. 1036° C. × 420.
FIG. 9.—85·5 at.-% Mn Q. 796° C. × 130.

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THE NUCLEATION OF CAST METALS AT THE MOULD FACE* 1328

By J. A. REYNOLDS,† B.Sc., Student Member, and
C. R. TOTTLE,‡ M.Met., A.I.M., Member

SYNOPSIS

The nucleation of some cast metals and alloys effected by applying metal powders to the face of a standard mould, has been investigated by comparing the grain-size of ingot surfaces produced with and without the coating.

In general, powdered metals having the same crystallographic form as the metal being cast are effective as nucleating agents, provided that the degree of lattice " misfit " does not exceed 5%, or in some cases 10%. This simple mechanism is found to apply to the solidification of tin, lead, zinc, antimony, aluminium, magnesium, and copper. Where the cast metal readily formed an oxide film, reduction of this film to the metal was found to occur when some coatings were applied, giving rise to simple homogeneous nucleation by the particles produced.

Homogeneous and heterogeneous nucleation of alloys can take place under the same conditions as with pure metals, the crystal structure of the solid first formed from the melt being the ruling factor. Reduction of oxide films also occurred, and in the case of an 18 : 8 austenitic steel resulted in isomorphous nucleation of the δ solid solution by chromium particles reduced from the film.

I.—INTRODUCTION

1. General

The mechanism of solidification of cast metals has received considerable attention in recent years, chiefly from the thermal standpoint, in terms of rates of heat extraction and crystal growth. Contributions to the subject include a study of the thermal properties of some commercial moulding materials [1] and a bibliography [2] of experimental investigations. Two papers on the grain-refinement of aluminium alloys [3, 4] have dealt with the formation of nuclei in the body of the melt, and Tammann's [5] experiments with organic and aqueous solutions have been extended to aluminium alloys.

In America, Turnbull [6] has investigated the mechanism of nucleation by supercooling droplets of liquid metals (mercury, gallium, and tin). His results support the " critical-size " concept of homogeneous nucleation, and have enabled the interfacial free energies between liquid metal and solid nuclei to be calculated. The thermodynamical aspects of nucleation have been discussed in a recent symposium.[7, 8]

The nucleation of crystals at a mould face is mentioned in almost every text-book on founding, and in many on metallography, but little experimental evidence has been brought forward in support. Turnbull [9] states that " for liquid–solid transformations in relatively large volumes of liquid the evidence seems decisive that at least the first few nuclei that appear originate by a heterogeneous mechanism at the surfaces of the containing vessel or of colloidal inclusions in the system."

The work described below was undertaken with a view to establishing the connection between the physical and/or chemical properties of the mould face and the solidification of metals in contact with that face.

2. Present Investigation

From the " critical-size " concept of nucleation,[6] it would appear that any particle acting as a nucleating agent must be of a specific size before growth can occur upon it. The addition of atoms to, or their subtraction from, a particle of critical size, results in a decrease of free energy; hence particles of diameter less than the critical size tend to vanish, while those of greater diameter tend to grow. The provision of nucleating particles would therefore demand a size greater than this critical value.

The requisite conditions are met by powders passing a 300-mesh sieve, as according to Turnbull the critical size is less than 10^{-3} mm. radius.

The use of powdered metal to nucleate the same metal in the liquid state (homogeneous nucleation) can be extended to powders of dissimilar metals. These must have the same crystal structure, though the lattice spacing may differ to a certain extent (heterogeneous nucleation). No evidence was available on the permissible difference in lattice spacing between the nucleating particle and the metal to be cast, but in the experiments to be described it was assumed that a favourable spacing-factor might be essential.

In order to assess the lattice " misfit " involved in various combinations of powder and liquid metal, it was necessary to know the lattice constants at the operative temperatures. Initially, the values were

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† British Steel Founders' Research Association Research Student, Department of Metallurgy, King's College, Newcastle-upon-Tyne.

‡ Ministry of Supply, Division of Atomic Energy (Production); formerly Lecturer in Metallurgy, King's College, Newcastle-upon-Tyne.

calculated up to the freezing point of the cast metal but experiment showed that the powder on the mould face did not reach such a high temperature. The maximum temperature of the mould face was frequently of the order of 70% of the freezing point of the cast metal, and this was reached only some time after solidification had begun. It appeared that no great error was introduced by comparing the unit cell of the cast metal at its freezing point with that of the nucleating agent at room temperature. The crystal structure and lattice constants of the metals investigated are shown in Table I.

TABLE I.—*Crystal Structure and Lattice Constants of Possible Mould Coatings.*

Metal	Structure	a_0, Å.[a]	c_0, Å.[a]	Axial Ratio or Angle
Strontium .	Face-centred cubic	6·075	...	...
Calcium .		5·56	...	...
Cerium . .		5·143	...	...
Thorium .		5·077	...	...
Lead . .		4·939	...	...
Silver . .		4·532	...	...
Gold . .		4·0701	...	...
Aluminium .		4·0414	...	...
Copper . .		3·608	...	...
γ-Iron . .		3·564 (910° C.)	...	...
Nickel . .		3·5167	...	...
Tungsten .	Body-centred cubic	3·1585	...	...
Molybdenum .		3·1409	...	...
Vanadium .		3·0338	...	...
δ-Iron . .		2·93 (1425° C.)	...	...
Chromium .		2·8786	...	...
α-Iron . .		2·8600	...	...
Magnesium .	Close-packed hexagonal	3·2022	...	...
Titanium .		2·953	...	...
Cobalt .		2·507	...	...
Beryllium .		2·268	...	...
Graphite .	Hexagonal	2·456	6·691	2·72
Zinc . .		2·6595	4·9368	1·86
Cadmium .		2·927	5·607	1·84
Tellurium .		4·4469	5·9149	1·33
Antimony .	Rhombohedral	4·4974	...	57° 6·5′
Bismuth .		4·7361	...	57° 14·2′
Manganese .	Complex cubic	6·83	...	...
Silicon . .	Diamond cubic	5·417	...	...
Tin . .	Body-centred tetragonal	5·819	3·175	...

[a] Room-temperature values unless otherwise stated.

II.—EXPERIMENTAL METHODS

The principal difficulties in casting metals under specified conditions are to eliminate complicating variables and to provide rigid controls to simplify comparison. In the experiments described the mould coating was originally applied to one side only of a square-section pattern, thus allowing comparison to be made with three untreated surfaces. With the initial casts made in sand moulds, the mould cavity was duplicated, a common runner to both sections being employed. This proved an unnecessary precaution, and subsequently two coated faces were compared with two untreated ones, prepared in either single or double moulds. Casting temperatures were measured by means of immersion thermocouples, and dried moulds were used throughout, whatever the mould material.

1. MOULDS

(a) *Sand*

Northallerton naturally bonded sand was milled dry for 10 min., adjusted to a 5% moisture content, and milled for a further 5 min. The patterns were hand-rammed in this sand, the ingate of the runner being so disposed as to avoid sand wash or erosion of the powder-coated mould surface. The general arrangement is illustrated in Fig. 1. This shows a double mould, which produced an ingot 13 cm. high × 4 cm. square at the top, and 3 cm. square at the base. After moulding, the box was dried in an air-circulation oven at 150° ± 10° C. and allowed to cool to room temperature immediately before casting. The surface finish of ingots cast in sand moulds was rough in most cases, and this caused difficulty in assessing the degree of grain refinement.

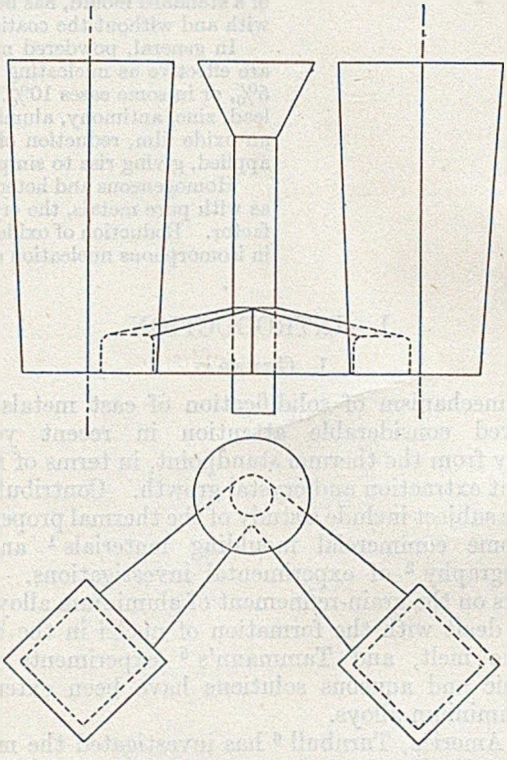


FIG. 1.—Mould Arrangement.

(b) *Synthetic*

For synthetic moulds the Osborn–Shaw precision-casting process [10] was used, on the same patterns as for sand. The moulds are prepared by pouring over the pattern an unstable slurry of finely divided sillimanite in a suspension of ethyl silicates, which are undergoing a controlled hydrolysis to silica gel and ethyl alcohol. After setting, the pattern is withdrawn, the alcohol ignited, and the mould is heated to 600°–700° C. for a few minutes to remove residual alcohol. The final product is a mould of high refractoriness and resistance to thermal shock, with approximately 45% porosity.

To simplify moulding, and to permit examination of both mould surface and casting after solidification, single moulds were employed; these were split along a diagonal, and were top poured. Owing to the porosity of the synthetic mould, cooling was very slow, and hence the grain-size of the cast metal when nucleation did not occur was large, which simplified comparison with the treated faces.

(c) *Metal*

A rectangular " finger " mould of cast iron or steel, with a cavity measuring $3 \times 1 \times \frac{3}{4}$ in., was used to investigate the effect of rapid chilling.

2. Application of Coatings

Metal powder passing a 300-mesh sieve was not always available, but it was found that coarse particles resulted in a poor surface finish on the ingots and rendered examination difficult. The purity of the powder employed was in general not less than 99·5%, and in some cases it was higher. The metals constituting the main impurities in any one powder were in the majority of cases themselves employed as powdered coatings during the investigation, which helped in determining the responsible nucleating agent. The magnitude of the effects produced in most cases suggested that impurities played only a minor role.

Where compounds of the metals were used, these were of analytical-reagent quality. The chief difficulty with the metal powders was to avoid oxidation during their preparation, handling, and application to the mould face. Strontium and calcium powders oxidized to such a degree that they were of little value, but it was possible to produce cerium powder by mechanical means without excessive oxidation.

For use with sand or chill moulds, a standard weight of powder (just sufficient to cover the mould face completely) was suspended in a weak solution of cellulose acetate in acetone, the viscosity of the solution being adjusted to allow uniform spreading of the final suspension. Application was by camel-hair brush, the acetone being allowed to evaporate in a warm atmosphere just before casting. As a periodic check, the cellulose solution alone was coated on to one face of the mould, but this was never found to influence the ingot grain-size.

The synthetic moulds were swabbed with ethyl silicate, the surplus solution allowed to evaporate, and the face to be treated dusted with the powder, any excess of which was removed by shaking or light tapping. A very thin coating, of the order of one particle thickness, was obtained by this means, and adhered well to the mould face. Iron and nickel powders agglomerated rapidly, and orientated themselves in the earth's magnetic field, thus preventing perfect removal of any excess powder.

3. Melting Practice

Commercial-purity metals were melted under flux according to normal practice in each case, and degassed where necessary. Gas-fired or electric-resistor crucible furnaces were employed for all metals other than steel, for which an indirect-arc furnace was used. Since untreated faces of each individual ingot were compared with treated faces of the same ingot, any peculiarities due to melting conditions applied equally to both. In the case of magnesium castings made in sand moulds, sulphur was added to the moulding material, but this proved unnecessary with synthetic moulds. The sand moulds were bottom-poured, the remainder top-poured, turbulence being minimized in the latter case by careful pouring.

4. Examination of Ingots

Immediately after casting and cooling to room temperature, the ingots and mould faces were examined for any sign of change in appearance of the coating. The castings were then lightly pickled in appropriate reagents, details of which are given in Table II, without preparation. Sand castings, and certain others with a poor surface finish, were often unsuitable for examination in the pickled state and from these a transverse slice was taken at a height two-thirds of the distance from the base of the ingot, lightly ground, and pickled as for the whole ingot.

Table II.—*Pickling Reagents Used to Reveal Grain-Size.*

Cast Metal	On Ingot	On Transverse Slice
Aluminium and its alloys	5% HCl, HNO_3, H_2SO_4 in water	...
Antimony	10% HCl, HNO_3 in water (hot)	...
Copper and its alloys	10% HNO_3 (hot)	$N/20$ Iodine solution
Lead and its alloys	...	10% Nitromolybdic acid
Magnesium	...	60% Acetic acid + 10% ammonium acetate
Tin and its alloys	15% HCl	...
Zinc and its alloys	10% HCl (hot)	2% Chromic acid + sodium sulphate
18 : 8 Austenitic steel	Aqua regia (hot)	...
Mild steel	5% HCl	...

After pickling, the grain-size was assessed by counting, with the naked eye or a binocular microscope, on all four faces of an ingot, and the four edges of a transverse slice. The number of grains per unit area (or unit length) was then calculated, and the nucleation effect expressed as the ratio of grains per unit on treated faces to the average number on untreated faces. In all cases where refinement had occurred, this was immediately obvious. Early casts, employing one treated to seven untreated faces (double mould) showed that 2–5% difference in ingot grain-size could be expected on the normal mould faces, whereas 50% or more reduction in grain-size was common for a treated face when nucleation had occurred.

III.—DISCUSSION OF RESULTS

1. Direct Nucleation

The results obtained by using as coatings those metals whose crystal structure might be expected to influence nucleation are recorded in Table III. Those coatings which did not promote nucleation are also included in the table. The term "homogeneous" nucleation is used where a powdered metal has refined the grain-size of the same metal cast against it, and "heterogeneous" nucleation where the two metals are different. In the latter case, the percentage difference in lattice spacing is given in the last column.

These results show that the general principle of homogeneous nucleation can be applied to cast zinc, aluminium, magnesium, tin, lead, copper, and antimony. As regards heterogeneous nucleation, the most marked effects arose with the combinations lead/thorium, lead/cerium, copper/nickel, and antimony/bismuth (the cast metal being placed first); positive results were also obtained with zinc/graphite, zinc/cadmium, magnesium/titanium, copper/aluminium, and copper/iron, though the results were not so perfectly reproducible. It appears that where the difference in lattice spacing between the coating and the cast metal is less than 5% simple heterogeneous nucleation will take place; with between 5 and 10% difference in spacing the results were variable, and beyond 11% no positive results were obtained.

TABLE III.—*Nucleation by Isomorphous Coatings.*

No. of Casts	Cast Metal	Casting Temp., °C.	Mould Material	Coating	Grain-Size Refinement Ratio *	Type of Nucleation	Lattice-Spacing Difference, %
4	Zinc	550–580	Sand	Zinc	2 : 1	Homogeneous	...
2		580	Synthetic	,,	1·5 : 1	,,	...
1		550	Steel	,,	2 : 1	,,	...
2		550	Sand	Graphite	1·5 : 1	Heterogeneous	8
2		560	,,	Cadmium	1·2 : 1	,,	9
2		560	,,	Tellurium	1	Nil	>10
5	Aluminium	760	Sand	Aluminium	4 : 1 to 9 : 1	Homogeneous	...
3		760	Synthetic	,,	10 : 1 to 15 : 1	,,	...
1		750	Cast iron	,,	10 : 1	,,	...
1		730	Sand	Coarse aluminium	5 : 1	,,	...
2		750	,,	Lead	1	Nil	>10
2		750	,,	Thorium	1	,,	>10
2		750	,,	Nickel	1	,,	>10
2	Magnesium	730	Sand	Magnesium	2 : 1	Homogeneous	...
2		750	Synthetic	,,	1·5 : 1	,,	...
3		750	,,	Titanium	1·2 : 1	Heterogeneous	8·5
1		750	Sand	Cobalt	1	Nil	>10
2		750	,,	Beryllium	1	,,	>10
2	Tin	400	Steel	Tin	20 : 1	Homogeneous	...
2	Lead	450–500	Steel	Lead	4 : 1 to 6 : 1	Homogeneous	...
3		500	,,	Thorium	10 : 1	Heterogeneous	2·8
2		450	,,	Cerium	2 : 1	,,	4
2		450	Sand	Aluminium	1	Nil	>10
2		450	,,	Copper	1	,,	>10
2	Copper	1180	Synthetic	Copper	10 : 1	Homogeneous	...
2		1320	Steel	,,	5 : 1	,,	...
2		1220	Synthetic	Nickel	50 : 1	Heterogeneous	2·6
2		1220	Sand	,,	25 : 1	,,	2·6
2		1200	Synthetic	Aluminium	1·2 : 1	,,	11
2		1200	Sand	γ-Iron	1·2 : 1	,,	1
2		1150	,,	α-Iron	1	Nil	...
2		1200	,,	Lead	1	,,	>10
2	Antimony	770	Steel	Antimony	5 : 1	Homogeneous	...
2		750	,,	Bismuth	2 : 1	Heterogeneous	5·4

* Expressed as number of grains per unit area on treated face to average number on untreated faces.

In the case of copper cast against iron powder, no effect was observed with a casting temperature below 1200° C, and it is presumed that the γ state is not reached by the iron coating under those conditions.

Fig. 2 (Plate XXII) illustrates refinement by a homogeneous coating and Fig. 3 (Plate XXII) demonstrates the absence of refinement where the lattice spacings differ by more than 10%.

2. NUCLEATION INVOLVING CHEMICAL REACTION

During the stripping of ingots cast against certain coatings (not specified in Table III), several instances occurred in which the coating had changed in colour and general appearance. The metal powders involved did not satisfy the principle of isomorphous nucleation, but the grain refinement was reproducible at will under a wide variety of conditions, and was in general greater in magnitude than the examples quoted in Table III.

Chemical and spectrographic analysis of residues scraped from the mould face showed that the oxide(s) of the metal originally applied as powder pre-

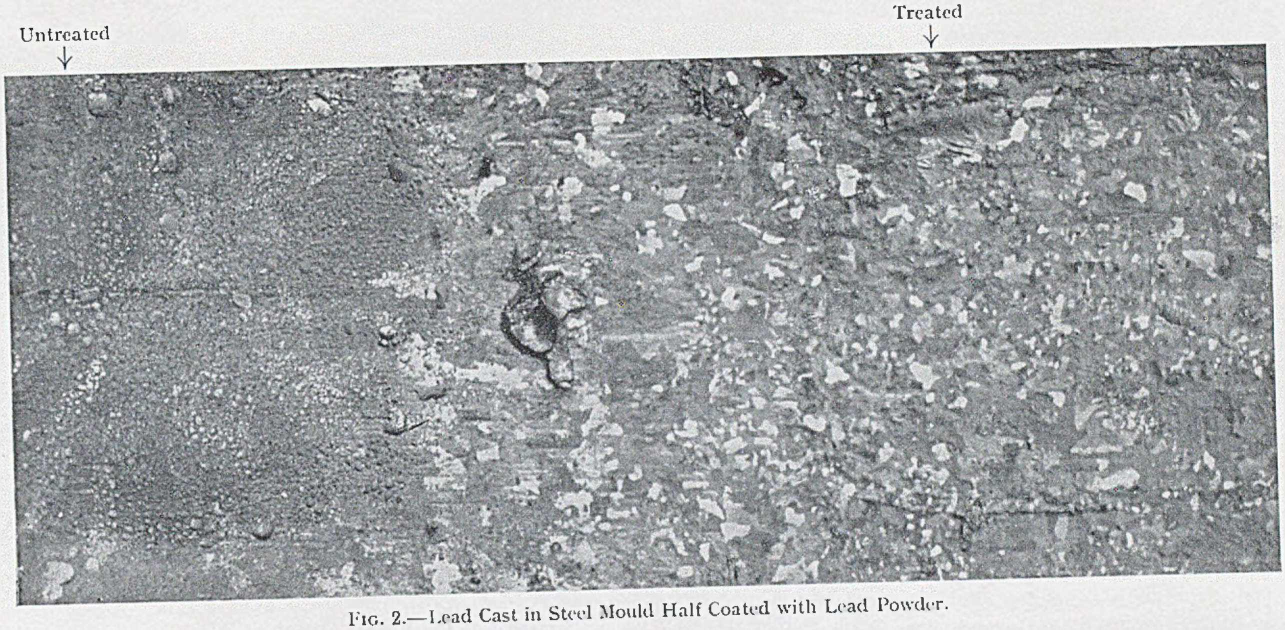
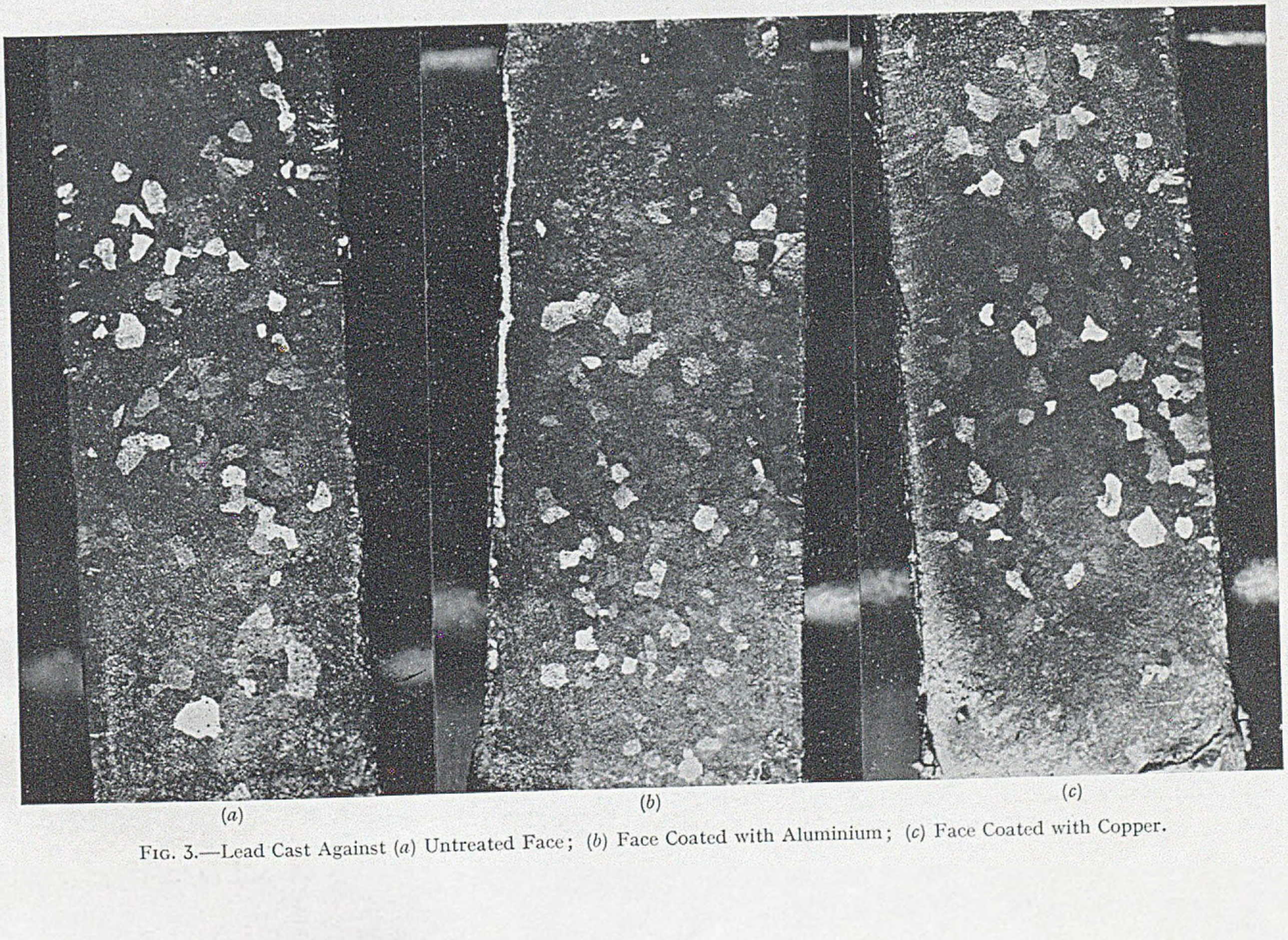


FIG. 2.—Lead Cast in Steel Mould Half Coated with Lead Powder.



(a) (b) (c)

FIG. 3.—Lead Cast Against (a) Untreated Face; (b) Face Coated with Aluminium; (c) Face Coated with Copper.

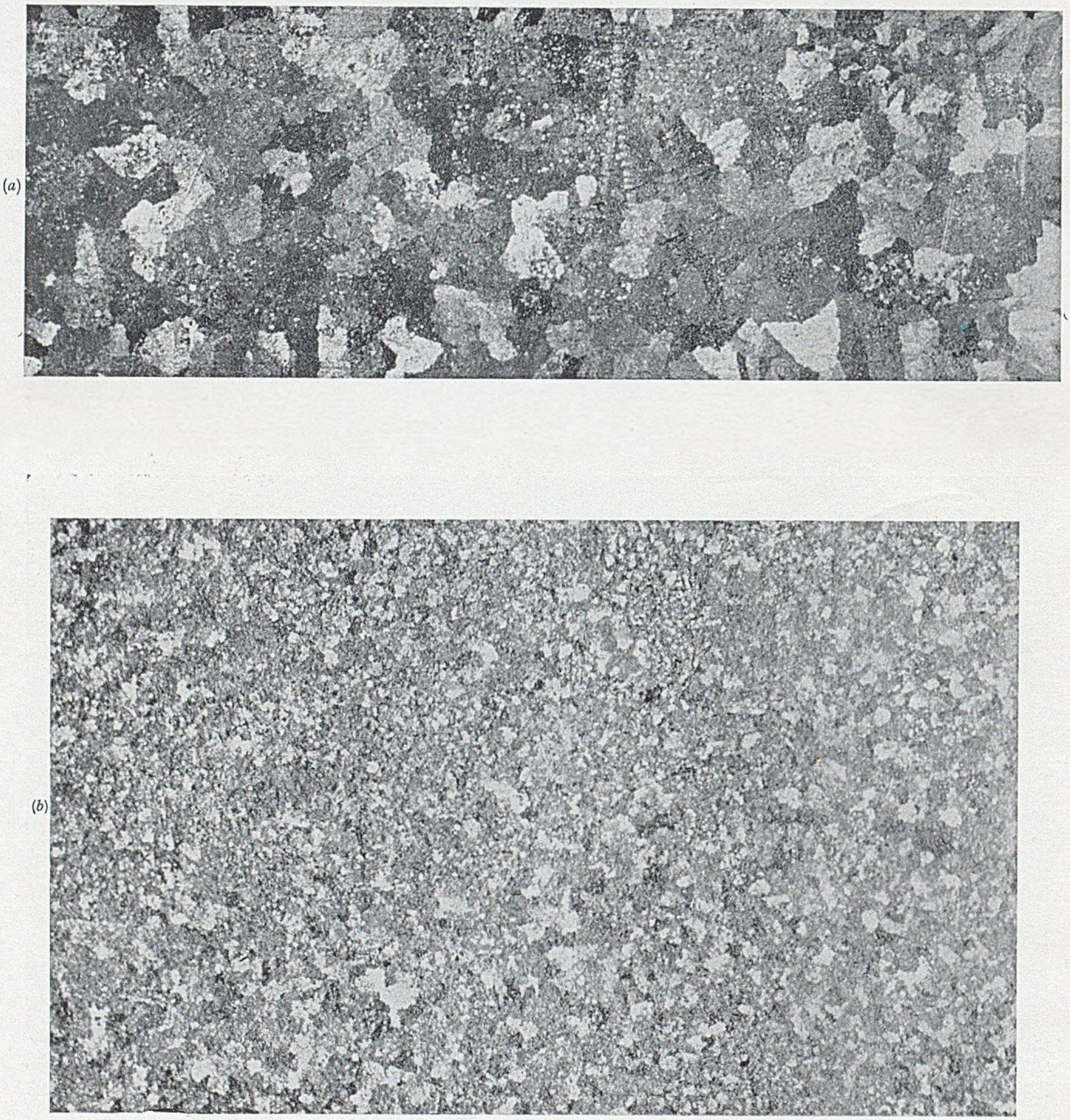


Fig. 4.—Aluminium Cast Against (*a*) Untreated Face; (*b*) Face Coated with Titanium.

dominated. It was therefore concluded that in such cases the powder had reduced the oxide film on the metal being cast, thus precipitating fine particles of the cast metal, which then acted as homogeneous nuclei.

TABLE IV.—*Free Energy of Formation* ($-\Delta G$) *of Metallic Oxides at Room Temperature.*[11–14]

Metal	Oxide	$-\Delta G$, kg.cal./g.mol. oxygen (at 17° C.)	Accuracy ± kg.cal.
Calcium . . .	CaO	289	7
Thorium . . .	ThO_2	281	25
Beryllium . .	BeO	281	6
Magnesium . .	MgO	273	1·5
Calcium silicide (Ca_2Si)	$2CaO + SiO_2$	260	?
Titanium . . .	TiO	257	8
Aluminium . .	Al_2O_3	244	10
Titanium . . .	Ti_2O_3	236	15
,, . . .	TiO_2	206	6
Vanadium . .	VO	199	8
Silicon . . .	SiO_2	196	7
Vanadium . .	V_2O_3	186	7
Manganese . .	MnO	174	3
Chromium . .	Cr_2O_3	167	4
Vanadium . .	VO_2	163	7
Zinc . . .	ZnO	153	4
Vanadium . .	V_2O_5	141	5
Tin	SnO	128	?
Iron . . .	FeO	115	3
Nickel . . .	NiO	103	2
Antimony . .	Sb_2O_3	101	?
Lead . . .	PbO	91	4
Bismuth . . .	Bi_2O_3	84	?
Copper . . .	Cu_2O	71	1

In determining which metals should be capable of reducing the oxide film on the cast metal, the values of free energy of oxide formation given in recent publications were used.[11–14]

The rate of such reactions is governed by the activation energy (E), and the free energy of formation ($-\Delta G$) is only a guide to the possibility of deoxidation occurring. However, the results obtained do correspond to the expected behaviour based on the values of $-\Delta G$ given in Table IV. Table V records the effects observed with grain-refined castings where oxidation of the coating took place and others where no refinement occurred. Fig. 4 (Plate XXIII) shows a typical example of nucleation involving oxide reaction.

It will be seen that aluminium provides the best example of a cast metal in which oxidation of the coating occurred. This is to be expected from the tenacious oxide film normally present on pouring the liquid metal. Magnesium behaves similarly, but the number of metals with a higher value of $-\Delta G$ is limited, and of these, beryllium powder was not available at the time, and calcium powder was found difficult to produce without oxidation of the particles. Calcium silicide was used as a possible source of calcium, and has been included in Table IV. The compound Ca_2Si was considered as a single component, and the free energy of oxide formation was made additive on a basis of the molecular ratios of the two constituents. Although this may not be justified, the calculated value is less than that of aluminium (see Table IV) and, as shown in Table V, calcium oxide is formed when aluminium reacts with the coating, and nucleation results to a limited degree.

The films remaining on titanium-coated mould faces after casting may contain TiO and Ti_2O_3, since both

TABLE V.—*Nucleation Involving Deoxidation of the Cast Metal.*

No. of Casts	Cast Metal	Casting Temp., °C.	Mould Material	Coating	Grain-size Refinement Ratio.*	Appearance of Coating After Casting
2	Aluminium	760	Sand	Magnesium	50 : 1	White; MgO confirmed
2		750	,,	Titanium	50 : 1	Intense blue
2		750	,,	Vanadium	2 : 1	Yellowish white
2		750	Cast iron	Titanium	50 : 1	Intense blue
2		750	,, ,,	Magnesium	10 : 1	White; MgO confirmed
4		720	Synthetic	Titanium	20 : 1	Intense blue
2		780	,,	Calcium silicide	1·5 : 1	White; CaO confirmed
2		800	,,	Vanadium	1·5 : 1	Yellow
2		740	,,	Thorium	2 : 1	White
2		750	Sand	Iron	1	Metallic
2		750	,,	Zinc	1	,,
2		750	,,	Antimony	1	,,
2		750	Synthetic	Chromium	1	,,
2		750	,,	Silicon	1	,,
2		750	Sand	TiO_2	1	White
2		750	,,	MgO	1	,,
2		750	,,	Al_2O_3	1	,,
2	Magnesium	760	Synthetic	Thorium	2 : 1	White
2		760	,,	Aluminium	1	Metallic
2		760	,,	Silicon	1	,,
2		770	Sand	Bismuth	1	,,
2	Copper	1200	Synthetic	Aluminium	1·2 : 1	Some white specks
2		1180	,,	Titanium	1·2 : 1	Slight blue film
2		1220	Sand	Antimony	1	Metallic
2		1220	Synthetic	Iron	1	,,

* Expressed as number of grains per unit area on treated face to average number on untreated faces.

these are blue in colour.[15] From Table V, it will be seen that the oxides of titanium and magnesium produce no grain refinement. Moulds heated to 200° C. after application of the coatings were also found to give negative results owing to oxidation of the powder at the surface.

Some attempt has been made to obtain X-ray back-reflection photographs of the treated surfaces of the castings, particularly where oxidation of the coating had occurred at the mould face. No traces of diffraction rings due to any metal other than the cast one were ever obtained. Reaction at the mould face, promoting turbulence, was also taken to be insignificant, after experimental casts had been made in which portions only of the mould face were coated. In these examples, the grain-refined areas showed sharp lines of demarcation, following exactly the pattern of the original coating.

3. Nucleation of Alloys

In the nucleation of alloys the metal forming the basic constituent seems to exert the greatest influence, but where oxide films are involved, the composition of these films may prove the most important factor. When the crystal structure of the first crystals to form from the alloy is identical with that of the principal constituent, isomorphous nucleation follows, as in the case of a pure metal. This was confirmed for some aluminium-, copper-, zinc-, lead-, and tin-base alloys. When the crystal structure of the first solid differs from that of the pure metal forming the basis of the alloy, then the 5% lattice-distortion rule appears to apply (see Section III, 1). Few results were obtained for such conditions, and they are not recorded here.

The most interesting example of alloy nucleation was provided by 18 : 8 austenitic steel. Here, the oxide film has been stated[16] to contain 90% chromium sesquioxide, 10% iron oxide, and a trace of nickel oxide. If a mould coating that will reduce chromium oxide is used, then the chromium particles formed may act as heterogeneous nuclei for the δ solid solution which solidifies first from the melt, the lattice distortion being of the order of 2%. From the values of $-\Delta G$ given in Table IV it appeared that thorium, magnesium, calcium silicide (as discussed above), aluminium, titanium, silicon, and possibly vanadium, should deoxidize the film on 18 : 8 austenitic steel at 1750° C, assuming this to consist mainly of chromic oxide. Table VI shows that these metals do, in fact, nucleate the steel.

As a practical test, a precision casting in this steel, containing sections tapering from $\frac{1}{16}$ in. to a knife edge (necessitating high casting temperatures) was made, with and without coatings of aluminium powder. The pouring temperature of 1750° C. led to solidification in the thin section of one single grain, occupying the whole of the cross-section, when no powder was coated on to the synthetic mould. Using aluminium powder, the grain-size was refined at the surface, and solidification of the interior so modified as to produce from 5 to 100 grains across the narrow tapering section.

Table VI.—*Nucleation of* 18 : 8 *Austenitic Steel.*

No. of Casts	Casting Temp., °C.	Mould Material	Coating	Grain-Size Refinement Ratio *	Oxide-Film Coating After Casting
4	1720	Synthetic	Titanium	100 : 1	Blue
3	1700		Silicon	2 : 1	White SiO_2
4	1720		Aluminium	50 : 1	White Al_2O_3
3	1700		Calcium silicide	20 : 1	White CaO
2	1750		Thorium	5 : 1	White
4	1750		Magnesium	4 : 1	White MgO
2	1720		Vanadium	1·5 : 1	Yellow

* Expressed as number of grains per unit area on treated face to average number on untreated faces.

Note: Some ingots of 18 : 8 austenitic steel were cast using vanadium coatings in which refinement was negligible. No refinement was ever observed with iron, nickel, or zinc powder.

The improvement in mechanical properties was no doubt considerable, but the size of the casting prevented any direct measurement of these. No attempt was made to draw any conclusions from the experimental ingots as to the grain-size of the centre sections when nucleation at the surface had occurred, but it was observed that considerable influence was exerted on the crystals growing from the surface layer, often reaching to the centre of the ingot.

Further work is necessary to confirm the mechanism of nucleation in alloys.

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REFERENCES

1. R. W. Ruddle and A. L. Mincher, *J. Inst. Metals*, 1949–50, **76**, 43.
2. R. W. Ruddle, "The Solidification of Castings", *Inst. Metals Monograph and Rep. Series* No. 7, 1950.
3. M. D. Eborall, *J. Inst. Metals*, 1949–50, **76**, 295.
4. A. Cibula, *J. Inst. Metals*, 1949–50, **76**, 321.
5. G. Tammann, "A Textbook of Metallography". New York: **1925** (Chemical Catalog Co., Inc.).
6. D. Turnbull, *J. Appl. Physics*, 1949, **20**, 817; 1950, **21**, 1022. *Trans. Amer. Inst. Min. Met. Eng.* (*in J. Metals*), 1950, **188**, 1144.
7. D. Turnbull, *Thermodynamics in Physical Metallurgy* (*Amer. Soc. Metals*), **1950**, 282.
8. J. H. Hollomon, *ibid.*, 161.
9. D. Turnbull, *J. Chem. Physics*, 1950, **18**, 198.
10. J. J. Marais, *Found. Trade J.*, 1949, **86**, 501.
11. M. de Kay Thompson, "The Total and Free Energies of Formation of the Oxides of Thirty-Two Metals". New York: **1943** (Electrochemical Society).
12. H. J. T. Ellingham, *J. Soc. Chem. Ind.*, 1944, **63**, 125.
13. F. D. Richardson and J. H. E. Jeffes, *J. Iron Steel Inst.*, 1948, **160**, 261; 1949, **163**, 397.
14. O. Kubaschewski and E. Ll. Evans, "Metallurgical Thermochemistry". London: **1951** (Butterworth-Springer).
15. J. W. Mellor, "Treatise on Inorganic and Theoretical Chemistry", Vol. VII, p. 28. London: **1927** (Longmans, Green and Co., Ltd.).
16. W. H. J. Vernon, F. Wormwell, and T. J. Nurse, *J. Iron Steel Inst.*, 1944, **150**, 81P.

METALLURGICAL ABSTRACTS AUTHOR INDEX

SYMBOLS AND ABBREVIATIONS FOR USE IN "METALLURGICAL ABSTRACTS"

Å.	Ångström units = 1×10^{-8} cm.
abs.	absolute
A.C.	alternating current
addn.	addition(s)
amp.	ampere(s)
amp.hr.	ampere-hour(s)
appn.	application(s)
approx.	approximate(ly)
aq.	aqueous
at.-%	atomic per cent.
at. wt.	atomic weight(s)
atm.	atmosphere(s) [pressure]
A.W.G.	American wire-gauge
B. & S.	Brown & Sharpe (gauge)
b.c.c.	body-centred cubic
°Bé.	degree Baumé [scale]
B.H.N.	Brinell hardness number(s)
b.h.p.	brake horse-power
b.p.	boiling point(s)
B.Th.U.	British thermal unit(s)
B.W.G.	Birmingham wire-gauge
°C.	degree Celsius (formerly centigrade) [scale]
C	coulomb(s)
cal.	calorie(s)
c.c.	cubic centimetre (s)
c.d.	current density(ies)
C.G.S.	centimetre-gramme-second (units)
chem.	chemical
cm.	centimetre(s)
coeff.	coefficient(s)
compn.	composition(s)
conc.	concentrated (adj.)
const.	constant (adj. or noun)
contg.	containing
cryst.	crystalline
crystn.	crystallization
c./s.	cycles per second
cwt.	hundredweight(s)
D.C.	direct current
d	density(ies)
detn.	determination(s)
dia.	diameter(s)
dil.	dilute (adj.)
dm.	decimetre(s)
D.P.N.	diamond pyramid (hardness) number(s)
elect.	electric, electrical(ly)
electrochem.	electrochemical(ly)
e.m.f.	electromotive force(s)
estn.	estimation(s)
eV.	electron volt(s)
°F.	degree Fahrenheit [scale]
F	Faraday's constant
f.c.c.	face-centred cubic
f.p.	freezing point(s)
ft.	foot; feet
ft.-lb.	foot-pound(s)
g.	gramme(s)
g.-atom	gramme-atom(s)
g.-mol.	gramme-molecule(s)
gal.	gallon(s)
grav.	gravimetric(ally)
H.F.	high-frequency
h.c.p.	hexagonal close-packed
H-ion	hydrogen-ion
h.p.	horse-power
h.p.-hr.	horse-power-hour(s)
hr.	hour(s)
in.	inch(es)
in.-lb.	inch-pound(s)
indust.	industrial
°K.	degree Kelvin absolute [temperature scale]
kc.	kilocycle(s)
kc./s.	kilocycle(s) per second
kg.	kilogramme(s)
kg.cal.	kilogramme-calorie(s)
kg.m.	kilogramme-metre(s)
km.	kilometre(s)
kV.	kilovolt(s)
kVA.	kilovolt-ampere(s)
kW.	kilowatt(s)
kWh.	kilowatt-hour(s)
kX	Crystal Ångström(s) = 1000 Siegbahn X-units
l.	litre(s)
lb.	pound(s)
L.F.	low-frequency
liq.	liquid (adj.)
M	molar [solution]
m.	metre(s)
m.amp.	milliampere(s)
math.	mathematical(ly)
max.	maximum (adj. or noun)
Mc.	megacycle(s)
Mc./s.	megacycle(s) per second
mech.	mechanical
met.	metallurgical(ly)
mg.	milligramme(s)
ml.	millilitre(s)
min.	minute(s); minimum (adj. or noun).
mm.	millimetre(s)
m.m.f.	magnetomotive force(s)
mol.	molecule(s)
m.p.	melting point(s)
mμ	millimicron(s) = 1×10^{-7} cm. = 10 Å.
mV.	millivolt(s)
N	normal [solution]
N.P.L.	National Physical Laboratory (Teddington, Middlesex)
No.	number(s)
N.T.P.	normal temperature and pressure
Oe.	oersted(s)
opt.	optical(ly)
oz.	ounce(s)
P.C.E.	pyrometric cone equivalent
p.d.	potential difference [electric]
*p*H	hydrogen-ion concentration
phys.	physical(ly)
p.p.m.	part(s) per million
prepn.	preparation
prodn.	production
pptn.	precipitation
P.S.	proof stress
qual.	qualitative(ly)
quant.	quantitative(ly)
°R.	degree Réaumur [scale]
recrystn.	recrystallization
ref.	reference(s)
resp.	respective(ly)
r.p.m.	revolution(s) per minute
sci.	scientific
sec.	second(s)
sepn.	separation(s)
soln.	solution(s)
sp.	specific
sp. gr.	specific gravity(ies)
spectrochem.	spectrochemical(ly)
sq.	square
S.W.G.	standard wire-gauge (Imperial)
tech.	technical(ly)
temp.	temperature(s)
t.p.i.	threads per inch
U.T.S.	ultimate tensile stress(es)
V.	volt(s)
VA.	volt-ampere(s)
vol.	volume(s)
W.	watt(s)
Wh.	watt hour(s)
wt.-%	weight per cent.
yd.	yard(s)
Y.P.	yield point(s)
%	per cent.
γ	microgramme = 1×10^{-6} g.
λ	wave-length
μ	micron(s) = 1×10^{-4} cm.
$\mu\mu$	1 millionth micron = 1×10^{-10} cm. = 0·01 Å.
Ω	ohm(s)
$^{\circ}$	degree(s) arc
$'$	minute of the arc
$''$	second of the arc
/	per
$<$	less than
$>$	greater than
$\nless$	not less than
$\ngtr$	not greater than
$\leqslant$	equal to or less than
$\geqslant$	equal to or greater than
$\neq$	not equal to
$\equiv$	identically equal to
$\cong$	approximately (or essentially) equal to
$\sim$	about
$\propto$	varies as
$\parallel$	parallel
$\perp$	perpendicular

METALLURGICAL ABSTRACTS

GENERAL AND NON-FERROUS

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1—PROPERTIES OF METALS

***Creep of High-Purity Aluminium.** William D. Jenkins (*J. Research Nat. Bur. Stand.*, 1951, **46**, (4), 310–317).—Tension tests were made on cold-drawn Al at 105° F. (40° C.) at various creep rates. In the first stage of creep Andrade's equation for transient flow was obeyed within a limited range of strain rates. Plastic extension at the beginning of the third stage increased with increasing load. Cyclic temp. variation between 95° and 105° F. (35° and 40° C.) appeared to increase the ductility. Evidence of discontinuous flow was obtained by plotting log creep rate against extension. The mechanism of flow is discussed qual. in terms of the crystallite theory. 14 ref.—J. W. C.

***Combined Tension–Torsion Creep–Time Relations for Aluminium Alloy 2S-O [Commercially Pure Aluminium].** Joseph Marin, J. H. Faupel, and L. W. Hu (*Proc. Amer. Soc. Test. Mat.*, 1950, **50**, 1054–1070; discussion, 1071–1072).—See *Met. Abs.*, 1950–51, **18**, 481.

***The New Element Berkelium (Atomic Number 97).** S. G. Thompson, A. Ghiorso, and G. T. Seaborg (*Phys. Rev.*, 1950, [ii], **80**, (5), 781–790).—The properties of the new element Berkelium (Bk) are described. The mass number is probably 243 as indicated by chemical separation. Its oxidation states are anticipated to be tri- and quadrivalent.—T. G.

†A Review of Beryllium and Beryllium Alloys. John T. Richards (*J. Metals*, 1951, **3**, (5), 379–386).—A comprehensive review dealing with: (1) Be—its extraction and prodn; crystal structure; alloy formation; elect., mech., and corrosion-resistance properties; methods of processing; appn. (2) Cu-base alloys contg. Be, with and without addn. of Co or Ni; crystal structure; the phys., mech., and chem. properties of 7 commercial wrought and casting alloys; methods of processing; appn. (3) Be addn. to various metals and alloys: Ni–Be (2·7%); ferrous alloys; light metals—the ternary system Be–Cu–Al, addn. of 0·005–0·3% Be to Al–Mg casting alloys in order to control oxidation or prevent reaction with moisture in sand moulds, and an Al–Be alloy for electron-microscope replicas; the effects of small addn. (0·03–0·37%) of Be on the precious metals Au, Ag, and Re; the systems Ti–Be and Zr–Be; the hardening effect of small quantities of Be on Mo within the temp. range 1400°–1600° F. (760°–870° C.). 91 ref.—E. N.

***The Production of Thin Beryllium Foils.** H. Bradner (*U.S. Atomic Energy Commission Publ.*, 1948, **(AECD-1949)**, 4 pp.).—B. describes the procedure for making Be foils from 10^{-5} to 10^{-3} cm. thick and with dia. up to 1·5 in., and also the method of mounting foils. The Be was evaporated *in vacuo* and deposited on a metal collector plate. Although of fine grain-size, the foils were very brittle, but could be loosened from the collector plate by differential thermal expansion when heated. For mounting, the surface on which they were to be held was moistened with a small amount of thinned collodion and lowered on to the film. Surface tension pulled the foils flat even if they had previously curled up on stripping.—B. W. M.

***Evaluation of the Stiffness Coefficients for Beryllium from Ultrasonic Measurements in Polycrystalline and Single-Crystal Specimens.** L. Gold (*U.S. Atomic Energy Commission Publ.*, 1949, **(AECD-2644)**, 25 pp.).—The stiffness coeff. were based on: (1) compressional and shear-wave velocities in polycryst. metal and (2) compressional wave velocities in single-crystal specimens in various directions. Apparatus for the pulsed-ultrasonic measurements was similar to that described by Pellam and Galt (*J. Chem. Physics*, 1946, **14**, 608). The theory and method of calculation are given in detail, the following results being obtained for Be: $C_{11} = 30{\cdot}8$; $C_{13} = 35{\cdot}7$; $C_{44} = 11{\cdot}0$; $C_{12} = -5{\cdot}8$; $C_{33} = 8{\cdot}7 \times 10^{12}$ dynes/cm.^{2}, resp. These results are compared with published data on other h.c.p. metals, such as Mg, Zn, and Cd, and it is shown that they are in conformity with a number of experimental facts and basic principles.—B. W. M.

***The Change of Electric Resistance of Bismuth Crystals in Strong Magnetic Fields. IV.—Discussion and Interpretation of the Experimental Results.** Yasaku Tanabe (*Sci. Rep. Research Inst. Tôhoku Univ.*, 1950, [A], **2**, (3), 531–548).—[In English]. Cf. *ibid.*, (2), 341; *Met. Abs.*, 1950–51, **18**, 641. Experimental results obtained in Parts II. and III. are discussed and interpreted. The marked anisotropy revealed by the change of resistance of Bi crystals in magnetic fields up to 200 kOe. is explained in terms of the special character of the conduction electrons in the crystals in accordance with Jones's model of energy levels in Bi crystals (*Proc. Roy. Soc.*, 1936, [A], **155**, 653; *Met. Abs.*, 1936, **3**, 440). Satisfactory agreement is found between experimental and calculated results. Complicated changes of resistance attributable to direction of the magnetic field, direction of current, and crystal orientation cannot, as yet, be completely explained quant. Conductivity phenomena in Bi crystals are more sensitive to impurities and imperfections of the crystal lattice than is the case with any other metal crystals.—J. S. G. T.

***The Electrical Conductivity of Bismuth Fibres. II.—Anomalies in the Magneto-Resistance.** B. Donovan and G. K. T. Conn (*Phil. Mag.*, 1950, [vii], **41**, (319), 770–782).—Cf. *ibid.*, 1949, [vii], **40**, 283; *Met. Abs.*, 1948–49, **16**, 625. The variation of elect. resistivity with magnetic field was investigated for several Bi fibres, known to behave effectively as single crystals. A significant proportion showed an anomalous, small initial decrease of elect. resistivity, leading to a min. with fields of 100–800 Oe. The anomalous fibres are believed to have their main cleavage plane inclined at a small angle to the fibre axis. The results are explained on the basis of a hitherto undiscovered longitudinal Hall effect.
—P. C. L. P.

***Magneto-Resistance of Bismuth at 3000 Megacycles.** C. W. Heaps (*Phys. Rev.*, 1950, [ii], **80**, (5), 892–893).—The magneto-resistance of a Bi cavity resonating at a frequency of 3000 Mc./s. was found to be $<\frac{1}{2}$ the D.C. magneto-resistance. This is consistent with the possibility that the mean free

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* Denotes a paper describing the results of original research. † Denotes a first-class critical review.

path is comparable with the skin depth. Etching the surfaces with HNO_3 decreased the magneto-resistance compared with that given by freshly machined surfaces.—P. C. L. P.

***The New Element Californium (Atomic Number 98).** S. G. Thompson, K. Street, Jr., A. Ghiorso, and G. T. Seaborg (*Phys. Rev.*, 1950, [ii], **80**, (5), 790–796).—Definite identification has been made of an element with atomic number 98, and it is thought to have the mass number 244. The experiments so far have revealed only the tripositive oxidation state.—T. G.

***Chemical Properties of Californium.** K. Street, Jr., S. G. Thompson, and G. T. Seaborg (*U.S. Atomic Energy Commission Publ.*, 1950, (**AECD-2891**), 13 pp.; also (**UCRL-752**)).—From ion-exchange adsorption data, Cf apparently fits in as the 9th actinide element. So far only the tripositive oxidation state has been revealed, and this makes the element similar in nature to eka-dysprosium.—B. W. M.

Recent Progress in the Metallurgy of Copper and Copper Alloys. Maurice Cook (*Proc. Fourth Empire Min. Met. Congr.*, **1950**, (II), 1029–1046; discussion, 1047–1093).—See *Met. Abs.*, 1949–50, **17**, 241.

The Energy Level of the Valence Electron in Copper, Viewed from Its X-Ray Spectrum [—II. (1)]. Mitsuro Satô (*Sci. Rep. Research Inst. Tôhoku Univ.*, 1950, [A], **2**, (3), 424–428).—Cf. *ibid.*, 1949, [A], **1**, 51; *Met. Abs.*, 1950–51, **18**, 151. [In English]. Energy levels expected to be associated with the valence electron in Cu are deduced from the *L*-satellites in the X-ray spectrum of Cu determined by Gwinner (*Z. Physik*, 1938, **108**, 523) and actual values of the levels calculated. —J. S. G. T.

***High-Frequency Vacuum Breakdown Tests for Copper and Copper-Plated Ceramic Surfaces.** P. C. Bettler (*U.S. Atomic Energy Commission Publ.*, 1948, (**MDDC-1695**), 4 pp.).—H.F. breakdown tests were evolved in a vacuum between Cu-plated zircon surfaces in connection with the use of Cu-plated ceramics for the teeth of the rotary capacitor for a frequency-modulated cyclotron. Solid Cu electrodes were tested for comparison, and data were obtained on both outgassed and fresh surfaces. Cu-plated ceramic gave similar results to solid Cu, the amount of sputtering not being too serious.—B. W. M.

***Pressure Dependence of Resistance of Germanium.** Julius H. Taylor (*Phys. Rev.*, 1950, [ii], **80**, (5), 919–920).—A letter. The effect of hydrostatic pressures up to 4500 lb./in.^{2} on the resistivity of 4 samples of Ge has been measured in the temp. range 297°–475° K. The results when reduced to 300° K. give a pressure coeff. of $10{\cdot}2 \pm 0{\cdot}4 \times 10^{-6}$/atm. Taken in conjunction with the compressibility and the coeff. of linear expansion, this leads to a change in the energy gap of $-0{\cdot}87 \times 10^{-4}$ eV./°C., which result is compared with those obtained by other methods.—P. C. L. P.

***On the Mechanism of Impurity-Band Conduction in Semi-Conductors [Germanium].** Cavid Erginsoy (*Phys. Rev.*, 1950, [ii], **80**, (6), 1104–1105).—A letter. It is pointed out that broadening of the discrete impurity states is to be expected with increasing impurity concentration, leading to a low-temp. activation energy lower than the normal activation energy. The existence of this activation energy is supported by the slopes of the log resistivity against $1/T$ and log Hall coeff. against $1/T$ curves at low temp. reported by Hung and Gliessmann for Ge (*ibid.*, **79**, 726; *Met. Abs.*, 1950–51, **18**, 418). The model can also be used to explain the resistivity/temp. curves of Ge without assuming the presence of more than one type of impurity.—P. C. L. P.

***Changes in Conductivity of Germanium Induced by Alpha-Particle Bombardment.** W. H. Brattain and G. L. Pearson (*Phys. Rev.*, 1950, [ii], **80**, (5), 846–850).—The effects on the elect. conductivity of a single crystal of high-purity *n*-type Ge during bombardment by α particles from Po have been investigated. The first effect was to eliminate the conduction electrons at the rate of 78/incident particle, the conductivity attaining a min. value. The mechanism causing this is discussed. Subsequently, conducting holes are introduced at an initial rate of 8·6/α particle; of these 6·6 decay with time if the bombardment is discontinued. After decay, the distribution of conducting holes with depth was investigated by etching off successive layers. The max. number of holes occurred at a depth of $1{\cdot}3 \times 10^{-3}$ cm., dropping to zero at the max. penetration depth of the α particles of $1{\cdot}9 \times 10^{-3}$ cm.—P. C. L. P.

***A Study of Thermoelectric Effects at the Surfaces of Transistor Materials [Germanium].** J. W. Granville and C. A. Hogarth (*Proc. Phys. Soc.*, 1951, [B], **64**, (6), 488–494).—Measurements were made of the photo-voltaic effect, the rectification and the polarity of the thermoelect. power, for Ge and PbS crystals, ground, polished, or cleaved. The thermoelect. power was the only effect which was not in agreement with the predictions made from the type (*n* or *p*) of each semi-conductor. A surface exploration showed there to be a point-to-point variation of thermoelect. polarity. This irregularity could be entirely removed by high-temp. baking. From electron diffraction and other subsidiary tests it was proved that the irregularities were due entirely to the quasi-amorphous surface film produced by polishing (cleaved surfaces behaved normally). The mechanism responsible for the irregularities is not understood.—R. W. C.

***Intergranular Energy of Iron and Some Iron Alloys.** Lawrence H. Van Vlack (*J. Metals*, 1951, **3**, (3), 251–259).—Using experimental techniques which are described in detail, the energy of the γ-Fe grain boundary has been determined and found to be 850 ergs/cm.^{2} at 1105° C. The α/α and α/γ boundaries were found to possess somewhat less energy. 24 ref.—E. N.

***The Plastic Deformation of Pure Single Crystals of Lead and Copper.** Peter W. Neurath and James S. Koehler (*J. Appl. Physics*, 1951, **22**, (5), 621–626).—Precision experiments on the creep of Pb and Cu crystals were carried out, by an experimental procedure which was carefully defined. The steady-state creep was negligible for Cu at 23° or −190° C., for resolved shearing stresses $<0{\cdot}6$ kg./mm.^{2}, and for Pb at −190° C., for resolved stresses $<0{\cdot}13$ kg./mm.^{2}. Stress/strain curves were taken under conditions excluding steady-state creep, and a resolved Y.P. of 219 ± 21 g./mm.^{2} found for Cu at both the above temp. For Pb at −190° C. the resolved Y.P. was 96 ± 8 g./mm.^{2}. Annealing effects were studied: 600° C. did not suffice, but 1000° C. did, to soften all Cu crystals to a const. Y.P. Steady-state creep measurements with Pb showed that the steady rate at a given temp. increased exponentially with stress.—R. W. C.

***The Influence of Vibration on the Creep of Lead [and Lead Alloys].** J. Neill Greenwood (*Proc. Amer. Soc. Test. Mat.*, 1949, **49**, 834–850; discussion, 851–856).—Creep tests were made on two very pure indust. Pb and Pb–0·03% Ag and Pb–0·07% Cu alloys, with stresses between 100 and 350 lb./in.^{2} and with a gentle 50 c./s. vibration superposed on the direct tensile stress. Vibration increased the rate of creep and accelerated the recrystn. under stress. Thus one specimen of Pb recrystallized after an extension of 4–5% without vibration and after 2·5% extension with vibration. During recrystn. the grain-size and the rate of creep both increased. Detailed comparisons are made of the behaviour of the various materials under creep with and without vibration, and also of the differences between the materials under the same conditions. The effect of Ag and Cu addn. on the creep of Pb depends on the circumstances, but both reduce the susceptibility of Pb to recrystallize under stress. Annealing at 120° C. for 24 hr. stabilizes the Pb, reduces the creep rate under given conditions, and reduces the susceptibility to recrystn. under stress.—P. T. G.

***A Surface Effect in the Creep Behaviour of Polycrystalline Lead.** E. N. da C. Andrade and A. J. Kennedy (*Proc. Phys. Soc.*, 1951, [B], **64**, (4), 363–366).—A letter. Creep experiments were carried out with pure Pb contg. 0·05% Te; this alloy undergoes transient creep only, i.e. the creep equation is $l = l_0 (1 + \beta t^{1/3})$. Plots of β against stress were prepared for wires of different dia. The plots did not coincide, but could be made to do so if corrected on the assumption that a thin surface layer was offering effectively no resistance to creep. To achieve coincidence of the plots, this layer had to be taken as $0{\cdot}301a$ in depth, where a = mean grain-size (const. and

= 0·2 mm. for all specimens). This surface effect is interpreted as being due to a lesser constraint of the surface region as regards slip. X-ray studies revealed a change in type of structure ~0·1 mm. below the surface.—R. W. C.

***Solubility of Hydrogen in Molten Lead.** W. R. Opie and N. J. Grant (*J. Metals*, 1951, **3**, (3), 244–245).—The solubility of H in pure (99·999+%) molten Pb, inductively melted in an alundum crucible, was determined as a function of temp. and pressure. The results show that the solubility (*S*, in c.c./100 g. Pb) of H in molten Pb : (1) at a pressure of 760 mm. Hg. is ~0·12, 0·25, 0·45, 0·8, and 1·3 at 500°, 600°, 700°, 800°, and 900° C., resp., and this may be represented by the equation: $\log_{10}S = \frac{-2450}{T} + 2{\cdot}19$, where $T = {}^\circ$K.; and (2) obeys Sievert's law ($S \propto \sqrt{P}$ at const. temp, where $P =$ pressure) within the temp. and pressure (100–784 mm. Hg) ranges studied. The heat of soln. was found to be 22·3 kg.cal./mole H. 4 ref. [For correction, see *ibid.*, (7), 528]. —E. N.

***Anomalous Heat Flow in Supraconductors [Lead and a Lead–Bismuth Alloy].** K. Mendelssohn and J. L. Olsen (*Phys. Rev.*, 1950, [ii], **80**, (5), 859–862).—The thermal conductivity of Pb has been measured at 4·6° and at 2·7° K., and of a Pb alloy with 0·02% Bi at 5·3° and at 2·9° K. An anomaly was found in the thermal conductivity of the alloy at 2·9° K. On applying a magnetic field, thus tending to bring the specimen back to the normal state, the thermal conductivity in the transverse direction was found to be less in the intermediate than in either the supraconducting or the normal state. The explanation suggested is based on the possibility of a circular flow of heat.—P. C. L. P.

***On the Cohesive Energy of Metallic Lithium.** R. A. Silverman and W. Kohn (*Phys. Rev.*, 1950, [ii], **80**, (5), 912–913).—A letter. The cohesive energy of Li has been calculated as 35·2 kg.cal./mole in comparison with the experimental value of 39·0 kg.cal./mole. The discrepancy is believed to lie in the atomic-sphere approximation used, which cannot be expected to be reliable near the top of the energy band. —P. C. L. P.

The Truth About Magnesium. George W. Orton (*Modern Metals*, 1951, **7**, (2), 52–53).—O. considers that Mg has proved itself as a constructional material, and discusses some of the criticisms, such as liability to cracking, lack of corrosion-resistance, and ease of ignition, which have been directed at Mg. He concludes that any difficulties are problems of technique in handling, processing, or finishing.—R. J.

Extraction, Alloying, and Fabrication of Magnesium. C. J. P. Ball (*Proc. Fourth Empire Min. Met. Congr.*, **1950**, (II), 1004–1028; discussion, 1047–1093).—See *Met. Abs.*, 1949–50, **17**, 242.

***The Heat of Combustion of Magnesium and Aluminium.** C. E. Holley and E. J. Huber (*U.S. Atomic Energy Commission Publ.*, 1951, (**AECU-1172**), 10 pp.).—Precise measurements of the heats of combustion of Mg and Al were made in an O bomb calorimeter, correcting for the elect. energy used in firing the charge and determining the completeness of combustion. For Mg, heat of combustion in 25 atm. O was 5896·0 ± 3·6 cal./g. and heat of formation of MgO was $\Delta H_{299^\circ \text{K.}} = -143{\cdot}73 \pm 0{\cdot}09$ kg.cal./mole. Heat of combustion of Al = 7405·6 ± 5·2 cal./g. and for Al_2O_3, $\Delta H_{299^\circ \text{K.}} = -400{\cdot}41 \pm 0{\cdot}28$ kg.cal./mole. These values are in substantial agreement with those published previously.—B. W. M.

***Heat Capacity of Liquid Mercury Between 0° and 450° C.; Calculation of Certain Thermodynamic Properties of the Liquid and Vapour.** Thomas B. Douglas, Anne F. Ball, and Defoe C. Ginnings (*J. Research Nat. Bur. Stand.*, 1951, **46**, (4), 334–348).—The enthalpy of liq. Hg from 0° to 450° C. was measured by the drop method. The results were combined with previously published vapour-pressure measurements near the normal b.p. and experimental values of fundamental const. to calculate the thermodynamic properties. Tables are given of the following functions between the triple point and 500° C.: relative enthalpies of liquid and vapour, heat of vaporization, compressibility factor of vapour, heat capacities of liquid and vapour, abs. entropies, relative free energies, and vapour pressure. The accuracy of the results is discussed, and comparison is made with experimental vapour-pressure results and entropies calculated from low-temp. data on solid Hg. 56 ref.—J. W. C.

Research on Nickel and Its Alloys. —— (*Metal Treatment*, 1950, **17**, (61), 29–34).—A review, under 9 sectional headings, of research work at the Birmingham laboratories of The Mond Nickel Co., Ltd.—J. W. C.

A Note on Thin Nickel Foils. Stanley Bashkin and Gerson Goldhaber (*Rev. Sci. Instruments*, 1951, **22**, (2), 112–113).—A note on the prepn. and properties of Ni foils 250–1000 Å. thick.—E. J.

***The Use of Low Temperatures for the Granulometry of Powdered Ferromagnetic Materials [Nickel and Iron].** Louis Weil and René Pauthenet (*J. Phys. Radium*, 1951, **12**, (4), 23S–24S).—The significance of using different temp., more especially low temp., for determining grain-sizes of ferromagnetic materials is explained in some detail. W. used temp. of 291° (room temp.), 77° (liq. N), and 20·4° K. (liq. H) (*Compt. rend.*, 1949, **229**, 584; *Met. Abs.*, 1949–50, **17**, 325). Illustrative examples derived from magnetic results obtained at these three temp. in the cases of Raney Ni and Fe show how it is possible, by such procedure, to determine approx. the distribution of grain-sizes in agglomerates, in cases where X-ray analysis can provide only a mean value.—J. S. G. T.

***The Electrical Properties of Selenium Coatings.** Paul H. Keck (*J. Opt. Soc. Amer.*, 1951, **41**, (1), 53–55).—The dark- and photo-conductivities of thin Se coatings deposited at 30°–75° C. upon substrates *in vacuo* were studied. At coating temp. <50° C. only vitreous Se is formed; at higher temp. the formation of monoclinic Se is favoured, commencing at ~50° C. The rectifying properties of the deposits indicate that, in vitreous Se, hole conduction is predominant over electron conduction, i.e. conduction is chiefly of the *p*-type, whereas the monoclinic form of deposit exhibits the *n*-type of conduction discussed by Gudden and Pohl (*Z. Physik*, 1926, **35**, 243; *J. Inst. Metals* (Abstracts), 1926, **35**, 486).—J. S. G. T.

***Some Optical Properties of Evaporated Layers of Silver, Copper, and Tin.** D. G. Avery (*Phil. Mag.*, 1950, [vii], **41**, (321), 1018–1031).—A detailed account is given of the interferometric method of determining phase changes occurring on reflection by metallic films. Films of Ag, Cu, and Sn were produced by evaporation under a pressure of $< 5 \times 10^{-5}$ mm. Hg. The variations of the ellipticity, ρ, and the relative phase change Δ, on reflection by a 400–500 Å. thick Ag film, with the angle of incidence θ, were found to agree closely with theoretical expectations. The variations of ρ and Δ with film thickness (100–500 Å.), at a const. θ of 65°, agreed with theory for thicknesses >150 Å. Similar measurements of the variations of ρ and Δ with θ were made with thick Cu films. The measurements with Sn films were made with $\theta = 65^\circ$ only, and gave results different from those given by a plane surface prepared by allowing a drop of molten Sn to fall on a glass surface. Three possible explanations of these differences are suggested.—P. C. L. P.

***Grain-Boundary Energies in Silver.** A. P. Greenough and Ronald King (*J. Inst. Metals*, 1951, **79**, (6), 415–427).—The variation of grain-boundary energy with the angle between the crystals meeting at the boundary has been investigated for Ag by examination of boundary grooves formed during thermal etching. The results are found to be in fair agreement with theoretical prediction based on the treatment of the boundary as a region of transition represented by an assembly of dislocations. 16 ref.—Authors.

***Lattice and Grain-Boundary Self-Diffusion in Silver.** R. E. Hoffman and D. Turnbull (*J. Appl. Physics*, 1951, **22**, (5), 634–639).—The theoretical background for deriving grain-boundary self-diffusion rates by comparative measurements on single-crystal and polycryst. specimens, either by a sectioning technique or by a surface-activity decay method is concisely discussed. Experiments based on these ideas were carried out with Ag. The plot of log $D/(1/T)$ was identical for single and polycrystals down to 700° C.; below this the polycrystal rate was faster. From the data the grain-boundary diffusion coeff. is calculated as $D_B = 0{\cdot}03 \exp(-20{,}200/RT)$ cm.^{2} sec.$^{-1}$ and $0{\cdot}12 \exp(-21{,}500/RT)$ cm^2. sec.$^{-1}$ for pure and super-pure Ag, resp. The coeff. for single crystals are given by $D_L = 0{\cdot}895 \exp(-45{,}950/RT)$ cm^2. sec^{-1}. An autoradiograph taken after a low-temp. diffusion anneal is reproduced as further evidence that $D_B \gg D_L$. It was found that D_L was independent of the thermal history of the crystals.—R. W. C.

***Preparation of Silver Colloid.** Gentaro Matsumura (*J. Amer. Chem. Soc.*, 1951, **73**, (5), 2375).—A note. A colloidal soln. of Ag prepared by heat-treatment of the fine powder obtained on drying a kneaded mixture of powdered MgO and dil. aq. $AgNO_3$ soln., followed by treatment with excess dil. acetic acid, filtration, and electrodialysis, was shown by spectral analysis to consist of metallic Ag with a trace of Mg.—J. R.

***An Absolute Measurement of the Susceptibility of Tantalum and Other Metals.** F. E. Hoare and J. C. Walling (*Proc. Phys. Soc.*, 1951, [B], **64**, (4), 337–341).—The magnetic (mass) susceptibility of spectrochem. pure Ta was determined abs. by the Gouy method, to a high precision. The susceptibilities of spectrochem. pure Pt, Pd, and Rh were determined by comparison with Ta. Values at 20° C. are: $\chi_{Ta} \times 10^6 = 0{\cdot}8490 \pm 0{\cdot}0006$, $\chi_{Pt} \times 10^6 = 0{\cdot}9712 \pm 0{\cdot}0007$, $\chi_{Pd} \times 10^6 = 5{\cdot}231 \times 0{\cdot}0004$, $\chi_{Rh} \times 10^6 = 0{\cdot}9903 \pm 0{\cdot}0008$.—R. W. C.

***Periodic Deviations in the Schottky Effect for Tantalum.** R. J. Munick, W. B. LaBerge, and E. A. Coomes (*Phys. Rev.*, 1950, [ii], **80**, (5), 887–891).—The thermionic emission from a Ta filament, investigated as a function of applied collecting voltage, shows systematic periodic deviations from the Schottky law. The phase and amplitude of the deviations are in approx. agreement with a theory neglecting the quantum-mech. tunnel effect. In agreement with the theory, the amplitude of the deviations at 1200° K. was greater than at 1500° K., though the phase remained the same. Successive depositions of Th on to the emitting surface, which lowered the work-function from 4·03 to 3·83 and then to 2·73 eV., caused a shift of the deviations to lower voltages and the amplitudes to decrease.—P. C. L. P.

***The Vapour Pressure of Tellurium and Selenium.** L. S. Brooks (*U.S. Atomic Energy Commission Publ.*, 1948, (**AECD-2546**), 12 pp.).—The vapour pressure of Te and Se were measured with quartz Bourdon gauges at pressures up to $\frac{1}{3}$ atm. for Te and just >1 atm. for Se. In the equation $\log p = A - B/T$, if A and B are const., p is the pressure in mm. Hg, T is the abs. temp. and if T_b = b.p. in °C. and L_v = latent heat of vaporization in kg.cal./mole, the properties found were as follows:

Element	A	B	T_b	L_v
Te	$7{\cdot}5999 \pm 0{\cdot}0136$	$5960{\cdot}2 \pm 14{\cdot}5$	$989{\cdot}8 \pm 3{\cdot}8$	$27{\cdot}26 \pm 0{\cdot}07$
Se	$8{\cdot}0886 \pm 0{\cdot}0048$	$4989{\cdot}5 \pm 4{\cdot}5$	$684{\cdot}9 \pm 1{\cdot}0$	$22{\cdot}82 \pm 0{\cdot}02$

These results are in good agreement with those obtained by de Selincourt (*Proc. Phys. Soc.*, 1940, **52**, 348; *Met. Abs.*, 1940, **7**, 276).—B. W. M.

†Thallium: Properties, Sources, Recovery, and Uses of the Element and Its Compounds. William H. Waggaman, Gladys G. Heffner, and Edwin A. Gee (*U.S. Bur. Mines Inform Circ.*, 1950, (**7553**), 50 pp.).—The properties and prepn. of Tl and its compounds, the sources, and extraction of Tl are reviewed. The uses of Tl, Tl-bearing alloys, and Tl compounds are discussed, and methods of analysis, both chem. and spectrographic, are described. *Bibliography* of 369 ref. —E. C. P.

***Internal Friction and Grain-Boundary Viscosity of Tin.** L. Rotherham, A. D. N. Smith, and G. B. Greenough (*J. Inst. Metals*, 1951, **79**, (6), 439–454).—The internal friction of high-purity Sn has been studied over the temp. range 15°–150° C. by measurements made on bars vibrating transversely at audio frequencies in the "free–free" mode. Both polycryst. specimens and bars consisting of a very few crystals have been examined. A peak in the curve of internal friction plotted against temp., ascribed to viscous relaxation at the grain boundaries, is found to exist for the former type, but not for the latter. The activation energy associated with the relaxation has been measured, and is consistent with that for steady-state creep of single crystals, but differs greatly from the value for self-diffusion in Sn. This is contrary to a theory put forward by Kê that all three values should be the same (*Phys. Rev.*, 1948, [ii], **73**, 267; *Met. Abs.*, 1947–48, **15**, 503). The variation of Young's modulus with temp. has been observed over the same range for both types of specimen. The results are similar to those obtained by Kê for Al (*ibid.*, 1947, [ii], **71**, 533; *Met. Abs.*, 1947, **14**, 305), although the magnitude of the effect observed is smaller. 15 ref.—AUTHORS.

***Magnetic Moments and Eddy Currents in Spheres of Supraconducting Tin.** James J. Fritz, Oscar D. Gonzalez, and Herrick L. Johnston (*Phys. Rev.*, 1950, [ii], **80**, (5), 894–899).—The magnetic moment frozen in solid and hollow supraconducting Sn spheres as a result of demagnetization from fields sufficient completely or partially to destroy their supraconductivity has been studied. With solid Sn spheres a small effect was found which was attributed to trace impurities. The hollow spheres remained partly in the intermediate state after demagnetization, giving rise to an appreciable magnetic moment. The effects of remagnetization on the magnetic moment and on the eddy-current damping characteristics gave interesting evidence on the nature of the intermediate state.—P. C. L. P.

***Solubility of Argon in Uranium.** I. B. Johns (*U.S. Atomic Energy Commission Publ.*, 1946, (**MDDC-290**), 1 p.; also (**LADC-202**)).—At room temp. the amount of A absorbed by U is <1 part in 3 × 10^5 by weight and at 600° C., <1 part in 3 × 10^4, which is the magnitude of the uncertainties of measurement of the gas vol.—B. W. M.

***The Use of Uranium as a Pump for Hydrogen.** R. C. Cornelius and S. E. Stephanou (*U.S. Atomic Energy Commission Publ.*, 1950, (**LA-1129**), 28 pp.).—The use of U as an absorbing medium for H in a Zinn-type ion source was studied for an investigation of the extent of the recovery of H and the resulting decrease in pressure in the accelerating tube attached to the ion source. With various geometries a max. recovery of ~70% was obtained and accelerating tube pressures were decreased by a factor of 2 or more, depending on the rate of flow of H into the ion source. These preliminary results suggest the method may have appn. under actual operating conditions of the ion source.—B. W. M.

***Rate of Evaporation of Zinc at Low Pressures.** H. W. St. Clair and M. J. Spendlove (*U.S. Bur. Mines Rep. Invest.*, 1950, (**4710**), 13 pp.).—The rate of evaporation of Zn was measured by heating molten Zn in a graphite crucible, allowing the vapour to condense in a water-cooled condenser, and measuring the weight of condensate and the loss in weight of the metal in the crucible. The results showed that the observed rate of evaporation was always less than the max. theoretical rate, the difference being a function of the residual gas pressure in contact with the metal. At temp. below the b.p. of Zn, 907° C., the rate of evaporation was slow, but when the gas pressure was reduced to <1 mm. Hg rapid distillation occurred at 500° C., e.g. at 5 mm. and 25 μ Hg the rates of evaporation were 0·09 and 0·30 g./cm.^{2}/min., resp. At low rates of evaporation the condensate appeared as dendritic crystals, while at high rates the condensate was more dense, having smooth, rounded surfaces suggesting that it had passed through the liq. phase before solidifying. Owing to the large heat of evaporation, temp. differences of 35°–100° C. between the surface and main mass of metal were observed. Agitation made the temp. of the metal more uniform and increased the rate of evaporation ~10%. The rate of evaporation was decreased and the temp. of the metal increased by constriction of the path of the vapour to the condenser.—E. C. P.

Zirconium Production, Properties, and Alloys. G. L. Miller (*Metallurgia*, 1951, **43**, (259), 209–214, 220).—The prodn. of Zr by the van Arkel and Kroll processes is described, and the properties of the metal and its alloys are reviewed. 9 ref.—F. M. L.

***A Metallurgical Investigation of Silver Chloride.** R. D. Moeller, F. W. Schonfeld, C. R. Tipton, Jr., and J. T. Waber (*Trans. Amer. Soc. Metals*, 1951, **43**, 39–66; discussion, 66–69).—See *Met. Abs.*, 1950–51, **18**, 344; and this vol., col. 6.

The Theory of the Degassing of Molten Metals Under Reduced Pressure. L. Fekete (*Acta Techn. Acad. Sci. Hungar.*, 1950, **1**, (1), 33–49).—[In German]. A simple, straightforward, hydrostatic theory, taking no account of diffusion, of the degassing of metals under reduced pressure, is developed.—J. S. G. T.

†**The Cold Working of Metals.** Cecil H. Desch (*Proc. Fourth Empire Min. Met. Congr.*, 1950, (II), 978–1003; discussion, 1047–1093).—See *Met. Abs.*, 1949–50, **17**, 245.

†**Creep of Metals.** J. D. Lubahn (*Cold Working of Metals* (*Amer. Soc. Metals*), 1949, 223–247).—The possible mechanism of creep is discussed. It is first emphasized that the view that the creep rate during the secondary stage of creep is const. is, fundamentally, not justified. Ref. is made to the conception of a " mech. equation of state ", and the two explanations of strain-hardening—that it is due to interference by previous deformation or to the " using up " of the sites of easiest slip—are reviewed. It is suggested that under const. stress the overall deformation is the sum of anelastic and plastic components and that the slope of a strain/stress curve should continuously decrease, as a result only, after the initial stages, of strain-hardening. At low temp, the stress required for continuing flow is primarily a function of strain, but at high temp. it depends markedly on the rate and much less on the strain.—J. C. C.

The Importance of Plasticity in Rupture Phenomena of Metals. O. L. Bihet (*Rev. Soudure*, 1950, 6, (1), 20–30).—A review of some of the more recent work on plasticity and fracture; the need for more critical laboratory tests is emphasized. 17 ref.—W. D. B.

Report of [A.S.T.M.] Committee E-9 on Fatigue. —— (*Proc. Amer. Soc. Test. Mat.*, 1950, **50**, 497–508).—See *Met. Abs.*, 1950–51, **18**, 155.

***On the Inhomogeneous Deformation of a Plastic Lamina in a Compression Test.** R. Hill (*Phil. Mag.*, 1950, [vii], **41**, (319), 733–744).—A theory is proposed for the calculation of the stresses and displacements in a thin sheet plastically compressed between parallel plates, taking into account inhomogeneous distortion caused by friction and edge constraints. The equations enable the coeff. of friction to be estimated from observations on the change of shape of a rectangular sheet. Experimental results are presented on the deformation of isotropic Cu specimens using various solid and liq. lubricants, including thin Pb foil, and on the deformation of Al and of a Pb alloy with pure Pb and In foils resp., which are in satisfactory agreement with the theory.
—P. C. L. P.

***Some Quantitative Data Bearing on the Scabbing of Metals Under Explosive Attack.** John S. Rinehart (*J. Appl. Physics*, 1951, 22, (5), 555–560).—Experiments were carried out on the local tension failure near one surface of a plate subjected to an explosive force on the other surface (scabbing). The development of the pressure wave and the reflected tension wave was studied by indirect means, and the corresponding normal fracture stress was computed. Results are given for two steels, an Al alloy, Cu, and brass. The results support the mechanism proposed to explain the phenomenon.
—R. W. C.

The Evaluation of Macroscopic Residual Stresses in Cylindrical Bars. H. Föppl (*J. Iron Steel Inst.*, 1951, **168**, (1), 15–23).—F. reviews the basic assumptions of the math. treatments used to evaluate internal stresses in bars and discusses some recent advances in stress–strain theory, which permit calculation of the limiting values of residual stress at each point in a body. Previous theories of residual stress have postulated a body consisting of a plastically deformed surface layer and an elastically deformed core. In an endeavour to simplify the theory, F. postulates the existence of an intermediate layer in which the plastic deformation is opposite in sign to the elastic deformation of the core. F.'s theory of a stress reversal in the intermediate layer is confirmed experimentally by results obtained with steel specimens. F. gives a new math. treatment for the evaluation of the principal stresses in a cylindrical bar: by the introduction of strain ratios by which the relationship of the principal strains are expressed, the treatment enables all the principal stresses to be calculated accurately, even though the strains be measured in only one direction. F.'s treatment is compared with those of Heyn and Sachs. The stress-reversal theory implies a discontinuity in the gradual transition from the plastic deformation of the surface to the elastic deformation of the core. The mainly bi-axially stressed surface layer should therefore be able to behave independently of the tri-axially stressed core; this was confirmed by experiment. A method is described in an Appendix for determining the length changes in a bar, the plastically deformed layers of which are removed by successive etching treatments. 24 ref.—R. W. R.

***Static Friction Tests with Various Metal Combinations and Special Lubricants.** H. S. White and Dino Zei (*J. Research Nat. Bur. Stand.*, 1951, **46**, (4), 292–298).—Inclined-plane tests for metal combinations involving stainless steels, C steels, cast Fe, Al alloys, and Cr plate were undertaken with various lubricants. The heat-treated stainless-steel combinations gave the lowest friction, and combinations which included cast Fe or Al alloy gave the highest friction. Lubricants contg. MoS_2 gave the best results.—J. W. C.

Erratum: Dry Metallic Friction as a Function of Temperature Between 4·2° and 600° K. I. Šimon, H. O. McMahon, and R. J. Bowen (*J. Appl. Physics*, 1951, 22, (6), 848).—Cf. *ibid.*, (2), 177; *Met. Abs.*, 1950–51, **18**, 695. Some ref. are given in connection with priorities for the adhesion hypothesis for friction, and the formula for the coeff. of static friction, advanced in the authors' recent paper.—R. W. C.

***Internal Friction of Ferromagnetic Materials.** A. Ferro and G. Montalenti (*J. Appl. Physics*, 1951, 22, (5), 565–568).—These experiments were made to test the hypothesis that the internal-friction component attributable to magnetic effects is due to an orientation oscillation of the domains with associated hysteresis. Experimentally, the magnetic component of internal friction was isolated by comparing the latter with the specimen (*a*) unmagnetized and (*b*) saturated. The value of this " magneto-elastic " friction was measured at various stresses (for Ni and several steels), and parallel measurements were taken, under static stressing, of the ratio residual induction : saturation intrinsic induction, which is a measure of domain rotation. The plots against stress of the two measured quantities had closely similar shapes for each substance, and this was taken as evidence in favour of the above hypothesis.—R. W. C.

On the Volume Diffusion of Metals. G. J. Dienes (*J. Appl. Physics*, 1951, 22, (6), 848).—A letter. A difference in the theoretical treatments of the diffusion frequency factor D_o, due to D. and to Zener (*ibid.*, (4), 372; *Met. Abs.*, this vol., col. 9), is commented upon in the light of some recent experimental data on diffusion. It is concluded that results on self-diffusion in Sn support D.'s views rather than Zener's.
—R. W. C.

A Note on the Theory of Coercive Force. Hiroshi Watanabe (*Sci. Rep. Research Inst. Tôhoku Univ.*, 1950, [A], 2, (3), 429–436).—[In English]. A math. theory of the origin of coercive force, based on experiments by Williams and Shockley (*Phys. Rev.*, 1949, [ii], **75**, 178; *Met. Abs.*, 1948–49, **16**, 478) is developed. Domain boundaries are not considered to be plane, and coercive force is determined by the sum of the gradients of boundary energy of cavities situated on concave parts of the boundary in the direction of advance of the boundaries.—J. S. G. T.

Empirical Correlation Among Supraconductors. J. G. Daunt (*Phys. Rev.*, 1950, [ii], **80**, (5), 911–912).—A letter. It is pointed out that, in general, the electronic sp. heat coeff. γ for the normal state of soft supraconductors varies linearly with the transition temp. T_c. If V is the atomic vol., a plot of γ/V against T_c leads to two straight lines, one for hard and the other for soft supraconductors. Al appears to fall on the hard supraconductor line.—P. C. L. P.

Two Comments on the Limits of Validity of the P. R. Weiss Theory of Ferromagnetism. P. W. Anderson (*Phys. Rev.*, 1950, [ii], **80**, (5), 922–923).—A letter. It is pointed out that: (i) the criterion for ferromagnetism on the Bloch spin-wave theory (*Z. Physik*, 1930, **61**, 206) is different in principle from that of Weiss (*Phys. Rev.*, 1948, [ii], **74**, 1493; *Met. Abs.*, 1948–49, **16**, 474); and (ii) on the Weiss theory, ferromagnetism must vanish at a sufficiently low temp. if the low-energy spin-wave states are taken into account.—P. C. L. P.

Note on the Inertia and Damping Constant of Ferromagnetic Domain Boundaries. C. Kittel (*Phys. Rev.*, 1950, [ii], **80**, (5), 918).—A letter. The theory treating a domain boundary moving under a magnetic field as having an effective mass, is generalized to include the case where the intrinsic relaxation frequency of the substance is high.—P. C. L. P.

***Studies of the Propagation Velocity of a Ferromagnetic Domain Boundary.** H. J. Williams, W. Shockley, and C. Kittel (*Phys. Rev.*, 1950, [ii], **80**, (6), 1090–1094).—A hollow rectangular specimen possessing a simple domain structure was cut from a single crystal of a 96 : 4 Fe–Si alloy. Experiments in weak magnetic fields ($\sim$0·003 Oe.) at 78°, 194°, and 293° K. gave results in approx. agreement with those calculated from the eddy-current losses arising from the uniform motion of a plane domain wall. In fields of 5–83 Oe., voltage pulses were measured which arose from the collapse of domain walls. The results agreed with calculations based on the eddy-current losses from a collapsing cylindrical boundary. The fact that observed eddy-current losses in ferromagnetic materials are always greater than those calculated, is attributed to the high local values of the permeability at the domain boundaries. A comparison of the losses calculated on the classical and domain models is given for a sq. rod in an A.C. field $\|$ its axis.—P. C. L. P.

***Energies and Widths of Domain Boundaries in Ferromagnetics.** B. A. Lilley (*Phil. Mag.*, 1950, [vii], **41**, (319), 792–813).—A general method of calculating the energies and widths of domain boundaries in ferromagnetic crystals is described, allowance being made for the effects of magnetostriction. The theoretical values of the energies and widths of the more important boundaries in Ni, Fe, and Co are calculated; for 180° boundaries at room temp. the theoretical widths are 2060, 1410, and 160×10^{-8} cm., resp.—P. C. L. P.

***Screening of Electronic Interactions in a Metal.** David Bohm and David Pines (*Phys. Rev.*, 1950, [ii], **80**, (5), 903–904).—A letter. It is shown from a consideration of the interactions in a high-*d* electron gas how the Coulomb forces come to be effectively screened.—P. C. L. P.

Secondary-Electron Emission and Atomic-Shell Structure. E. J. Sternglass (*Phys. Rev.*, 1950, [ii], **80**, (5), 925–926).—A letter. An empirical correlation is pointed out between the max. value of the yield of true secondary electrons and the position of the element in the Periodic Table.—P. C. L. P.

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2 — PROPERTIES OF ALLOYS

Aluminium Alloys—1940 to 1950. E. H. Dix, Jr. (*Metal Progress*, 1950, **58**, (4), 484–489).—An outline of the met. situation as it was in 1940 is given, and subsequent developments are traced. The most important single development of the war years was the acceptance by the aircraft industry of artificially aged wrought Al alloys for structural members. Brazing alloys and bearing alloys are also touched upon; high output is the present main preoccupation.—F. A. F.

Aluminium [Alloys] and Their Nomenclature. W. G. R. de Jager (*Metalen*, 1951, **6**, (5), 69–76).—[In Dutch]. An explanation is given of the descriptive terminology for Al alloys, and of the code of abbreviations now used in Holland, Great Britain, the U.S.A., France, Germany, and Switzerland. A table for converting in these languages the names or short titles of alloys in the six countries is given, comprising 36 wrought alloys and 51 cast alloys.—E. van S.

***Tension–Compression Biaxial Plastic Stress–Strain Relations for Aluminium Alloys 24S-T and 2S-O.** Joseph H. Faupel and Joseph Marin (*Trans. Amer. Soc. Metals*, 1951, **43**, 993–1012).—See *Met. Abs.*, 1950–51, **18**, 505.

***Tensile Properties of Some Aircraft Structural Materials [Aluminium Alloys] at Various Rates of Loading.** Richard F. Klinger (*Proc. Amer. Soc. Test. Mat.*, 1950, **50**, 1035–1050; discussion, 1051–1053).—See *Met. Abs.*, 1950–51, **18**, 505.

***The Influence of Fluctuations in Stress Amplitude on the Fatigue of Metals [Steels and 75S Alloy].** T. J. Dolan, F. E. Richart, Jr., and C. E. Work (*Proc. Amer. Soc. Test. Mat.*, 1949, **49**, 646–679; discussion, 680–682).—Fatigue tests were carried out on 3 steels and 75S-T Al alloy using small high-speed rotating cantilever-beam machines and notched and unnotched specimens. The specimens were subjected to repeated stresses fluctuating between two different predetermined amplitudes. In some tests the amplitude of stress was varied gradually and continuously between two limits, and in others it was alternated abruptly at regular intervals between two values. Results are given for a number of such tests, the stresses applied being in some cases above the normal fatigue limit and in others below. The greatest emphasis was on tests in which each kc. of a higher stress amplitude was followed by 9 kc. of a lower stress amplitude. 21 ref.
—P. T. G.

***Fatigue in Light Alloy Assemblies.** R. Boccon-Gibod (*Rev. Mét.*, 1951, **48**, (5), 369–375; discussion, 375).—Although it is important to know the intrinsic fatigue properties of metals and alloys, it is also necessary, especially when replacing steel by Al, to know the fatigue properties of assemblies of alloys. B.-G. studied the fatigue properties of a T-shaped girder joint, effected by welding, sticking with synthetic resins, and riveting, making the usual assumptions as to homogeneity and taking the max. stress as the imposed stress. He carried out similar tests on a structural steel and found that the use of light alloys in mech. structures leads to appreciable gains in weight-saving and strength.—J. H. W.

***Fatigue Tests [on Cast Aluminium Alloys].** J. Wood (*Metal Ind.*, 1951, **78**, (23), 459–462).—W. determined the fatigue limits of sand- and chill-cast D.T.D. 424 and Intal 305 (Cu 3·7, Si 5·2%) and Intal 308 (Cu 3·9, Si 9·1%). The values for D.T.D. 424 were higher than those previously published, partly owing to the difference in the section of the castings from which the test-pieces were machined. The fatigue limits of the Intal alloys were very similar to those of D.T.D. 428, and in three out of four cases actually higher than those for D.T.D. 424. It was not possible to account for the scatter of the S/N curve for the chill-cast D.T.D. 424 test-pieces.—J. H. W.

***[I.—] Solidification Textures of Aluminium and Light Alloys Produced by Immersion in Water [Water-Bath Quenching]. [II.—] Electron-Microscope Study of Age-Hardened Aluminium Alloys.** (Nishimura and Toyohara). See col. 119.

***Recovery and Cold Working of 52S Commercial Aluminium Alloy.** Gerard H. Boss (*Trans. Amer. Soc. Metals*, 1951, **43**, 122–140; discussion, 140–141).—See *Met. Abs.*, 1950–51, **18**, 349.

***On the Ageing of Aluminium–Magnesium Alloys.** H. Jolivet and M. Armand (*Rev. Mét.*, 1951, **48**, (5), 376–378; discussion, 378).—Vachet and Lacombe have observed an important break in the dilatometric curve between 50° and 100° C. during the heating of Al–Mg alloys that had been quenched in cold water and attributed this to the elimination of the internal stress caused by the quenching. This anomaly did not occur—or occurred only slightly—after less severe quenching. As no experimental confirmation was brought forward to support this hypothesis, J. and A. undertook a dilatometric study of the 11% Mg alloy after heating to 425° C. for 1 hr., quenching in cold water, and heating at 30° C. for up to 60 days. They found that the alloy thus treated showed at room temp. ageing accompanied by a contraction, a considerable increase in hardness, a slight increase in the tensile strength and in the elastic limit, and by an appreciable diminution in the elongation and impact resistance. Ageing is not accompanied by any visible micrographic alteration, and the effects disappear between 50° and 100° C.—J. H. W.

***Some Observations on the Occurrence of Stretcher-Strain Markings in an Aluminium–Magnesium Alloy.** R. Chadwick and W. H. L. Hooper (*J. Inst. Metals*, 1951–52, **80**, (1), 17–22).—Detailed observations have been made of the appearance of and dimensional distortion associated with surface markings developed by the progressive stretching of Al–3% Mg alloy sheet in different conditions of cold working and annealing. In material of 0·025 mm. grain-size, markings develop with a very small strain and are at first normal to the tension axis but subsequently become random in direction, reaching max. intensity at ~1%, and decaying within a 2% extension. These random markings consist of a series of kinks, and there is no thinning of the sheet. Parallel, intersecting bands or shallow grooves at a definite angle to the direction of stretching, first appear at ~2% extension and increase progressively in intensity up to the point of fracture. Parallel bands, which are caused by local thinning or necking, are of much less intensity than the random markings, which are the main cause of defects in pressing operations. When the grain-size exceeds 0·05 mm., random markings do not occur in stretching nor are they obtained in partially annealed or temper-rolled sheet irrespective of grain-size. Parallel bands are found in all these materials. When the grain-size is increased substantially, the well-known " orange-peel " effect develops on stretching and completely masks any other effect which might be present, the degree of roughening from this cause in sheet of 1·0 mm. grain-size being comparable with that produced by random markings in fine-grained sheet.—Authors.

***The Grain Refinement of Aluminium Alloy Castings by Additions of Titanium and Boron.** A. Cibula (*J. Inst. Metals*, 1951–52, **80**, (1), 1–16).—Previous work (*ibid.*, 1949–50, **76**, 321; *Met. Abs.*, 1949–50, **17**, 914) showed that the grain refinement produced by the addn. of Ti or B to Al casting alloys is primarily caused by nucleating particles in the melts; the nuclei in Ti-contg. alloys were found to be TiC crystals, though only a small proportion of the added Ti was present in this form. The main objects of the present work were to identify the nuclei in other fine-grained Al alloys and to find ways of increasing the proportion of the refining elements present as nucleating compounds. By centrifuging the particles from molten alloys contg. B but no Ti and observing the change in grain-size produced, evidence was obtained that the nuclei in these alloys are Al boride crystals. The min. B addn. for adequate refinement of these alloys therefore depends mainly on the solubility of Al boride in molten Al. Attempts to increase the concentration of TiC in alloys contg. Ti achieved no useful results, owing to difficulties in forming or dispersing the carbide as fine particles. The addn. of B instead of C was more effective in producing refinement, nucleating particles of Ti boride being formed at very low concentrations of Ti and B; moreover, B was more easily added than C, as Al–B master alloys could be used. The boride formed was isomorphous with pure TiB_2, but had smaller lattice dimensions and a range of compn. ($a_0 =$ 3·010–3·016, $c_0 =$ 3·235–3·240 Å.). The grain refinement of some commercial casting alloys by simultaneous addn. of Ti and B was studied in detail; grain coarsening due to high casting temp., repeated remelting, or gravity segregation during solidification was less than in alloys contg. much larger % of Ti alone. Alternative methods of adding Ti and B and the refinement produced by borides of transition metals other than Ti were investigated. As the B additions required when Ti was present were small, the mould reaction previously encountered in alloys of high B content was largely avoided. 18 ref.—Author.

History and Development of Aluminium–Silicon Alloys. E. Scheuer (*Found. Trade J.*, 1951, **90**, (1813), 569–578).—Read before the Slough Section of the Institute of British Foundrymen. A detailed historical review. S. also discusses the theoretical aspects of the modification process and the general characteristics and appn. of alloys of the Al–Si group. 36 ref.—J. E. G.

†A Review of Beryllium and Beryllium Alloys. (Richards). See col. 97.

***The Ternary System Chromium–Molybdenum–Iron.** J. W. Putman, R. D. Potter, and N. J. Grant (*Trans. Amer. Soc. Metals*, 1951, **43**, 824–847; discussion, 847–852).—See *Met. Abs.*, 1950–51, **18**, 352.

***The Effect of Various Treatments on the Fatigue Strength of Notched S-816 [Cobalt–Chromium–Nickel] and Timken 16-25-6 [Iron–Chromium–Nickel–Molybdenum] Alloys at Elevated Temperatures.** W. E. Jones, Jr., and G. B. Wilkes, Jr. (*Proc. Amer. Soc. Test. Mat.*, 1950, **50**, 744–760; discussion, 761–762).—See *Met. Abs.*, 1950–51, **18**, 505.

***Changes in Free Energy and Heat Content During the Reactions of Formation of Cobalt Arsenides.** M. I. Kochnev (*Zhur. Priklad. Khim.*, 1949, **22**, (9), 970–977).—[In Russian]. K. measured the ignition temp. (T) and the dissociation pressure at a temp. T' near to T (P_{As_4}) for six arsenides ($CoAs_2$, Co_2As_3, $CoAs$, Co_3As_2, Co_2As, Co_5As_2); $T = 540°$, 610°, 775°, 785°, 790°, and 805° C. resp., and $P_{As_4} \times 10^5 = 380$, 145, 7·21, 6·97, 5·82, and 5·61 mm. Hg, resp. From these results, the free energies of formation ($\Delta\Phi$) and heats of formation (ΔH), at T' and other temp. were calculated, and equations for the variation in equilibrium const. with temp. deduced. It is shown that at the same temp. the values of $\Delta\Phi$ and ΔH, per g.-atom Co, increase from Co_5As_2 to $CoAs_2$. —G. V. E. T.

***Effect of Neutron Bombardment on Order in the Alloy Cu_3Au.** S. Siegel (*U.S. Atomic Energy Commission Publ.*, 1949, (**AECD-2465**), 2 pp.).—Cf. *Phys. Rev.*, 1949, [ii], **75**, 1823; *Met. Abs.*, 1949–50, **17**, 65.—B. W. M.

***Re-Solution of Precipitated Silver in Copper–Silver Alloys.** Harold Margolin and Walter R. Hibbard, Jr. (*J. Metals*, 1951, **3**, (3), 247).—Cf. *Trans. Amer. Inst. Min. Met. Eng.*, 1951, **191**, 174; *Met. Abs.*, this vol., col. 18. During the ageing of the binary Cu–Ag (3 at.-%) alloy, a specimen which had been overaged 24 hr. at 550° C. and then heated in a N–H atmosphere for 30 hr. at 600° C., showed re-soln. of the Ag-rich precipitate. It is concluded, therefore, that the matrix–transition–precipitate pptn. sequence is reversible; a suggestion is made as to the mechanism of the process. 3 ref.—E. N.

High-Tensile Special Bronzes. E. Weisner (*Metall*, 1951, **5**, (1/2), 5–8).—A review of the compn., tech. properties, appn., and economics of special high-tensile bronzes conforming to various German specifications.—E. N.

†Analogies Between Steels and Aluminium Bronzes. H. Laplanche (*Proc. Inst. Brit. Found.*, 1950, **43**, A224–A250).—Exchange paper from L'Association Technique de Fonderie to the 1950 Conference of the Institute of British Foundrymen. A full and critical discussion in which are discussed the analogies between the thermal diagrams of the binary systems $Fe–Fe_3C$ and Al–Cu, the microstructure of these two groups of alloys, the martensitic transformations occurring therein and the additive isothermal decompn. of the γ-phase in steels and the β-phase in Al bronze. 149 ref.—J. E. G.

***The Ternary System Indium–Cadmium–Zinc.** S. C. Carapella, Jr., and E. A. Peretti (*Trans. Amer. Soc. Metals*, 1951, **43**, 853–872).—See *Met. Abs.*, 1950–51, **18**, 353.

***Grain and Grain-Boundary Compositions: Mechanism of Temper-Brittleness [in Steel].** J. W. Spretnak and Rudolph Speiser (*Trans. Amer. Soc. Metals*, 1951, **43**, 734–748; discussion, 748–758).—See *Met. Abs.*, 1950–51, **18**, 357.

***Intergranular Energy of Iron and Some Iron Alloys.** (Van Vlack). See col. 100.

***Creep and Stress Rupture [of an Iron–Cobalt–Chromium–Nickel Alloy] as Rate Processes.** Italo S. Servi and N. J. Grant (*J. Inst. Metals*, 1951–52, **80**, (1), 33–37).—Creep data for an Fe–Co–Cr–Ni alloy reported by G. and Bucklin (*Trans. Amer. Soc. Metals*, 1950, **42**, 720; see *Met. Abs.*, 1949–50, **17**, 336) are analysed according to the rate-process theory of plastic flow. The data indicate that the theory can be applied over only a limited range of creep rates. An empirical equation, which relates the applied stress and the temp. to the min. creep rate, is suggested for the analysis of creep data. This equation is valid only in the absence of structural instabilities.—Authors.

***Constitution of Iron–Chromium–Molybdenum Alloys at 1200° F. [650° C.].** Spencer R. Baen and Pol Duwez (*J. Metals*, 1951, **3**, (4), 331–335).—The phase boundaries of the Fe–Cr–Mo system have been determined at 1200° F. (650° C.)

by X-ray-diffraction measurements and, occasionally, microscopic observations, on some 180 ternary alloys, which were prepared by powder-metallurgy methods, and covered all possible ranges of compn. The results are discussed and shown in two diagrams, one with compn. in at.-% and the other in wt.-%. The largest single-phase, solid-soln. region, α, is located near the Cr corner of the diagram, while the brittle σ and ε phases extend over a major portion of the remainder. At 1200° F. up to 8 at.-% Fe is soluble in Mo, and up to 4 at.-% Mo is soluble in Fe; the ε-phase of this binary system is located near the Fe_3Mo_2 compn. and has a rhombohedral structure. 13 ref.—E. N.

***The Dimensional Behaviour of Invar.** B. S. Lement, B. L. Averbach, and Morris Cohen (*Trans. Amer. Soc. Metals*, 1951, **43**, 1072–1097).—See *Met. Abs.*, 1950–51, **18**, 506.

***The Influence of Vibration on the Creep of Lead [and Lead Alloys].** (Greenwood). See col. 100.

***Anomalous Heat Flow in Supraconductors [Lead and a Lead-Bismuth Alloy].** (Mendelssohn and Olsen). See col. 101.

***Evolution of Heat During the Precipitation in a Lead–Tin Alloy.** G. Borelius and K. M. Säfsten (*Arkiv Mat. Astron. Fysik*, 1948, [A], **36**, (5), 5 pp.).—[In English]. Homogenized samples of a Pb–Sn alloy contg. 6 wt.-% Sn were rapidly cooled to just above a temp. $T°$ C. and placed in a precision calorimeter at $T°$ C. This was done for different values of T. By comparing the max. gradient of the rate/time curve taken at diff. temp. the dependence of pptn. rate on temp. was deduced, and found to agree with earlier calculations. By integrating the rate/time curves, the heat of pptn. at different temp. was obtained. This was always lower than the calculated values, the differences, of the order of 100 cal./mole, being attributed to pptn.-induced stresses. This energy compares with that retained in certain metals after heavy cold working.—R. W. C.

***Evolution of Heat During Precipitation in Lead–Tin Alloys.** Josef Nyström (*Arkiv Fysik*, 1949, **1**, (18), 359–367).—[In English]. Homogenized wire samples of Pb–Sn alloys contg. 6–15 wt.-% Sn were allowed to precipitate in a special calorimeter (described) which settles down very quickly and so makes it possible to obtain the earlier parts of the curve of heat evolved against time. From the shape of this it is argued that precipitates at low temp. grow two-dimensionally, but three-dimensionally at higher temp. The analysis of the late portions of the curve often leads to two time const., which are interpreted as corresponding to the formation of Preston–Guinier " knots ", and to pptn. proper resp. The theoretical heat of pptn. at low Sn contents is obtained thermodynamically from the data for Sn–Ag alloys; comparison with observed heat evolutions show that only a fraction of the expected amount of pptn. takes place at lower temp.—R. W. C.

***The Determination of Solidus Temperatures in Magnesium Alloys by Dilatometric Measurements.** Heinrich Adenstedt and Jay R. Burns (*Trans. Amer. Soc. Metals*, 1951, **43**, 873–886; discussion, 887).—See *Met. Abs.*, 1950–51, **18**, 354.

***The Application of Chemical Thermodynamics to the Study of Metallic Alloy Formation of Certain Magnesium–Cadmium Alloys at 25° C.: Progress Report for Oct. 1950–Jan. 1951.** W. E. Wallace and T. M. Buck (*U.S. Atomic Energy Commission Publ.*, 1951, (**NYO-751**), 13 pp.).—The heats of formation at 25° C. of 4 Mg–Cd alloys (25·19, 47·36, 50·50 and 74·73 at.-% Cd) were found to be 775 ± 72, 2197 ± 70, 1960 ± 53, and 1595 ± 60 cal./g.-atom, resp., compared with 950, 1910, 1950, and 1510, resp., obtained by Trumbore from e.m.f. measurements at 270° C. (Thesis, Pittsburgh: **1950**). The difference in the results is explained by the fact that the alloys are ordered at 25° C., but disordered at 270° C. The discrepancy between the above values and those expected from the quasi-chem. theory (Hildebrand and Scott, " The Solubility of Non-Electrolytes." New York; **1950**), which indicates that the heats of formation of $MgCd_3$ and Mg_3Cd should be equal, is discussed.—B. W. M.

***Magnesium–Cerium Wrought Alloys for Elevated-Temperature Service.** K. Grube, J. A. Davis, and L. W. Eastwood (*Proc. Amer. Soc. Test. Mat.*, 1950, **50**, 965–987; discussion, 988).—See *Met. Abs.*, 1950–51, **18**, 21.

***Preparation and Properties of Gadolinium–Magnesium Alloys Rich in Magnesium.** (Mme) Françoise Gaume-Mahn (*Compt. rend.*, 1951, **232**, (20), 1815–1816).—The Gd–Mg diagram has not yet been determined; but, on the basis of Vogel's work on the Ce–Mg and La–Mg alloys, the occurrence of the compounds: $GdMg_9$ (41·7% Gd), $GdMg_3$ (68·2% Gd), $GdMg_2$ (76·3% Gd), GdMg (86·6% Gd), and, perhaps Gd_4Mg (96·3% Gd) may be predicted. G.-M. investigated the presence or absence of these compounds by thermomagnetic methods. The prepn. of the alloys was difficult because the m.p. of Gd (~1200° C.) is higher than the b.p. of Mg at atmospheric pressure (1120° C.), and also because the diffusion of solid Gd into molten Mg is slow owing to the great difference in the sp. gr. of the two metals (Mg 1·74, Gd 7·94). A range of alloys contg. 0–30% Gd was easily obtained, however, by displacing GdF_3 by Mg. Richer Gd alloys were prepared from the 30% alloy by distilling off the Mg in an atmosphere of pure A at ordinary pressure between 1000° and 1100° C. G.-M. found that the Mg-rich alloys contg. 0–65% Gd are purely paramagnetic, and that from 70 to 100% Gd there is a series of alloys presenting very marked ferromagnetism at low temp. She confined her investigations to the magnetic properties of alloys contg. up to 65% Gd. Amongst the results she established the existence of the compound, $GdMg_9$. The compn. at the transition between the purely paramagnetic alloys and those ferromagnetic at low temp. corresponds to the compound $GdMg_3$.—J. H. W.

Magnesium–Zirconium Alloys: Development of Alloying and Fluxing Technique. A. E. Williams (*Metal Treatment*, 1950, **17**, (62), 73–77).—An account of the development of Mg–Zr alloys and a brief survey of their mech. and phys. properties.—J. W. C.

Research on Nickel and Its Alloys. (——). See col. 102.

***Nickel–Aluminium–Molybdenum Alloys for Service at Elevated Temperatures.** H. V. Kinsey and M. T. Stewart (*Trans. Amer. Soc. Metals*, 1951, **43**, 193–219; discussion, 219–225).—See *Met. Abs.*, 1950–51, **18**, 355.

***Effects of Various Elements on the Resistivity of Nickel–Molybdenum–Chromium Alloys to Hydrochloric Acid.** (Endô and Ishihara). See col. 129.

***Preparation of Metals [and Nickel Alloys] in a Finely Divided State for Use as Catalysts.** W. J. C. de Kok and H. I. Waterman (*J. Appl. Chem.*, 1951, **1**, (5), 196–198).—Investigations with the object of obtaining finely divided 80 : 20 Ni–Cr alloy, Monel metal, and steel for use as catalysts in chem. processes are described. The materials, in the form of wire, were atomized by a method similar to that employed in the Schoop pistol, the atomized particles being directed perpendicularly into a non-inflammable liquid. Test results showed that the metals and alloys so prepared were, after activation by treatment with NaClO, effective as catalysts for hydrogenation.—J. R.

***The Coercive [Magnetic] Field of Ferro-Nickel Powders: Effect of Compression and Fritting.** Louis Weil (*J. Phys. Radium*, 1951, **12**, (4), 520–526).—The coercive magnetic field of ferro-nickel agglomerates contg. >50% Ni was studied, more especially in relation to the proportion of voids in the agglomerates, at +300° to −193° C. At ordinary temp. magnetic anisotropy vanishes at a compn. corresponding to ~68% Ni, and magnetostriction disappears at ~81% Ni. Moreover, magnetic anisotropy is nil at all temp. down to −190° C. for the alloy contg. 68% Ni. In agreement with Néel's theory (*Compt. rend.*, 1947, **224**, 1488), aggregates characterized by feeble magnetic anisotropy have a coercive force proportional to the voids, and the limit of the coercive field for isolated grains is proportional to the magnetization. These two simple laws apply only when the agglomerates have been subjected to a preliminary fritting and heating at 300° C. Provided they have not been heated, the coercive field of almost all the agglomerates obeys the laws established by Néel for cases in which anisotropy of form controls magnetic rotation. Anomalies observed in the behaviour of heated agglomerates are explained by assuming the formation of Bloch boundaries at the surfaces of grains in contact, and taking into account Brownian motion thereat. 21 ref.—J. S. G. T.

***The Large Barkhausen Jump and Internal Stress in Perminvar Wire.** Tokutaro Hirone, Shiro Ogawa, and Takasi Huzimura (*Sci. Rep. Research Inst. Tôhoku Univ.*, 1950, [A], **2**, (3), 491–497).—[In English]. Changes of (1) reversible permeability at the remanence point, (2) residual magnetization, and (3) energy of magnetization from the remanence point to magnetic saturation, due to change of external tension are discussed math. and applied to the discussion of experimental results obtained using Perminvar wire. Internal stress in the wire was studied by observing the large Barkhausen jumps accompanying magnetization of the wire. Theory and experimental results agree in indicating that the internal stress in the wire is $\sim$7 kg./mm.^{2}.—J. S. G. T.

***Discontinuous Displacement of Magnetic Domain Boundaries of a Ferromagnetic Substance [Perminvar Wire] Due to Tension.** Tokutaro Hirone and Shiro Ogawa (*Sci. Rep. Research Inst. Tôhoku Univ.*, 1950, [A], **2**, (3), 498–502).—[In English]. Two kinds of domain boundaries exist in ferromagnetic substances, viz. the 180°-boundary and the 90°-boundary, so called because the directions of spontaneous magnetization on both sides of the boundary differ resp. by 180° and 90°. Discontinuous expansion of a magnetic reversal nucleus occurs in a uniformly magnetized Perminvar wire subjected to change of external tension, and is attributable to change of surface energy at the nuclear boundary accompanying change of tension. Calculated and experimental values of the critical minor axis a_s of the nuclear ellipsoid agree very closely, each having very approx. values ranging from 0·08 to 0·11 for values of the external applied tension between 31·61 and 30·45 kg./mm.^{2} at which the large Barkhausen jump occurs. It is concluded that the 180° magnetic domain boundary is displaced discontinuously by change of external tension.—J. S. G. T.

***Preparation of Ag–Mg Alloys in Thin Films by the Simultaneous Evaporation of the Constituents in Vacuum.** Stanislas Goldsztaub and Pierre Michel (*Compt. rend.*, 1951, **232**, (20), 1843–1845).—The two metals were placed in the usual manner in the interior of helices of W wire for Mg and Mo wire for Ag and were heated by an elect. current to a suitable temp. The Ag melted to a globule before vaporizing, while the Mg sublimed. The metallic vapours were condensed on brass grids covered with a film of collodion, and the deposits were studied by electron diffraction. According to the proportion of the constituents the diffraction diagrams showed lines of the different phases already revealed by X-rays, except the γ-phase. To vary the compn. of the deposits it was sufficient to interpose more grids on a convenient surface surrounding the two filaments, so that different alloys could be prepared in one operation. The lines obtained and the corresponding lattice parameters are tabulated for Ag, the f.c.c. α-phase, the b.c.c. β-phase, the hexagonal δ-phase, and Mg. The parameters of the δ-phase are very close to those of pure Mg, as was shown by Ageev and Kuznetsov (*Izvest. Akad. Nauk S.S.S.R.*, **1937**, [Khim.], 289; *Met. Abs.*, 1937, **4**, 613); but the precision of the measurements was not sufficient to reveal the extent of the difference.—J. H. W.

***X-Ray Measurement of Long-Range Order in β-AgZn.** L. Muldawer (*J. Appl. Physics*, 1951, **22**, (5), 663–665).—It is impossible to measure the critical temp. T_c for the order–disorder change in β-AgZn (50 at.-% Zn), because of the ζ-phase stable at lower temp. This was overcome by adding small amounts of Au to the alloy to eliminate the interfering compound, and extrapolating T_c to zero Au content. The result of a (long) extrapolation was $T_c = 272°$ C. The order parameter S was measured as a function of temp., by a Geiger-counter technique, for an alloy with 3·5% Au, assuming $S = 1$ at room temp. The data are reproduced graphically.—R. W. C.

***Titanium–Boron Alloys.** H. R. Ogden and R. I. Jaffee (*J. Metals*, 1951, **3**, (4), 335–336).—The Ti-rich end of the Ti–B system has been studied, using alloys prepared—by arc melting in an atmosphere of A—from iodide-type Ti, and B of high purity produced by the H reduction of BBr_3. The solubility of B in both α and β Ti was found to be very low, probably well below 0·4%, and B has no observable effect on the transition temp. of Ti. The h.c.p. structure of the second phase appears to be of the M_2X type or Ti_2B. The addn. of B increases the strength of Ti by only a moderate amount, but it also decreases the ductility progressively, a type of effect which is expected when a dispersed intermetallic phase is present in the structure. Alloys contg. up to 1% B could be hot rolled at 850° C.; those contg. 2% or more of B could not. 5 ref.—E. N.

***Preparation and Properties of Titanium–Chromium Binary Alloys.** D. J. McPherson and M. G. Fontana (*Trans. Amer. Soc. Metals*, 1951, **43**, 1098–1125).—See *Met. Abs.*, 1950–51, **18**, 509.

***The Uranium–Manganese and Uranium–Copper Alloy Systems.** H. A. Wilhelm and O. N. Carlson (*U.S. Atomic Energy Commission Publ.*, 1949, (**AECD-2717**), 20 pp.).—Cf. *Trans. Amer. Soc. Metals*, 1950, **42**, 1311; *Met. Abs.*, 1950–51, **18**, 27.—B. W. M.

***Studies on the Uranium–Oxygen System.** H. R. Hoekstra and J. J. Katz (*U.S. Atomic Energy Commission Publ.*, 1950, (**AECD-2954**), 7 pp.).—The results of various workers on the U–O system in the range UO_2–UO_3 are reviewed and compared with the phase relationships for the alkaline-earth diuranates. The percentage of solid soln. encountered increased with increase in the molecular weight of the alkaline-earth atom and the diuranate most nearly resembling the U–O system appeared to be the Sr compound.—B. W. M.

Zirconium Production, Properties, and Alloys. (Miller). See col. 104.

***Constitution and Mechanical Properties of Zirconium–Iron Alloys.** E. T. Hayes, A. H. Roberson, and W. L. O'Brien (*Trans. Amer. Soc. Metals*, 1951, **43**, 888–904; discussion, 904–905).—See *Met. Abs.*, 1950–51, **18**, 357.

***The Melting Point/Composition Diagram of the Zirconium–Oxygen System.** Daniel Cubicciotti (*J. Amer. Chem. Soc.*, 1951, **73**, (5), 2032–2033).—A phase diagram of the Zr–O system, reproduced from the results of a study of the m.p. of wires of the oxidized metal, indicates that only two solid phases, namely Zr metal and ZrO_2, are stable near the m.p. The metal phase is capable of dissolving O to form a solid soln. of 55 at.-% O.—J. R.

***The Development of Low-Carbon N-155 Alloy for Gas-Turbine Construction.** W. O. Binder (*J. Iron Steel Inst.*, 1951, **167**, (2), 121–134).—B. reviews the development of low-C N-155 alloy (C 0·12, Ni 20, Cr 20, Co 20, W 2, Nb 1, Mo 3, N 0·12%, balance Fe) and describes the results of a study of the effects of "hot" cold working and heat-treatment on the structure, room-temp. strength, ductility, and impact-resistance of the alloy. The elevated-temp. properties of the material are discussed and the results are given of creep tests extending up to 32,000 hr. in the temp. range 650°–815° C. The strength of the alloy at up to 650° C. may be improved by "hot" cold working, which should preferably be preceded by a soln. anneal at 1060°–1150° C. At temp. $>$650° C. the best properties are obtained by soln. treating at 1090°–1230° C. Some pptn.-hardening occurs at 650°–980° C., and this somewhat increases the room-temp. strength, while decreasing the ductility and toughness; pptn.-treatment improves the hot ductility. The properties of N-155 alloy sheet are shown to be similar to those of bar material. An assessment made of the formability of the sheet showed it to be comparable to the formability of Nb-stabilized 18 : 8 stainless-steel sheet. 19 ref. plus *bibliography*.—R. W. R.

Gas-Turbine Alloys, 10 Years Later. Howard Scott (*Metal Progress*, 1950, **58**, (4), 503–511).—Mainly concerned with ferrous materials; however, operation at 1700° F. (925° C.) calls for new types of alloys, and these may well be based on Mo, Cr, or Ti. Oxidation and hot-working difficulties of Mo and its alloys are discussed.—F. A. F.

***Hot Hardness of Hard-Facing Alloys.** Howard S. Avery (*Weld. J.*, 1950, **29**, (7), 552–577).—The hot hardness of a number of hard-facing alloys has been surveyed, using a Rockwell diamond tester at temp. up to 650° C. Some indication of creep-resistance was obtained by incorporating a time factor in the test. At 535° C. alloys having secondary hardening properties are markedly superior, but at 650° C. creep is prominent, especially in the Fe-base alloys, though less so in the Cr–Ni and Cr–Co alloys. WC inserts provide the highest available hot hardness; composite WC electrodes give deposits with excellent hot hardness, but are sensitive to welding technique. 39 ref.—W. D. B.

***The Mechanism of Plastic Deformation in Homogeneous Metallic Alloys at High Temperatures.** K. A. Osipov (*Izvest. Akad. Nauk S.S.S.R.*, 1949, [Tekhn.], (9), 1372–1377).—[In Russian]. From theoretical considerations and experimental results O. concludes that the increase of inhomogeneity of distribution of alloying elements in solid soln. of metals is one of the most significant factors in plastic deformation. This inhomogeneity is produced by the diffusion of dissolved atoms into preferred planes and in the direction of the applied stress. In the extreme case a new phase may result. It is to be expected that those alloys in which the solubility of the alloying element changes little with rise of temp. will be the most resistant to plastic deformation. The resistance to plastic deformation is proportional to the sp. energy required for the formation of a new phase which can exist at a given temp. according to the phase diagram. For these experiments alloys of Ni–Cr and Fe–Cr–Ni–Mn were used, contg. different % of alloying elements. The specimens were tested by bending at 800° and 1100° C.—W. S.

***The Time Delay for the Initiation of Plastic Deformation at Rapidly Applied Constant Stress.** D. S. Clark and D. S. Wood (*Proc. Amer. Soc. Test. Mat.*, 1949, **49**, 717–735; discussion, 736–737).—The design and construction of a special rapid-load testing machine are described, with which tensile loads may be applied to a specimen within 5 millisec. and upwards. Tests made on an annealed low-C steel are discussed in detail, and it is shown that a definite time delay is required for the initiation of plastic deformation in this material, the time delay depending on the applied stress and varying from 5 millisec. at a stress of 51,000 lb./in.^{2} to 6 sec. at a stress of 37,000 lb./in.^{2}. No such time delay was observed for several materials for which the stress/strain curve exhibits no definite Y.P. These included three alloy steels and 24S-T and 75S-T Al alloys. It is concluded that a definite time delay in the initiation of plastic deformation is associated only with materials for which the static stress/strain curve exhibits a definite Y.P. 19 ref.—P. T. G.

***Interface and Marker Movements in Diffusion in Solid Solutions of Metals.** Luiz C. Corrêa da Silva and Robert F. Mehl (*Trans. Amer. Inst. Min. Met. Eng.* (in *J. Metals*), 1951, **191**, (2), 155–173).—Cf. *ibid.*, 1950, **188**, 1219; *Met. Abs.*, 1950–51, **18**, 510. Double diffusion couples were prepared by sandwiching a disc (1–1$\frac{1}{4}$ in. dia., $\frac{1}{2}$–$\frac{1}{4}$ in. thick) of one material, which had been wound with several turns of marker wire or having two pieces of foil at opposite ends of a dia. on each of its flat surfaces, between two discs of a second material. They were then initially compressed, and welded together under pressure, by heating in H or an inert atmosphere for ~1 hr. at ~600° C. The assemblies studied were: (1) 70 : 30 and 80 : 20 α-brass between Cu, and the reverse, (2) 97 : 3 α-Cu–Al solid soln. between Cu, (3) 90 : 10 α-Cu–Sn between Cu, (4) Ni between Cu, (5) Au between Cu, and (6) Au between Ag. Interface markers of many types were used, including Mo, Ni, Pt, and W wires, Fe, Ni, Pt, and Ta foils, and C, Al_2O_3, and Fe_2O_3 powders. The welded sandwiches were given a diffusion-treatment at 733°–1054° C. for 2–695 hr. in lampblack, H, or N, according to the nature of the sandwich. Four methods were used for the measurement of marker position and marker shift: (*a*) direct method (the most unreliable)—the distance between the wires on each side of the central disc, on each of two opposite planes accurately machined ⊥ to the welding zone and the marker, was measured before and after diffusion; (*b*) diffusion curve method—thin layers were machined off to determine the diffusion/penetration curves, and these served to place the markers upon the curve, for when the markers were encountered during machining they could be easily recognized and thus associated with the compn. of the layer machined at that point; (*c*) foil ref. method—impermeable foils inserted in the original join served as ref. points for other, mobile markers; and (*d*) foil bend method—based upon the new observation that the edge of a foil placed at the join will bend progressively as diffusion occurs around it; the middle portion of the " impermeable " foil remains fixed as long as it is not affected by the diffusion occurring near the edges. The results—which are in agreement with and confirm the earlier observations of Smigelskas and Kirkendall (*ibid.*, 1947, **171**, 130; see *Met. Abs.*, 1947, **14**, 94) showed that: (1) all markers, whatever their compn., i.e. whether soluble (Fe, Ni, Pt) or insoluble (Mo, W), moved or bent by exactly the same amount in any given system, thus showing that the phenomenon is not a property of the marker; (2) the direction of marker movement is, (*a*) away from the Cu towards the Cu alloy, i.e. towards the brass, Sn bronze, or Al bronze, (*b*) towards the Cu in the Cu/Ni system, (*c*) towards the Au in the Cu/Au system, and (*d*) towards the Ag in the Ag/Au system; (3) the amount of marker shift varies from system to system; it is large in the Cu/α-brass and Cu/Ni systems, and smaller in the others; it is not apparently directly related to diffusion coeff.; (4) the dimensions of the markers do not influence the amounts by which they move; and (5) Cu/α-brass couples exhibit apparent traces of porosity in the diffusion zone—and anomalously low hardness there—while no such porosity could be found in other systems. It is considered that only the vacancy mechanism of diffusion appears to be useful in explaining these phenomena, though several theoretical points remain to be solved. Inasmuch as the amount of shift is independent of the nature of the external surface of the diffusion couple, and of the dimensions of the couple, it appears that operating vacancies cannot come from external surfaces but must be generated within the diffusion zone itself. A dislocation mechanism is proposed to explain the marker shift. 19 ref.—E. N.

***On the Theory of the Electrical Resistance of Metallic Solid Solutions.** J. O. Linde (*Arkiv Mat. Astron. Fysik*, 1948, [A], **36**, (10), 22 pp.).—[In English]. A treatment, for alloys between the Cu sub-group and either *B* sub-group or transition metals, of the atomic increase of resistivity, and of the pressure and temp. coeff. of resistivity. The basic assumption of the treatment is that with *B* sub-group solutes, the conduction electrons are liable mostly to " ordinary " scattering (i.e. decreasing scattering with increasing electron velocity); and that with transition-metal solutes, the scattering is mostly " extraordinary " and has the reverse dependence on electron velocity. The theory is worked out on semi-classical lines. The expression for the atomic resistivity increase is identical with that derived by wave mechanics. From a comparison between theory and experimental values of the pressure coeff., a new expression is suggested for the electron velocity at the surface of the Fermi distribution as a function of atomic vol.—R. W. C.

***The Saturation Magneto-Resistance of Ferromagnetic Alloys.** R. Parker (*Proc. Phys. Soc.*, 1951, [A], **64**, (5), 447–452).—It is suggested that the magneto-resistance coeff. of a ferromagnetic alloy is composed of two terms, one associated with the temp.-dependent contribution to the elect. resistance, (thermal vibrations), the other, with the temp.-independent contribution (in this case lattice aperiodicities due to solute atoms). An equation for the coeff. in terms of temp. and solute content is derived on this hypothesis. Good agreement with experiment is found for Fe–Si alloys. —R. W. C.

Intermittent Progressive Activation in Some Series of Ferromagnetic Alloys. R. Forrer (*J. Phys. Radium*, 1951, **12**, (4), 31S).—F. differentiates between intermittent activation towards the resp. states 4*s* and 4*p*, of which the latter permits the possibility of effecting interactions producing ferromagnetism. An experimental direct verification of intermittent activation towards the 4*p* state appears to be, at present, impossible, but the possibility of verifying F.'s contention indirectly appears to be afforded by the alloys γ-Fe–Ni, γ-Pt–Co, Pt–Cr, and Pt–Mn.—J. S. G. T.

Investigation of Alloy Systems : Trends in Present-Day Thought. —— (*Metal Treatment*, 1951, **17**, (61), 42–46).—A brief report of a symposium organized by the Birmingham Local Section of the Institute of Metals. See abstracts below.—J. W. C.

Phase Diagrams. E. H. Bucknall and P. Hersch (*Metal Ind.*, 1951, **78**, (22), 443–446).—Contribution to a Symposium on the Investigation of Alloy Systems, organized by the Birmingham Local Section of the Institute of Metals. B. and H. describe the construction of phase diagrams for 1- and 2-component systems. In 2- to 4-component systems it is convenient to make the diagrams isobaric, so that they may be represented in two dimensions; but the pressure should not be neglected when any of the components is volatile. B. and H. discuss the appn. of phase diagrams to distillation and refining and to the dependence of creep-resistance on the constitution. 15 ref.—J. H. W.

Thermal Analysis. C. Sykes (*Metal Ind.*, 1951, **78**, (19), 379–381; (20), 409).—Contribution to a Symposium on the Investigation of Alloy Systems, organized by the Birmingham Local Section of the Institute of Metals. S. explains how thermal effects can be used to detect changes in the state of metals and alloys and considers the two cases: (*a*) when latent heat is evolved, and (*b*) when the sp. heat increases substantially at the transformation temp. A change of state should not be assumed on thermal evidence alone, since there may be hysteresis or the presence of a metastable state. He discusses the usual methods of plotting thermal arrests and describes an apparatus for accurately measuring the sp. heat. 7 ref.—J. H. W.

The Approach of Alloys to Equilibrium. D. McLean (*Metal Ind.*, 1951, **78**, (20), 399–402).—Contribution to a Symposium on the Investigation of Alloy Systems, organized by the Birmingham Local Section of the Institute of Metals. The approach of alloys to equilibrium involves the appearance and disappearance of phases that exist under certain conditions, and is, therefore, intimately associated with pptn. and re-soln. Reactions in metals often proceed by the formation and growth of nuclei, and factors that influence nucleation and growth rate are diffusion, statistical fluctuation, and binding energy. McL. discusses the effects of strain-induced segregation and the critical nucleus size, and illustrates the discussion by the case of the tempering of martensite. 9 ref.—J. H. W.

Practical Thermodynamics. G. V. Raynor (*Metal Ind.*, 1951, **78**, (21), 419–421, 429).—Contribution to a Symposium on the Investigation of Alloy Systems, organized by the Birmingham Local Section of the Institute of Metals. The practical investigation of thermodynamic quantities is of considerable importance in the development of the alloy theory, and much quant. information can be derived from it. R. gives examples of the calculations involved and describes methods of determining the relation of the activity curves to the equilibrium diagram.—J. H. W.

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3 — STRUCTURE

(Metallography ; Macrography ; Crystal Structure.)

[For all abstracts on the constitution of alloy systems, including X-ray studies, see 2—Properties of Alloys.]

***Recovery and Recrystallization in Highly-Stressed [Compressed] Aluminium.** E. A. Owen and Y. H. Liu (*Proc. Phys. Soc.*, 1951, [B], **64**, (5), 386–396).—A precision back-reflection method was used to measure the lattice parameters for several planes of Al specimens as a function of time at room temp. after heavy compression, and of the angle between the X-ray beam and the direction of stress. The parameter in all cases approached a metastable value representing a strain of $\sim 13{\cdot}7 \times 10^{-5}$ with respect to the equilibrium value. The strain/time curve depended upon the angle of incidence, as did the initial value of the strain; but there was no variation in either for different planes with a given angle of incidence. Partial recrystn. took place, as evidenced by sharp diffraction spots; these grew to a certain size, then stopped. Some speculations on the dislocation mechanisms involved are included in the discussion. —R. W. C.

***Effect of Strain Rate on the Plastic Deformation of Aluminium Single Crystals.** Hideo Nishimura and Jin-ichi Takamura (*Mem. Fac. Eng. Kyoto Univ.*, 1950, **13**, (1), 1–20).—[In English]. The fine structures of slip bands produced in electrolytically-polished Al single crystals subjected to various rates of tensile water-loading stress at room temp. were studied by the electron microscope and X-rays. Crystals subjected to a slow rate of strain generally show wider slip bands, wider spacings between the slip bands, and a larger number of sets of active gliding planes than do crystals subjected to rapid strain. Slowly-deformed crystals generally give rise to marked X-ray asterism and plastic flow. Sub-microscopic slip takes place between the obvious slip bands. Notch lines on the crystal surface affect the behaviour of the slip bands. Thus, a line marked on the crystal surface $\parallel$ the slip bands tends to hold up propagation of slip, so that slip bands are not observed near the line nor in the corresponding region on the back surface of the crystal. 18 ref.—J. S. G. T.

***Particular Aspects of Slipping Produced by Alternating Stress in Annealed Aluminium Crystals.** Paul Henri Laurent (*Rev. Mét.*, 1951, **48**, (6), 441–446; discussion, 446).—By means of alternating torsion L. obtained slip bands different from those usually observed in tension, and their explanation is not obvious. They fell into three classes: (1) two very close and regular crossed networks. The first explanation that the equidistance of the slip bands is a result of static phenomena produced in metal submitted to vibration is not satisfactory; the appearance is probably due to the characteristic texture of the grain examined, the criss-crossing appearing under the microscope to be not perfectly regular over the whole surface of the grain. (2) Slip bands in chevron form. Many explanations are possible for this, such as: (*a*) the stress applied is not the cause of this appearance; (*b*) the regular arrangement is due to a particular orientation of the direction of slip compared with those of max. stress; but neither of these suffices; (*c*) the crystal texture is not homogeneous throughout the grain, and it is the sub-structure of the crystal that is shown up by the periodic development of the slip bands. (3) Grouping of the slipping in large-spaced bands. As in case (2) there are two directions of slip, but here the slip bands occur in only one of these directions. It will be seen from these examples that there is probably a very marked periodicity of the cryst. texture in the grain. 7 ref.—J. H. W.

***The Relations Between Deformation and Recrystallization of Aluminium Single Crystals.** (Mlle) A. Lalœuf and Ch. Crussard (*Rev. Mét.*, 1951, **48**, (6), 462–469; discussion, 470).—In general there is a more or less close relationship between the orientations of a deformed crystal; and this is undoubtedly true for severe deformation, but less certain for deformation just above critical work-hardening. L. and C. carried out experiments on 99·99% Al, with some tests on 99·996% Al by way of comparison, which showed that the relation held good in the latter case. Single-crystal specimens were drawn to 15–25% in a Chevenard micro-machine, and the stress/elongation curve was photographically recorded. In certain cases, the test was interrupted from time to time and the specimen examined by X-rays for asterism. The deformation of Al single crystals differs according as the crystal is oriented so as to show a single system or two systems of slip. Kinked bands appear in the first but not in the second case. Consolidation or work-hardening and asterism show corresponding variations, differing from one type to another. It may be concluded from this that the rotation of parts of the crystal constitutes an essential factor in work-hardening. L. and C. discuss the results of their work in further detail. 14 ref.—J. H. W.

***[I] Solidification Textures of Aluminium and Light Alloys Produced by Immersion in Water [Water-Bath Quenching]. [II] Electron-Microscope Study of Age-Hardened Aluminium Alloys.** Hideo Nishimura and Tomihiro Toyohara (*Mem. Fac. Eng. Kyoto Univ.*, 1950, **12**, (4), 41–46, 47–52).—[I.—]. [In French]. The effect of slow immersion of molten Al or Al alloys contained in a soft-steel crucible upon the texture of the metal or alloy was studied, the effects of the temp. of the fused mass and the speed of quenching being, more especially, investigated. The macrostructure of quenched Al ingots is found to depend considerably upon the speed of quenching: at a certain quenching speed, columnar crystals are produced along the length of the ingot. When the quenching is slow or fast, i.e. when cooling is effected in a furnace or in water, equi-axed crystals are formed. Corresponding to intermediate conditions, mixtures of equi-axed and columnar crystals are formed. Metal which has been heated above 750° C. always shows a columnar macrostructure, whatever the speed of quenching. Metal heated to 670° C. gives ingots having slightly elongated equi-axed cryst. structures. Quenching from between these two temp. gives either an equi-axed or columnar structure, according to the speed of quenching. Addn. of Cu to the metal produces grain-refining whether the crystals are equi-axed or columnar. A study of the water-quenching of 13 Al alloys contg. 1·18–41·20% Cu, heated to 20°, 60°, 140°, and 240° C. above the resp. liquidus temp. shows that alloys contg. little Cu, and the eutectic alloy have a tendency, on quenching, to develop a columnar macrostructure. The velocity of crystn. depends on the proportion of eutectic present in the alloy, and the resp. alloys show corresponding tendencies to the development of equi-axed or columnar macrostructures. Hypereutectic alloys show a tendency to develop the columnar macrostructure owing to the influence of $CuAl_2$. The alloy contg. 30% Cu shows superposed layers of columnar and equi-axed macrostructures due to gravitational segregation. [II.—]. [In English]. Pptn. in age-hardened Al alloys is first observable in electron micrographs when the particles are of dimensions of the order 100 Å. Coherent transition structures can be clearly recognized during their growth. These structures comprise one in which a string structure is well developed, and another showing a well-developed plate structure. The former occurs in the Al–Zn–Mg and Al–Mg–Si systems, the latter in the Al–Cu and Al–Cu–Mg systems. The characteristics depend upon the lattice structures of the precipitates.
—J. S. G. T.

***Fractographic Registrations of Fatigue [in 75S Aluminium Alloy and Steel].** C. A. Zapffe and C. O. Worden (*Trans. Amer. Soc. Metals*, 1951, **43**, 958–969; discussion, 969).—See *Met. Abs.*, 1950–51, **18**, 359.

***A New Anodic-Film Method for Studying Orientation in Aluminium.** André Hone and E. C. Pearson (*Metal Progress*, 1950, **58**, (5), 713–715).—A modified anodizing electrolyte is given, differing from that quoted by H. and P. in an earlier paper (*ibid.*, 1948, **53**, 363; *Met. Abs.*, 1948–49, **16**, 60). The new electrolyte consists of: H_3PO_4 (85%) 800 ml., diethylene glycol monoethyl ether (carbitol) 1660 ml., HF (48%) 320 ml., H_3BO_3 (anhydrous) 128 g., oxalic acid (crystals) 72 g., distilled H_2O 1012 ml. Anodizing at 35° ± 1° C., with ~40 V. and a c.d. of 5 down to ½ amp./in.^{2} gives a definite depth of coating and a clear striated structure with super- and commercial-purity Al. The film can be examined for information similar to that provided by macro-etching, and is useful in indicating the progress of recrystn.—F. A. F.

***The Crystal Structures of CeB_4, ThB_4, and UB_4.** A. Zalkin and D. H. Templeton (*U.S. Atomic Energy Commission Publ.*, 1950, (**AECD-2762**), 2 pp.).—These borides crystallize with a tetragonal lattice of space-group $D_{4h}^5 - P4/mbm$. The cell const. are as follows:

Compound	CeB_4	ThB_4	UB_4
a, Å.	7·205 ± 0·008	7·256 ± 0·004	7·075 ± 0·004
c, Å.	4·090 ± 0·005	4·113 ± 0·002	3·979 ± 0·002
X-ray *d*, g./c.c.	5·74	8·45	9·38

The metal atoms are in planes, each atom having 5 nearly equidistant neighbours in the same plane and 2 in adjacent planes at a greater distance. The holes between the layers are filled with B atoms to give the compn. XB_4, and there are three types of positions. Atoms of the first type are in holes bounded by 6 metal atoms at the corners of a triangular prism as in the case of the compounds XB_2, where X is Al, Zr, Ti, Cr, Nb, V, or U. The B atoms of the other two types are grouped together as octahedra in holes bounded by metals at the corners of sq. prisms as in the compounds CaB_6, CeB_6, ThB_6, &c., in which a simple cubic array of metal atoms is interpenetrated by B octahedra. Thus the XB_4 structure can be considered as a combination of the MB_2 and MB_6 structures.—B. W. M.

The Borides of Some Transition Elements. Roland Kiessling (*J. Electrochem. Soc.*, 1951, **98**, (4), 166–170).—Previous work (*Acta Chem. Scand.*, 1950, **4**, 209; *Met. Abs.*, 1950–51, **18**, 460) on the crystal structure of the borides of the transition elements is summarized, and it is shown that borides may be classified according to the B arrangement. The phases are regarded as metal lattices with B atoms in the interstices. The B atoms form an increasing number of B–B contacts with increasing B content of the intermediary phases of a system, from isolated B atoms to a three-dimensional B network. It is shown that the borides resemble in many ways the hydrides, nitrides, and carbides, though differing from them by virtue of the presence of B–B bonds. It was not found possible to apply Pauling's theory of the metallic state to these compounds. 24 ref.
—G. T. C.

***Deformation and Recrystallization Texture of Cold-Drawn O.F.H.C. Copper Wire.** W. A. Backofen (*J. Metals*, 1951, **3**, (3), 250).—Using the conventional X-ray transmission diffraction technique, the deformation texture of cold-drawn O.F.H.C. copper wire, after a reduction in area of 97·3%, was found to be a composite of [111] and [100] directions—the former more predominant than the latter—aligned || the wire axis. Recrystn. of such wire at 300°–450° C. was accompanied by practically complete elimination of the [111] component of the deformation texture, and the final structure consisted almost entirely of a [100] alignment. 4 ref.—E. N.

***Branching of Slip Lines in Alpha-Brass.** Harry Czyzewski (*J. Appl. Physics*, 1951, **22**, (6), 846).—A letter. Electron-microscopic evidence is presented to show that: (1) slip may occur along a twin interface, and (2) the slip may branch at crystallographic discontinuities, such as annealing twin interfaces. The processes described took place on a very fine scale and were caused by abrasion with a diamond point.—R. W. C.

***Microscopic Observation of the Solidification of Cu–Ni Alloy Droplets.** R. E. Cech and D. Turnbull (*J. Metals*, 1951, **3**, (3), 242–243).—Using a high-temp. microscopic technique, a study was made of the solidification behaviour, in a H atmosphere, of droplets of Cu–Ni alloys as a function of compn. (0–100% Ni). The ratio of the amount of supercooling $(\Delta T-)_{\max.}$ to the abs. liquidus temp. (T_1 °K.) was found to be 0·18 ± 0·01, which checked closely with the value of 0·18 ± 0·02 for the ratio of $(\Delta T-)_{\max.}$ to the abs. m.p. found for pure metals. The degree of segregation exhibited by cross-sections of super-cooled particles indicated that the heat of fusion was probably sufficient to raise the droplets to the equilibrium temp., and allow a portion of each particle to solidify by normal dendritic growth. The temp. dependence of the nucleation rate was about as large as for pure metals. 5 ref.—E. N.

***Precipitation and Diffuse Scattering in an Fe–[17%] Mo–[12%] Co [Permanent Magnet] Alloy.** A. M. Geisler and F. E. Steigert (*J. Metals*, 1951, **3**, (3), 259–260).—A report of X-ray observations on the orientation relationships of the various lattice structures which occur during the pptn. reactions in this alloy.—E. N.

***Some Notes on the "Barley Shell" Structure in Iron–Silicon Alloys.** R. V. Riley (*J. Metals*, 1951, **3**, (5), 412).—With ref. to the paper by Wachtell (*Trans. Amer. Inst. Min. Met. Eng.*, 1950, **188**, 354; *Met. Abs.*, 1950–51, **18**, 263) it is pointed out that although the HF in the etchant is primarily responsible for the "barley shells," silica or its compounds must also be present either in the etching reagent or in the specimen. Thus even pure electrolytic Fe may be etched in the reagent to give the barley shell structure provided the etchant has been either: (1) stored in contact with glass or silica, or (2) used previously for etching an Fe-contg. Si. Therefore, although W.'s method of utilizing the etch is novel, and in the hands of the investigator gave a useful differentiation between the high- and low-Si areas in the specimens, the technique cannot be regarded as of universal appn. Fresh, pure reagents only must be used each time, and the greatest care must be exercised in the interpretation of the results, which may be regarded as qual. only and may, in fact, not be sp. to Si alloys. 7 ref. —E. N.

***[Effect of] Roller Levelling [on the Structure] of Magnesium Alloy Sheet.** E. A. Calnan and A. E. L. Tate (*J. Inst. Metals*, 1951, **79**, (6), 455–464).—It has been widely suggested that the loss of P.S. in Mg alloy sheet after roller levelling is due to the untwinning of material twinned by bending in the roller-levelling process. In the present work, X-ray-diffraction evidence of additional twinned material in roller-levelled sheet has been found. It has been deduced that the loss in P.S. will be avoided by levelling either under tension in the levelling direction or by compression normal to the sheet and that the loss will be reduced by roller levelling in the $[10\bar{1}0]$ direction when this has a sp. direction in the sheet. From an investigation of the influence of previous heat-treatments and deformation it has been found that previous cold working reduces the loss in P.S. on roller levelling.—AUTHORS.

***The Crystal Structure of PuN and PuC.** W. H. Zachariasen (*U.S. Atomic Energy Commission Publ.*, 1948, (**AECD-2195**), 2 pp.).—PuN and PuC have the NaCl-type of structure and are isomorphous with PuO ($a = 4{\cdot}948 \pm 0{\cdot}002$ Å.). The lattice const. for PuN and PuC are $4{\cdot}895 \pm 0{\cdot}001$ and $4{\cdot}910 \pm 0{\cdot}005$ Å. resp. and the calculated d $14{\cdot}23 \pm 0{\cdot}01$ and $13{\cdot}99 \pm 0{\cdot}05$ g./c.c. X-ray-diffraction data are included. —B. W. M.

***Crystallization of Supercooled Liquid Selenium.** G. Borelius *et al.* (*Arkiv Fysik*, 1949, **1**, (13), 305–322).—[In English]. The kinetics of the rate of advance of the crystn. front of supercooled pure Se were studied experimentally, as was the appearance of the solid phase. It is concluded that the growth rate is a marked min. || the hexad axis. The variation with temp. of the crystn. rate is discussed in terms of theory.—R. W. C.

***The Crystal Structures of ThH_2 and ZrH_2.** R. E. Rundle, A. S. Wilson, R. Nottorf, and R. F. Raeuchle (*U.S. Atomic Energy Commission Publ.*, 1948, (**AECD-2120**), 5 pp.).—The strong reflections from ThH_2 correspond to a body-centred tetragonal lattice with $a = 4{\cdot}09 \pm 0{\cdot}03$ and $c = 5{\cdot}02 \pm 0{\cdot}03$ kX, two mol./unit cell and $d = 9{\cdot}20$ g./c.c. A few weak lines indicate that this is probably a pseudo-unit. ThH_2 is pseudo-isomorphous with ZrH_2, ZrC_2, and ThC_2. ZrH_2 gave no reflections which were not expected on the basis of a body-centred tetragonal lattice with $a = 3{\cdot}513 \pm 0{\cdot}003$ and $c = 4{\cdot}441 \pm 0{\cdot}003$ kX. Hence ZrH_2 and ThH_2 may not be completely isomorphous, and since ThC_2 produces extra reflections which do not correlate with those from ThH_2, these may not be completely isomorphous. The possible H positions in the hydrides are discussed, and it seems probable that the structures contain M–H–M bridges. The bond distances for this structure are in fair agreement with predictions from Pauling's rule, with the exception of rather long Th–Th distances in ThH_2.—B. W. M.

***Metallography of Electro-Tinplate.** J. E. Davies and W. E. Hoare (*J. Iron Steel Inst.*, 1951, **168**, (2), 134–140).—The metallography of flow-brightened electro-tinplate samples from 7 different sources was studied by (*a*) micro-examination of oblique-mounted specimens and (*b*) macro- and micro-examination of specimens etched with alcoholic $FeCl_3$ or anodically de-tinned in 5 or 10% NaOH soln. The oblique-mounted specimens were prepared in the following way. The specimen is first plated with 0·025 in. Fe or Cu to provide edge support and is then mounted in a specially shaped Bakelite block. The specimen is ground on coarse emery paper in the usual way, and final grinding is carried out on emery paper or lead impregnated with carborundum powder. Polishing is done on Selvyt cloths impregnated with diamond dust (2 grades are used), and a final polish is given on Selvyt impregnated with alumina. The examinations showed the presence of a layer of $FeSn_2$ in all the specimens. A study of the commercial specimens and of others prepared in the laboratory revealed that the rate of formation of the alloy layer depends on the orientation of the underlying ferrite grains. The structure of the layer depends on the temp. reached during flow-brightening and on the time elapsing between fusion of the coating and quenching. When the flowing temp. was $<250°$ C. the $FeSn_2$ layer was characterless; in the range 260°–320° C. a duplex structure was produced which developed into a nodular kind of structure if the fusion time was long. It is estimated that the temp. attained at the reacting interfaces during flow-brightening may be as high as 300° C. Numerous photomicrographs illustrate the structures found. 19 ref. —R. W. R.

***Eutectic Solidification in Metals [Tin-Lead Alloys].** W. C. Winegard, S. Majka, B. M. Thall, and B. Chalmers (*Canad. J. Chem.*, 1951, **29**, (4), 320–327).—Previous theories of the solidification of eutectics are reviewed, and experimental work on the mechanism of freezing near-eutectic Sn–Pb alloys contg. 30 and 45% Pb is described. Specimens were made under conditions which enabled the speed and direction of solidification to be controlled, the graphite-boat and movable-furnace technique developed by C. being used (*Proc. Roy. Soc.*, 1940, [A], **175**, 100; *Met. Abs.*, 1940, **7**, 277). The macrostructures of the specimens were examined, and the microstructures were studied with the ordinary microscope, the phase-contrast microscope, and the electron microscope. It is shown that freezing takes place by the simultaneous edgewise growth of the eutectic lamellæ, whose thicknesses depend on the rate of solidification. Experiments in which specimens were half-solidified and the residual liquid then quickly drained away, revealed that the solidification front is corrugated, the Sn-rich lamellæ leading the Pb-rich. Measurements made with the phase-contrast microscope showed that in one instance the depth of the corrugations was 0·2–0·5 μ, the lateral spacing of the lamellæ being ~2 μ. The fact that one phase leads the other during solidification is attributed to differences in thermal conductivity of the two phases. A detailed mechanism for the solidification of eutectics in general is proposed.—R. W. R.

***An X-Ray Study of Tin–Nickel Electrodeposits.** H. P. Rooksby (*Electrodepositors' Tech. Soc., Advance Copy*, 1951, **27**, (5), 8 pp.).—The crystal structure and textural properties of Sn–Ni deposits were studied. The deposits are characterized by a metastable NiAs-type structure similar to that of γ'-Ni_3Sn_2. The hexagonal cell const. ($a_0 = 4{\cdot}15$, $c_0 = 5{\cdot}10$ Å., $c/a = 1{\cdot}23$) differ from the established values for Ni_3Sn_2, and the reason for this is considered to be the change in compn. of Ni_3Sn_2 towards NiSn which is induced by the conditions of deposition. The alloy deposit is stable up to 300° C., but decomposes at higher temp. A strongly marked preferred orientation is found in the bright deposits, comparable with that in speculum deposits. 9 ref.—G. T. C.

***The Structure of $TiBe_{12}$.** R. F. Raeuchle and R. E. Rundle (*U.S. Atomic Energy Commission Publ.*, 1951, (**ISC-125**), 39 pp.).—The structure is hexagonal with $a_0 = 29{\cdot}44 \pm$

0·01, $c_0 = 7{\cdot}33 \pm 0{\cdot}01$ Å., and 48 mol./unit cell. The even-layer Weissenberg and precession diagrams, considered independently of the odd layer, may be indexed on the basis of a smaller hexagonal pseudo-cell, the c_0 axis being identical with the c_0 axis of the true cell and the a_0 axis = 4·23 Å. The pseudo-cell has one mol. with the Ti atom alternating between the 000 and $00\frac{1}{2}$ positions. The structure is disordered, and though the general reason for disorder is apparent, the particular type of disorder observed has not been explained. The pseudo-cell structure indicates that there are linear Be–Ti–Be groups arranged ‖ c_0 with a Be–Ti distance of 2·13 Å. About each group there are 18 Be atoms arranged hexagonally in 3 layers, with 6 at the level of Ti and 6 at approx. the level of the 2 Be atoms. The Be atoms in the $TiBe_2$ groups form 2 types of hexagonal nets, one at $z = 0$ and $\frac{1}{2}$ consisting of hexagons with shared edges, and the other at $z = \frac{1}{4}$ and $\frac{3}{4}$, consisting of smaller hexagons with shared corners. Centres of the hexagons coincide in the (001) plane. The $TiBe_2$ groups are placed within these hexagons, so that a Ti atom occurs at the centre at $z = 0$ or $\frac{1}{2}$ and Be atoms occur near $z = \frac{1}{4}$ or $\frac{3}{4}$. The fit is apparently close enough to ensure that if a Ti atom is at $z = 0$ in the pseudo-cell, it lowers the probability that neighbouring Ti atoms will be at $z = 0$.—B. W. M.

The Electrolytic Polishing and Etching of Uranium. B. W. Mott and H. R. Haines (*Metallurgia*, 1951, **43**, (259), 255–257).—A variety of soln. for electrolytically polishing and etching U are quoted, and the methods for their use described. The advantages and limitations are indicated.—F. M. L.

***The Study of Recrystallization in Zinc by Direct Observation.** G. Brinson and A. J. W. Moore (*J. Inst. Metals*, 1951, **79**, (6), 429–438).—Specimens of deformed Zn have been observed under a polarizing microscope while being heated. The changes in the crystal structure during recrystn. were continuously observed and photographed. B. and M. show that the growth of new crystals is very erratic, and although grain and twin boundaries, inclusions, and local inhomogeneities sometimes markedly affect growth, at other times they have no influence on it. This erratic behaviour implies that a small crystal will not grow uniformly when it is of about the same size as the matrix crystals. The relevance of this to the conclusions reached by previous workers is discussed. The method of observation can be applied to any non-cubic metal that will undergo structural alterations at a temp. at which it can be observed by means of a microscope. 11 ref.—AUTHORS.

Metallography of Zirconium. H. P. Roth (*Metal Progress*, 1950, **58**, (5), 709–711).—Metallic Zr is ground with emery paper to 3/0 fineness and then electrolytically polished. The electrolyte used consists of one part 60% $HClO_4$ to 10 parts glacial acetic acid, the cathode is stainless steel, the voltage 12 to 18, the current taken being 0·02–0·5 amp. Polishing is accomplished in ~45 sec. 15 photomicrographs are reproduced; chem. etching is not necessary after electropolishing.—F. A. F.

***Preferred Orientation in Zirconium.** R. K. McGeary and B. Lustman (*U.S. Atomic Energy Commission Publ.*, 1950, (**AECD-2951**), 43 pp.).—The preferred orientation in Zr was investigated after deformation by various types of cold rolling, cross rolling, and hot rolling, both before and after recrystn. above and below the transformation temp. A crystal bar produced by thermal decompn. of the iodide behaved similarly to a Zr ingot from the Bureau of Mines in the form of sponge melted in a graphite crucible. The results obtained from transmission Laue photographs are presented as pole figures which show that the textures obtained are fundamentally different from those given by other h.c.p. metals of similar axial ratio, viz. Mg and Be. The cold-rolled texture indicated an ideal orientation with the basal plane ‖ the rolling direction, but inclined from the rolling plane of the sheet ~30° in the transverse direction, with a $[10\bar{1}0]$ direction in the rolling direction. After recrystn. of this material, the average position of the basal planes was as before, but a $[11\bar{2}0]$ direction was in the rolling direction. In this case, twinning on first-order pyramidal planes $\{10\bar{1}1\}$ represented the recrystn. pole figures. The hot-rolled texture had a preferred orientation similar to that of cold-worked material, but with a greater degree of scatter, especially in the transverse direction. A specimen alternatively cross rolled and straight rolled at room temp. had a sharp orientation texture with the basal planes ‖ the rolling plane, the $[10\bar{1}0]$ direction in one rolling direction and the $[11\bar{2}0]$ direction ⊥ this. After recrystn. the $[11\bar{2}0]$ direction was in the $[10\bar{1}0]$ rolling direction and the $[10\bar{1}0]$ direction in the $[11\bar{2}0]$ rolling direction. For the cold-rolled material, the orientation of the surface layers was of the same type as that of the rest, and was more marked. Evidence was obtained to show that the transformation from the b.c.c. form to the h.c.p. form is reversible, the equivalent planes being $\beta\{110\}$ and $\alpha\{0001\}$ and equivalent directions, $\beta[111]$ and $\alpha[11\bar{2}0]$.—B. W. M.

Report of [A.S.T.M.] Committee E-4 on Metallography. —— (*Proc. Amer. Soc. Test. Mat.*, 1950, **50**, 440–443).—See *Met. Abs.*, 1950–51, **18**, 179.

***Attack-Polish Method of Metallographic Preparation.** D. Boyd-Metz and H. W. Woods (*U.S. Atomic Energy Commission Publ.*, 1950, (**SEP-42**), 14 pp.).—Electrolytic polishing is unsuitable for specimens with voids and very fine wires and since ordinary mech. polishing works the surface, B.-M. and W. describe a method in which the polishing powder is suspended in a dil. etching reagent. For U, final polishing is carried out on a wax-covered bronze disc, using silk and alumina in a soln. contg. HF and HNO_3. This results in a surface suitable for examination of the grain structure under polarized light. Be can be polished in the same way as U, using a dil. soln. of oxalic acid, and Zr in dil. HF. The method is very quick and gives excellent definition of voids and inclusions, which is illustrated.—B. W. M.

Metallography in Industry and Research. June A. McNicol (*Australasian Eng.*, **1951**, (Jan.), 73–78).—The various methods used for examining metals are discussed under the following headings: direct visual examination, opt. microscopy, X-ray diffraction, radiography, electron microscopy, and electron diffraction. 7 ref.—T. A. H.

Phase-Contrast Metallography. R. L. Seidenberg and J. R. Benford (*Metal Progress*, 1950, **58**, (5), 725–728).—A phase-contrast system suitable for metallography developed by the Bausch and Lomb Optical Co. is described. It permits metallographic microscopes to be converted into phase-contrast microscopes by addn. of an annular stop in the illuminating system and a phase-retarding annulus located in the image of the annular stop.—F. A. F.

Further Development of Metallographic Grain-Size Measurement. Cord Petersen (*Metall*, 1951, **5**, (1/2), 8–13).—Various methods of determining grain-sizes by comparison with standard networks are critically reviewed, and their sources of error are discussed. 15 ref.—E. N.

A Bridge Method for the Control of Electrolytic Polishing. (Bates and Mee). See col. 147.

***Structure of Permanent-Magnet Alloys.** A. H. Geisler (*Trans. Amer. Soc. Metals*, 1951, **43**, 70–101; discussion, 101–104).—See *Met. Abs.*, 1950–51, **18**, 365.

***Colloidal Phenomena in Metals. XI.—On the Macromolecular and Micellar Structure of Metals.** Yu. A. Klyachko (*Zhur. Priklad. Khim.*, 1949, **22**, (5), 455–466).—[In Russian]. Cf. *ibid.*, 1941, **14**, 342; *Met. Abs.*, 1943, **10**, 237. K. discusses the evidence for the existence of "macromolecules" in metals, the nature of the various types of forces between these macromolecules, the relation between the compressibility of a metal and its photoelect. work-function, and the mechanism of conduction of electricity through metals. The size of the temp. interval between m.p. and b.p. is a criterion for the plasticity of a metal. Departure from Dulong and Petit's law at high temp. is due to the macromolecular and micellar structure.—G. V. E. T.

†Work-Hardening Under Combined Stresses. L. R. Jackson (*Cold Working of Metals* (*Amer. Soc. Metals*), **1949**, 210–222).—The plastic stress/strain curve of a polycryst. metal can probably be described by concepts of shear-strain

energy, whatever is the stress system, provided that anisotropy is taken into account. The elastic moduli of most metal crystals vary very greatly along the various crystal axes. The effect of hydrostatic tension (produced by notches) is discussed at length, and it is shown that while the shape of the work-hardening curve is predictable from considerations of shear-strain energy, the termination of the curve depends on the hydrostatic stresses present.
—J. C. C.

***Plastic After-Effects.** J. G. Leschen (*Cold Working of Metals* (*Amer. Soc. Metals*), **1949**, 197–209).—When a slip-band is propagated in one crystal of a bicryst. specimen, growth usually stops when a grain boundary is reached. The probability that slip may then be initiated in the adjacent crystal is discussed. If the probability is low, the second slip may appear after some delay and produce a plastic after-effect. If the material in a slip plane behaves viscously, the probability may change with time, and comparatively large after-effects may then occur. In a polycryst. metal, the stress distributions are more complex and a statistical analysis of the problems then arising is suggested. Part of the total deformation is considered to be made up of a multiplicity of plastic after-effects, since many of the slips are secondary ones initiated by previous slips. An average quantity $\bar{K}$ is introduced to represent the relative contributions of the initial and secondary slips to the total deformation.—J. C. C.

***A Simple Method of X-Ray Microscopy and Its Application to the Study of Deformed Metals.** R. W. K. Honeycombe (*J. Inst. Metals*, 1951–52, **80**, (1), 39–43).—A method of obtaining images from metal crystals, using a line source of characteristic X-rays, is described. The images can be enlarged to at least 50 dia. to reveal significant microscopic phenomena, in particular distortions arising from slight plastic deformation which are not readily observed by opt. micrography. However, opt. micrography is of great assistance in the initial interpretation of the X-ray images, and the two techniques should be regarded as complementary. The scope of the method in the study of plastic deformation is illustrated by a series of X-ray and opt. micrographs obtained from metal crystals deformed under varying conditions.—AUTHOR.

***A New X-Ray Diffraction Method for Studying Imperfections of Crystal Structure in Polycrystalline Specimens.** Alfred J. Reis, Jerome J. Slade, Jr., and Sigmund Weissmann (*J. Appl. Physics*, 1951, **22**, (5), 665–672).—A high-precision double-crystal spectrometer for determining the angular range of reflection from individual particles in a Debye–Scherrer specimen, is described. This is achieved by rotating the specimen by small increments and moving the film at each stage. The specimen rotation is known to within $1''$. Necessary corrections are explained. The range is given in terms of a half-width, and the requisite analysis is instanced on data from commercial Si powder. Here the average half-width $\sim 1{\cdot}5'$, and differs little between lines except for (111) lines (and their higher orders). It is concluded that the (111) plane in Si has a unique function in connection either with the growth mechanism or with the cohesion.
—R. W. C.

Some New Functions of Interest in X-Ray Crystallography. M. J. Buerger (*Proc. Nat. Acad. Sci.*, 1950, **36**, (7), 376–382).—Math. Starting from the theory of fundamental point sets, vector sets and their interrelation, it is shown how a three-dimensional Fourier series resembling the standard electron *d* summation can be computed, on certain assumptions, from a three-dimensional map of Patterson functions.
—R. W. C.

Absorption Edges on Debye–Scherrer X-Ray Photographs. R. Brooks (*Brit. J. Appl. Physics*, 1951, **2**, (3), 76–77).—When working at high kilovoltages the residual short-wavelength "tail" may give rise to sharp-edged bands, often misleadingly like low-angle powder lines, which are associated with the absorption edges of Ag and Br (0·48 and 0·92 Å.). Measurements on patterns made from NaCl confirm this interpretation of the bands.—R. W. C.

***The Principles of the Experimental Study of Pseudo-Symmetric Crystalline Lattices by Means of Debye–Scherrer Diagrams : Application to Certain Solid Solutions.** René Faivre (*Rev. Mét.*, 1951, **48**, (6), 447–461).—F. discusses the principles of the interpretation of the Debye–Scherrer diagrams of a substance possessing pseudo-symmetry, and applies the conception of variable pseudo-symmetry to the study of the Pb–O system and to the solid soln. of C in α-Fe (martensite). 22 ref.—J. H. W.

***[Electron-Diffraction Study of] Oriented Crystals in the Rolled Plates of Alloys.** Shigeto Yamaguchi (*J. Appl. Physics*, 1951, **22**, (5), 680–681).—A letter. Electron-diffraction photographs were taken of etched stainless steels with rolling texture, which were then oxidized by high-pressure steam. The oxide film was stripped off and used as a replica for electron microscopy. From diffraction and microscopical data together it was possible to find the indices of the faces bounding etch pits.—R. W. C.

***Precise Determination of Lattice Constants by Electron Diffraction and Variations in the Lattice Constants of Very Small Crystallites.** F. W. C. Boswell (*Proc. Phys. Soc.*, 1951, [A], **64**, (5), 465–476).—Precision electron-diffraction photographs were made of thin films of several alkali halides, Au, Ag, and Bi, evaporated on to a Formvar film *in situ*. A film of TlCl was used as parameter standard. For the halides, a deviation from X-ray parameters was found for crystallite dimensions <100 Å.; the max. deviation was a decrease of 0·05%. For Au, the parameter was 0·2% less than the X-ray value for crystallites 40 Å. across, and 2% less for those 20 Å. across. Similar figures were found for Ag and Bi. The results for halides contradict some previous work, but B. is confident that his method is more reliable than previous ones. A brief theoretical discussion is attached.
—R. W. C.

Studies of Thin Films by Electron Diffraction. N. R. Mukherjee and Oliver Row (*J. Appl. Physics*, 1951, **22**, (5), 681–682).—A letter. A technique is described, involving a water-soluble plastic substrate, for making evaporated metal films in the form of unbacked diffraction specimens. Such films give much better patterns than samples with unremoved substrate. Some patterns are reproduced.—R. W. C.

Modification of a Positive-Replica Technique for Electron Microscopy. A. E. Austin and C. M. Schwartz (*J. Appl. Physics*, 1951, **22**, (6), 847–848).—A letter. A method of making positive replicas from negative Formvar replicas, using a Zapon lacquer film and differential solvents. Handling technique is described.—R. W. C.

Summarized Proceedings of a Conference on the Development and Application of Fourier Methods in Crystal Structure Analysis, London, November 1950. A. J. C. Wilson (*Brit. J. Appl. Physics*, 1951, **2**, (3), 61–70).—New techniques, both math. and instrumental, for Fourier syntheses of various kinds were described, mostly by their inventors. The accent was on apparatus such as the EDSAC calculator and an electric summing machine which projects a contoured electron-*d* projection on a cathode-ray screen. The theoretical parts include new devices for finding the phase of structure factors. 23 ref.—R. W. C.

5 — POWDER METALLURGY

***The Powder Metallurgy of Beryllium.** Henry H. Hausner and Norman P. Pinto (*Trans. Amer. Soc. Metals*, 1951, **43**, 1052–1069; discussion, 1069–1071).—See *Met. Abs.*, 1950–51, **18**, 527.

***A Process for Hot Pressing Beryllium Powder.** A. U. Seybolt, R. M. Linsmayer, and J. P. Frandsen (*U.S. Atomic Energy Commission Publ.*, 1949, (**AECU-1029**), 12 pp.).—Cf. *ibid.*, (**AECD-2679**); *Met. Abs.*, 1949–50, **17**, 575. A method of making Be compacts of theoretical *d* up to 12 in. dia. and 4 in. thick by pressing at 1100° C. The fine-grained material produced is more suitable for extruding, forging, and rolling and is more easily machined than cast billet. The cleanliness of the product depends on the original powder, but is not as good as that made by the vacuum-casting extrusion process.—B. W. M.

Metals and Refractories Combined in High-Temperature Structural Parts. John B. Campbell (*Materials and Methods*, 1950, **31**, (5), 59–63).—C. discusses the properties and prodn. of oxide–metal, boride–metal, and carbide–metal combinations. Al_2O_3–Fe, CrB–Ni, and TiC–Ni or Co combinations are described in some detail. Appn. of these metal–ceramics are discussed.—E. C. P.

Powder Metallurgy. P. Braillard (*Ingénieurs et Techniciens*, 1950, (24), 223–226; (25), 269–273).—Review of the methods of prodn. of the powders and of the finished parts. Types of powders and their propn., sintering and the theory of sintering including that of Kuczynski (*Trans. Amer. Inst. Min. Met. Eng.*, 1949, **185**, 169; *Met. Abs.*, 1948–49, **16**, 551) are discussed. Examples of sintering and appn. of powder metallurgy are given. A *bibliography*.
—G. A. K.

Recent Developments in Powder Metallurgy. H. W. Greenwood (*Metall*, 1951, **5**, (1/2), 16–18).—A brief review of developments during 1949–50.—E. N.

Preparation of Coated and Porous Powders for Powder Metallurgy. Tatsuo Matsukawa (*Technol. Rep. Osaka Univ.*, 1951, **1**, (6), 89–97).—[In English]. Methods of producing porous alloy powders and Cu-coated and Ag-coated powders are briefly described. Among the powders prepared are: (1) Cu-coated graphite, W, Mo, Ni, and Pb, and (2) Ag-coated graphite, Ni, Mo, and WC. Porous alloy powders are produced by alloying the metal with an easily-removable metal and decomposing the alloy so that one metal remains as powder. Thus, e.g. Zn–Cu alloy contg. 20–30% Cu may be treated with a soln. of $CuSO_4$, whereby the ε phase is attacked and decomposed, while the γ phase remains unattacked, the resulting structure being porous. From the powders produced by these methods, elect. contacts, metallic C brushes, and porous bronze bearings can be manufactured.—J. S. G. T.

***On the Mechanism of Sintering in Metal Powders.** W. Dawihl (*Schweiz. Arch. angew. Wiss. Techn.*, 1951, **17**, (3), 91–96).—[In German]. D. advances a new theory of sintering in metal powders, in which it is assumed that an intermediate layer is formed between the particles, this layer possessing higher strength than the metal within the particles; the formation of the layer may be facilitated or hindered by the presence of impurities, and its strengthening effect is greatest in compacts of fine particle size. It is suggested that it should be possible to produce powder-metal compacts of very fine grain, which should possess strength properties superior to those of cast metal of the same compn. The results of some measurements of the adhesion temp. of Cu, Co, Fe, W, and WC are given in support of the theory; some results showing the variation of hardness with grain-size of WC bonded with 6% Co are also given. In contrast to Jones ("Principles of Powder Metallurgy", **1937**; *Met. Abs.*, 1938, **5**, 66), D. concludes that it is likely that a temp. limit exists, below which no sintering occurs.—R. W. R.

***Note on Surface Diffusion in Sintering of Metal Particles.** N. Cabrera (*Trans. Amer. Inst. Min. Met. Eng.* (in *J. Metals*), 1950, **188**, (4), 667–668).—The rate at which the crack between a spherical particle and a metallic plane of the same material heated at temp. near the m.p. fills up is discussed. Kuczynski (*ibid.*, 1949, **185**, 169; *Met. Abs.*, 1948–49, **16**, 551) proposed two laws for the rate of growth of the radius of contact, *x*, between the plane and the sphere with time, *t*, according as to whether the nature of the mechanism of transport of material is: (1) the vol. diffusion of vacant lattice sites, where $x^5 = At$, where the const. *A* contains as a factor the self-diffusion coeff. of the metal, or (2) surface diffusion, when $x^7 = At$. C. shows, from theoretical considerations, that K.'s second law is incorrect, and that even when the mechanism of transport is surface diffusion the first equation, $x^5 = At$, is obeyed, but with a different const. *A*, and is valid for both small (<40μ dia.) and large particles.—E. N.

***Surface Diffusion in Sintering of Spheres on Planes.** P. Schwed (*J. Metals*, 1951, **3**, (3), 245–246).—Cf. *Trans. Amer. Inst. Min. Met. Eng.*, 1950, **188**, 667; preceding abstract. It is considered that the laws proposed by Kuczynski and by Cabrera for the rate of growth of the radius of contact, *x*, between a spherical particle and metallic plane of the same material, are incorrect, owing to erroneous assumptions of the limits of the region of surface diffusion. Accordingly, S. has performed the necessary calculations without restrictions on the region of diffusion, and has obtained two equations, the deciding factor as to which is applicable being whether $\rho \ll \frac{y_s}{\pi}$ or $\gg \frac{y_s}{\pi}$, where ρ is the radius of curvature of the neck at any given stage in the sintering process, and $y_s = (D_s t_s)^{1/2}$, where D_s is the coeff. of surface diffusion and t_s is the relaxation time for condensation. S.'s results, in comparison with those of K. and of C., are:

	$\rho \ll \frac{y_s}{\pi}$	$\rho \gg \frac{y_s}{\pi}$
Kuczynski	$x^7 \sim At$	$x^7 \sim At$
Cabrera	$x^5 \sim At$	$x^5 \sim At$
Schwed	$x^3 \sim At$	$x^6 \sim At$

It follows, therefore, that on the revised theory it is possible to distinguish surface diffusion from vol. diffusion by experiments on small particles.—E. N.

6 — CORROSION AND RELATED PHENOMENA

***Study of the Methanol Corrosion of Copper by Electron Diffraction.** Shiro Ogawa and Denjiro Watanabe (*Sci. Rep. Research Inst. Tôhoku Univ.*, 1950, [A], **2**, (3), 462–470).—[In English]. The methanol corrosion of Cu made either in America or Japan was studied by electron diffraction. Japanese Cu suffered heavier corrosion than American Cu, and showed a lead-grey colour. In the early stages of corrosion Cu_2O and CuO were produced, but CuO alone was formed in the final stage, and the latter is readily formed after a short period of immersion in methanol. Corrosion is due mainly to H_2O absorbed by the methanol. Specimens immersed in H_2O do not experience such violent corrosion as in methanol. It is therefore considered that unknown impurities, present in methanol and soluble in H_2O, affect the corrosion.—J. S. G. T.

***The Determination by Interference Colours or by Polarized Light of the Rate of Oxidation of Certain Faces of a Single Crystal of Copper.** Allan T. Gwathmey and Frederick W. Young (*Rev. Mét.*, 1951, **48**, (6), 434–440).—G. and Y. undertook measurements of the rates of oxidation of certain faces of a single Cu crystal by means of interference colours and the ellipticity of polarized light as a part of a general study of the surface properties of single crystals. The first method has the advantage of simplicity and the second of greater precision; together they enable continuous measurements of the thickness of the film on the different faces to be made. Qual. investigations have been carried out by several workers and show that at 200° C. the rate of oxidation is greatest on the (100) and (210) faces and least on the (311) face. In the present work measurements were first made of the refractive index of the film of Cu oxide on Cu by Winterbottom's graphical method. This was a most laborious study and resulted in values of 2·75–0·195, of no use for calculating the thickness. A comparison was next made of the thickness measured by the grav. method, by electrometric reduction, and by polarized light. The mean values of the three methods (which were in close agreement) were: lightly tarnished 79, tarnished 185, pale rose 265, purple 337, pale blue 405, and yellow 740 Å. The rates of oxidation at 150° C. were measured on the two faces having the highest (110) and the lowest (311) rates, being shown graphically as thickness against time. The relations between films of oxide and the substratum on which they are formed were studied with X-rays between 170° and 325° C. at atmospheric pressure and between 250° and 351° C. at 5·15 mm. Hg. In certain cases polycryst. structures, with or without preferred orientation, and in other cases, monocryst. films with considerable and variable disorientation occur. In general, the oxide is the more nearly monocryst. the higher the temp. of oxidation and the lower the pressure. At 200° C. and atmospheric pressure in the range of thickness of 500–3000 Å., the film corresponding to the (100) face was polycryst. Whether the film is mono- or polycryst. depends on the face considered. 11 ref. —J. H. W.

Plated [Brass] Coil Springs. H. H. Symonds (*Metal Ind.*, 1951, 78, (23), 469).—Isolated intergranular films of oxide were found to be the cause of cracking of Ag-plated brass coil springs.—J. H. W.

***The Effect of Phosphorus on the Corrosion-Resistance of Magnesium and Some of Its Alloys.** E. F. Emley, A. C. Jessup, and W. F. Higgins (*J. Inst. Metals*, 1951–52, **80**, (1), 23–32).—Corrosion tests have been carried out, by total immersion in salt soln, on samples of Mg alloys contg. various amounts of P and other impurities. It is concluded that the corrosion-resistance of pure Mg is reduced by the presence of P in amounts over ~0·002%, but that this effect can be suppressed by addn. of Mn. With a normal Fe content, the adverse effect of P is not suppressed by Mn. P also exerts a marked adverse effect on the corrosion-resistance of high-purity Elektron A8 (Al 8, Zn 0·4, Mn 0·25%, remainder Mg). The amount of P that can be introduced into Mg is much greater in the presence of Fe. P increases Fe pick-up in remelting A8 alloy. P is precipitated from Mg by Zr, and the corrosion-resistance of alloys contg. Zr is unaffected by P present in the metal used for their prepn.—AUTHORS.

***Effects of Various Elements on the Resistivity of Nickel–Molybdenum–Chromium Alloys to Hydrochloric Acid.** Hikizô Endô and Saburo Ishihara (*Sci. Rep. Research Inst. Tôhoku Univ.*, 1950, [A], 2, (4), 632–636).—[In English]. The effects of the addn. of ferro-V, ferro-Ta, ferro-Ti, ferro-Zr, Cu–Be, W, Co, and Cu to Ni–Mo–Cr alloys upon the resistance of these alloys to corrosion by 10, 20, or 30% HCl at room temp., and by 10 and 20% HCl at b.p., were studied. Ferro-V was the most effective in reducing corrosion, the Ni–30% Mo–15% Cr–0·5% ferro-V alloy being free from attack, at room temp., for 15 days in 10% HCl. All the other added materials gave rather bad results; ferro-Ti gave the worst results of all. —J. S. G. T.

***The Reaction Between Oxygen and Thorium.** Pascal Levesque and Daniel Cubicciotti (*J. Amer. Chem. Soc.*, 1951, 73, (5), 2028–2031).—The oxidation of Th in the range 250°–700° C. was studied. Above 450° C. the temp. of the sample rose considerably above the furnace temp., and below 450° C. the oxidations were essentially isothermal. The rate const. for the linear oxidation were measured, and the energy of activation was calculated to be 22 kg.cal./mole. From 250° to 350° C. the oxidation proceeded according to the parabolic law, and the calculated energy of activation for the reaction was 31 kg.cal./mole. Comparison of the results with those reported by Gulbransen (*Trans. Electrochem. Soc.*, 1947, **91**, 573; *Met. Abs.*, 1949–50, **17**, 502) and G. and Andrew (*Trans. Amer. Inst. Min. Met. Eng.*, 1949, **185**, 515; *Met. Abs.*, 1949–50, **17**, 153) for Zr and Ti, resp., shows that the oxidation rates increase uniformly with atomic number in the relevant group of the Periodic Table.—J. R.

***Studies in the Corrosion of Tin: Corrosion in [I.—] Hydrochloric Acid; [II.—] Sulphuric Acid Solution; [III.—] Nitric Acid Solution.** Hikizô Endô and Gorô Yokoyama (*Sci. Rep. Research Inst. Tôhoku Univ.*, 1950, [A], 2, (3), 449–455, 456–461; (4), 637–644).—[In English]. [I.—] Sn in HCl exhibits the passive state in concentrations <0·01%. Depolarizing corrosion increases with acid concentration from 0·01 to 3%, and is complete at 3–15% concentration. H_2-evolution corrosion increases at concentrations of 15–25%, and is complete at concentration >25%. The effect of casting and mould temp. on corrosion in 30% HCl was studied. [II.—] The corrosion mechanism of Sn in 5–30% H_2SO_4 soln. is of the depolarizing type, and that in 97% H_2SO_4 soln. is controlled by diffusion. The mechanism in the region of 60% acid concentration is not yet understood. [III.—] The corrosion of Sn in 10–64·5% HNO_3 soln. is of a mixed type, attributable to decompn. of the NO_3 ion and diffusion of HNO_3. The importance of diffusion increases gradually with increasing acid concentration. In all cases Sn remains passive for acid concentrations >80%. The passivity of α- and β-Sn in conc. HNO_3 soln. was studied at room and low temp. In all three papers the effects of stirring, temp., and various additives, e.g. $K_2Cr_2O_7$, $NaNO_3$, formaldehyde, Na_2SO_3, were studied. Corrosion velocity curves are given in all papers.—J. S. G. T.

***Effect of Chromium on the Oxidation-Resistance of Titanium Carbide.** J. D. Roach (*J. Electrochem. Soc.*, 1951, 98, (4), 160–165).—Cr added to TiC as Cr_2O_3 before recrystn. affects the oxidation-resistance of the carbide in a manner depending on the amount added. Small percentages (0·5–1% Cr calculated as wt.-% of the materials added) decreased the oxidation-resistance in a given time at 650°–1400° C., but larger percentages (up to 5% Cr) increased it. The max. oxidation-resistance occurs at 5% Cr, and the extent of oxidation at a given temp. with this amount of Cr is considerably less than that obtained on straight TiC. Experiments were made with addn. of 10 and 20% Cr, but double recrystn. was necessary to obtain satisfactory specimens, and therefore the amount of oxidation-resistance was not comparable. Cr in recrystallized TiC probably occurs as a solid soln. and not as a binder. Figures were obtained showing the effect of Cr addn. in increasing the *d* of the TiC and in decreasing the amount of free C. 18 ref.—G. T. C.

The Corrosion-Resistance of Galvanized Coatings, with Special Reference to Galvanized Window Frames. E. F. Pellowe (*Internat. Conf. Hot-Dip Galvanizing* (*Copenhagen*), 1950, 122–133; discussion, 134–138).—Details are presented of the galvanizing process used for window-frames. Brief information is given on the resistance of the galvanizing on window-frames to mech. damage during transit and erection, and it is concluded that areas where such damage occurs are adequately protected from corrosion by the galvanic action of the Zn. Exposure tests at present in hand appear to indicate that the anodic protection given by the alloy layer is at least as effective as that given by the Zn layer and, moreover, that the corrosion losses of the alloy layer are somewhat less than those of the Zn layer. Previous work on the corrosion-resistance of galvanized coatings is reviewed. Results are reported of a survey of the behaviour of galvanized window-frames in a number of types of particularly severe indust. service. Brief information is included on the behaviour of riveted galvanized window-frames.—G. T. C.

Report of [A.S.T.M.] Committee A-5 on Corrosion of Iron and Steel [and Galvanized Products]. —— (*Proc. Amer. Soc. Test. Mat.*, 1950, 50, 100–117).—See *Met. Abs.*, 1950–51, **18**, 186.

Report of [A.S.T.M.] Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys. —— (*Proc. Amer. Soc. Test. Mat.*, 1950, **50**, 137–138).—T. G.

Report of [A.S.T.M.] Committee B-6 on [the Corrosion of] Die-Cast Metals and Alloys. —— (*Proc. Amer. Soc. Test. Mat.*, 1950, **50**, 167–172).—See *Met. Abs.*, 1950–51, **18**, 186.

How to Avoid Galvanic Corrosion. —— (*Steel*, 1951, **128**, (21), 82–83, 104, 106, 109, 110).—The phenomenon of galvanic corrosion is described and a review given of various methods used to combat it, particularly in Mg and Al alloy assemblies. —E. J.

Materials for Chemical Apparatus. L. Piatti (*Schweiz. Arch. angew. Wiss. Techn.*, 1951, **17**, (3), 80–91).—[In German]. P. reviews the different types of corrosion which are commonly encountered in chem. engineering practice and discusses their avoidance. The resistance to corrosion of the available materials is considered in fair detail. 10 ref. —R. W. R.

Corrosion Cut in T.C.C. Gas Plant [for Oil Refining]. C. A. Murray and M. A. Furth (*Petroleum Processing*, 1951, **6**, (5), 504–506).—The use of Admiralty metal and 70 : 30 cupro-nickel heat-exchanger tubes in various units is mentioned.—G. T. C.

Corrosion Mitigation Viewed by Management. H. H. Anderson (*Petroleum Eng.*, 1951, **23**, (4), D30, D32–D34).—A general paper dealing with the economics of corrosion control, the position and function of the corrosion engineer and his relationship with management.—G. T. C.

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7 — PROTECTION

(Other than by Electrodeposition.)

Continuous Anodizing of Aluminium Coiled Sheet. Charles J. Simon (*Metal Finishing*, 1951, **49**, (5), 63–64).—A brief description is given of an actual plant for this purpose. The machine takes four sheets from 0·008 to 0·020 in. thick and 6–12 in. wide. Brief details are included on the operation of the plant and of the pretreatment and anodizing baths. —G. T. C.

Sealing of Anodized Aluminium Increases Corrosion-Resistance. A. E. Durkin (*Iron Age*, 1951, **167**, (19), 96–98).—Anodized Al sealed in high-purity water with a *p*H controlled at or near 6 has approx. 35% greater corrosion-resistance than when it is sealed in ordinary tap-water without any *p*H control.—J. H. W.

The Chromizing Process. T. Gibson (*Sheet Metal Ind.*, 1951, **28**, (290), 569–571, 575).—The present-day shortage of high alloy steels and non-ferrous metals emphasizes the importance of suitable substitutes. G. describes a method of chromizing Fe or steel to produce good corrosion- and heat-resisting surfaces. Chromizing is claimed to be of established use in many industries, including the chem. industries, oil refineries, carburizing plant, &c.—R. J.

***Molybdenum Plating by Reduction of the Pentachloride Vapour.** W. J. Childs, J. E. Cline, W. M. Kisner, and John Wulff (*Trans. Amer. Soc. Metals*, 1951, **43**, 105–121).—See *Met. Abs.*, 1950–51, **18**, 372.

Hot-Dip Tinning of Cast Iron. W. E. Hoare (*Found. Trade J.*, 1950, **89**, (1786), 411–414, 427).—An account of the "iron plating", the T.R.I. nitrate, the T.R.I. chloride (Tin Research Institute), and the wipe-tinning processes for the hot-dip tinning of cast-iron.—J. E. G.

***The Influence of Aluminium on the Alloying Action in Hot-Dip Galvanizing.** M. L. Hughes (*Internat. Conf. Hot-Dip Galvanizing* (*Copenhagen*), **1950**, 31–43; discussion, 44–49).—Cf. *J. Iron Steel Inst.*, 1950, **166**, 77; *Met. Abs.*, 1950–51, **18**, 373. Experimental work is described which had for its objects the detn. of the min. amount of Al which needs to be present in the galvanizing bath to prevent the usual alloying action and also to examine the existing explanations for the action of the Al. Four types of material were used: (*a*) Armco iron, (*b*) commercial low-C steel, cold reduced sheet of rimming quality, (*c*) killed steel contg. 0·10% C and only a trace of Si, and (*d*) steel contg. 0·22% C and 0·10% Si. Immersion times varied from 5 to 60 sec., but in some instances were 2 min. The temp. varied from 430° to 490° C., and the Al addn. from 0·05 to 3%. The following conclusions were reached: (1) Under favourable conditions 0·10% Al is sufficient to produce coatings free from the usual alloy layers, whilst 0·05% is sufficient to promote good blending properties. (2) Under average conditions, 0·18% Al prevents normal alloying. (3) The presence of Si enhances the effect of the Al. (4) In the presence of relatively large addn. of Al appreciable amounts of alloy different from the usual alloys in appearance, compn., properties, and mechanism of formation, may be formed. It is of ternary compn., and the Al present is in the form of $FeAl_3$. (5) The presence of this alloy explains peculiar results sometimes obtained when Al is added to the bath. (6) It is shown that neither the "membrane" nor the "non-adherent alloy" theory adequately explains why small amounts of Al prevent visible formation of alloy, and it is concluded that Al acts by the initial formation of a very thin layer of the alloy referred to in (4). (7) Evidence was obtained of a secondary alloying process which occurs under the ternary alloy and causes the latter to break away and form loose crystals in the coating. This secondary alloying is erratic, and more information is required about it. (8) The ternary alloy has excellent bending properties, and no tendency to flaking was observed. (9) The simultaneous presence of much Al and Pb in the coatings makes them liable to suffer severe attack by hot water and steam. —G. T. C.

Wet Galvanizing versus Dry Galvanizing. H. Bablik (*Internat. Conf. Hot-Dip Galvanizing* (*Copenhagen*), **1950**, 76–81; discussion, 85–88).—In the wet galvanizing process a flux layer is maintained on the surface of the bath, but in the dry process the work is first dipped in a $ZnCl_2$ soln. and then dried in an oven at 120°–200° C. It is shown that use of the latter process results in a smaller Zn consumption than the former and also in less dross formation. There is less flux residue on the work than with the wet process. It is also claimed that the quality of the coating is better. The main disadvantage of the dry process is the fact that special equipment, i.e. a drying oven, &c., is required. Moreover, it is only practicable when articles are of a uniform shape and size. It is less suitable for the prodn. of heavy coats than for that of thinner ones.—G. T. C.

Some Notes on the Relative Merits of Flux Galvanizing and Dry Galvanizing. T. Liban (*Internat. Conf. Hot-Dip Galvanizing* (*Copenhagen*), **1950**, 82–84; discussion, 85–88).—The relative merits of the two processes are summarized as follows: (1) in flux galvanizing the skimmings and Zn ash together account for 6·9% Zn loss, whereas in dry galvanizing the average Zn loss in Zn ash is 27% of the total Zn used. Owing to the chloride content, the value of the ash is relatively low and even after the metallic Zn has been removed, at considerable expense, there still remains in it ~11% of the total Zn used. (2) Maintenance and service of the drying oven is an expensive item in dry galvanizing. (3) Flux galvanizing allows a higher output than dry galvanizing. (4) Under similar conditions of operation, both processes lead to about the same amount of hard Zn formation. —G. T. C.

A Survey of the Formation of Zinc Ash, Dross, and Flux Skimmings from Galvanizing Baths During 1949. R. W. Bailey (*Internat. Conf. Hot-Dip Galvanizing* (*Copenhagen*), **1950**, 90–101; discussion, 116–120).—A survey is described of a cross-section of galvanizing baths typical of those operating in the general galvanizing industry during 1949.

20 baths were kept under observation in the tests. The classes of work covered by these baths were very varied, but as far as possible each bath chosen was devoted to one particular class of article. The survey covered flux- and dry-galvanizing; prefluxed work and work dipped straight from HCl pickling without washing; some baths operating with, and some without, Al; baths heated by several different methods, some with and others without thermostatic control. The figures obtained are tabulated in detail, and in addn. the following tentative conclusions were reached: (1) losses from ash formation and flux skimmings depend on the extent to which the surface of the bath is flux-covered and on the extent to which the surface is disturbed both during operation and during the removal of Zn ash. Careful skimming and controlled speed of immersion and withdrawal of the work are probably more important with dry baths than any other single factor, both in determining the extent to which oxidation occurs and the amount of metallic Zn entrained in the ash. (2) The most important factors in controlling dross formation are: (*a*) The washing of Fe salts from the work after pickling and before dipping. (*b*) Bath heating. Different bath-heating systems vary considerably in effectiveness. Although thermostatically controlled baths seem to give lower dross figures than coke-fired baths, the latter can hold their own if properly tended. (*c*) The through-put of work. The highest efficiency is obtained by maintaining a high through-put, though this must not exceed a certain optimum figure, as an over-worked bath produces an excessive amount of dross.—G. T. C.

Can Dross be Eliminated in Hot-Dip Galvanizing? W. G. Imhoff (*Internat. Conf. Hot-Dip Galvanizing* (*Copenhagen*), **1950**, 102–113; discussion, 116–120).—The various sources of dross are each briefly discussed, and the steps which have been taken to eliminate dross formation are described. The conclusion is reached that at present it is impossible to carry out hot-dip galvanizing without forming some dross.
—G. T. C.

Notes on New Methods of Drossing. H. Arnason (*Internat. Conf. Hot-Dip Galvanizing* (*Copenhagen*), **1950**, 114–115; discussion, 116–120).—Portable equipment, of which no details are included, has been developed into which the mixture of Zn and hard Zn is fed on removal from the bath. In this way hard Zn with 4·5–5·0% Fe and Zn with $\ngtr$0·05% Fe are obtained. This procedure results in a reduction in loss of $\sim$20% and allows drossing to be done during the lunch break. Experiments have been carried out with removing dross by means of a pump instead of by the spoon method. The dross removed in this way is less conc., and a method has been developed for continuously separating the superfluous Zn. A hard Zn with 3·50% Fe is obtained in this way. In a second stage, extraction of further Zn takes place and the hard Zn finally obtained contains 4·5–5·0% Fe. Using a small pump, 500–600 kg. of conc. dross can be removed in ½ hr.—G. T. C.

Some Thermal Considerations in the Heating of Galvanizing Baths. R. W. Bailey (*Internat. Conf. Hot-Dip Galvanizing* (*Copenhagen*), **1950**, 51–62; discussion, 63–72).—The main principles involved in heating a galvanizing bath are discussed, and various heating methods are described. The extent to which the latter fulfil the requirements of galvanizing-bath heating is evaluated. No information is given on furnace construction, burner design, or temp. control and measurement.—G. T. C.

Twenty Years of Research for the Galvanizing Industry. W. L. Hall and D. N. Fagg (*Internat. Conf. Hot-Dip Galvanizing* (*Copenhagen*), **1950**, 14–29; discussion, 44–49).—Work carried out by the British Non-Ferrous Metals Research Association is reviewed. The reaction between the Zn and the steel is considered from the points of view of the effect of temp., the effect of immersion time and rate of withdrawal, the effect of addn. to the galvanizing bath of Al, Sn, Sb, Cd, alone or in combination, and the effect of the basis steel. Dross formation due to pickling products and to the action of the flux is described, and suggestions are made for limiting it. Various researches on the corrosion-resistance of galvanized coatings are also described.—G. T. C.

The Outlook for General Galvanizing. R. Lewis Stubbs (*Internat. Conf. Hot-Dip Galvanizing* (*Copenhagen*), **1950**, 140–148; discussion, 149–156).—Some brief statistics on the amount of Zn used for galvanizing, &c., in the more important countries are presented. Possible rival coatings to galvanizing, including other Zn coatings, other metallic coatings, and various non-metallic coatings, are discussed. Future developments are considered from the point of view of improvements in the process itself and that of extended uses of galvanized products.—G. T. C.

Sendzimir Process Broadens Galvanized Sheet Applications. K. Oganowski (*Steel*, 1950, **126**, (24), 102, 104, 107, 110, 113).—A plant is described for the continuous prodn. of galvanized sheets and coils by the Sendzimir process.
—E. J.

***A Study of Some Metals for Use as Permanent Anodes in Water-Tank Cathodic Protection Systems.** A. L. Kimmel (*Corrosion*, 1950, **6**, (11), 353–358; discussion, 358–359).—Corrosion-rate data are given on the following commercial metals and alloys used as permanent anodes: Al (99·99%), Al alloy (3S), black Fe (hot rolled), Ni, Monel, Inconel, and stainless steel (302 and 316). The stainless steels appear to be the most suitable, and Al is also suitable and inexpensive. 6 ref.—W. A. M. P.

Combating Corrosion in a Chemical Plant with Magnesium Anodes. Oliver Osborn (*Corrosion*, 1951, **7**, (1), 2–9).—The use of Mg anodes for the cathodic protection of steel and cast Fe in a chem. plant is described and discussed.
—W. A. M. P.

Corrosion Programmes for a Pipe Line [Cathodic Protection by Magnesium Anodes]. Marshall E. Parker (*Petroleum Eng.*, 1951, **23**, (5), D68, D70, D72, D74, D76–D80).—The planning of anti-corrosion measures for steel pipe-lines is described, with particular ref. to cathodic protection. The use of Mg anodes for the latter purpose is briefly mentioned.—G. T. C.

Corrosion and Protective Coatings of Metals. M. Ballay (*Ingénieurs et Techniciens*, 1950, (21), 113–118).—The introduction to a series of articles on the various protective coatings for metals. The subject is dealt with under the headings: (*a*) forms of corrosion; (*b*) protective coatings which do not affect or only very slightly modify the surface of the treated metal; and (*c*) coatings which do modify the surface.
—G. A. K.

The Preparation of Metallic Surfaces Before Anti-Corrosive Treatment. Jean Bary (*Ingénieurs et Techniciens*, 1950, (22), 173–175).—Different methods of cleaning the surfaces preparatory to anti-corrosive treatment are described. A table gives the appropriate method for various metals.
—G. A. K.

Metallic Coatings by Hot Dipping. J. Grilliat (*Ingénieurs et Techniciens*, 1950, (25), 283–286).—A review of various methods of hot-dip coating and a description of two methods of standardized control of the coatings, one French and the other American.—G. A. K.

The Protection of Metals by Metal Spraying. J. Grilliat (*Ingénieurs et Techniciens*, 1950, (23), 201–206).—A critical discussion of this method. The tools, prodn. of the metallic coatings, treatment and finishing of the coatings, their properties, choice of a suitable coating, and its thickness, are considered. Indust. appn. in marine engineering, bridges and public buildings, metallic structures, railway engineering, chem. and food industries, are described.—G. A. K.

***A New Method for the Protection of Metals Against Pitting, Tuberculation, and General Corrosion.** H. Lewis Kahler and Charles George (*Corrosion*, 1950, **6**, (10), 331–340).—The di-anodic method for the control of tuberculation and pitting of metals in water-side corrosion is described. It employs a dual treatment of molecularly-dehydrated phosphates and chromates over selective *p*H ranges, and its use in relatively low concentrations gives benefits not obtainable with single chromate and phosphate treatments at considerably higher concentrations. It appears from experimental evidence that the two anodic treatments used together under the conditions of the di-anodic method act as a single agency and not as two separate treatments. Experimental and plant data are given. 16 ref.—W. A. M. P.

Preventing Corrosion with Protective Paint Coatings. George Diehlman and E. L. Beenfeldt (*Corrosion*, 1951, **7**, (3), 88–92).—The use and appn. of red Pb paint for preventing corrosion is fully discussed.—W. A. M. P.

Selection of Protective Coatings for Chemical Fume Resistance. Clarence C. Harvey (*Corrosion*, 1950, **6**, (10), 323–326).—Laboratory screening tests and panel tests located in the plant are described for the selection of organic coatings to withstand sp. fume conditions, such as SO_2, SO_3, H_2S, Cl_2, HCl, and nitric oxides which are found present in varying proportions in chem. plants and refineries. —W. A. M. P.

Porcelain Enamel as a Corrosion-Resistant Coating for Metals. G. H. McIntyre (*Corrosion*, 1951, **7**, (4), 118–122; discussion, 122–123).—The use of porcelain enamel as a decorative finish is compared with its more recent use as a corrosion-resistant coating. The properties of different enamels are enumerated, and some sp. appn. for corrosion-resistance are discussed. Resistance to impact damage is also discussed. 10 ref.—W. A. M. P.

8 — ELECTRODEPOSITION

***The Electrodeposition of Aluminium from Non-Aqueous Solutions at Room Temperature.** Frank H. Hurley and Thomas P. Wier, Jr. (*J. Electrochem. Soc.*, 1951, **98**, (5), 207–212).—A satisfactory plating soln. consists of a mixture of 2 moles $AlCl_3$ (anhydrous) with 1 mole ethyl pyridinium bromide, preferably in a dry, O-free atmosphere, to produce a liquid to which benzene or toluene is added until a second layer, principally excess benzene or toluene, forms on top of the plating soln. Using an Al anode, plating may be carried out in a protective atmosphere at ~1 V. and with a cathode c.d. of ~1 amp./dm.^{2}. White shiny deposits of Al may be produced on various metals by this method. Other pyridinium compounds also produced satisfactory results. The use of A.C. superimposed on D.C. gave greatly improved adhesion. A Cu wire plated in this way could be repeatedly flexed without breaking the coating. The use of A.C. superimposed on D.C. also increased the thickness that could be obtained without brittleness, increased the max. c.d., somewhat lowered the voltage requirements, gave the same efficiency, based on D.C., and changed the appearance of the deposit from shiny to satiny. 8 ref. —G. T. C.

***Electrodeposition on Aluminium: A Study of the Zincate Process.** G. L. J. Bailey (*Electrodepositors' Tech. Soc., Advance Copy*, 1951, **27**, (10), 22 pp.).—Recently published investigations on this subject are briefly reviewed, and new work is then described on the influence on adhesion of variation in compn. of the zincate soln. and in the time and temp. of immersion in this soln. The influence of variation in the "conditioning" procedure, alloy compn., and heat-treatment condition was also studied. The rate of formation of Zn deposits was investigated, and studies were made of their structure by electron diffraction and X-rays. A theory of the mechanism of adhesion is proposed. 8 ref.—G. T. C.

Chromium Plating in Barrels. Alan Whittaker (*Mech. World*, 1951, **129**, (3351), 309–311, 314).—The principles of Cr plating are briefly described, and a short but detailed account is given of the barrel plating of small articles. —R. W. R.

***Effect of Chromium Plating on the Endurance Limit of Steels Used in Aircraft.** Hugh L. Logan (*Proc. Amer. Soc. Test. Mat.*, 1950, **50**, 699–713; discussion, 714–716).—See *Met. Abs.*, 1949–50, **17**, 449.

***Cathode Polarization Potential During Electrodeposition of Copper. I.—Non-Reproducibility in Acid Copper Sulphate Solutions.** L. L. Shreir and J. W. Smith (*J. Electrochem. Soc.*, 1951, **98**, (5), 193–202).—It is shown that the const.-state polarization potential (C.S.P.) of the cathode during the electrodeposition of Cu from $CuSO_4$–H_2SO_4 soln. depends on the conditions of prepn. and storage of the soln. and the time that it is kept after prepn. A fresh soln. has a high C.S.P. and usually gives fine-structure deposits, but as the soln. ages the C.S.P. value decreases and the structure of the deposits coarsens. The C.S.P. value can again be raised either by treating the soln. with oxidizing agents or else by prolonged electrolysis and again fine-grained deposits are obtained. There was evidence that the fall in C.S.P. on storage is associated with small traces of impurity either present in the reagents or formed during the ageing of the soln., this impurity having the property also of increasing the crystal size of the deposited metal. It is shown that the formation of this impurity is associated with traces of contamination in ordinary distilled water, and a method is described for the prepn. of the soln. in such a way that they remain stable for several months. This involves the use of "AnalaR" H_2SO_4 and $CuSO_4$ twice recrystallized from freshly distilled water contg. a little H_2SO_4. The distilled water used to make up the soln. was freshly distilled from $KMnO_4$ and NaOH. After prepn. the soln. was boiled, cooled in a stream of N, and then stored under N. 24 ref.—G. T. C.

***Conditions for the Electrodeposition of the Alloy Mn–Ni.** D. N. Gritsan and N. S. Tsvetkov (*Zhur. Priklad. Khim.*, 1949, **22**, (6), 600–604).—[In Russian]. Preliminary experiments established that unless NH_4^+ ions are present it is impossible to obtain satisfactory Mn–Ni deposits. A bath contg. Mn, Ni, and NH_4 sulphates was therefore used, with a brass (in some experiments, Cu or Fe) cathode (working area 35 cm.^{2}) and two graphite anodes, the bath temp. being 20° C. After electrolysis the cathode was washed, dried, and the deposit dissolved in a mixture of H_2SO_4 (1 : 20) 320, HNO_3 (1 : 4) 100, H_3PO_4 (21%) 50 ml. Mn was determined in this soln. by a persulphate/nitrite method; the Ni content of the deposit was calculated by difference. With baths contg. $MnSO_4.5H_2O$ 50–300, $NiSO_4.7H_2O$ 40, $(NH_4)_2SO_4$ 75 g./l., and cathodic c.d. (D_k) = 2·8 amp./dm.^{2}, the Mn content of the deposit rose sharply to ~7% at 150 g./l. $MnSO_4.5H_2O$, but remained const. at higher $MnSO_4$ concentrations; the coatings were mirror-bright and possessed high chem. stability. With $MnSO_4.5H_2O$ 150, $NiSO_4.7H_2O$ 10 g./l., and D_k = 2·8 amp./dm.^{2}, the Mn content of the deposit fell from 15 to 9% as the $(NH_4)_2SO_4$ compn. of the bath rose from 20 to 75 g./l., the quality of the deposits being best at 60–75 g./l. Variation in D_k from 2·7 to 17·0 amp./dm.^{2} with this type of bath had little effect on the Mn content of the deposits, which were dense and bright; above 17·0 amp./dm.^{2} the deposits were dark. A five-fold lowering of the Ni concentration in the bath resulted in only a 2% increase in the Mn content of the deposit. Further experiments were made using a diaphragm cell, the catholyte contg. $MnSO_4.5H_2O$ 150, $(NH_4)_2SO_4$ 75 g./l., and the anolyte $NiSO_4.(NH_4)_2SO_4.7H_2O$ saturated, $(NH_4)_2SO_4$ 75 g./l. For each value of D_k there is a definite catholyte *p*H at which there is a sudden jump in the Mn content of the deposit; with an increase in D_k, the jump in the % Mn/*p*H curve shifts into the more acid region. Thus at 5·4 amp./dm.^{2}, there is a jump from 15 to 87% Mn at a catholyte *p*H = ~8; at 10·8 amp./dm.^{2}, from 32 to 98% Mn at a *p*H = ~5. This rise is accompanied by a rise in the cathodic potential (ϕ)/*p*H curve. Deposits with <35% Mn were only slightly less resistant to corrosion than those of Ni; deposits with >35% Mn quickly lost their lustre, but were more resistant than those of pure electrolytic Mn. Treatment of Mn-rich deposits with 5% $K_2Cr_2O_7$ soln. preserved their lustre and increased their corrosion-resistance.—G. V. E. T.

***Electrodeposition of Nickel in the Bores of Tubes Using Insoluble Anodes.** A. W. Hothersall and G. E. Gardam (*Electrodepositors' Tech. Soc., Advance Copy*, 1951, **27**, (7), 15 pp.).—The method described is one which requires special apparatus and experience, but it has been used successfully on an indust. scale for the rectification of the bores of expensive tubes which have been over-bored. Smooth deposits of Ni, having good mech. properties, can be built up to a considerable thickness by the use of an insoluble anode which is made of or coated with Pb. A $NiSO_4$–H_3BO_3 soln. free from Cl was used and continuously circulated through the tube. Const. small addn. of $Ni(OH)_2$ were necessary in order to maintain the compn. of the soln. const. By this method, deposits 0·05 in. thick have been built up in tubes 6 ft. long and 4 in. dia. These deposits had a D.P.N. of $\sim$200, a U.T.S. of $\sim$30 tons/in.^{2}, and an elongation of $\sim$30%. Full details of the equipment and the method of operation are included.—G. T. C.

***Protective and Decorative Nickel Plating of Articles Without Subsequent Polishing.** N. T. Kudryavtsev, O. M. Korol'kova, and V. V. Fedurkin (*Zhur. Priklad. Khim.*, 1949, **22**, (6), 586–591).—[In Russian]. Deposits of Ni on brass, Cu, and steel were made from sulphate baths contg. a mixture of salts of naphthalene 2 : 6- and 2 : 7-disulphonic acids. These were prepared by heating naphthalene with H_2SO_4, cooling and adding the product to the plating bath, excess H_2SO_4 being neutralized with $NiCO_3$ or alkali to give $pH = 5{\cdot}8$–$6{\cdot}3$. Baths contg. $NiSO_4$ 140–300, H_3BO_3 30, KF or NaF 5–6, KCl or NaCl 3–15, and naphthalene disulphonic acids 2–4 g./l. are recommended for bright deposits. The presence in the bath of up to 1 g./l. Fe (as sulphate) did not affect the brightness or other properties of the deposits. Optimum conditions were 20°–40° C., and 1·5–5·0 amp./dm.^{2} (with agitation) or 0·2–1·0 amp./dm.^{2} (without agitation). X-ray examination of deposits prepared at high c.d. showed that they had highly orientated Ni crystals, whereas in the absence of naphthalene disulphonic acids there was random orientation. The deposits were also less porous and more uniform than matt deposits prepared from ordinary baths. The brittleness of the deposits became noticeable only at thicknesses $>$25–30 μ.
—G. V. E. T.

Nickel Salts from Nickel Anodes : Porous-Pot Method. Nathaniel Hall and G. B. Hogaboom, Jr. (*Metal Finishing*, 1951, **49**, (5), 66–67, 70).—In view of the difficulty of obtaining at the present time $NiSO_4$ and $NiCl_2$ of adequate purity for making up plating baths, it is suggested that scrap Ni anode residues be dissolved electrolytically in H_2SO_4 or HCl contained in porous pots during the periods when the plating bath is not in use. Full details are presented on how to arrange the apparatus and on the current required, &c. Information is also included on operating costs.—G. T. C.

Inorganic Chromatography on Cellulose : Some Applications to the Analysis of Nickel-Plating Baths. F. H. Burstall, N. F. Kember, and R. A. Wells (*Electrodepositors' Tech. Soc., Advance Copy*, 1951, **27**, (11), 12 pp.).—A cellulose column sepn. method is described which enabled a number of metals to be extracted together from a Ni-plating soln. The behaviour of various metals which might be present as impurities in Ni-plating soln. is described, and methods of estn. are given in detail for Co, Cu, Fe, Zn, Mn, and Cd. 8 ref.—G. T. C.

Phosphorus Alloys Used in New Plating Method. —— (*Steel*, 1950, **126**, (25), 99, 102).—See Brenner, Couch, and Williams, *Plating*, 1950, **37**, 36, 161 and *J. Research Nat. Bur. Stand.*, 1950, **44**, 109; *Met. Abs.*, 1949–50, **17**, 582, 669, 738.
—E. J.

***Silver Plating on Inconel.** L. J. Cook (*U.S. Atomic Energy Commission Publ.*, 1950, (**UCRL-1068**), 5 pp.).—C. describes a process for cleaning the metal before laying down a flash of Ni, followed by Ag plating in a bath contg. AgCN, KCN, and K_2CO_3. The deposits had excellent adherence, were fine grained, and even when they were as thin as 0·0002 in., the elect. conductivity was $\sim$95% of the value for pure bulk Ag.—B. W. M.

New Electrolytic Line Boosts Nation's Tinplate Capacity. —— (*Steel*, 1950, **126**, (20), 104, 106).—A new plant of Weirton Steel Co., Weirton, W. Va., for electro-tinning is described.—E. J.

***Metallography of Electro-Tinplate.** (Davies and Hoare). See col. 122.

***The Electrodeposition of Bright Tin–Nickel Alloy Plate.** N. Parkinson (*Electrodepositors' Tech. Soc., Advance Copy*, 1951, **27**, (4), 23 pp.).—A method of depositing bright coatings of Sn–Ni alloy is described. The soln. used is : $SnCl_2.2H_2O$ 50, $NiCl_2.6H_2O$ 300, NaF 28, NH_4HF_2 35 g./l. The effect of varying the compn. of the soln. was studied and is described in detail. The compn. of the deposit is almost independent of variations in c.d. or temp. Variations of 20–30% in the content of either metal in the soln. do not appreciably affect the compn. of the alloy deposited. The chief factor which does affect the alloy compn. is the total F content of the electrolyte. Operated at 65° C. and an average cathode c.d., a bright deposit is obtained over irregular objects at a current efficiency of nearly 100%. The broadest bright-plating range is obtained at high rather than low temp. The presence of free HF and of alkali metal ion, preferably Na, is necessary for the best results. Alloy anodes may be used or else separate Ni and Sn anodes supplied at different voltages. The disadvantage of the use of separate anodes is the fact that special precautions are necessary to minimize the effect of chem. reaction between the Sn anodes and the soln. The deposit obtained is hard and extremely resistant to atmospheric tarnishing. It is almost immune from attack by inhibited conc. HCl and by conc. HNO_3. The deposit has none of the blueness of Cr nor the yellowness of Ni; 0·0005 in. is considered to be an adequate thickness on non-ferrous metals, but on steel a Cu undercoat of this thickness should be put on first, followed by an equally thick deposit of the alloy plate. 6 ref.—G. T. C.

***An X-Ray Study of Tin–Nickel Electrodeposits.** (Rooksby). See col. 122.

Continuous Electrogalvanizing. —— (*Metal Ind.*, 1951, **78**, (24), 484–489).—A description is given of the construction and operation of an electrogalvanizing plant at Hamilton, Lanarkshire. This is probably the largest plant of its kind in the world.—J. H. W.

Metallic Coatings by Electrodeposition. Paul Pic (*Ingénieurs et Techniciens*, 1950, (28), 385–388).—P. describes three methods for the electrodeposition of Cu, and a method for Ni.—G. A. K.

***†Study of Electrolytic Deposition in Very Dilute Solution. —I.–II.** André Coche (*J. Chim. Phys.*, 1951, **48**, (3/4), 135–145; 146–149).—[I.—] Conditions controlling the electrodeposition of radio-elements or of other elements using radioisotopes as indicators, from very dil. soln. are discussed in much detail. Amongst matters studied are : (1) the effects of concentration of the radio-element and the nature of the electrode upon the critical deposition voltage; (2) the critical potential for the deposition of Po upon Mo, W, and Ta; (3) the critical potential for the deposition of Pb upon various metals, e.g. Cu, Bi, Ni, Ta, Au, Ag, and Pt at dilutions of 10^{-10} *N*; (4) the critical potential for the deposition of Bi upon Au, Pt, and Ta, at dilutions of 10^{-13} *N*; (5) the critical potential of Po at 10^{-13} *N* dilution; and (6) variation of the deposition potential of Pb upon Pt with concentration. [II.—] Apparatus for maintaining the cathode voltage const. over long periods of electrolysis of very dil. soln. of radio-elements, &c., is described. Appn. of the device are indicated. Au electrodes used in the electrolysis of HNO_3 soln. using a current $\ngtr 1{\cdot}5$ m.amp. have been maintained const. to within $\pm$0·005 V. for 15–20 hr.—J. S. G. T.

Brighter Finishes with "PR" Plating. George W. Jernstedt (*Steel*, 1950, **126**, (18), 86–88, 108, 110, 113–114, 116).—Cf. *Met. Abs.*, 1950–51, **18**, 770. The PR plating process consists essentially of periodically reversing the D.C. plating current for a definite time interval. With the use also of a proprietary addn. agent, the prodn. of bright Cu finishes of high lustre and uniform *d* is described. J. also quotes some useful information on plating baths in general.—E. J.

Plating and Finishing Tubing at Spang-Chalfant. —— (*Metal Finishing*, 1951, **49**, (5), 65).—A brief description is given, with very little detailed information.—G. T. C.

Electroforming. —— (*Product Eng.*, 1950, **21**, (8), 145–147).—The prodn. of components, e.g. capillary tubing, by electrodeposition is described. Electrodeposition on non-metallic bases is also discussed.—E. C. P.

***Studies in the Discontinuities of Electrodeposited Metallic Coatings.—III.** S. C. Shome and U. R. Evans (*Electrodepositors' Tech. Soc., Advance Copy*, 1951, **27**, (1A), 30 pp.).—Cf. *ibid.*, (1); *Met. Abs.*, 1950–51, **18**, 715. The available methods of assessing the porosity of metallic coatings are briefly reviewed, and it is shown that none of them is entirely satisfactory. The electrochem. basis of the methods is discussed, and a new method is described in detail which employs a soln. contg. 3% NaCl by weight and 1·5% by vol. of H_2O_2 (20 vol.). A small quantity of this soln. is poured on to the specimen, and after 10 min., it is washed off with a stream of tap-water, the specimen dried in air, and the rust spots counted. It is shown that pure Ni foil is not attacked at all by this reagent after 10 min. exposure. The test is shown to reveal a smaller number of pores than the ferroxyl test, and possible reasons for this are put forward. A number of potential measurements were carried out, and it was shown that the potential of Ni-plated steel moves in the cathodic direction as the thickness of the coatings increases. The change in potential is small in the presence of NaCl or ferroxyl soln., but is marked if the NaCl soln. also contains H_2O_2. 7 ref.—G. T. C.

Report of [A.S.T.M.] Committee B-8 on Electrodeposited Metallic Coatings. —— (*Proc. Amer. Soc. Test. Mat.*, 1950, **50**, 181–183).—T. G.

***Some Applications of Interferometry to the Examination of an Electrodeposited Film.** (Tolansky). See col. 150.

Quality Control in the Making of Plating Rectifiers. Walter A. Raymond (*Metal Finishing*, 1951, **49**, (5), 68–70).—Various recommendations are made on elect. testing and other miscellaneous tests which should be carried out.—G. T. C.

Publicizing the Plating Industry [in U.S.A.]. W. M. Phillips (*Metal Finishing*, 1951, **49**, (5), 62, 73).—A brief survey of what is being done to promote public appreciation and understanding of the metal-finishing industry.
—G. T. C.

9—ELECTROMETALLURGY AND ELECTROCHEMISTRY

(Other than Electrodeposition.)

***The Electrochemical Behaviour of Aluminium. II.—In Solutions of Iron Sulphate.** J. V. Petrocelli (*J. Electrochem. Soc.*, 1951, **98**, (5), 183–186).—Cf. *ibid.*, 1950, **97**, 10; *Met. Abs.*, 1949–50, **17**, 585. The kinetics of oxidation–reduction electrode reactions are discussed, and it is shown that the electrode reactions may be treated as rate processes and a general expression for the electrode potential/c.d. relationship is derived. This equation shows how the polarization curves may behave under conditions where concentration overvoltage and activation overvoltage are involved. The view is expressed that the use of this equation may allow such factors as "exchange current" and "transfer coeff." to be evaluated. 11 ref.—G. T. C.

***The Temperature Coefficient of the Electrode $Cu|Cu^{++}$.** Maurice Bonnemay and James Fairbank (*J. Chim. Phys.*, 1951, **48**, (3/4), 152–160).—The measurement of the temp. coeff., at const. activity, of a metal electrode in a soln. of one of the salts of the metal, presents difficulties owing to: (1) the variation with temp. of the activity of the soln., (2) the temp. coeff. of the potential at liq. junctions, and (3) variation with temp. of the potential at the reference electrode. A method of eliminating these difficulties is discussed (B., *ibid.*, 1949, **46**, 176). Experimental results relating to a Cu amalgam electrode in $Cu(NO_3)_2$ and $CuSO_4$ soln. to which various small amounts of H_2SO_4 or Na_2SO_4 are added, are given. These confirm results previously obtained with H, Cd, and Cd amalgam electrodes. The whole of the experimental results, plotted as a graph having temp. as abscissa and e.m.f. as ordinate, fall on one straight line, irrespective of the nature of the anion, in marked contradistinction to results with pure Cu salt soln. contg. no additional substances, in which case different straight lines are obtained according as $Cu(NO_3)_2$ or $CuSO_4$ is used as electrolyte.—J. S. G. T.

***Cathode Polarization Potential During Electrodeposition of Copper. I.—Non-Reproducibility in Acid Copper Sulphate Solutions.** (Shreir and Smith). See col. 135.

***Electrolytic Dissolution of Copper in Ammoniacal Electrolytes.** Fred A. Schimmel (*J. Phys. Colloid Chem.*, 1950, **54**, (6), 841–847).—Ammoniacal electrolytes have been used successfully in industry for Cu recovery. S. has investigated the relation between the c.d. and the valency at which Cu enters and leaves various ammoniacal soln. Some detn. of potentials of Cu in ammoniacal electrolytes were also made; the cathode potential becomes more negative, the anode potential more positive, with increasing c.d.—D. M. D.

***Oxide-Film Formation on the Surface of Metals in Aqueous Solutions and the Evaluation of Their Standard Potentials. II.—The Mercury Electrode.** S. E. S. El Wakkad and T. M. Salem (*J. Phys. Colloid Chem.*, 1950, **54**, (9), 1371–1383).—In continuation of work by Tourkey and El W. (cf. *J. Chem. Soc.*, 1948, 740; *Met. Abs.*, 1948–49, **16**, 569), El W. and S. discuss the probable existence of an oxide film on the Hg electrode. They measured the potential of Hg out of contact with air and obtained the value 0·7971 V. at 25° C. 25 ref.
—D. M. D.

***Hydrogen Exchange Current and Hydrogen Overvoltage on Smooth Platinum.** V. L. Kheifets and N. E. Polyakova (*Zhur. Priklad. Khim.*, 1949, **22**, (8), 801–808).—[In Russian]. Kh. and P. deduce that for strong soln. of acids the overvoltage $\eta = b \log \frac{i}{i_{0a_{H^+}}}$, where b is a const., i the c.d., and $i_{0a_{H^+}}$ the exchange current. Hence, the const. a in Tafel's equation ($\eta = a + b \log i$) depends on the activity of H ions in the soln. (a_{H^+}) and on the potential of zero discharge of metal (ϕ_0). Also, the exchange currents in soln. of different activity are related by the equation: $\frac{i_{0a'_{H^+}}}{i_{0a''_{H^+}}} = \left[\frac{a'_{H^+}}{a''_{H^+}}\right]^{\beta}$, where β is a const. These relations were confirmed by saturating Pt electrodes with H by polarization in H_2SO_4 for 7–12 days at 25° C. and then measuring η at various c.d., without interrupting the current. On plotting η against $\log i$, straight lines were obtained with 1·0, 2·0, and 4·0N-H_2SO_4; from the above equations, $i_{0a_{H^+}} = 0{\cdot}117$, $0{\cdot}116$, and $0{\cdot}224 \times 10^{-5}$ amp./cm.², resp., and $\beta \cong 0{\cdot}5$. It was shown that at const. c.d., $\eta = \text{const.} - \beta b \log a_{H^+}$, and at const. η, $i = \text{const.} \sqrt{a_{H^+}}$. Measurements of η in ~2N-H_2SO_4 confirmed that there is a linear relationship between η and $\log i$, between $\log i_{0a_{H^+}}$ and $1/T$ (T being the abs. temp.), and between η and T.—G. V. E. T.

The Electrolytic Zinc Industry. Harry Hey (*Proc. Fourth Empire Min. Met. Congr.*, **1950**, (II), 809–823; discussion, 1047–1093).—See *Met. Abs.*, 1949–50, **17**, 282.

***Electrodeposition of Metals from Fused Quaternary Ammonium Salts.** Frank H. Hurley and Thomas P. Wier, Jr. (*J. Electrochem. Soc.*, 1951, **98**, (5), 203–206).—Fused mixtures of ethyl pyridinium bromide and metallic chlorides were used, and it was found that the following metals could be electrodeposited from such mixtures: Ag, Cu, Bi, Pb, Sn, Ni, Co, Cd, Fe, Zn, and Al. Metals could also be deposited from other mixtures, except where non-ionic complex compounds were formed. Such mixtures are fused benzyl pyridinium bromide with metallic nitrates or sulphates. The mixtures were held at 135° ± 5° C. and a voltage of

6–12 V. used. No deposit could be obtained when $MnCl_2$ was used. The chlorides of Na, Li, Ba, and Ce were practically insoluble in the quaternary salts used. A mixture contg. $BeCl_2$ produced only a brown deposit on the cathode. More extensive investigation was carried out on the deposition of Al. It was shown that a compound $EtPyBr.AlCl_3$ is formed and that a low-melting eutectic (−40° C.) is formed at 67 mol.-% $AlCl_3$. Al could be deposited from mixtures in the compn. range 54–70 mol.-% $AlCl_3$. Smooth plating of Al on Fe, steel, Cu, bronze, brass, Pt, Pb, and Sn could be obtained using an Al anode. The best deposits were obtained when the bath was near the eutectic compn. and at a temp. of 125° C., using a c.d. of 0·5 amp./dm.². Similar results were obtained using ethylene dipyridinium dibromide mixtures. The state of the ionization of the compound $EtPyBr.AlCl_3$ in C_6H_6 was investigated by means of migration and conductivity experiments. 7 ref.—G. T. C.

[Electrolytic] Production of Metal Powders. E. Mehl (*Metal Treatment*, 1950, **17**, (62), 118–122, 124, 126, 128).—Electrolytic deposition of metal powders is discussed and details are given for the prodn. of Cu, Fe, Zn, Ni, Sn, and Pb by this method. 36 ref. J. W. C.

10 — REFINING

***Experiment in the Metallurgy of [Reduction of] Chromium by Hydrogen in the Solar Furnace.** F. Trombe and M. Foëx (*Rev. Mét.*, 1951, **48**, (5), 359–362).—T. and F. point out the difficulties encountered in the reduction of Cr_2O_3 by H_2, owing to the small O_2 content of the gas permissible and the high temp. required. Working at 2000° C. and 150 kg./cm.² or at 2500° C. and 5 kg./cm.² did not produce very pure metal. They experimented with a solar furnace having a fixed parabolic mirror 2 m. in dia. and a focal length of 85 cm. (the arrangement is described in another paper (*ibid.*, p. 353)). The Cr_2O_3 is placed in a silica boat, supported on a silica stand, and enclosed in an inverted Pyrex flask of 2 l. capacity, purified H_2 being led direct on to the oxide. The sun's rays are so concentrated on to the specimen that the Pyrex container only reaches a temp. of ~300° C. The resulting metal was in the form of a bead ½ mm. thick. As a test of purity, its elect. resistance was measured from −195° to +100° C. and showed the same anomaly as electrolytically prepared 99·99% Cr. The metal is so pure because it is in contact only with its oxide and, in any case, the silica boat is only slightly heated. 11 ref.
—J. H. W.

A Process for the Recovery of Gold from Activated Carbon by Leaching and Electrolysis. J. B. Zadra (*U.S. Bur. Mines Rep. Invest.*, 1950, **(4672)**, 47 pp.).—Z. describes a process for the extraction of Au from Au-laden C which enables the activated C to be used again. The process consists of leaching the Au-laden C with a caustic Na_2S soln. contg. 30 g. Na_2S and 40 g. NaOH/l. and subsequently recovering the Au from the soln. by electrolysis. The process is rapid and the C can be re-used 11 times without reactivating. A circular-type electrolytic cell was used consisting of stainless-steel anodes and cathodes. Stainless steel and charred excelsior were both used as Au-collecting media, though it was found that the latter had the disadvantage that because of its high elect. resistance it was necessary to precoat it with Au before use. Full details of the electrolytic cells are given. The Au-collecting media were reduced to Au by burning off the excess excelsior in a graphite crucible or melting furnace and adding while hot a suitable flux. 46 ref.—E. C. P.

Refining Metals and Alloys by Filtration. H. W. St. Clair (*U.S. Bur. Mines Rep. Invest.*, 1949, **(4614)**, 16 pp.).—St. C. describes work carried out in Germany during the war on the refining of metals and alloys by filtering the molten metal through suitable filters such as very fine brass gauze, porous stone, &c., thereby removing solid particles of intermetallic compounds formed by the impurities. Results are given for the removal of: (1) Sb, Sn, and Cu from Pb alloys, (2) Fe from Zn alloys, (3) Fe from Sn, (4) Fe from Al alloys, and (5) non-metallic impurities from Al alloys.—E. C. P.

11 — ANALYSIS

***Studies in the Determination of Arsenic. V.—Volumetric Determinations of Reduced Metallic Arsenic.** Yachiyo Kakita (*Sci. Rep. Research Inst. Tôhoku Univ.*, 1950, [A], **2**, (3), 483–490).—[In English]. In previous work, K. had studied the sepn. and detn. of As using NaH_2PO_2 and $SnCl_2$, the reduced As being dissolved in excess of standard KI soln. and the excess titrated against standard $Na_2S_2O_3$ soln. The appn. of $K_2Cr_2O_7$, $KMnO_4$, chloramine, $Ce(SO_4)_2$, and KIO_3 to the detn. of As reduced by $SnCl_2$ or NaH_2PO_2 was now examined. In the dichromate method, As is dissolved in $K_2Cr_2O_7$ soln. acidified with H_2SO_4; H_3PO_4 and diphenylamine (indicator) are added, and the soln. titrated against $FeSO_4.(NH_4)_2SO_4$. In the chloramine method, As is dissolved in chloramine, after washing with 6*N*-HCl and H_2O. After soln. *N*/30 neutral arsenite soln., ~0·5 g. $NaHCO_3$, a small amount of KI, and starch soln., are added, and the excess of arsenite titrated against *N*/30 chloramine soln., until the soln. becomes blue. Both methods are recommended. The other methods studied are also accurate, but are rather difficult to carry out.
—J. S. G. T.

The Analytical Chemistry of Beryllium: Bibliography. C. V. Banks, J. H. McClure, and H. V. Meek (*U.S. Atomic Energy Commission Publ.*, 1948, **(AECU-10)**, 23 pp.; also, **(ISC-29)**).—An exhaustive *bibliography* of 266 ref. in the literature from 1798 to 1948, dealing with studies on the analysis of Be and Be compounds.—B. W. M.

Electro-Analysis of Copper. S. Skowronski (*Amer. Soc. Test. Mat. Bull.*, 1951, **(174)**, 60–65).—The economic importance and the historical development of the method are outlined. Types of electrodes and compn. of electrolyte and the times of electrolysis necessary at various c.d. are discussed. The effects of interfering elements are then considered, particularly Ag, Se, Te, As, Sb, Bi, Mo, Sn, Fe, Mn, Cr, and also halogens, nitrous oxides, and organic matter. It is shown that excessive amounts of a number of these materials can cause inaccurate results. The effect of variation of temp. of electrolysis and the detn. of small quantities of Cu are mentioned.—P. T. G.

The Photometric Determination of Copper in Aluminium Alloys with Sodium Diethyldithiocarbamate. E. C. Mills and S. E. Hermon (*Analyst*, 1951, **76**, (902), 317–318).—A note. It is pointed out that Na diethyldithiocarbamate, in spite of its disadvantages (cf. Williams, *ibid.*, 1950, **75**, 425; *Met. Abs.*, 1950–51, **18**, 202). can be and is successfully used for the routine detn. of Cu in Al alloys, and that interference by Ni can readily be overcome by suitable adjustment of the NH_3 concentration in the final soln.—F. M. L.

***The Analysis of Manganese Bronze.** George Norwitz (*Analyst*, 1951, **76**, (902), 314–316).—The sample is dissolved in a cold mixture of HNO_3 and $HClO_4$, and H_2O_2 is added to the soln. to oxidize lower N oxides. Cu and Pb are determined electrolytically in the soln., using a Pt gauze cathode and a Pt spiral anode. Mn, Ni, Fe, Al, and Sn are determined in known manner in aliquots of the electrolyte.—F. M. L.

***A Rapid Polarographic Determination of Copper, Cadmium, and Zinc in Silver-Base Alloys.** E. G. Ford (*Canad. J. Technol.*, 1951, **29**, (2), 61–65).—Cu, Cd, and Zn in Ag-base alloys may be determined in $<\frac{1}{2}$ hr. with an accuracy of

$> \pm 1\%$ by using the following method. A 0·1-g. sample of the alloy is dissolved in 10 ml. of $8M$-HNO_3 and 5 ml. of $3M$-H_2SO_4. The soln. is heated until SO_3 fumes appear and then cooled and diluted to 250 ml. exactly. A 25-ml. aliquot is transferred to a 100-ml. flask and the following are added: 20 ml. $5M$-NH_4Cl, 20 ml. $5M$-NH_4OH, 5 ml. 0·2% gelatin, 1 g. Na_2SO_3. The soln. is made up to the mark with H_2O, and a portion is transferred to a polarographic cell. The soln. is polarographed from −0·1 to −1·6 V. against the standard calomel electrode. The waves at −0·24 and −0·50 V. are measured for the Cu^{++} and Cu^{+} complexes, resp. The waves at −0·81 and −1·35 are measured for Cd and Zn, resp. If Ni is present, its wave may be measured at −1·10 V. The wave-heights are converted to current values and then to weights or percentages by direct calibration against standard soln. or by ref. to published diffusion current const.—R. W. R.

Colorimetric Determination of Iron: A Review of Known Methods.—II. T. S. West (*Metallurgia*, 1951, **43**, (259), 260–261, 263–264).—Cf. *ibid.*, (258), 204; *Met. Abs.*, this vol., col. 50. The detn. of Fe with thiocyanate, ferron, tiron, salicylic acid, sulphosalicylic acid, and salicylaldoxime are described in detail. 60 ref.—F. M. L.

***Catalytic Analysis: Microdetermination of [X.—] Osmium by a Reaction Between Ceric Sulphate and Arsenious Acid; [XI.—] Osmium by a Reaction Between Potassium Permanganate and Arsenious Acid; [XII.—] Vanadium with Pulfrich's Photometer.** Takanobu Shiokawa (*Sci. Rep. Research Inst. Tôhoku Univ.*, 1950, [A], **2**, (3), 443–445; 446–448; (4), 613–617).—Cf. *ibid.*, (2), 293; *Met. Abs.*, 1950–51, **18**, 723. [In English]. [X.—] The catalytic action of Os upon the reaction between soln. of $Ce(SO_4)_2$ and As_2O_3, in which the yellow ceric ion is reduced to the colourless cerous ion was studied colorimetrically in Ukena's colorimeter. The detn. of 0·3–1·8 γ of Os is possible by the method developed; 10 mg. or less of Cu^{++}, Fe^{3+}, Hg^{++}, or Ti^{4+} had no effect on the detn., but >5 mg. of Co^{++}, Ni^{++}, Mo^{6+}, or Mn^{++} affected the time measurements involved. The method is applicable to the detn. of Os in the presence of (unspecified) metallic ions. [XI.—] The catalytic action of Os on the reduction of $KMnO_4$ to the colourless state by As_2O_3 was studied by Ukena's colorimeter. The detn. of 1·5–7·5 γ of Os is possible by the method developed. Suitable concentrations and conditions are specified. The effects of added metallic ions were the same as in [X.]. [XII.—] V^{4+} catalyses sensitively the reaction between $KClO_3$ and KI in H_2SO_4 soln. The relation between the time required for a definite coloration due to iodine–starch and the amount of V was determined by Pulfrich's photometer, and a method was developed for determining as little as 5–50 γ of V. Effects of temp. and various ions were studied.—J. S. G. T.

***The Determination of the Oxygen Content of Metals by the Carbon Monoxide Method.** D. Lipkin and M. L. Perlman (*U.S. Atomic Energy Commission Publ.*, 1946, (**MDDC-294**), 15 pp.; also (**LADC-142**)).—A detailed description of the technique and apparatus for determining the O_2 content of small samples by vacuum fusion in graphite and measuring the O_2 as CO using a micro-gas-analysis apparatus as designed by Prescott (*Indust. and Eng. Chem.* (*Analyt. Edn.*), 1939, **11**, 230). The results obtained on various steel samples, U, and oxides of U and Ta are included, the size of the specimen used varying from 0·14 to 40 mg.—B. W. M.

***The Determination of Palladium and Nickel with Alpha-Furildioxime.** S. A. Read and C. V. Banks (*U.S. Atomic Energy Commission Publ.*, 1948, (**AECD-1819**), 8 pp.).—α-Furildioxime is more sensitive for detecting Pd and Ni than dimethylglyoxime, 1 part Pd in 20×10^6 and 1 part Ni in 6×10^6 being easily detected in aq. soln. The reagent may be used for estimating Pd, and can be used to separate the metal from Ni, Pt, Co, Fe, and many others with a single pptn. from strong mineral acid soln. The Pd precipitate may be dried and weighed or ignited to the metal. The reagent may not be used for estimating Ni, since the compn. of the Ni α-furildioxime complex varies with the *p*H of the soln. from which it is precipitated.—B. W. M.

***The Colorimetric Determination of Palladium with 2-Mercapto-4 : 5-dimethylthiazole.** D. E. Ryan (*Analyst*, 1951, **76**, (902), 310–313).—Small amounts of Pd are determined absorptiometrically using 2-mercapto-4 : 5-dimethylthiazole. Pt interferes in the detn. and may be separated by extracting into, preferably, ethyl acetate the Pd complex of *p*-nitrosodiphenylamine (cf. *ibid.*, (900), 167; *Met. Abs.*, 1950–51, **18**, 675).—F. M. L.

***Determination of Scandium with 8-Quinolinol [Oxine].** Lewis Pokras and Peter M. Bernays (*Analyt. Chem.*, 1951, **23**, (5), 757–759).—A precipitate with a very favourable factor (7·24%) is given with Sc and oxine.. Other elements precipitated by oxine must be absent.—F. M. L.

***Determine Silicon, Copper, and Iron in Monel Metal.** W. L. Miller and George Norwitz (*Amer. Foundryman*, 1951, **19**, (5), 80).—A 1-g. sample is dissolved in 10 c.c. HNO_3 (70%), 20 c.c. $HClO_4$ (70%), and 5 c.c. H_2O and evaporated to fumes of $HClO_4$. After dilution with 60 c.c. H_2O, SiO_2 is filtered off; 7 c.c. HNO_3 (70%) are added, the filtrate is diluted to 200 c.c. and electrolysed for Cu at c.d. of 2 amp./dm.². The electrolyte is further diluted to 250 c.c., and 5 c.c. of it transferred into a 100-c.c. flask. The Fe is determined colorimetrically after adding 1 c.c. H_2O_2 (3%) and diluting to 100 c.c. with NaCNS (4%).—V. K.

***Determination of Silica in Beryllium Metal Powder.** J. F. O'Leary (*U.S. Atomic Energy Commission Publ.*, 1948, (**MDDC-1707**), 2 pp.).—The powder is dissolved in *aqua regia* and the insoluble residue filtered off, washed, and ignited. The residue is fused with anhydrous Na_2CO_3, dissolved in HCl and the soln. added to the filtrate from the insoluble residue. The whole is evaporated to dryness at <120° C. and the residue taken up in HCl and decanted through an ashless filter-paper. The insoluble residue is again treated with acid and finally transferred to the filter, which is washed thoroughly with hot water and ignited to const. weight in Pt. The residue is treated with a few drops of dil. H_2SO_4 and 5 ml. HF, this treatment being repeated once and the crucible re-ignited to const. weight. The loss in weight is total SiO_2 and the residue is BeO.—B. W. M.

***Separation of Thorium from Uranium.** K. V. S. Krishnamurty and Bh. S. V. Raghava Rao (*Rec. Trav. Chim.*, 1951, **70**, (5), 421–424).—Sepn. may be achieved with anisic acid. From neutral soln. Th may be freed from twice its quantity of U in a single pptn. and ~8 times as much U in 2 pptn.
—J. W. C.

***Determination of Tin in Ferro-Tungsten and Tungsten Metal.** —— (*J. Iron Steel Inst.*, 1951, **168**, (1), 51–56).—A report of the British Iron and Steel Research Association Methods of Analysis Committee. Two methods for the detn. of Sn in ferro-W and in W powder are described and discussed. (1) The powdered sample (2 g.) is dissolved in a covered Pt crucible in 5 ml. H_2SO_4 (1 : 1), 5 ml. HF, and enough HNO_3 (added dropwise) to complete the dissoln. 10 ml. H_2SO_4 (1 : 1) are then added and the sample is evaporated to SO_3 fumes; fuming is continued for 1 hr. The residue is extracted in 100 ml. citric acid (20%) and the citric acid then neutralized with NH_4OH ($d = 0·880$); 10 ml. excess NH_4OH are added. After heating to ensure soln. of the tungstic acid, the soln. is acidified with H_2SO_4 (1 : 1) and 5 ml. excess added. The soln. is diluted to 450 ml. and boiled; 2 ml. $(NH_4)_2MoO_4$ (1%) are added, and a brisk stream of H_2S is passed for 1 hr. After standing, the precipitated sulphides are filtered off and washed with sulphide wash. The precipitate is dissolved in 25 ml. HNO_3 ($d = 1·42$), 10 ml. H_2SO_4 ($d = 1·84$) and evaporated to SO_3 fumes. The fumed soln. is extracted with 100 ml. H_2O and 15 ml. HCl ($d = 1·16$) and boiled. Any tungstic acid precipitated is filtered off and washed with HCl (1 : 19). 5 ml. $FeCl_3$ soln. are added and NH_4OH (1 : 1) is added to the boiling liquid until it is alkaline. The precipitate is filtered off through a No. 541 paper and washed with hot H_2O. The precipitate is dissolved in hot HCl (1 : 1) and placed in a special flask. This soln. is diluted to 200 ml. after addn. of 2 ml. $SbCl_3$ soln. When nearly boiling 1 g. Al is added. A bung and delivery tube are fitted to the flask and the soln. boiled for 30 min. the condensate being discarded. The end of the delivery tube is then immediately immersed in 200 ml. $NaHCO_3$ soln. (10%), which is thus sucked back into the flask. The flask is cooled in running H_2O. 10 ml. KI soln. (10%), and 20 ml. starch soln. are then added, and the soln. is titrated with KIO_3 soln. ($N/100$). (2) The sample is dissolved and fumed for 1 hr. as in Method 1. After cooling, the contents of the crucible are transferred to a beaker by means of a jet of H_2O; the crucible is rinsed with HCl ($d = 1{\cdot}16$) and H_2O and the total vol. adjusted to 100 ml. After boiling for 10 min. the soln. is allowed to settle overnight. The tungstic acid precipitate is filtered through a No. 540 paper and washed with HCl (1 : 19). The filtrate is neutralized with NH_4OH (1 : 1) and 10 ml. excess is added. The soln. is boiled for 2 min., filtered through a No. 541 paper, and the precipitate washed with hot H_2O. The precipitate is dissolved in 140 ml. hot HCl (1 : 1). 2 ml. $SbCl_3$ are then added and the soln. treated as in Method 1. Method 1 is more accurate but Method 2 is better suited to routine use.—R. W. R.

***Colorimetric Determination of Tungsten: Study of Variables Involved in Stannous Chloride–Thiocyanate Method.** Harry Freund, Mark L. Wright, and Robert K. Brookshier (*Analyt. Chem.*, 1951, **23**, (5), 781–784).—The effect of acid and chloride concentration on the results obtained in the detn. spectrophotometrically of small amounts of W have been investigated. Consistent and accurate results may be obtained by careful control of conditions. The sample contg. 1–1·5 mg. W in 15 c.c. is placed in a 100-c.c. flask and 10 c.c. conc. H_2SO_4 are added. After cooling, 10 c.c. HCl and 5 c.c. $2M$-$SnCl_2$ are added and the soln. is warmed; after again cooling 10 c.c. $2M$-KCNS is added and the soln. made up to 100 c.c. After 15 min. the extinction is determined at 400 mμ.—F. M. L.

***Determination of Uranium in Solution by X-Ray Absorption.** T. W. Bartlett (*Analyt. Chem.*, 1951, **23**, (5), 705–707).—0·1–10 g./l. U can be determined with reasonable accuracy using an X-ray photometer with special 19-mm.-deep cells. Interference of other elements increases with increasing at. no. and, generally, a chem. sepn. of U is necessary. 10 ref.—F. M. L.

***Analysis of Uranium Solutions by X-Ray Fluorescence.** L. S. Birks and E. J. Brooks (*Analyt. Chem.*, 1951, **23**, (5), 707–709).—X-ray fluorescence analysis is shown to be applicable to low-concentration U soln. Sepn. of impurities, except heavy elements, e.g. Pb in large amount, is unnecessary.—F. M. L.

***Polarographic Characteristics of +2 and +3 Vanadium. II.—Polarography in Complexing Solutions.** James J. Lingane and Louis Meites (*J. Amer. Chem. Soc.*, 1951, **73**, (5), 2165–2170).—Cf. *ibid.*, 1948, **70**, 2525; *Met. Abs.*, 1949–50, **17**, 285. Ionic states and polarographic behaviours of complexes formed by vanadous and vanadic ions in a wide variety of media are discussed. Data on the half-wave potentials and diffusion current const. are presented.—J. R.

***New Spot Tests for Zinc.** R. Belcher, A. J. Nutten, and W. I. Stephen (*Analyst*, 1951, **76**, (903), 378–379).—The presence of Zn is denoted by a red-violet coloration when a soln. of $K_3Fe(CN)_6$ and naphthidine hydrochloride or 3 : 3′-dimethylnaphthidine hydrochloride is added to the test soln. Ions that form coloured precipitates with $K_3Fe(CN)_6$ or oxidize the reagents must be absent.—F. M. L.

Chemical Analysis of Zinc Dust (Metallic Zinc Powder). —— (*Proc. Amer. Soc. Test. Mat.*, 1950, **50**, 237–243).—Part of Report of A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products. See *Met. Abs.*, 1950–51, **18**, 204.

***Electrolytic and Polarographic Determination of Zinc in Thorium.** J. H. Patterson and C. V. Banks (*U.S. Atomic Energy Commission Publ.*, 1948, (**MDDC-1708**), 13 pp.).—Cf. *Analyt. Chem.*, 1948, **20**, 897; *Met. Abs.*, 1948–49, **16**, 355. —B. W. M.

***The Quantitative Spectrographic Analysis of the Rare-Earth Elements.** V. A. Fassel and H. A. Wilhelm (*U.S. Atomic Energy Commission Publ.*, 1947, (**MDDC-1512**), 16 pp.).—Cf. *J. Opt. Soc. Amer.*, 1948, **38**, 518; *Met. Abs.*, 1948–49, **16**, 422.—B. W. M.

The Quantitative Spectrographic Analysis of the Rare-Earth Elements. III.—Determination of Major Constituents in Complex Mixtures. V. A. Fassel and H. A. Wilhelm (*U.S. Atomic Energy Commission Publ.*, 1948, (**MDDC-1777**), 8 pp.).—Cf. F., *J. Opt. Soc. Amer.*, 1949, **39**, 187; *Met. Abs.*, 1949–50, **17**, 96.—B. W. M.

***Amperometric Titrations with *m*-Nitrophenylarsonic Acid.** I. M. Kolthoff and Ralph A. Johnson (*J. Electrochem. Soc.*, 1951, **98**, (4), 138–145).—The polarography of certain arsonic acids has been studied in connection with the amperometric titration of metal ions with these acids. The acids are reduced to substituted arsines in a six-electron reduction. *m*-Nitrophenylarsonic acid is shown to be a suitable reagent for the amperometric titration of certain cations, which precipitate as arsonates. In these titrations the excess of reagent line is determined at potentials at which the nitro group is reduced to the hydroxylamino or to the amino. The effect of HCl concentration on these reductions is discussed, and concentrations are given at which the arsonic group is reduced. Procedures are given for the amperometric titration of uranyl, Th, Zr, and Sn^{IV}. Qual., Ti^{IV} behaves like Sn^{IV}. 14 ref.—G. T. C.

Analysis for Industry. R. Belcher and R. Goulden (*Indust. Chemist*, 1951, **27**, (316), 204–205; (317), 249–251).—Recent developments in electro-analytical techniques are reviewed. B. and G. discuss some newly developed electrodes and describe recently introduced methods for the detn. of Sb (from H_2SO_4/HCl soln. contg. hydroxylamine hydrochloride), Bi (from $HClO_4$ contg. hydrazine sulphate), and Cd (from H_2SO_4, $HClO_4$, or HCl/HNO_3 soln. contg. gelatin). New developments in the detn. of Cu are also discussed, including the detn. of the element in the presence of Ag and Sn. 36 + 25 ref.—R. W. R.

Report of [A.S.T.M.] Committee E-3 on Chemical Analysis of Metals. —— (*Proc. Amer. Soc. Test. Mat.*, 1950, **50**, 433–439).—See *Met. Abs.*, 1950–51, **18**, 205.

Sampling of Ferrous and Non-Ferrous Alloys: A Bibliography. Albert C. Holler (*Amer. Soc. Test. Mat. Bull.*, 1951, (174), 66–67).—A *bibliography* prepared under the auspices of A.S.T.M. Committee E-3, on Chemical Analysis of Metals, contg. 52 ref. 19 of these refer to the theory of sampling, 5 to Al-base alloys, 9 to Cu-base alloys, 1 to precious metals, 3 to Sn- and Pb-base alloys, 2 to Zn-base alloys, and the remainder to ferrous metals.—P. T. G.

12—LABORATORY APPARATUS, INSTRUMENTS, &c.

(See also "Testing" and "Temperature Measurement and Control".)

[Equipment for] Producing Single Crystals of Metals. M. A. Steinberg (*J. Metals*, 1951, **3**, (5), 387–388).—The following equipment is described: (1) a furnace and elect. control system, and moulds for the prodn. of single crystals—0·25–1 in. dia. and 12–14 in. long—of Al, Sb, Bi, Pb, Mg, Sn, Zn, and the brasses, by the Bridgman method; and (2) a furnace for the prodn. of single crystals of refractory metals and alloys, i.e. Mo, Ni, W, &c., by a modification of the Andrade method—growth of an existing grain of a polycryst. specimen to a single crystal by movement through a temp. gradient without recourse to the nucleation of a new phase or recrystn. 5 ref. —E. N.

Vacuum Furnace for Metal Single Crystals. D. Lazarus and D. R. Chipman (*Rev. Sci. Instruments*, 1951, **22**, (3), 211–212).—An apparatus is described which has been used for the prodn. of single crystals of Cu.—E. J.

Determination of Small Thermal Expansion Coefficients by a Micrometric Dilatometer Method. B. S. Lement, C. S. Roberts, and B. L. Averbach (*Rev. Sci. Instruments*, 1951, 22, (3), 194–196).—A method is described for the measurement of small thermal-expansion coeff. in the vicinity of room temp.—E. J.

Apparatus for Measuring Young's Modulus and Decrement of Graphite and Metals. J. W. Michener and J. S. Handloser (*U.S. Atomic Energy Commission Publ.*, 1947, (**MDDC-1428**), 12 pp.).—The instrument is essentially an impedance bridge for measuring the A.C. resistance between the two electrodes of a quartz crystal which is part of a composite oscillator. The latter is made up of a quartz bar to which is glued, with beeswax, a specimen of the material under test, the cross-sections of both the quartz and the specimen being small compared with their lengths. The length of the quartz bar is such that when vibrating longitudinally its resonant frequency is near 35 kc./s. while the length of the specimen is adjusted to one-half wave-length at the resonant frequency of the crystal. The electronic circuits and method of working out the results are discussed in detail.—B. W. M.

Tungsten Wire Straightener. A. A. Jarrett (*U.S. Atomic Energy Commission Publ.*, 1948, (**MDDC-1646**), 2 pp.).—An apparatus for straightening W wire of 5–10 mils dia. by alternately heating and cooling the wire while under tension, is described. The wire is mounted on a wooden dowel, resistance-heated for periods of ~1 sec., the slack on heating being taken up by hand control.—B. W. M.

A Bridge Method for the Control of Electrolytic Polishing. L. F. Bates and C. D. Mee (*J. Sci. Instruments*, 1950, 27, (11), 317–318).—The use of a Wheatstone bridge circuit for electrolytic polishing enables optimum polishing conditions to be accurately controlled and maintained. To avoid irregularities in the c.d. distribution the anode is rotated slowly. A typical graph is shown of the cell resistance/cell voltage for a Si–Fe specimen with a chromic acid–acetic acid electrolyte. —P. C. L. P.

An Equipment for the Microscopic Examination of Metals and Crystals in Polarized Light at Temperatures from −130° to +35° C. J. C. Monier and R. J. Hocart (*J. Sci. Instruments*, 1950, 27, (11), 302).—An apparatus is described by which a microscopical specimen can be cooled by a current of cold air obtained from a flask of liq. air contg. an immersion heater. The specimen temp. was controlled to within 1° C. for 1 hr. at −45° C.; at lower temp. closer control was possible.—P. C. L. P.

A Low-Temperature Microscope Stage. R. G. Rhodes (*J. Sci. Instruments*, 1950, 27, (12), 333–334).—The same method of cooling the specimen is used in this apparatus as that described by Monier and Hocart (*ibid.*, (11), 302; preceding abstract). Temp. down to −100° C. can be readily attained and those down to −140° C. with more difficulty. Control to within ±0·1° C. is claimed. The use of liq. H to reach lower temp. is suggested.—P. C. L. P.

An X-Ray Method for the Study of Phase Changes at High Temperatures. H. T. Heal and H. Mykura (*Metal Treatment*, 1950, 17, (63), 129–135).—H. and M. describe an arrangement which enables the total intensity from one diffraction line of a given phase to be automatically recorded as a function of time. A bent-crystal monochromator and Seeman–Bohlin focusing camera are used, and the intensity is measured by a stationary Geiger–Müller counter, monitored to compensate for variations in tube output. Isothermal transformation curves for a 2½% Ni steel are given and suggestions made for other appn.—J. W. C.

A New Type of High-Temperature X-Ray Camera for Chemical Reactions in the Solid State. Teruichiro Kubo and Hiroshi Akabori (*J. Phys. Colloid Chem.*, 1950, 54, (8), 1121–1131).—K. and A. describe an X-ray camera in which the powdered sample is supported on a Pt-wire heating element. 14 ref.—D. M. D.

A Sheet Specimen Scanner for X-Ray Diffraction. D. J. Neil (*Canad. J. Technol.*, 1951, 29, (2), 84–86).—A simple design is described for an X-ray-diffraction scanner for determining the degree and type of preferred orientation in metal sheet specimens. The instrument consists essentially of a frame, an electric motor, the level winding mechanism from a fishing-reel (this imparts the horizontal motion), and a simple elevating mechanism. Back-reflection and transmission photographs at distances of 3 and 5 mm., resp., can be taken simultaneously. The glancing angle may be varied between 0 and 90°. The area scanned is ~2 cm.^{2}, and two scanning rates are available—2 or 4 cm.^{2}/hr.—R. W. R.

***An X-Ray Microbeam Technique. I.—Collimation.** P. B. Hirsch and [the late] J. N. Kellar (*Proc. Phys. Soc.*, 1951, [B], 64, (5), 369–374).—Experimental details are given of an apparatus designed for taking X-ray-diffraction photographs of cold-worked metals with very small beams (so as to investigate the fine structure). Pb glass capillaries were mostly used; the geometry of these, and various relevant factors, such as absorption and optimum divergence, are treated, and figures are given. 12 ref.—R. W. C.

***An X-Ray Microbeam Technique: II.—A High-Intensity X-Ray Generator.** P. Gay, P. B. Hirsch, J. S. Thorp, and [the late] J. N. Kellar (*Proc. Phys. Soc.*, 1951, [B], 64, (5), 374–386).—A high-intensity generator was built to reduce exposure time when using microbeams. It incorporates a rotating anode and an electron gun with adjustable bias, so that foci from 1 mm. to 50 μ width, and with 50× greater brightness than in commercial stationary anode tubes, can be obtained. The effects of varying shield bias, high tension and shield geometry on the focal dimensions and brightness were analysed experimentally, and various filament shapes are discussed. The mechanism of focusing is briefly treated. 12 ref.—R. W. C.

The Philips 100-kV. Electron Microscope. A. C. van Dorsten, H. Nieuwdorp, and A. Verhoeff (*Philips Tech. Rev.*, 1950, 12, (2), 33–51).—A comprehensive account of the construction and use of a 100-kV. electron microscope.—D. K. W.

A Geiger–Müller Photo-Counter Having a Ferro-Nickel Cathode, for Use with Ultra-Violet Radiation. J. Labeyrie (*J. Phys. Radium*, 1950, 12, (4), 569–570).—A letter. A photo-counter having a central longitudinal anode of Platinite (a Ni–Fe alloy), and a cathode deposited on the wall of the counter by evaporation and deposition of the anode *in vacuo*, very sensitive to ultra-violet radiation between 2000 and 3000 Å. and practically insensitive above 3600 Å., is described. 16 ref.—J. S. G. T.

13 — PHYSICAL AND MECHANICAL TESTING, INSPECTION, AND RADIOLOGY

The Significance of Mechanical Testing. E. W. Blumer (*Australasian Eng.*, **1951**, (Jan.), 65–69).—The value of tensile, impact, and bend tests is reviewed.—T. A. H.

The Mullen Bursting-Strength Test as a Means of Determining the Strength of Annealed Aluminium Foil. W. C. Aber and F. M. Howell (*Proc. Amer. Soc. Test. Mat.*, 1950, **50**, 425–429; discussion, 429).—Report of A.S.T.M. Committee E-1 on Methods of Testing. See *Met. Abs.*, 1950–51, **18**, 209.

***A Method for Making Tension Tests of Metals Using a Miniature Specimen.** R. L. Templin and W. C. Aber (*Proc. Amer. Soc. Test. Mat.*, 1950, **50**, 1188–1194; discussion, 1195).—See *Met. Abs.*, 1950–51, **18**, 296.

***The Influence of Specimen Dimension and Shape on the Results in Tension Impact Testing.** D. S. Clark and D. S. Wood (*Proc. Amer. Soc. Test. Mat.*, 1950, **50**, 577–585; discussion, 586).—See *Met. Abs.*, 1950–51, **18**, 548.

Report of the Task Group on Effect of Speed of Testing on Fatigue Test Results. —— (*Proc. Amer. Soc. Test. Mat.*, 1950, **50**, 421–424).—Report of A.S.T.M. Committee E-1 on Methods of Testing. See *Met. Abs.*, 1950–51, **18**, 210.

***Microhardness Tester for Metals at Elevated Temperatures.** Abner Brenner (*J. Research Nat. Bur. Stand.*, 1951, **46**, (2), 126–131).—See *Met. Abs.*, this vol., col. 55.—J. W. C.

***A Theoretical Investigation of the Effect of Specimen Size in the Measurement of Hardness.** R. Hill (*Phil. Mag.*, 1950, [vii], **41**, (319), 745–753).—The manner in which a rectangular specimen deforms if indented by a wedge is considered theoretically. Critical specimen widths and thicknesses are found for this type of hardness test to give a valid result, and it is explained why further increases in specimen size do not affect the apparent hardness within experimental error. The bearing of these results on the min. specimen size for hardness testing with a 136° pyramid is briefly indicated.—P. C. L. P.

New Methods of Non-Destructive Testing and Their Basic Principles. F. Förster (*Metall*, 1951, **5**, (1/2), 20–21).—F. gives brief notes of methods based on chem. impregnation, acoustics, thermal conductivity, elect. resistivity, and electromagnetic induction.—E. N.

Non-Destructive Testing in Austria. H. Krainer (*Metall*, 1951, **5**, (1/2), 18–20).—K. gives brief descriptions of the methods employed in Austria for the identification and the inspection of materials and finished products. 8 ref.—E. N.

Practical Consequences of the Skin-Effect at High Frequencies. H. Aberdam (*Ingénieurs et Techniciens*, 1950, (20), 77–81).—The localization of elect. currents at the surface of a conductor increases with the frequency. Its practical manifestation takes the form of an increase in the resistance of the conductor, and this effect is suitable for determining the shape and state of a metallic surface. A. gives a critical review of research in this field, including recent German and American studies.—G. A. K.

Some Industrial Applications of the Skin-Effect at High Frequencies. H. Aberdam (*Ingénieurs et Techniciens*, 1950, (21), 125–130).—The utilization of the skin effect in the detection of superficial fissures in metal wires and thin tubes, as well as for the detn. of the nature of the surfaces of parts to be used in H.F. instruments, is described. A test instrument arrangement is discussed, and an example of its appn. is given.—G. A. K.

Surface Finish Control. C. R. Lewis (*Product Eng.*, 1950, **21**, (8), 91–95).—L. describes the method of taper-sectioning for the measurement of surface finish and discusses the effect of surface-finishing operations on the structure of metallic materials at or immediately below the finished surface. The effect of surface finish on the frictional and fatigue properties of metals is also discussed.—E. C. P.

Surface Finish and the Designer. Roy P. Trowbridge (*Product Eng.*, 1950, **21**, (9), 122–127).—T. discusses where and when surface finish should be controlled, and methods of measuring are described; stylus-type instruments, taper-sectioning, and opt. methods are referred to. Methods of producing a specified machined surface finish are discussed. —E. C. P.

***Some Applications of Interferometry to the Examination of an Electrodeposited Film.** S. Tolansky (*Electrodepositors' Tech. Soc., Advance Copy*, 1951, **27**, (6), 8 pp.).—It is shown that multiple-beam interferometry offers a means of studying electrodeposits. A description is given of one particular method which can be used for examination of surface roughness, local small features, distortions produced elastically, and for the measurement of the thickness of deposits. Some results obtained on Sn–Ni deposits are briefly described. —G. T. C.

Visual and Optical Evaluation of Metal Surfaces. Helmut Thielsch (*Metal Finishing*, 1951, **49**, (5), 54–61).—Cf. *ibid.*, 1950, **48**, 66; *Met. Abs.*, 1950–51, **18**, 452. The more important methods are reviewed and summarized. These include examination by sight, microscopic examination, various replica techniques, electron-microscopic methods, several sectioning methods, and interferometer methods. A table is included which summarizes the appn. and characteristics of the various opt. methods. 15 ref.—G. T. C.

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14 — TEMPERATURE MEASUREMENT AND CONTROL

Temperature Measurements in Metallurgical Operations. P. Desemery (*Ingénieurs et Techniciens*, 1950, (22), 157–160; (23), 189–192; (24), 239–242).—A review of old and new methods of temp. measurement. Differences between indust. and sci. measurements and instruments are pointed out. Pyrometric scales, general principles of pyrometry, degrees of precision and approximation of measurements are considered, and thermo-elect., radiant-heat, and opt. pyrometers are described.—G. A. K.

†The Measurement of High Temperatures. Pierre Rodicq (*Fonderie*, 1951, (64), 2423–2438).—Immersion pyrometers are much to be preferred to opt. pyrometers for temp. up to 1200° C.; but the life of the thermocouples is shortened by the formation of silicides of Pt in the presence of S that may arise from traces of oil. In addn. to careful degreasing, the N.P.L. and T. Land use an alumina-base sheath instead of the normal silica one and, to reduce the partial volatilization of Rh, a 13% Rh wire in place of the usual 10% wire. R. discusses the use of this thermocouple and the method of testing it; describes and compares the Tinsley amplifier, the CUG 11 converter, and the Speedomax recording apparatus, and describes an indicating device. He also discusses the construction, use, and accuracy of the Leeds–Northrup Rayotube for measuring the temp. of molten steel. He compares the temp. readings obtained by opt. methods with those obtained with the immersion pyrometer, and suggests that, if 50° C. is added to the values given by the disappearing-filament pyrometer, one has a 47% chance of getting the true temp. ±15° C. —J. H. W.

Some Aspects of Automatic Control of Heat-Treatment Furnaces. A. Linford (*Metal Treatment*, 1950, **17**, (61), 53–62).—A review of the basic principles of automatic temp. control as applied to indust. heat-treatment furnaces. Deals mainly with pneumatic control for fuel-fired furnaces and describes proportional, proportional + integral, and first-derivative control.—J. W. C.

***The Control of Electric Element Furnaces to Fine Limits at High Temperatures for Research Purposes.** T. W. Lomas, M. D. Jepson, and J. R. Rait (*J. Iron Steel Inst.*, 1951, **168**, (2), 126–134).—The authors discuss the temp. control needed for the accurate detn. of high-temp. creep data and conclude that an accuracy of ±0·1° C. is desirable. The merits of 3 types of proportional controller are considered: (1) the D.C. amplifier with saturable reactor controller; (2) the resistance thermometer/thyratron controller; and (3) the resistance thermometer/reactor controller. It is concluded that the (Pt) resistance thermometer/thyratron controller is the most suitable on grounds of cost and performance; a commercially available form of this instrument is described in some detail. The instrument as supplied was found to be unstable at temp. above 800° C. and experiments were carried out in an attempt to extend its range. Part of the instability was shown to be caused by capacitance effects in the resistance bulb; this was ameliorated by balancing out the stray capacity by means of a variable condenser placed in the other side of the bridge, and good control was obtained up to 1000° C. In further experiments the capacity effect was virtually eliminated by redesigning the bulb, or by replacing the alumina cement which held the Pt wire in place, by a magnesite cement. With the apparatus thus modified, temp. up to 1100° C. can be held within ±0·05° C., provided the supply voltage to the instrument is stable within ±2 V. It was further found that stable control up to 1600° C. could be obtained with the magnesite-cemented resistance bulbs, except in the temp. range 1275°–1335° C. In this range the resistance of the magnesite falls to a value comparable with that of the Pt wire, and above 1335° C. the bulb functions as a magnesite thermistor. At 1600° C. the accuracy of control is ±1·0° C.—R. W. R.

Calibration Furnaces for Total-Radiation Pyrometers. E. J. Burton and R. Mayorcas (*J. Iron Steel Inst.*, 1951, **168**, (2), 151–155).—B. and M. define the essential requirements of a furnace for calibrating total-radiation pyrometers: (*a*) the radiation emitted must be substantially black-body; (*b*) the source provided must be at least 3 in. in dia.; and (*c*) the temp. must be capable of being changed rapidly. Two furnaces meeting these requirements are described in detail. The first, a graphite-tube resistance furnace 36 in. long and 3 in. in dia. was satisfactory, but required too large a current (1400 amp.) for convenience. A second furnace 17 in. long and 3 in. in dia. was made, in which the graphite tube was slit longitudinally into a number of sections each insulated from its neighbours. This increased the resistance of the tube ~20 times and reduced the current required to 150 amp. Two kinds of radiating sources are described for use with these furnaces: (i) a graphite disc grooved to increase the surface emissivity—the emissivity of this source in the furnace is computed to be 0·999; (ii) a source consisting of a bundle of small graphite tubes backed by a graphite disc. The method of carrying out calibrations of total-radiation pyrometers is described. —R. W. R.

Designing Bimetal Control Devices. U. Savolainen (*Product Eng.*, 1950, **21**, (8), 115–119).—Design formulæ are given for 6 different types of bimetallic elements and the thermostatic properties of the most common bimetals are tabulated. Suitable alloys for bimetallic elements and appn. are discussed.—E. C. P.

15—FOUNDRY PRACTICE AND APPLIANCES

***The Grain Refinement of Aluminium Alloy Castings by Additions of Titanium and Boron.** (Cibula). See col. 109.

Pressure-Cast Aluminium Matchplate Production. —— (*Found. Trade J.*, 1950, **89**, (1791), 563–565; correspondence, 1951, **90**, (1793), 49).—An abstract on pressure-cast Al matchplate prodn. taken from the Grey Ironfoundry Productivity Report. A note is also included on the compn. of the plaster used for making the mould.—J. E. G.

On the Design of Sand, Gravity, and Pressure-Die Castings in Light Alloys. Philipp Schneider (*Giesserei* (Sonderausgabe *Konstrukteur und Giesser*), **1951**, 70–75).—The main features of sand, gravity, and pressure-die castings in light alloys are described, and illustrated by means of a number of examples of castings of each type.—V. K.

Making Special-Duty Castings in Aluminium Alloys. J. Caven and H. W. Keeble (*Proc. Inst. Brit. Found.*, 1950, **43**, B25–B34).—See *Met. Abs.*, 1949–50, **17**, 849.

Casting a Large Pulley in Aluminium Alloy. W. Wilson and A. Talbot (*Proc. Inst. Brit. Found.*, 1950, **43**, B1–B10; discussion, B10–B13).—See *Met. Abs.*, 1949–50, **17**, 761.

***[Some Experiments in] Sand Casting of Conductivity Copper.** W. H. Glaisher (*Proc. Inst. Brit. Found.*, 1950, **43**, A110–A113; discussion, A113).—See *Met. Abs.*, 1950–51, **18**, 552.

***Sand-Cast Test-Bars for Copper-Base Alloys.** O. R. J. Lee and B. W. Peck (*Proc. Inst. Brit. Found.*, 1950, **43**, A104–A107; discussion, A107–A109).—See *Met. Abs.*, 1950–51, **18**, 552.

Guidance in the Application of Gravity Die-Castings in Copper-Base Alloys. C. A. Piel (*Giesserei* (Sonderausgabe *Konstrukteur und Giesser*), **1951**, 76–78).—Basic principles of die and casting design and the technique of manufacturing gravity die-castings in Cu-base alloys are briefly reviewed. For Al brasses the following properties can be obtained in normal prodn.: B.H.N. = 80–110; elongation = 15–20%; U.T.S. = 22–25 tons/in.^{2}.—V. K.

Making Aluminium Bronze Alloys. Marcel Cirou (*Found. Trade J.*, 1950, **89**, (1781/82), 349–350).—Translated from *Fonderie*, 1950, (55), 2131–2132; *Met. Abs.*, 1950–51, **18**, 128.—J. E. G.

[Defects in] Cast [Brass and Gun-Metal] Letter Plates. H. H. Symonds (*Metal Ind.*, 1951, **78**, (22), 447–448; correspondence, 1951, **79**, (2), 33).—Surface defects in cast brass and gun-metal letter plates were found in nearly every case investigated to be due to the fact that the metal used did not comply with the specified compn.—J. H. W.

Manufacture of Bronze Boiler Plugs. B. F. Kline and J. R. Davidson (*Amer. Foundryman*, 1951, **19**, (3), 34–37).—The full procedure for casting, machining, and assembling bronze boiler drop plugs is described.—V. K.

General Considerations in the Founding of Copper Alloys. W. F. Walker (*Mech. World*, 1951, **129**, (3351), 312–314).—The design of moulds and the running and feeding procedures suitable for the manufacture of Cu-base alloy castings are discussed in general terms. Mention is also made of melting procedures and moulding sands.—R. W. R.

Practical Details in the Founding of Copper Alloys. W. F. Walker (*Mech. World*, 1951, **129**, (3352), 337–340).—Cf. preceding abstract. Brief details are given of moulding, running, feeding, melting, and pouring procedures suitable for the manufacture of castings in Cu, Cu–Be alloys, brass, gun-metal, high-tensile brass, Al bronze, Cu–Pb alloys, cupro-nickel, amd nickel silver.—R. W. R.

" Soro " Process for the Casting of Bars [in Copper Alloys]. R. Genders (*Found. Trade J.*, 1950, **89**, (1778), 305–310, 312).—A detailed account is given of a casting process in which the bar is cast in the form of a ring or hoop. The cast ring is machined to remove the sink head and to make the bar the correct shape, after which the ring is slit and pulled straight on a draw-bench. Subsequently, the bar is fully straightened by hammering or by pressing, and where necessary " scalped " by drawing through a sharp-edged die. The process is applicable to the prodn. of round bars, squares, hexagons (up to 3 in.), and various widths and thicknesses of strip (up to 6 in. wide). Alloys cast by the process include 70 : 30 brasses, 58 : 40 : 2 free-cutting brass, 85 : 5 : 7 : 3 bearing bronze, 88 : 10 : 2 bearing bronze, 89 : 11 Al bronze, and 18 : 8 corrosion-resistant steel. It is also possible to cast bimetallic rings by this process.—J. E. G.

Die-Casting Magnesium Alloys. R. C. Cornell (*Amer. Foundryman*, 1951, **19**, (4), 75–77).—Melting procedure, die design, and casting technique used in prodn. of cold-chamber pressure die-castings in Mg-base alloys are described.—V. K.

Casting and Forging of Titanium. J. Bartlett Sutton, Edwin A. Gee, and William B. DeLong (*Metal Progress*, 1950, **58**, (5), 716–720).—Details are given of the construction and operation of an induction furnace for melting Ti in graphite and bottom pouring into a graphite mould. Ingots up to 650 lb. can be made, the energy consumption/lb. being ~0·6 kWh. The ingot metal may contain impurities such as 0·98% C, 0·06% N, 0·05% Fe: the ingot top may bulge, possibly owing to rejection of dissolved H. Commercially pure Ti contg. ~0·7% C is readily forgeable in the temp. range 1800°–1500° F. (980°–815° C.), and then has a hardness of ~240 D.P.N.; voids are welded up in forging. Conversion curves are also given relating hardness values on the different hardness scales.—F. A. F.

Cast Permanent Magnets. A. Braybrook and D. Hadfield (*Proc. Inst. Brit. Found.*, 1950, **43**, B112–B118; and (abridged) *Found. Trade J.*, 1950, **88**, (1743), 95–101).—Read before the Sheffield branch of the Institute of British Foundrymen. A review of the historical development of the permanent-magnet industry. Details are given of current foundry practice, melting and pouring techniques, and after-treatment processes as practised for the repetition prodn. of permanent magnets.—J. E. G.

Degas Molten Metals with Inert Gas. E. F. Kurzinski (*Amer. Foundryman*, 1951, **19**, (4), 78–81).—The technique of degassing molten alloys with N or A is described. Graphite tubes $\frac{1}{2}$ in. internal dia., 2 in. outer dia. are used to introduce the gas at an approx. rate of 30 ft.^{3}/hr., and a gas consumption of 8 ft.^{3}/ton of Cu-base and 25 ft.^{3}/ton of Al-base alloys. —V. K.

The Theory of the Degassing of Molten Metals Under Reduced Pressure. (Fekete). See col. 105.

The Effect of Liquid-Metal Properties on the Casting Fluidity of Alloys. V. Kondic (*Proc. Inst. Brit. Found.*, 1950, **43**, A153–A157; discussion, A158–A161).—See *Met. Abs.*, 1950–51, **18**, 554.

Investment Casting Conserves Critical Materials. Rawson L. Wood and Davidlee Von Ludwig (*Steel*, 1951, **128**, (20), 80–82).—The many advantages of precision casting are enumerated, with particular ref. to minimizing alloy waste.—E. J.

How to Set Up a Precision-Casting Foundry. W. F. Davenport and Adolph Strott (*Iron Age*, 1951, **167**, (16), 90–94).—D. and S. describe the installation of a small precision-casting foundry for the benefit of those with little experience of investment casting, indicating some new techniques as well as standard procedures.—J. H. W.

Recommended Practice for the Repair and Reclamation of Non-Ferrous Castings. —— (*Proc. Inst. Brit. Found.*, 1950, **43**, A123–A134; discussion, A135–A143; and *Found. Trade J.*, 1950, **89**, (1775/76), 243–253; discussion, (1778), 297–304).—Report of a Sub-Committee of the Institute of British Foundrymen. A detailed assessment is made of methods available for the repair and reclamation of non-ferrous castings. Processes discussed include burning-on, welding, brazing, soldering, porosity removal by annealing, impregnation, caulking and plugging, electroplating, and metal spraying. 48 ref.—J. E. G.

Castings for River and Coastal Craft. A. G. Thomson (*Found. Trade J.*, 1950, **89**, (1777), 269–272, 284).—A general illustrated account.—J. E. G.

Elements of Casting Design. Werner Trommer (*Giesserei* (Sonderausgabe *Konstrukteur und Giesser*), **1951**, 24–40).—The principles of design of castings in relation to patternmaking, moulding, and properties obtainable in a cast product are reviewed and illustrated by numerous examples mainly from the ferrous field.—V. K.

Selecting Components for Die-Casting. W. M. Halliday (*Found. Trade J.*, 1951, **90**, (1802), 287–290).—A general account of the requirements to be assessed when considering the fabrication of a component by a die-casting process. —J. E. G.

Ordering of Die-Castings. W. M. Halliday (*Found. Trade J.*, 1950, **89**, (1789), 501–503).—A discussion of the limitations and basic design requirements of the different types of die-castings. In order to increase prodn., a plea is made for closer co-operation between the die-caster and the customer. —J. E. G.

Do You Buy Castings? —— (*Mass Prod.*, 1951, **27**, (5), 48–50).—The design and prodn. factors to be considered by the purchaser when ordering castings are discussed.—R. W. R.

Gating Systems and the Production of Electrical and Machine-Tool Castings. W. Collinge (*Proc. Inst. Brit. Found.*, 1951, **43**, B119–B125; discussion, B125–B127).—See *Met. Abs.*, 1949–50, **17**, 762.

Symposium on Running Methods. J. F. Measures. P. A. Russell. D. W. Berridge (*Proc. Inst. Brit. Found.*, 1950, **43**, B63–B68; discussion, B68–B70).—See *Met. Abs.*, 1949–50, **17**, 381.

Metal-Penetration Test. S. L. Gertsman and A. E. Murton (*Amer. Foundryman*, 1951, **19**, (3), 32–33).—The procedure is described for making the cores, the mould, and carrying out the test for determining the sensitivity of a core-sand specimen to metal penetration.—V. K.

Evaluate Metal-Penetration Variables. S. L. Gertsman (*Amer. Foundryman*, 1951, **19**, (4), 94–99).—The metal penetration test, as described by G. and Murton (cf. *ibid.*, (3), 32; preceding abstract) was applied to a study of the effect of a number of mould properties on the penetration of the molten steel into the moulding sand. This penetration is reduced with low metal pouring temp., hard mould surface, fine-grained sand and silica flour addn. to the sand. Lime-silica mixture dressings are also effective in reducing metal penetration.—V. K.

***The Adherence of Sand on Steel Castings.** P. R. Beeley and H. T. Protheroe (*J. Iron Steel Inst.*, 1951, **167**, (2), 141–157).—The influence was studied of numerous factors on metal penetration and sand adherence during the manufacture of steel castings. It was shown that there is a critical temp. above which penetration and adherence occur; this critical temp. decreases with decrease in the permeability and grain-size of the sand. The depth of penetration is, however, smallest with fine-grained sands. The extent to which penetration and adherence occur can be related to the temp. of the metal/mould interface relative to the solidus temp. of the steel. The results are also reported of a study of the influence on penetration and adherence of the bonding material in the sand and of various mould washes. Cereal binders reduce penetration to some extent; best results were obtained with the following coatings: Corefrax, fused Al_2O_3, sillimanite, and zircon. 12 ref.—R. W. R.

***Colloidal Phenomena in Metals. XII.—Explanation of the Occurrence of Sand Pick-Up in the Casting of Metals on the Basis of the Electrochemical Theory of Slags.** Yu. A. Klyachko and L. L. Kunin (*Zhur. Priklad. Khim.*, 1949, **22**, (7), 707–715).—[In Russian]. Cf. *ibid.*, (5), 455; *Met. Abs.*, this vol., col. 124. Sand pick-up is ascribed to the adhesion to the metal of a new phase formed by reaction between the oxides of the metal and the mould materials, and it is suggested that adhesion depends upon the degree of similarity between the lattice of this intermediate phase and those of the metal oxide and the mould material. X-ray investigations of the compn. of the new phases formed with cast Fe and SiO_2 or Al_2O_3 confirmed predictions based on the electrochem. theory of slags advanced by Herasymenko (e.g. *Trans. Faraday Soc.*, 1938, **34**, 1245) *et al.*—G. V. E. T.

Moulding Technique. W. Pollock (*Found. Trade J.*, 1950, **89**, (1791), 551–557).—Read before the Scottish branch of the Institute of British Foundrymen. A detailed and illustrated account of the moulding techniques employed in the prodn. of a large bulkhead door-panel and frame and a dome-shaped casting. Details are also given of an adjustable moulding box and rules to follow when making core grids. —J. E. G.

Loam Moulding of Pump Casings and Impellers. E. Clipson (*Found. Trade J.*, 1951, **90**, (1811), 515–520; (1812), 547–550; discussion, 550–551).—Read before the Lincoln branch of the Institute of British Foundrymen. An illustrated account of the moulding techniques employed.—J. E. G.

Moulding Technique Aids Production. S. R. Bridger (*Proc. Inst. Brit. Found.*, 1950, **43**, B128–B133).—See *Met. Abs.*, 1949–50, **17**, 855.

Reorganization of a Moulding-Machine Section. R. F. Ottignon (*Found. Trade J.*, 1950, **89**, (1788), 467–472).—An account of a scheme of foundry reorganization, with special ref. to the moulding-machine section.—J. E. G.

Core-Blower Application and Operation. G. W. Fearfield (*Found. Trade J.*, 1951, **90**, (1799), 207–210; (1800), 229–232; discussion, 232–233).—Read before the Lancashire branch of the Institute of British Foundrymen. A detailed review of the subject of core-blowing.—J. E. G.

Zircon Sand: Properties and Uses in Cores and Facings. Arthur Smith (*Amer. Foundryman*, 1951, **19**, (5), 64–67).—Foundry practice based on $ZrSiO_4$ is described. The main advantages of this type of synthetic sand are higher heat diffusivity than that of SiO_2-base sand and greater chem. inertness resulting in low fettling costs.—V. K.

19—WORKING

A New Continuous Rolling Mill for Aluminium. —— (*Metal Treatment*, 1950, **17**, (63), 141–147).—Cf. *Met. Abs.*, 1950–51, **18**, 777. A description, with photographs, of the new rolling mill at the Rogerstone works of the Northern Aluminium Co., Ltd.—J. W. C.

***Hot Forming of Aluminium and Magnesium Alloys.** T. E. Piper (*Trans. Amer. Soc. Metals*, 1951, **43**, 1013–1029; discussion, 1029–1032).—See *Met. Abs.*, 1950–51, **18**, 558.

Aluminium Die-Forging Design for Quality and Economical Production. A. E. Favre and A. J. Orazem (*Product Eng.*, 1950, **21**, (8), 140–144).—The design of forgings is discussed and illustrated by a large number of block diagrams.
—E. C. P.

Aluminium Die-Forging Design Details That Promote Metal Flow. A. E. Favre and A. J. Orazem (*Product Eng.*, 1950, **21** (9), 130–134).—Factors which influence the dimensions of corner radii at section edges and of fillets in Al alloy forgings are discussed. Design details are given.—E. C. P.

New Aluminium Extrusion Process May Simplify Aircraft Construction. —— (*Steel*, 1950, **126**, (22), 63).—By extruding a tubular section which is then slit longitudinally, it is possible to produce skin and skin-stiffening elements as one unit for aircraft construction. Details are given.—E. J.

Free-Machining Copper-Rich Alloys. P. Mabb (*Metal Treatment*, 1950, **17**, (63), 154–160).—A review of the machining properties of brasses, bronzes, and nickel silvers, including the effects of addn. of Pb, Te, and Se.—J. W. C.

Revolution in Copper and Brass Mills. Daniel R. Hull (*Metal Progress*, 1950, **58**, (4), 473–477).—A general survey is given of changes in U.S. plant in the last ten years, which have been in the direction of increased mechanization.
—F. A. F.

***[Effect of] Roller Levelling [on the Structure] of Magnesium Alloy Sheet.** (Calnan and Tate). See col. 121.

Casting and Forging of Titanium. (Sutton, Gee, and DeLong). See col. 152.

New Method [Cavitron] Machines: [Cutting of] Sintered Carbides, Sintered Borides, Hardened Tool Steels. Alvin S. Cohan (*J. Metals*, 1951, **3**, (3), 216–217).—Cutting, threading, drilling, forming, and other machining operations on fully sintered cemented carbides and borides, Stellites, and hardened tool and die steels can be performed by a new machining method known as the Cavitron process, which uses ultrasonic vibrations. Unlike conventional machining methods, the Cavitron employs a blunt tool of any tough, malleable metal such as cold-rolled steel, in the shape of the cut or hole to be made in the work. It operates head-on into the work by vibrating at a frequency of 27,000 c./s., this high vibrating frequency imparting to the work tool accelerations in the order of 70,000 times the acceleration caused by the force of gravity. The actual cutting is done by a stream of inexpensive commercial abrasive which is fed into the work area in a liq. carrier. Holes, openings, and recesses having any arbitrary shape with dia. of 0·007–2 in. and tolerances of 0·002 in. can be formed in the hardest materials.—E. N.

Technological Plasticity of Metals Worked Under Pressure. S. I. Gubkin (*Izvest. Akad. Nauk S.S.S.R.*, **1949**, [Tekhn.], (8), 1242–1244).—[In Russian]. G. introduces a new factor into his previous conception of mean mech. plasticity by taking into account the difference between the properties of metal as tested in the laboratory and under working conditions in indust. practice. This factor, termed "technological plasticity", is defined as: $D_t = \frac{aD' + b}{c}$, where D' = mean mech. plasticity and a, b, and c are coeff. related in values to the different technological processes. Some experimental values for these coeff. and examples of the use of the new conception for the evaluation of plastic properties of metals, are given.—W. S.

***Calculation of the Deformation Resistance and of the Power Requirements in Rolling.** A. Geleji (*Acta Techn. Acad. Sci. Hungar.*, 1950, **1**, (1), 78–109).—[In German]. G.'s theory of rolling practice (*Schweiz. Arch. angew. Wiss. Techn.*, 1947, **13**, 336; *Met. Abs.*, 1947–48, **15**, 536) is clarified and amplified. The theory is based upon the relative cycloidal motion of rolls and rolled material. A formula is derived for the deformation resistance. Simple assumptions lead to the deduction of formulæ for the power requirements and roll-turning moments in rolling; in practical appn. these calculated data require multiplication by a factor dependent upon the percentage reduction of size produced by rolling. Experimental and calculated results for a large number of metals and alloys are in fairly good agreement. The theory of rolling with calliper rolls, i.e. non-cylindrical rolls, is briefly discussed.—J. S. G. T.

***Cold Rolling with Strip Tension. I.—A New Approximate Method of Calculation and a Comparison with Other Methods.** H. Ford, F. Ellis, and D. R. Bland (*J. Iron Steel Inst.*, 1951, **168**, (1), 57–72).—By making simplifying approximations to Orowan's general theory of rolling (*Proc. Inst. Mech. Eng.*, 1943, **150**, 140; *Met. Abs.*, 1944, **11**, 167) equations are derived for the roll force and torque, which can be solved by a simple and rapid graphical method. These equations enable the above quantities to be computed from the yield-stress characteristics of the material, the coeff. of friction, and the dimensions of the pass. Curves are presented of the functions used in solving the equations, from which the values of the functions can be read off against the pass dimensions. The use of the method is illustrated by the soln. of problems relating to the rolling of mild steel and Cu. The equations employ a "mean yield stress" (the yield stress varies with the degree of reduction) and the computation of this from the roll dimensions is discussed. The method is compared with the similar methods of Orowan (*loc. cit.*), Hill, Siebel, Tselikov, and Nádai, for a wide range of strip tensions, the agreement on the whole being good. The present method of calculation is considerably faster than any of the earlier methods. 10 ref.—R. W. R.

***The Effect of Tension on Torque and Roll Force in Cold Strip Rolling.** W. C. F. Hessenberg and R. B. Sims (*J. Iron Steel Inst.*, 1951, **168**, (2), 155–164).—Experiments were carried out on the B.I.S.R.A. experimental mill to determine the effect of strip tension on the roll force and torque. On the assumption that the energy of deformation is independent of the strip tension, a simple formula is proposed for the effect of tension on the torque; a second approx. equation giving the relation between tension and roll force is also suggested. The more elaborate treatments of other workers are discussed in detail. Comparison with the experimental results shows that the equation for both torque and roll force are adequate except at very high values of back tension. The results show that the total energy of rolling is substantially unaffected by the appn. of tension and that there is no advantage in applying tensions greater than that required to produce flat, properly coiled strip. The failure of tension to affect the energy of rolling is explained as follows. The energy of rolling is composed of the energy required to deform the strip and that absorbed in friction. Since the energy of deformation depends only on the amount of deformation and on the properties of the strip, any effect of tension must arise from changes in the frictional component. This component is relatively small, so that the total energy of rolling is hardly affected by small changes in it.—R. W. R.

***Distribution of Deformation Within a Rolled Strip.** T. M. Golubev (*Izvest. Akad. Nauk S.S.S.R.*, **1950**, [Tekhn.], (4), 582–589).—[In Russian]. G. describes investigations into the distribution of deformation in rolling. Thin strips of Pb (2·5 × 32·5 mm.) welded together at the ends to form a specimen 30 mm. wide and 32·5 mm. thick were used. The amount of deformation was checked at three points across the thickness of each strip and at three points across the width. The distribution of deformation within the specimens for different reductions of area, speeds of rolling, and friction at the roll surfaces, was determined. The experimental results show that the amount of deformation varies across the width, as well as through the thickness of the specimen. The material is more deformed at the outside surface than inside the specimen. This difference decreases at greater reductions and with increasing speed of rolling. With increasing coeff. of friction the amount of plastic deformation increases through the thickness of the specimen. Max. deformation is found at the centre of the specimens and decreases towards the edges. It also decreases with increase of reduction and speed of rolling and with decrease of the coeff. of friction between the metal and the roll surfaces. G. suggests that two mechanisms of deformation take place in specimens: compression at the outside surface and tension inside the specimen. The simultaneous presence of these can result in the formation of cracks in the material. To prevent them the use of heavier rolling reductions is advocated wherever possible.—W. S.

Theory of Hollow Sinking of Thin-Walled Tubes. S. Y. Chung (*Metallurgia*, 1951, **43**, (259), 215–218).—Using a modified criterion of plastic yielding C. discusses theoretical aspects of hollow sinking. The theoretical stresses are compared with previously published experimental results on highly pre-strained P-deoxidized Cu and soft brass, and predicted strains with recent research data on Al and brass.
—F. M. L.

A Review of Impact-Extrusion and Some Related Processes. G. Sachs (*Sheet Metal Ind.*, 1951, **28**, (290), 533–538, 546).—S. gives a general description of the impact extrusion process, extrusion forging, coining, and sizing.
—R. J.

*"**Ringing" in Wire-Drawing Dies.** J. G. Wistreich (*J. Iron Steel Inst.*, 1951, **167**, (2), 162–164).—W. describes experiments in which (*a*) soft Cu and (*b*) mild-steel wires were drawn through identical dies. One lot of each wire was drawn freely through the die; back-pull was applied during the drawing of the second lot of each wire. In all, 4 dies were thus used. At the end of the tests the dies were examined for evidence of "ringing" (grooving near the point of entry of the wire). Ringing was found to be substantially reduced by the appn. of back-pull. It was concluded that ringing is not caused by the abrasive action of the lubricant, but is the result of the steep stress gradients at the beginning of the working surface of the die.
—R. W. R.

For Best Forming Results Use the Right Lubricant. E. L. H. Bastian (*Steel*, 1951, **128**, (19), 116–119, 130, 133).—Lubricants used in press drawing, hot and cold forging, and hot and cold extruding of metals are described, and practical recommendations are made.—E. J.

The Economic Utilization of Mineral Oils in the Production and Working of Non-Ferrous Metals [in Germany]. J. M. Nestor Kuckhoff (*Metall*, 1950, **4**, (15/16), 341–344).—Lubricants and coolants suitable for use in the metal industry are discussed from an economic point of view.—E. N.

24 — BIBLIOGRAPHY

(Publications marked * have been received.)

***American Society for Metals.** *High-Temperature Properties of Metals.* A Series of Five Educational Lectures on High-Temperature Properties of Metals. Presented to Members of the A.S.M. during the Thirty-Second National Metal Congress and Exposition, Chicago, 23–27 October 1950. 9 × 6 in. Pp. [v] + 176, illustrated. 1951. Cleveland (3), O.: The Society, 7301 Euclid Avenue. ($4.00.)

[Contents: Earl R. Parker, "Creep of Metals"; N. J. Grant, "Stress Rupture Testing"; H. J. Grover, "High-Temperature Fatigue Testing"; Carl Wagner, "Methods of High-Temperature Oxidation Testing and Evaluation of Observations"; John J. B. Rutherford, "Some Experiences in Service (Power, Oil, and Chemical Plants)".]

***American Society for Metals.** *Interpretation of Tests and Correlation with Service.* A Series of Four Educational Lectures on Interpretation of Tests and Correlation with Service. Presented to Members of the A.S.M. during the Thirty-Second National Metal Congress and Exposition, Chicago, 23–27 October 1950. 9 × 6 in. Pp. [v] + 198, illustrated. 1951. Cleveland (3), O.: The Society, 7301 Euclid Avenue. ($5.00.)

[Contents: M. F. Garwood, H. H. Zurburg, and M. A. Erickson, "Correlation of Laboratory Tests and Service Performance"; M. Gensamer, "Limitations of Mechanical Testing"; J. T. Burwell, "Wear Tests and Service Performance"; F. L. LaQue, "Corrosion Tests and Service Performance".]

***American Society for Testing Materials.** *Symposium on the Nature, Occurrence, and Effects of the Sigma Phase.* Presented at the Fifty-Third Annual Meeting, American Society for Testing Materials, Atlantic City, N.J., 26–27 June 1950. (Special Technical Publication No. 110.) 9 × 6 in. Pp. [v] + 181, illustrated. 1951. Philadelphia (3), Pa.: The Society, 1916 Race Street. ($2.50.)

[Contents: E. J. Dulls and G. V. Smith, "Identification and Mode of Formation and Re-Solution of Sigma Phase in Austenitic Chromium–Nickel Steels" (see *Met. Abs.*, 1950–51, 18, 654); V. T. Malcolm and S. Low, "Sigma Phase in Several Cast Austenitic Steels" (see *Met. Abs.*, 1950–51, 18, 654); Pol Duwez and Spencer R. Baen, "X-Ray Study of the Sigma Phase in Various Alloy Systems" (see *Met. Abs.*, 1950–51, 18, 172); John W. Putnam, N. J. Grant, and D. S. Bloom, "Sigma Phase in Chromium–Molybdenum Alloys with Iron or Nickel"; L. Menezes, J. K. Roros, and T. A. Read, "The Tetragonality of the Sigma Phase in the Iron–Chromium System"; J. J. Heger, "The Formation of Sigma Phase in 17 per cent. Chromium Steel" (see *Met. Abs.*, 1950–51, 18, 654); G. N. Emmanuel, "Sigma Phase and Other Effects of Prolonged Heating at Elevated Temperatures on 25 per cent. Chromium–20 per cent. Nickel Steel" (see *Met. Abs.*, 1950–51, 18, 654); J. H. Jackson, "The Occurrence of the Sigma Phase and Its Effect on Certain Properties of Cast Fe–Ni–Cr Alloys"; Raymond S. Stewart and Stephen F. Urban, "The Formation of Sigma and Its Influence on the Behaviour of Stabilized 18 per cent. Chromium–8 per cent. Nickel Steels in Concentrated Nitric Acid"; W. O. Binder, "Some Notes on the Structure and Impact Resistance of Niobium-Bearing 18 : 8 Steels After Exposure to Elevated Temperatures"; F. W. Schmitz and M. A. Scheil, "Observations of the Effect of Sigma on the Mechanical Properties of Niobium-Stabilized Weldments in Austenitic Stainless Steels".]

Bastian, E. L. H. *Metal-Working Lubricants: Their Selection, Application, and Maintenance.* 9 × 6 in. Pp. 357, illustrated. 1951. New York: McGraw-Hill Book Co., Inc. ($6.00); London: McGraw-Hill Publishing Co., Ltd. (51*s.*)

Bozorth, Richard M. *Ferromagnetism.* 9 × 6 in. Pp. 968, illustrated. 1951. New York: D. Van Nostrand Co., Inc. ($17.50.)

***British Standards Institution.** *British Standards Institution Yearbook 1951.* 8½ × 5½ in. Pp. 399. 1951. London: The Institution, 24–28 Victoria Street, S.W.1. (7*s.* 6*d.*, post free.)

***British Standards Institution.** *Inspection and Testing Procedure for Aluminium and Aluminium Alloys.* (British Standard for Aircraft Material L100.) 13½ × 8½ in. 1951. London: The Institution, 24–28 Victoria Street, S.W.1. (7*s.* 6*d.*, post free.)

***Brode, Wallace R.** *Chemical Spectroscopy.* Twenty-fourth Edward Marburg Lecture 1950. Presented before the Fifty-Third Annual Meeting of the American Society for Testing Materials. 9 × 6 in. Pp. [iv] + 47, with 60 illustrations. [1951]. Philadelphia (3), Pa.: The Society, 1916 Race Street. ($1.35.)

[Reprinted from *Proc. Amer. Soc. Test. Mat.*, 1950, 50, 513–559.]

***Department of Scientific and Industrial Research.** *National Physical Laboratory: Report for the Year 1950.* 9½ × 6 in. Pp. viii + 71. 1951. London: H.M. Stationery Office. (2*s.* net.)

***Erdmann-Jesnitzer, Friedrich.** Bearbeitet und herausgegeben von. *Werkstoff und Schweissung. Handbuch für die Werkstoff- und werkstoffbedingte Verfahrenstechnik der*

Schweissung. Band I. 25 × 18 cm. Pp. xxiv + 1002, with 1117 illustrations and 196 tables. 1951. Berlin NW7 (Ostberlin): Akademie-Verlag G.m.b.H., Schiffbauerdamm 19. (Geb., Band I + II DM 160.—.)

***Fourth Empire Mining and Metallurgical Congress.** *Proceedings* of the Congress, held in Great Britain, 9–23 July 1949. Two volumes. 8½ × 5½ in. Vol. I. Pp. xx + 551; Vol. II. Pp. xx + 553–1139, illustrated. 1950. London: The Congress, 436 Salisbury House, Finsbury Circus, E.C.2. (£1 each vol.)

Gallasini, A. *Leghe metalliche e siderurgia.* Pp. 320. 1950. Milan: U. Hoepli. (L. 850.)

***Halla, Franz.** *Kristallchemie und Kristallphysik metallischer Werkstoffe. Eine Einführung für Ingenieure.* 2. Auflage. 23·5 × 16 cm. Pp. xvi + 638, with 346 illustrations. 1951. Leipzig: Johann Ambrosius Barth. (Br., DM 45.—; geb., DM 46.50.)

***Henisch, H. K.,** Edited by. *Semi-Conducting Materials.* Proceedings of a Conference held at the University of Reading, 10–15 July 1950. 9¾ × 7¼ in. Pp. xv + 281, illustrated. 1951. London: Butterworths Scientific Publications, Ltd. (40*s*., by post 1*s*. extra.)

[Contents: N. F. Mott, "Semi-Conductors"; G. W. Castellan and F. Seitz, "On the Energy States of Impurities in Silicon"; W. Shockley, "New Phenomena of Electronic Conduction in Semi-Conductors", W. H. Brattain, "Semi-Conductor Surface Phenomena"; K. Lark-Horovitz, "Nucleon-Bombarded Semi-Conductors"; P. C. Banbury, H. A. Gebbie, and C. A. Hogarth, "Crystal Triode Action in Lead Sulphide"; H. K. Henisch and J. W. Granville, "Recent Experiments on Lead Sulphide Contacts"; P. R. Aigrain, C. R. Dugas, and H. W. Etzel, "Electrical Characteristics and Anomalies of Germanium.—I.–II."; R. W. Pohl, "Electron Traps and Electron Conduction in Irradiated Alkali Halide Crystals"; L. P. Smith, "Motion of Electrons and Holes in Silver Chloride"; R. L. Sproull and W. W. Tyler, "Semi-Conduction and Photo-Conduction in Barium Oxide Crystals"; H. Y. Fan and M. Becker, "Infra-Red Optical Properties of Silicon and Germanium"; E. W. J. Mitchell and J. W. Mitchell, "Work-Function of Germanium"; E. J. W. Verwey, "Oxidic Semi-Conductors"; J. Volger, "Some Properties of Mixed Lanthanum and Strontium Manganites"; P. H. Miller, Jr., "Electrical and Optical Properties of Zinc Oxide"; T. J. Gray, "On the Properties of Semi-Conducting Oxides"; G. Busch, J. Wieland, and H. Zoller, "Electronic Properties of Grey Tin"; R. A. Smith, "Electrical and Optical Properties of Certain Sulphides, Selenides, and Tellurides"; R. P. Chasmar and E. H. Putley, "Measurements of the Temperature-Dependence of Conductivity and Hall Coefficient in Lead Sulphide and Lead Telluride"; A. E. Sandström, "Some Experimental Studies of the Resistance and Electromotive Force of Selenium Blocking Layer Cells"; H. K. Henisch and M. François, "On the Thermoelectric Properties of Selenium"; H. Krebs, "Crystallization of Semi-Metals and the Formation of Lattice Defects"; T. R. Scott and S. E. Mayer, "Engineering and Chemical Aspects of Semi-Conductors"; N. Mostovetch and B. Vodar, "Electrical Conductivity of Very Thin Metallic Films Evaporated in High Vacuum".]

Jacob, L. *An Introduction to Electron Optics.* (Methuen's Monographs on Physical Subjects). Pp. x + 150. 1951. London: Methuen and Co., Ltd. (8*s*. 6*d*.)

Kennel, Ernst. *Das Nieten im Stahl- und Leichtmetallbau.* 21 × 15 cm. Pp. 190, with 159 illustrations. 1951. Munich 27: Carl Hanser-Verlag. (DM 9.20.)

Locati, L. *La Fatica dei Materiali Metallici.* Pp. 333. 1950. Milan: U. Hoepli. (L. 1600.)

***Metal Statistics.** *Metal Statistics 1951.* Forty-fourth annual edition. 6 × 4 in. Pp. 808. 1951. New York (38): American Metal Market, 18 Cliff Street. ($2.00.)

[This edition contains similar information to the earlier ones on ferrous and non-ferrous metals and miscellaneous economic factors. Statistics are given of the production, consumption, prices, &c., of iron ore, pig iron, manganese ore and alloys, semi-finished and finished steel, iron and steel scrap, fuels, aluminium, antimony, cadmium, cobalt, copper, and copper alloys, gold, lead, magnesium, mercury, nickel, platinum metals, silver, tin, and zinc. Data are generally given up to the end of 1950.]

Ney, J. *Mécanique et Métallurgie. Cours rédigé pour l'enseignement des sciences dans les Centres d'apprentissage, les Cours professionnels et pour la préparation au Certificat d'aptitude professionnelle.* 22 × 14 cm. Pp. xiv + 294, with 224 illustrations. 1951. Paris (VI^e): Dunod, 92 Rue Bonaparte. (Br., 470 fr.)

***Quin's** *Metal Handbook 1950.* 4 × 6 in. Pp. 612. [1951]. London: Metal Information Bureau, Ltd., 27 Albemarle Street, W.1. (20*s*., post free.)

[This international reference book on metals is replete with statistical and other information. The new edition contains extensive details relating to prices, production, consumption, exports, imports, brands, and stocks of a wide range of ferrous and non-ferrous metals for most of the industrialized countries of the world. The memoranda sections include, *inter alia*, brief accounts of the physical properties and uses of the commercial minor metals, notes on the physical, chemical, and other characteristics of commercial grades of ore of the principal non-ferrous metals, diaries covering the main base metals, British and American standard classifications of non-ferrous scrap, and a summary of the British Iron and Steel Nationalization Bill. Also included is a useful multi-lingual dictionary of technical terms relating to the iron and steel industry.]

***Raynor, G. V.** *The Equilibrium Diagram of the System Iron–Zinc.* (Annotated Equilibrium Diagram No. 8). 4to. Pp. 8, with 2 illustrations. 1951. London: Institute of Metals, 4 Grosvenor Gardens, S.W.1. (2*s*. ($0.50), post free.)

***Raynor, G. V.** *The Equilibrium Diagram of the System Antimony–Lead.* (Annotated Equilibrium Diagram No. 9.) 4to. Pp. 6, with 1 illustration. 1951. London: Institute of Metals, 4 Grosvenor Gardens, S.W.1. (2*s*. ($0.50), post free.)

***Salmon, William H., and Eric N. Simons.** *Foundry Practice.* 8½ × 5½ in. Pp. xiii + 384, with 192 illustrations. 1951. London: Sir Isaac Pitman and Sons, Ltd. (30*s*., net.)

Sandell, E. B. *Colorimetric Determination of Traces of Metals.* Volume III. 2nd edition. Pp. xix + 673. 1951. New York 1: Interscience Publishers, Inc., 250 Fifth Avenue. ($9.00.)

Schwahn, C. *Handwörterbuch des Gold- und Silberschmieds und Metallarbeiters.* Pp. 125. 1950. Halle (Saale): Carl Marhold. (DM 3.80.)

***Shenker, Henry, John I. Lauritzen, Jr., and Robert J. Corruccini.** *Reference Tables for Thermocouples.* (National Bureau of Standards Circular 508.) 10¼ × 8 in. Pp. [ii] + 71, with 18 tables. 1951. Washington, D.C.: U.S. Government Printing Office. ($0.35.)

***Simons, Eric N.** *Cutting-Tool Materials.* 8½ × 5½ in. Pp. ix + 180, with 15 illustrations. 1951. London: Sir Isaac Pitman and Sons, Ltd. (21*s*., net.)

***Tin Research Institute.** *Report on the Work of the Tin Research Institute, 1950.* 8½ × 5½ in. Pp. 32, illustrated. 1951. Greenford, Middx.: The Institute, Fraser Road.

***U.S. Department of Commerce, National Bureau of Standards.** *Standard Samples and Reference Standards, Issued by the National Bureau of Standards.* (Supplement to National Bureau of Standards Circular 398.) 10¾ × 8 in. Pp. ii + 23. 1951. Washington, D.C.: U.S. Government Printing Office. ($0.20.)

Vest, Gottlieb. *Lehrbuch der zahnärztlichen Kronen- und Brückentechnik.* Band 1: *Kronenprothetik.* (Odonto-Stomatologie, Volume III.) Pp. 296, with 362 illustrations. 1951. Basel: Verlag Birkhäuser. (Geb., Swiss fr. 36.)

***Voce, E.** *Copper and Copper Alloys: A Survey of Technical Progress During 1950.* 8½ × 5½ in. Pp. 31. [1951]. Radlett, Herts.: Copper Development Association, Kendals Hall.

[Reprinted from *Metallurgia*, 1950, 42, 382; *Met. Abs.*, 1950–51, 18, 482.]

Werner, Eugen. *Metallische Überzüge.* 4. Auflage. Pp. 182. 1950. Munich 27: Carl Hanser-Verlag. (DM 6.80.)

NEW JOURNALS

Der Giesserei-Praktiker. Published twice a month by Fachverlag Schiele und Schön, Berlin SW 29, Boppstrasse 10. First issue appeared in January 1951.

Metallurgie und Giessereitechnik. Published monthly by Verlag Technik G.m.b.H., Berlin NW7, Dorotheenstrasse 41. First issue appeared in January 1951. (DM 4.80 a quarter.)

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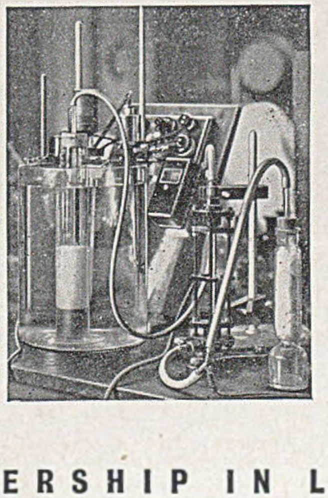

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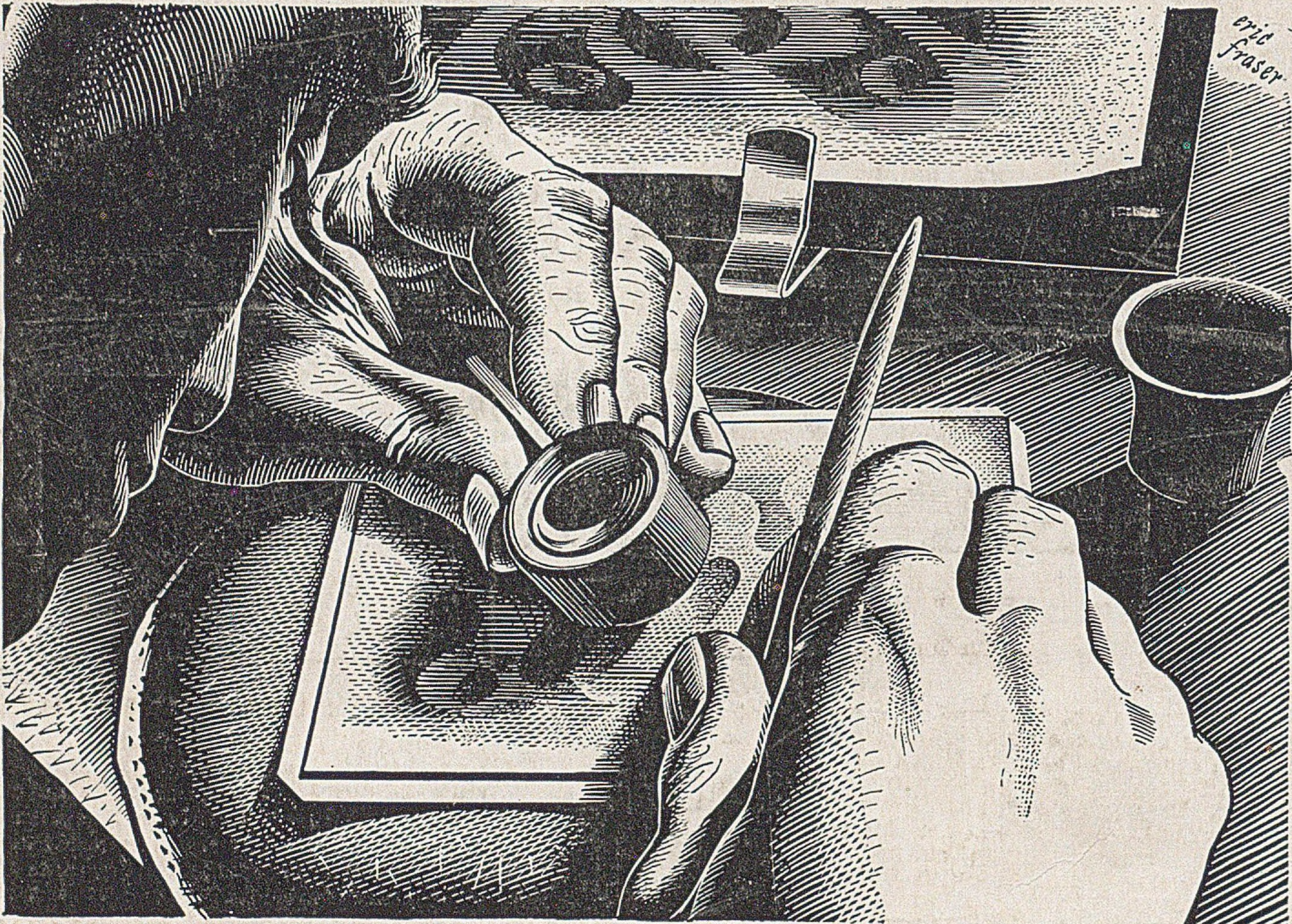

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