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P. 99/LXXIV

S.56

The Journal of the

# INSTITUTE OF METALS

and

## METALLURGICAL ABSTRACTS

17



### *In this Issue :*

PAGE

- 1108. The Application of Etch-Figures on Pure Aluminium (99.99%) to the Study of Some Micrographic Problems. *Paul Lacombe and Louis Beaujard* . . . . . 1
- 1109. On the Equilibrium and Kinetics of Order-Disorder Transformations in Alloys. *G. Borelius* . . . . . 17
- 1110. The Constitution of Alloys of Aluminium with Copper and Manganese. (*Miss*) *M. K. B. Day and H. W. L. Phillips* . . . . . 33

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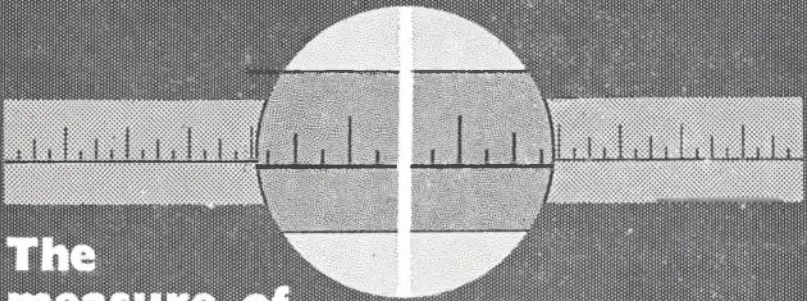
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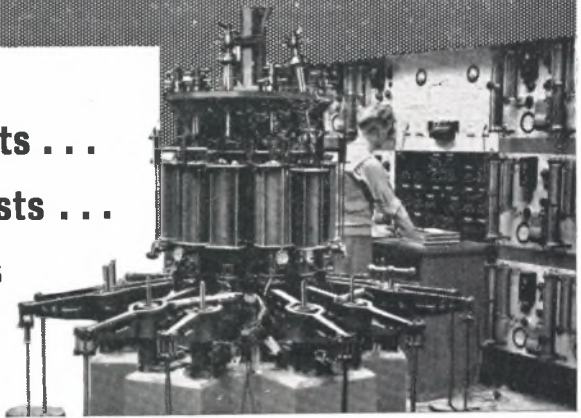
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P.99/LXXIV



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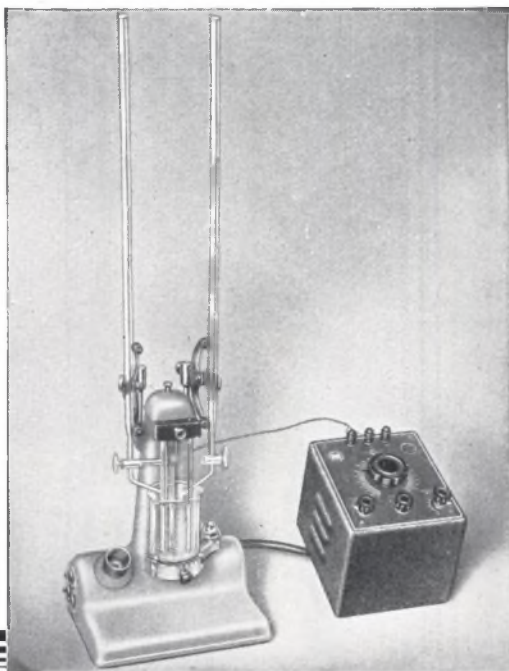
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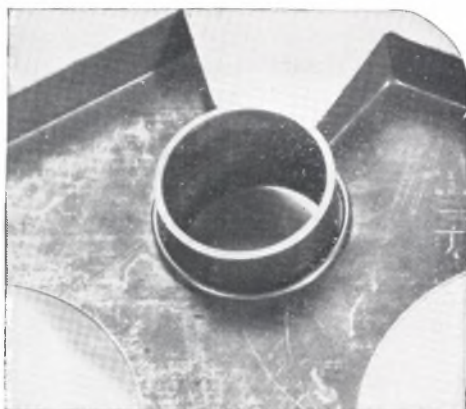
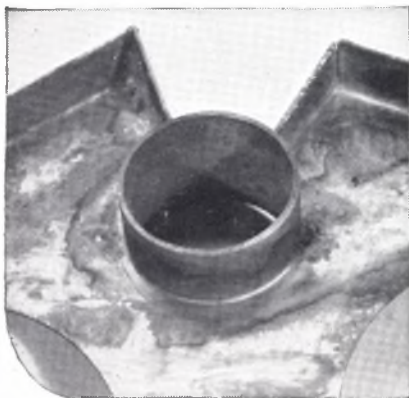
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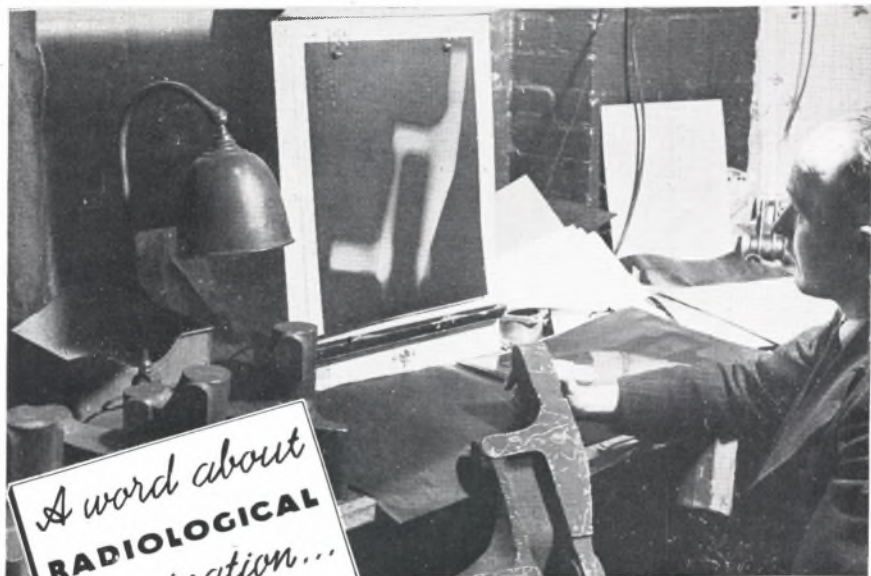
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I.M. 129



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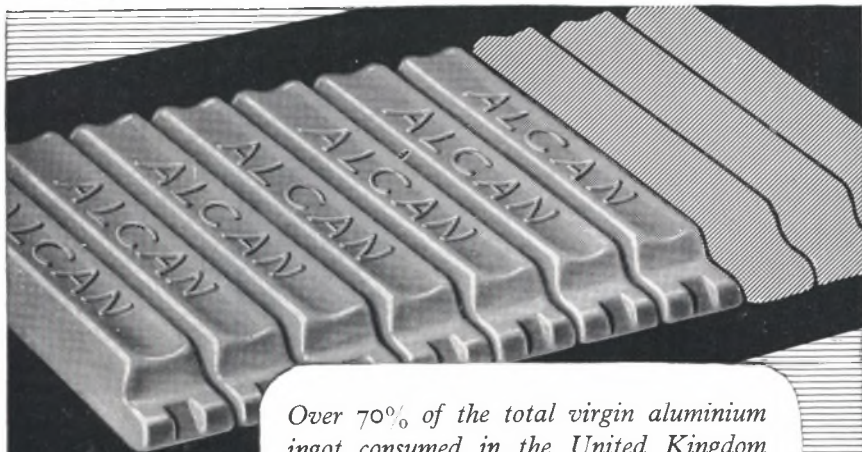
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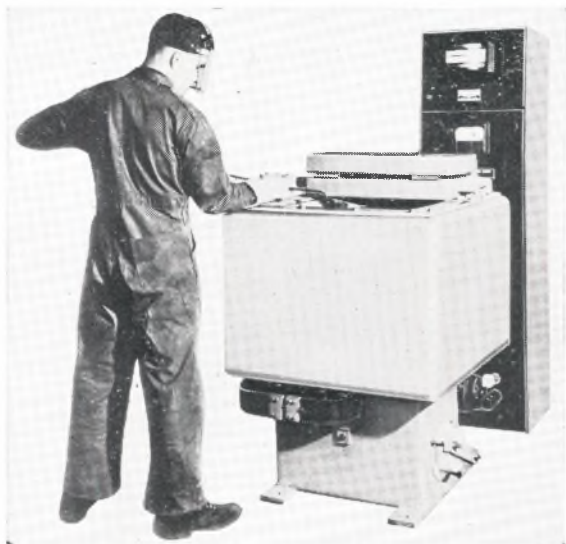
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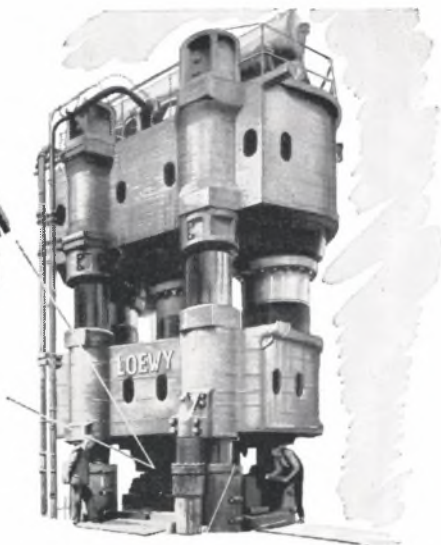
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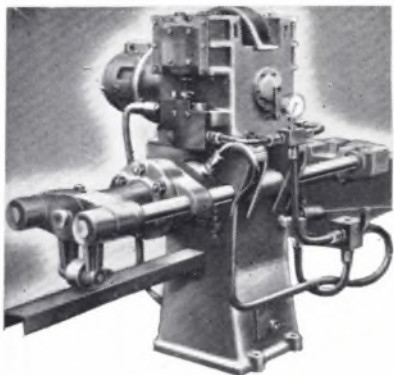
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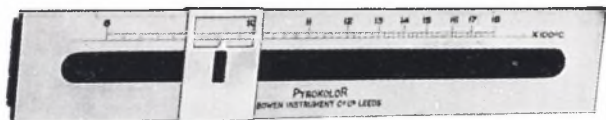
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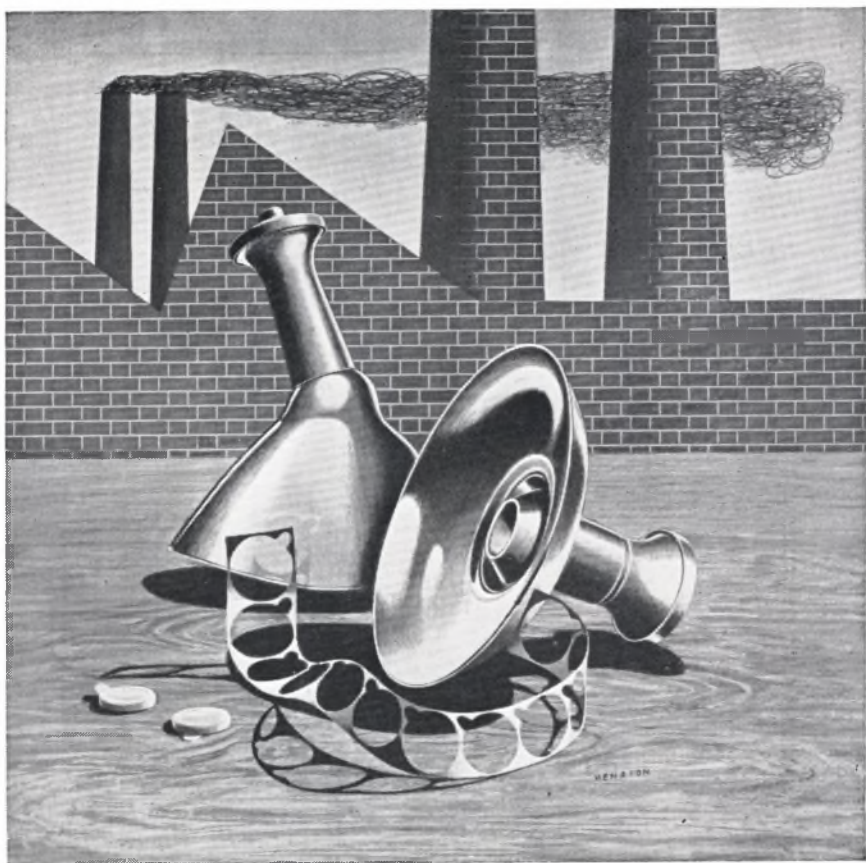
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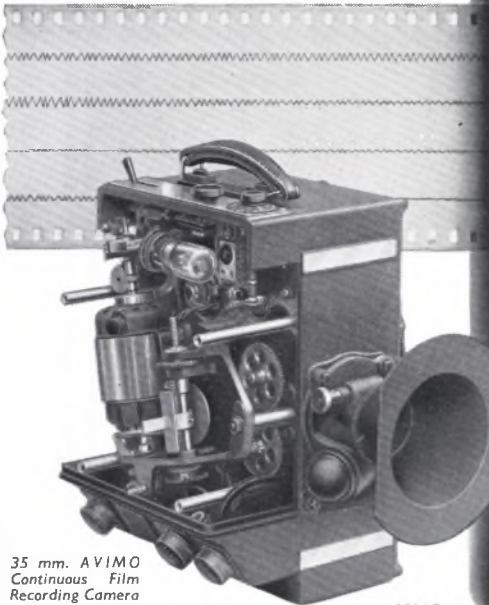
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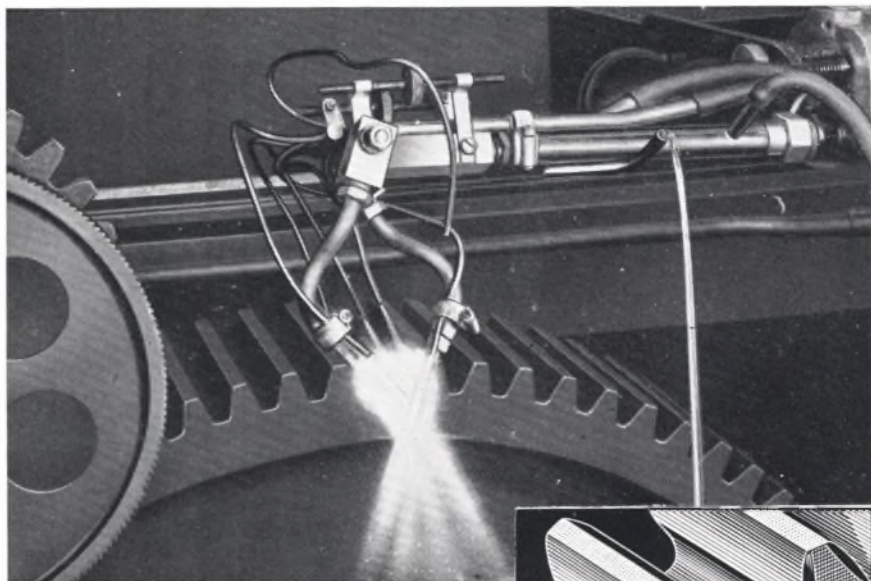


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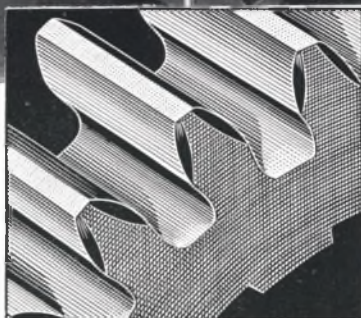
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### Contents

	PAGE
Institute News and Announcements . . . . .	XXIX
Local Sections News . . . . .	XXXV
Other News . . . . .	XXXVIII
Forthcoming Meetings . . . . .	XXXIX
Appointments Required and Vacant . . . . .	xl
1108. The Application of Etch-Figures on Pure Aluminium (99.99%) to the Study of Some Micrographic Problems. By Paul Lacombe and Louis Beaujard . . . . .	I
1109. On the Equilibrium and Kinetics of Order-Disorder Transformations in Alloys. By G. Borelius . . . . .	17
1110. The Constitution of Alloys of Aluminium with Copper and Manganese. By (Miss) M. K. B. Day and H. W. L. Phillips . . . . .	33
Metallurgical Abstracts . . . . .	I

### INDEX TO ADVERTISEMENTS

	Page		Page
Allen, Edgar, & Co., Ltd. . . . .	x	Ilford, Ltd. . . . .	—
Almin, Ltd. . . . .	—	Imperial Chemical Industries, Ltd. . . . .	vi
Aluminium Union, Ltd. . . . .	ix	Imperial Chemical Industries (Metals) Co., Ltd. . . . .	—
Avimo, Ltd. . . . .	xxiv	Imperial Smelting Corporation, Ltd. . . . .	xv
Baird & Tatlock (London), Ltd. . . . .	iv	International Alloys, Ltd. . . . .	—
Baker Platinum, Ltd. . . . .	xvi	International Electrolytic Plant Co., Ltd., The. . . . .	—
Bausch & Lomb Optical Co., Ltd. . . . .	—	Johnson, Matthey & Co., Ltd. . . . .	—
Beryllium Smelting Co., Ltd. . . . .	xxii	Kodak, Ltd. . . . .	(Inside front cover)
Birkett, T. M., & Sons, Ltd. . . . .	xx	Lewis, H. K., & Co., Ltd. . . . .	—
Birlec, Ltd. . . . .	xi	McGraw-Hill Publishing Co., Ltd. . . . .	xix
Bolton, Thos., & Sons, Ltd. . . . .	xii	McKiehn Bros., Ltd. . . . .	xii
Booth, James, & Co., Ltd. . . . .	xvii	Manganese Bronze & Brass Co., Ltd., The . . . . .	xxvi
Bowen Instrument Co., Ltd. . . . .	xxii	Mellows & Co., Ltd. . . . .	(Outside back cover)
Bristol's Instrument Co., Ltd. . . . .	—	Metallisation, Ltd. . . . .	xviii
British Aluminium Co., Ltd., The . . . . .	xxiii	Ministry of Fuel . . . . .	(Inside back cover)
British Gas Council . . . . .	—	Mond Nickel Co., Ltd., The . . . . .	iii
British Insulated Callender's Cables, Ltd. . . . .	xxviii	Mullard Wireless Service Co., Ltd., The . . . . .	—
British Oxygen Co., Ltd., The . . . . .	xxv	Northern Aluminium Co., Ltd. . . . .	—
British Rototherm Co., Ltd., The . . . . .	—	Park & Paterson, Ltd. . . . .	xviii
Bureau of Analysed Samples, Ltd. . . . .	xvi	Ridsdale & Co., Ltd. . . . .	xvi
Capper Pass & Son, Ltd. . . . .	xiv	Sheffield Smelting Co., Ltd., The . . . . .	—
Cooke Troughton & Simms, Ltd. . . . .	—	Siemens-Schukert (Great Britain), Ltd. . . . .	—
Copper Development Association . . . . .	—	Spear & Jackson, Ltd. . . . .	—
Derby & Co., Ltd. . . . .	(Outside back cover)	Stone, J., & Co., Ltd. . . . .	vii
Earle Bourne & Co., Ltd. . . . .	—	Sunday Times . . . . .	—
Eclipse Tool Co., Ltd., The . . . . .	xx	Sunvic Controls, Ltd. . . . .	xvii
Electric Furnace Co., Ltd. . . . .	—	Tyseley Metal Works, Ltd. . . . .	—
Electric Resistance Co., Ltd. . . . .	—	Vinten, W., Ltd. . . . .	xiv
Entores, Ltd. . . . .	xix	Watson, W., & Sons, Ltd. . . . .	xxii
Fisher & Ludlow, Ltd. . . . .	—	Wiggin, Henry, & Co., Ltd. . . . .	—
Foster Instrument Co., Ltd. . . . .	xxii	Wild-Barfield Electric Furnaces, Ltd. . . . .	—
Foyle, W. & G., Ltd. . . . .	—	Wolverhampton Die-Casting Co., Ltd. . . . .	xxiv
Gibbons Bros., Ltd. . . . .	v		
Heffer, W., & Sons . . . . .	xviii		
High Duty Alloys . . . . .	xxi		
Hind, E. (Southbank), Ltd. . . . .	viii		
Holroyd, John, & Co., Ltd. . . . .	—		
Hughes, F. A., & Co., Ltd. . . . .	xiii		

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**Major R. E. MOORE**

*Administrative and Editorial Offices :*

**4 GROSVENOR GARDENS, LONDON, S.W.1**

*Telephone :*

**SLOANE 6233**



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## INSTITUTE NEWS AND ANNOUNCEMENTS

SYMPOSIUM ON INTERNAL STRESSES IN METALS AND ALLOYS,  
LONDON, 15 AND 16 OCTOBER 1947

As previously announced, and as detailed in a circular which has been sent to all members, the Institute has organized a symposium on "Internal Stresses in Metals and Alloys" to be held on 15 and 16 October 1947 in the Hall of the Institution of Mechanical Engineers, Storey's Gate, Westminster, London, S.W.1. The meeting has been arranged in association with the Faraday Society, Institute of Physics, Institution of Mechanical Engineers (and its Automobile Division), Iron and Steel Institute, Physical Society, and Royal Aeronautical Society.

The object of this symposium—for which the Institute's Metal Physics Committee is responsible—is to bring together engineers, metallurgists, and physicists to discuss problems of common interest.

The 36 papers contributed to the symposium are available, bound in a paper cover, as a special "advance copy" issue (price 5s. each) for the purpose of stimulating discussion. Later, the papers, together with a full report of the discussions, will be published as Monograph No. 5, for wide distribution. Because of the present paper shortage, it is particularly requested that those who are unable to attend the symposium will await the publication of the cloth-bound Monograph.

The meeting is open (free) to all who are interested in the subject, but attendance at the buffet luncheons and the conversazione arranged in connection with the symposium is limited to members of the Institute and of the associated societies.

Authors of papers included in this symposium will not present their papers in person, but the discussion relative to each session will be opened by a Rapporteur, who will briefly review the papers allotted to his Section, and may also direct attention to points bearing on the subject of his Section that are included in other papers in the programme. The Rapporteurs may also indicate those points on which they consider that discussion will be most profitable.

It will greatly assist the Chairmen of the Sessions if those taking part in the discussions will confine their remarks to the specific subject under discussion.

## NEWS AND ANNOUNCEMENTS

### Programme

Wed., 15 Oct., 1947.

10 a.m. to 12 noon. Introductory Address by Colonel P. G. J. Gueterbock, C.B., D.S.O., M.C., T.D., D.L., J.P., M.A., A.D.C., President.

Section I.—*The Measurement of Internal Stresses*. Chairman: Dr. C. Sykes, F.R.S.; Rapporteur: Mr. D. A. Oliver, M.Sc.

12.30 p.m. Buffet luncheon (price 6s.) at the Institution of Mechanical Engineers.

2 p.m. to 5 p.m. Section II.—*The Origin, Control, and Removal of Internal Stresses*. Chairman: Professor L. Aitchison, D.Met., B.Sc.; Rapporteur: Professor H. O'Neill, D.Sc., M.Met.

8 p.m. Conversazione at 4 Grosvenor Gardens, London, S.W.1.

Thurs., 16 Oct., 1947.

10 a.m. to 12 noon. Section III.—*Effects Associated with Internal Stresses*: (a) *Effects on a Microscopic and Sub-Microscopic Scale*. Chairman: Dr. Maurice Cook; Rapporteur: Dr. A. G. Quarrell.

1 p.m. Buffet luncheon (price 6s.) at the Institution of Mechanical Engineers.

2.30 p.m. to 5 p.m. Section III.—*Effects Associated with Internal Stresses*: (b) *Effects on a Macroscopic Scale*. Chairman: Dr. H. J. Gough, C.B., M.B.E., F.R.S.; Rapporteur: Dr. N. P. Allen, M.Met.

### Papers

#### *Section I.—The Measurement of Internal Stresses.*

“Mechanical Methods for the Measurement of Internal Stresses”, by H. Ford, Wh.Sc., Ph.D. (British Iron and Steel Research Association, London).

“The Investigation of Internal Stresses by Physical Methods Other than X-Ray Methods”, by R. King, B.Sc. (Metallurgy Department, Royal Aircraft Establishment, Farnborough).

“Measurement of Internal Stresses by X-Rays”, by D. E. Thomas, M.A., B.Sc. (Principal Scientific Officer, Armament Research Department, Ministry of Supply, Woolwich).

“Some Fundamental Aspects of the Application of X-Rays to the Study of Locked-Up Stresses in Polycrystalline Metals”, by W. A. Wood, D.Sc. (Senior Research Fellow, Baillieu Laboratory, University of Melbourne, Australia).

“The Principles of the Interpretation of X-Ray Photographs of Imperfect Crystals”, by H. Lipson, D.Sc., M.A. (Head of the Physics Department, College of Technology, Manchester).

#### *Section II.—The Origin, Control, and Removal of Internal Stresses.*

##### (a) *Origin.*

“Classification and Nomenclature of Internal Stresses”, by E. Orowan, Dr.-Ing., F.R.S. (Cavendish Laboratory, Cambridge University).

## NEWS AND ANNOUNCEMENTS

- “ László's Papers on Tessellated Stresses : A Review ”, by F. R. N. Nabarro, M.B.E., M.A., B.Sc. (Royal Society Warren Research Fellow, H. H. Wills Physical Laboratory, University of Bristol).
- “ The Relation of Composition to Stress-Corrosion Cracking in Copper Alloys ”, by Maurice Cook, D.Sc., Ph.D. (Director, Imperial Chemical Industries, Ltd., Metals Division, Witton, Birmingham).
- “ Internal Stresses in Steel Castings ”, by H. Elliss, B.Met., Ph.D. (Chief Metallurgist, K. and L. Steelfounders and Engineers, Ltd., Letchworth, Herts).
- “ The Stresses in Large Masses of Steel Cooling from the Austenitic Region ”, by J. E. Russell, M.A. (Research Department, English Steel Corporation, Ltd., Sheffield).
- “ Stress in Electrodeposited Metals ”, by A. W. Hothersall, M.Sc.Tech. (Armament Research Department, Woolwich).
- “ Residual Stresses Due to Welding,” by R. Weck, Ing. (Department of Engineering, Cambridge University).
- “ Internal Stresses Produced by the Sliding of Metals ”, by F. B. Bowden, Sc.D., Ph.D., and A. J. W. Moore, B.Sc. (Department of Physical Chemistry, Cambridge University).
- “ Some Internal Stresses in Turbine Rotors ”, by M. C. Caplan, B.Sc., L. B. W. Jolley, M.A., and J. Reeman, B.Sc. (Research Laboratories, The General Electric Company, Ltd., Wembley).
- “ Residual Stresses in Beams After Bending ”, by G. Forrest, B.Sc. (Senior Research Engineer, Aluminium Laboratories, Ltd., Banbury).
- “ Internal Stresses in Glassware ”, by W. C. Hynd, M.Sc. (Physicist, Development Section, Chance Brothers, Ltd., Smethwick, nr. Birmingham).

### (b) *Control and Removal.*

- “ The Relief of Internal Stresses in Aluminium Alloys by Cold Working ”, by W. Betteridge, B.Sc., Ph.D. (late of the Bristol Aeroplane Company, Ltd., Filton, Bristol; now at The Mond Nickel Company, Ltd., Birmingham).
- “ Stress Relief Treatment of Iron Castings ”. Report of Subcommittee T.S. 17 of the Technical Council of the Institute of British Foundrymen.
- “ Stresses Induced by the Shot-Peening of Leaf Springs ”, by J. C. W. Humfrey, O.B.E., M.Sc., M.Eng., B.A. (Riley, Harbord, and Law, London; formerly S.T.A.M., Ministry of Supply).
- “ The Production of Favourable Internal Stresses in Helical Compression Springs by Pre-Stressing ”, by D. G. Sopwith, B.Sc.Tech. (Senior Principal Scientific Officer, Engineering Division, National Physical Laboratory, Teddington).
- “ Autofrettage ”, by A. G. Warren, M.Sc. (Armament Research Department, Ministry of Supply, Woolwich).

### *Section III.—Effects Associated with Internal Stresses.*

#### (a) *Microscopic and Sub-Microscopic Effects.*

- “ Effects Associated with Stresses on a Microscopic Scale ”, by Sir Lawrence Bragg, O.B.E., Sc.D., F.R.S. (Cavendish Laboratory, Cambridge University).

## NEWS AND ANNOUNCEMENTS

- “ Internal Stresses Arising from Transformations in Metals and Alloys ”, by Professor F. C. Thompson, D.Met., M.Sc. (Professor of Metallurgy, Manchester University).
- “ A Note on the Effect of Internal Stresses on the Rates of Transformation in Iron-Nickel Alloys ”, by C. C. Earley, B.Sc. (Metallurgy Division, National Physical Laboratory, Teddington).
- “ Diffusion and Precipitation in Alloys ”, by F. R. N. Nabarro, M.B.E., M.A., B.Sc. (Royal Society Warren Research Fellow, H. H. Wills Physical Laboratory, The University, Bristol).
- “ Note on the Effect of Cold Work on the Rate of Precipitation in Aluminium-7% Magnesium and Aluminium-8% Magnesium-1% Zinc Alloys ”, by E. C. W. Perryman, B.A. (Investigator, British Non-Ferrous Metals Research Association, London).
- “ Age-Hardening ”, by Marie L. V. Gayler, D.Sc. (Consultant, British Non-Ferrous Metals Research Association, London; formerly Metallurgy Division, National Physical Laboratory).
- “ Internal Stresses and the Formation of Hair-Line Cracks in Steel ”, by Professor J. H. Andrew, D.Sc. (Professor of Metallurgy, University of Sheffield), and Hsun Lee, Ph.D. (Senior Research Assistant, The University, Sheffield).
- “ The Experiments of Boas and Honeycombe on Internal Stresses Due to Anisotropic Thermal Expansion of Pure Metals and Alloys ”, by F. P. Bowden, Sc.D., Ph.D. (Department of Physical Chemistry, Cambridge University).
- “ A Photoelastic Approach to Stress Modifications Caused by Inhomogeneities ”, by B. Sugarman, M.Sc., D.I.C. (B.S.A. Group Research Centre, Sheffield).

### (b) *Macroscopic Effects.*

- “ Influence of Residual Stress on Chemical Behaviour ”, by U. R. Evans, Sc.D., M.A. (Reader in the Science of Metallic Corrosion, Cambridge University).
- “ Chemical Manifestations of Internal Stress ”, by F. H. Keating (Imperial Chemical Industries, Ltd., Billingham Division, Billingham, Co. Durham).
- “ Note on Stress-Corrosion Cracking of Steels in the Presence of Sulphur Compounds ”, by W. P. Rees, M.Sc. (Senior Principal Scientific Officer, Metallurgy Division, National Physical Laboratory, Teddington).
- “ Internal Stresses in Railway Materials ”, by Professor H. O'Neill, D.Sc. M.Met. (Professor of Metallurgy, University College, Swansea; formerly Chief Metallurgist, L.M.S. Railway, Derby).
- “ Delayed Cracking in Hardened Alloy Steel Plates,” by E. H. Bucknall, M.Sc. (The Mond Nickel Company, Ltd., Birmingham), W. Nicholls, B.Sc. (formerly Research Metallurgist, The Mond Nickel Company, Ltd., Birmingham), and L. H. Toft, B.Sc. (The Mond Nickel Company, Ltd., Birmingham).
- “ The Stress System Causing Hard-Zone Cracking in Welded Alloy Steels ”, by J. A. Wheeler, Ph.D. (Department of Metallurgy, The University, Birmingham).



## NEWS AND ANNOUNCEMENTS

### ANNUAL GENERAL MEETING 1948

The dates arranged for the next Annual General Meeting, which will be held in London, are Wednesday, 17 and Thursday, 18 March 1948. If it is found necessary to provide additional time for the discussion of papers, the meeting will commence on Tuesday, 16 March.

### MEMBERSHIP ADDITIONS

The undermentioned 13 Ordinary Members and 7 Student Members were elected on 9 September 1947 :

#### As Members

- ALEXANDER, Martin, B.Sc., Metallurgist, Ministry of Supply, Department of Atomic Energy, Risley, Warrington, Lancashire.
- BARR, William, A.R.T.C., Executive Director and Chief Metallurgist, Colvilles, Ltd., Motherwell.
- BEVER, Professor Michael B., Sc.D., Assistant Professor, Department of Metallurgy, Massachusetts Institute of Technology, Cambridge 39, Mass., U.S.A.
- EASDALE, Robert Muir, Metal Merchant and Refiner, 67 Washington Street, Glasgow, C.3.
- HANDS, Charles Henry, Chief Chemist, Hoover, Ltd., Perivale, Greenford, Middlesex.
- LACOMBE, Paul Jean Gilbert, Dr. es Sci., Laboratoire Central des Traitements Chimiques, Centre National de la Recherche Scientifique, Vitry-sur-Seine, France.
- MCLEAN, John Campbell, Superintendent-in-Charge, H.M. Assay Office, H.M. Mint, Strand Road, Calcutta, India.
- MURRAY, Herbert Spencer Stanley, Managing Director, Electroplaters, Ltd., and associated companies, 31 Townsend Street, Glasgow, C.4.
- MURRAY, Stanley Alexander James, Analytical Chemist; Director, Electroplaters, Ltd., and associated companies, 31 Townsend Street, Glasgow, C.4.
- ROBERTS, Samuel Thomas, Chemical Engineer, International Corrodeless, Ltd., Lockfield Avenue, Enfield, Middlesex.
- RUIMONTE, Professor Dr. Florentino Gomez, Profesor de Quimica Aeronáutica, Departamento de Materiales y Talleres, Instituto Nacional de Técnica Aeronáutica, Narvaez 59, Madrid, Spain.
- WHITELEY, Hubert Augustus, Technical Officer, National Association of Drop Forgers and Stampers, 245 Grove Lane, Handsworth, Birmingham.
- WILLIAMS, Urias, Chief Research Officer, Richard Thomas and Baldwins, Ltd., Light Alloys Section, Briton Ferry, Neath, Glamorganshire.

#### As Student Members

- BENNETT, Bruce Boyd, Chemist, Metropolitan Gas Company, Melbourne, Vic., Australia.
- BUTLER, Ronald, B.Met., Research Metallurgist, Research Laboratories, British Aluminium Company, Ltd., Chalfont Park, Gerrards Cross, Buckinghamshire.

## NEWS AND ANNOUNCEMENTS

- CHRISTIAN, John Wyrill, B.A., Research Assistant, Inorganic Chemistry Laboratory, Oxford.
- COLES, Bryan, B.Sc., Research Assistant (Ministry of Supply) at Oxford University.
- SALAMY, Stanley George, University of Melbourne, Vic., Australia.
- STEINBERG, Morris Albert, Graduate Student of Metallurgy, Massachusetts Institute of Technology, Cambridge 39, Mass., U.S.A.
- THOMPSON, David Laurance, 98 Acre Lane, Bramhall, Stockport, Cheshire.

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## PERSONAL NOTES

MR. K. V. AIYER, B.A., is now in Canada. His address is c/o The Aluminium Company of Canada, Shawinigan Falls, Que., Canada.

MR. R. W. BERRIMAN has left London and is now with the Australian Aluminium Company Pty., Ltd., Granville, Sydney, N.S.W., Australia.

MR. W. C. COPPINS has been awarded the M.Sc. degree of London University.

DR. H. J. GOUGH, C.B., M.B.E., F.R.S., has been honoured by receiving the award of the United States Medal of Freedom with Silver Palm. The presentation was made in London on 21 August 1947. The citation, which was read at the ceremony, was: "Doctor Herbert John Gough, United Kingdom, during the period of active hostilities in World War II, performed exceptionally meritorious service in the field of scientific research and development. An engineer-scientist and leading authority on strength of materials, as Director-General of Scientific Research and Development in the Ministry of Supply, he was responsible for much of the British programme for development of ground force weapons, actively co-operating in establishing and supporting throughout the war arrangements for the Anglo-American exchange of important relevant information."

MR. G. H. HALE is now Assistant Foundry Manager, Frederick Newman and Company, Ltd., Lever Street, Wolverhampton.

MR. G. HOYLE, B.Sc., has left Leeds University and has taken up an appointment with the British Iron and Steel Research Association.

MR. D. M. LEWIS, B.Sc., has taken up an appointment with the British Iron and Steel Research Association, Physics Laboratories, Battersea Park Road, London, S.W.11; he is in charge of high-frequency heating work.

MR. D. MCLEAN, B.Sc., is now at the Metallurgy Division, National Physical Laboratory, Teddington.

MR. G. J. METCALFE, M.Sc.Tech., has joined the staff of the Fulmer Research Institute, Ltd., Stoke Poges, where he is in charge of work on corrosion.

## NEWS AND ANNOUNCEMENTS

MR. T. F. PEARSON, M.Sc., has left Colvilles, Ltd., and is now with the Consett Iron Company, Ltd., Consett, Co. Durham.

MR. J. S. RODGERS, of the Wolverine Tube Division of the Calumet and Hecla Consolidated Copper Company, Detroit, arrived in England on 25 July and plans to return to the United States in the *Queen Elizabeth* on 11 October. He is visiting a number of British metallurgical plants.

DR. CARL H. SAMANS has been appointed Chief of the Metallurgical Section, Research Laboratory, American Optical Society, Southbridge, Mass., U.S.A., replacing Mr. W. J. Wrighton, who has retired after holding the position for 27 years.

DR. C. J. SMITHELLS is on a visit to the United States.

MR. P. VAJRAGUPTA, B.Sc., A.R.S.M., has left London and is now at the Government Mines Department, Bangkok, Siam.

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## DEATH

The Editor regrets to announce the sudden death, on Wednesday, 10 September 1947, of Mr. P. W. ROLLESTON, B.A., Vice-President of Aluminium Laboratories, Ltd., Banbury, Oxon.

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## LOCAL SECTIONS NEWS

### PROGRAMMES FOR THE SESSION 1947-48

Programmes already arranged are detailed below. Members are reminded that (free) membership of the Local Sections is a privilege of membership of the Institute and that those who desire to receive notices of Local Section meetings must notify the responsible Honorary Secretary to that effect.

#### Birmingham.

1947.

- |                  |   |
|------------------|---|
| Thurs., 25 Sept. | Dr. G. Schlesinger : "Machinability".   |
| Thurs., 2 Oct.   | Mr. F. E. Stokeld : Presidential Address to the Birmingham Metallurgical Society. |
| Thurs., 23 Oct.  | Open Discussion : "The Significance of Mechanical Testing".                       |
| Thurs., 6 Nov.   | Dr. G. V. Raynor, M.A. : "The Determination of Constitution Diagrams".            |
| Thurs., 27 Nov.  | Mr. W. E. Ballard : Film on "Metal Spraying" and talk on "Protective Coatings".   |
| Thurs., 4 Dec.   | Students' Evening.  |

## NEWS AND ANNOUNCEMENTS

1948.

- Thurs., 22 Jan. Symposium on "Chain Making".  
 Thurs., 5 Feb. Dr. W. Steven: "Modern Developments in Hardenability Testing".  
 Thurs., 26 Feb. Mr. C. A. H. Jahn: "The Metallurgy of Gold, Silver, and Platinum".  
 Thurs. 4 Mar. Mr. F. Hudson: "Precision Casting" (illustrated by a film).  
 Thurs., 25 Mar. Mr. L. F. Denaro: "The Metallurgy of Alloy Steel Welding".  
 Thurs., 22 Apr. Special meeting of the Local Section.

All meetings take place in the Lecture Hall of the James Watt Memorial Institute, Great Charles Street, Birmingham, at 6.30 p.m. The programme is arranged jointly by the Midland Metallurgical Societies, comprising—in addition to the Local Section of the Institute of Metals—the Birmingham Metallurgical Society and the Staffordshire Iron and Steel Institute.

The Honorary Secretary of the Local Section is Mr. E. H. Bucknall, M.Sc., 53 Halesowen Road, Quinton, Birmingham 32.

London.

1947.

- Thurs. 9 Oct. Mr. E. H. Bucknall, M.Sc.: "The Hardening of Non-Ferrous Alloys and Steels".  
 Thurs., 13 Nov. Professor C. W. Dannatt, A.R.S.M., D.I.C.: "Some Aspects of Metal Refining" (at the Royal School of Mines, South Kensington, S.W.7).  
 Fri., 5 Dec. Annual Dance, at 4 Grosvenor Gardens, London, S.W.1.  
 Thurs., 11 Dec. Mr. G. Skript, Dipl.-Ing.: "The Mechanization of Non-Ferrous Metal Foundries" (Joint Meeting with the London Branch of the Institute of British Foundrymen, at the Waldorf Hotel, Aldwych, W.C.2, at 7 p.m.)

1948.

- Thurs., 8 Jan. Dr. L. B. Hunt, M.Sc.: "Silver".  
 Thurs., 12 Feb. Dr. H. Sutton: "Metallurgical Problems of Importance in Aircraft".  
 Mon., 1 Mar. Major C. J. P. Ball, D.S.O., M.C.: "The Manufacture and Usage of Magnesium and its Alloys". (Joint Meeting with the London Section of the Society of Chemical Industry, at the Rooms of the Chemical Society, Burlington House, Piccadilly, W.1, at 6.30 p.m.).  
 Thurs., 8 Apr. Annual General Meeting (6 p.m.); Open Discussion on "The Oxidation of Metals" (7 p.m.). The subject will be introduced by short papers by Professor N. F. Mott, M.A., F.R.S., and Dr. J. H. Mitchell.



## NEWS AND ANNOUNCEMENTS

Except where stated otherwise, the meetings will be held at 4 Grosvenor Gardens, S.W.1, at 7 p.m.

The Honorary Secretary of the Local Section is Dr. E. C. Rhodes, c/o The Mond Nickel Company, Ltd., Development and Research Department, Bashley Road, London, N.W.10.

### Scottish.

1947.

- Mon., 13 Oct. Mr. G. L. Willan: "The Design and Use of Electric Melting Furnaces in the Non-Ferrous Industry".
- Mon., 10 Nov. Mr. A. Craig MacDonald, B.Sc.: "Commentary on a Visit to America".
- Mon., 8 Dec. Mr. R. E. Wilson: "Electroplating Practice and Engineering Application".

The meetings are held at the Institution of Engineers and Shipbuilders in Scotland, 39 Elmbank Crescent, Glasgow, C.2, at 6.30 p.m. A light snack meal is served before each meeting, from 6 to 6.30 p.m.

The Honorary Secretary of the Local Section is Mr. Matthew Hay, 132 Elliot Street, Glasgow, C.3.

### Sheffield.

1947.

- Fri., 31 Oct. Mr. A. W. Hothersall, M.Sc.Tech.: "Electroplating Problems".
- Fri., 21 Nov. Mr. A. J. Murphy, M.Sc.: "Magnesium Alloys".
- Fri., 19 Dec. Mr. H. Evans, Assoc.Met.: "Surface Finish and Electrolytic Polishing".

1948.

- Fri., 23 Jan. Mr. E. W. Colbeck, M.A.: "Some Metallurgical Problems in the Field of Atomic Energy". Joint meeting, to which members of the Sheffield Society of Engineers and Metallurgists are invited (at the Royal Victoria Hotel, Sheffield, at 6.15 p.m.).
- Fri., 20 Feb. Lecture on "Powder Metallurgy" (name of lecturer to be announced later).
- Fri., 19 Mar. Mr. J. F. B. Jackson, B.Sc.: "Centrifugal Castings". Joint meeting, to which members of the Sheffield Section of the National Trades Technical Societies are invited.

Except where otherwise stated, the meetings will be held in the Mining Lecture Theatre, Department of Applied Science, The University, St. George's Square, Sheffield, at 7.30 p.m.

The Honorary Secretary of the Local Section is Dr. W. R. Maddocks, Department of Metallurgy, The University, St. George's Square, Sheffield.

## NEWS AND ANNOUNCEMENTS

### Swansea.

1947.

- Tues., 14 Oct. Mr. W. E. Ballard: "Metal Spraying" (illustrated by a film) (at the Metallurgy Department, University College, Swansea).
- Wed., 19 Nov. Sir Arthur Smout, J.P.: "The Place of the Metallurgist in Industry" (at the Guildhall, Swansea).
- Tues., 2 Dec. Dr. R. Higgins, B.Sc., and Mr. D. W. Hopkins, B.Sc.: Accounts of visits to Canada and the United States under the auspices of the Nuffield Foundation Scheme (at the Y.M.C.A., Swansea).

1948.

- Tues., 13 Jan. Display of industrial films (at the Metallurgy Department, University College, Swansea).
- Tues., 10 Feb. Mr. W. H. Tait: "Bearings" (at the Y.M.C.A., Swansea).
- Tues., 9 Mar. Lecture on "Continuous Casting" (at the Y.M.C.A., Swansea).
- Tues., 20 Apr. Particulars to be announced later.

The meetings are held at 6.30 p.m.

The Honorary Secretary of the Local Section is Mr. D. W. Hopkins, B.Sc., Metallurgical Department, University College, Singleton Park, Swansea.

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## OTHER NEWS

### ROYAL SOCIETY OF ARTS

H.R.H. Princess Elizabeth has graciously accepted the invitation of the Royal Society of Arts to become its President.

The Royal Family has been closely connected with the Royal Society of Arts for over a hundred years. Prince Albert became the Society's President in 1843, and in that capacity initiated the Great International Exhibition held in Hyde Park in 1851. Other Royal Presidents have been their Royal Highnesses The Prince of Wales (later King Edward VII), The Prince of Wales (later King George V), and The Duke of Connaught.

The Society is the third oldest of the learned Societies in Britain, and will shortly prepare to celebrate its second centenary in July, 1954.

### REPORTS ON JAPANESE INDUSTRY: INDUSTRIAL STATISTICS

A number of reports on technical intelligence from Japan are being published in the B.I.O.S. series and a token free distribution made to Research and Trade Associations, Universities, Public Libraries, and Professional Societies. It should be emphasized,

## NEWS AND ANNOUNCEMENTS

however, that the British Intelligence Objectives Sub-Committee is only authorized to obtain information on processes, &c., developed during the war years and that, therefore, only information falling within this category will be included in the reports.

Certain post-war industrial statistics are, however, now becoming available from American sources, and extracts are being published monthly. These reports will not be given a free distribution, but may be purchased from H.M.S.O. The issues for January and February 1947 are available now, at prices ranging from 1s. 6d. to 2s. 6d. per copy, according to subject.

### METAL FINISHING JOURNAL

*Electroplating* is the title of a new technical monthly dealing with plating, anodizing, enamelling, pickling, lacquering, bronzing, polishing, and phosphating, the first issue of which is scheduled for November. This will be the first British journal to be devoted exclusively to metal finishing.

The editor will be Mr. C. R. Draper, who has been Editor of *Paint Manufacture*, and Assistant Editor of *The Metal Industry*. For the time being he will also be responsible for advertisements and general management. The publishing office is at 83 Udney Park Road, Teddington, Middlesex. The price of the new periodical will be 37s. 6d. per annum.

### DIGEST OF ARTICLES ON DIAMONDS

The Industrial Diamond Information Bureau, Industrial Distributors (1946), Ltd., St. Andrew's House, 32-34, Holborn Viaduct, London, E.C.1, publishes monthly a bulletin containing abstracts of articles dealing with properties and industrial applications of the diamond, together with notices of patents and patent applications in many countries. A copy of this bulletin may be obtained, free of charge, on application to the above address.

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### FORTHCOMING MEETINGS, ETC.

**Chemical Society.**—Professor F. G. Donnan, C.B.E., D.Sc., LL.D., F.R.S. Ernst Julius Cohen Memorial Lecture. (Burlington House, Piccadilly, London, W.1, Thursday, 2 October 1947, at 7.30 p.m.)

**Chemical Society.**—Professor E. G. Cox, D.Sc.: "Crystallographic Technique and its Chemical Significance". (Burlington House, Piccadilly, London, W.1, Thursday, 23 October 1947, at 7.30 p.m.)

**Chemical Society (at Edinburgh).**—Professor W. Wardlaw, D.Sc.: "Modern Ideas of Valency". (Joint meeting with the Local Sections of the Royal Institute of Chemistry and Society of Chemical Industry, North British Station Hotel, Edinburgh, Thursday, 16 October 1947, at 7.30 p.m.)

## NEWS AND ANNOUNCEMENTS

**Chemical Society (at Glasgow).**—Professor H. Nicol, Ph.D.: “From the Highest to the Lowest”. (Joint meeting with the Alchemists’ Club and the Andersonian Chemical Society, Chemical Lecture Theatre, Glasgow University, Friday, 31 October 1947, at 3.45 p.m.)

**Institute of Welding.**—Presidential Address by Mr. J. L. Adam, C.B.E. (Institution of Civil Engineers, Great George Street, London, S.W.1, Wednesday, 29 October 1947, at 6 p.m.)

**Swansea and District Metallurgical Society.**—Presidential Address by Mr. E. A. Davies. (Royal Institution of South Wales, Swansea, Saturday, 18 October 1947, at 6.30 p.m. Members of the Institute of Metals are invited.)

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### APPOINTMENT REQUIRED

**POSITION** required by Technical Sales Metallurgist either at works, home, or abroad. 20 years’ experience in all types of non-ferrous metals, both raw and semi-finished. Box No. 212, Institute of Metals, 4 Grosvenor Gardens, London, S.W.1.

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  - W. Hofmann and W. Jäniche, *Z. Metallkunde*, 1936, **28**, 1 (i.e. year, volume, page).
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# THE APPLICATION OF ETCH-FIGURES ON PURE ALUMINIUM (99.99%) TO THE STUDY OF SOME MICROGRAPHIC PROBLEMS.\*

By PAUL LACOMBE,† D.Sc., MEMBER, and LOUIS BEAUJARD,‡ L. es. Sc.

(TRANSLATED BY NORMAN STUART, Ph.D.)

## SYNOPSIS.

By using an electrolytic polishing method on refined aluminium, it has been possible to develop a method of micrographic attack which reveals etch-figures in a particularly clear manner. The perfection of the geometric form of these figures has enabled a rapid determination to be made of the orientation of the metal crystals from a study of their elements of symmetry.

This has enabled the importance of the relative orientations between adjacent crystals to be related to the nature of the grain boundaries, to the recrystallization due to heat-treatment, and to the mutual plastic deformations of two adjoining crystals. This method of attack has shown, above all, the imperfections in the natural structure of large metal crystals, regardless of the methods by which they are produced, whether by recrystallization or by slow solidification.

Single crystals of aluminium are, in fact, shown by the polyhedral alignments of the etch-figures to be composed of an aggregate of little crystalline "blocks" whose orientations are very slightly, but perceptibly, different. This mosaic of microscopic blocks constitutes the assemblage into a monocrystal such as is revealed by the classical methods of macrographic attack, and examination of a monocrystal by X-rays has enabled this hypothesis to be confirmed and a determination made of the importance of the disorientations.

## I.—INTRODUCTION.

In general, it is known that specular polishing, by either mechanical or electrolytic means, for the purpose of a micrographic examination of metals does not itself suffice to elucidate their structure. It is necessary that polishing should be followed by an appropriate attack to reveal, for example, the state of granulation. The actions of these micrographic reagents differ according to their concentrations and composition, and in the case of a pure and homogeneous metal one may either observe a uniform dissolution of the crystals constituting the polycrystalline aggregate, or a preferential attack of the intercrystalline boundaries or even of certain crystals, depending on their orientation.

\* Manuscript received 15 July 1946.

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One particular class of reagents has a very localized action on the metal, and is characterized by the formation of etch-figures having geometric forms which depend on the crystallographic orientation of the surface being examined. In this work it is intended to show that the interest of this type of micrographic reagent is not limited merely to a determination of crystalline orientations.

A number of earlier workers had already begun to study etch-figures. In particular, the classic work of Osmond and Cartaud<sup>1</sup> should be cited on etch-figures and percussion figures on iron, since the object of their work was to determine what structural modifications were undergone by iron in the course of its allotropic transformations. The more general study made by Portevin in 1923 should also be quoted,<sup>2</sup> since which time, etch-figure reagents have been chiefly used for examining the crystalline orientation of large metal crystals by macrographic methods. These are based on the fact that the facets of the etch-figures are all similarly oriented on the surface of the same crystal and form a system of small mirrors which will reflect a collimated beam of incident light in one definite direction. The orientation of the crystal is thus determined by means of the usual crystallographers' goniometer. The studies by Weerts,<sup>3</sup> and Barrett and Levenson<sup>4</sup> are examples of this method. In their measurements an attempt was made to increase the number of etch-figures on the same crystal by prolonging the duration of the action of the reagent in order to increase the number of small mirrors taking part in the reflection and so increase the brightness of the reflection, and, thus, the precision of the measurement of orientation. But the etch-figures then mask one another in such a way that it is difficult to determine their individual elements microscopically. In the present work, however, we have tried to obtain fewer and larger etch-figures, the geometric proportions of which enable a microscopic examination to be carried out at all magnifications. Furthermore, we have been able to show that there is not a random distribution of etch-figures on the surface of any given crystal, but that this is related directly to its structure.

It has only been possible to complete this study of the etch-figures by taking advantage of the improvements already developed in micrographic technique. For a correct interpretation of the results, the characteristics of etch-figures demand an initial surface as perfectly polished as possible. In the preparation of the metal specimens all scratches or pits are liable to modify, by local deformation, not only the form, but also the more or less regular distribution of the etch-figures. It was in this way that Portevin<sup>5</sup> was able to demonstrate, very clearly, the influence of local deformations due to polishing scratches, on the



etch-figures on an iron crystal, and was also able to show striations due to an accumulation of figures forming right above the old scratches which had been more or less removed by polishing. We have confirmed that the distribution of etch-figures is also very sensitive to the existence of locally cold-worked regions. In order to do this, we made a deep pin-scratch on the surface of a sheet of refined aluminium of coarse grain-size, kept at a temperature of 400° C. In the course of rapid cooling, the most deformed region, corresponding to the centre of the scratch, recrystallized as is shown in Fig. 1 (Plate I), where, on either side of a central band of small crystals corresponding to the bottom of this scratch, the etch-figures which have collected are more or less unorientated and badly formed. It should be mentioned that this observation was made after a slight levelling of the surface had been effected by electrolytic polishing, in order to permit its microscopic examination.

This influence of even a slight cold working on the distribution of the figures demonstrates clearly that any mechanical polishing of the specimens should be forbidden in such a case as the present, because refined aluminium is so soft that it would be difficult to polish it properly without the inclusion of abrasive particles. The new electrolytic methods of polishing, however, obviate these difficulties and above all, avoid producing in the surface an amorphous cold-worked layer due to mechanical polishing, which can disturb the crystalline orientation by its effect on the metal substrate and so alter the form of the etch-figures. Electrolytic polishing is really indispensable in the case of monocrystalline specimens and even in the case of the very large crystals employed in this study. For the depth of the layer disturbed by mechanical polishing is actually greater than that of the amorphous Beilby layer recognized as present in the case of metals having a finer crystal size. In collaboration with Chaudron and Bénard <sup>6</sup> we have recently shown, by the use of X-rays, that on single crystals of aluminium and iron the depth of the disturbance can extend to between 20 and 50  $\mu$ .

Further, by using metals of the greatest possible purity (refined aluminium 99.99%, distilled zinc, sublimed magnesium, electrolytic copper, &c.) one prevents that too rapid multiplication of etch-figures which leads to a mutual encroachment, unfavourable to their regular growth. It is clear that the presence of inclusions, or of finely divided undissolved constituents, will also contribute to those accidental defects in the etch-figures which have no visible connection with the true crystal structure.

This work has been mostly devoted to single crystals, or to specimens composed of large crystals of aluminium, refined to a purity of 99.99%,

prepared by Carpenter and Elam's method <sup>7</sup> of "critical deformation", as well as by a new method of fusion which will be the subject of a further paper. Reference will be made, incidentally, to the results obtained with such other metals as copper and zinc and they will be compared with aluminium.

## II.—CHOICE OF REAGENTS USED FOR DEVELOPING ETCH-FIGURES AND METHODS OF EMPLOYING THEM.

Every etch is preceded by as perfect an electrolytic polishing as possible, for which we have used Jacquet's <sup>8</sup> electrolyte having the composition :

Perchloric acid ( <i>d.</i> 1.55)	. . . . .	345 c.c.
Acetic anhydride	. . . . .	655 c.c.

Aluminium specimens are polished directly in this bath without any previous mechanical treatment. Polishing takes place at a potential of between 22 and 25 V., using the potentiometric set-up recommended by Jacquet. In this way it is easily possible to polish large metal surfaces working at a current density of 0.8–2.5 amp./dm.<sup>2</sup>, which is considerably lower than that necessary in the original series circuit <sup>9</sup> (4–6 amp./dm.<sup>2</sup>).

It is important to note that the perfection of the polishing here obtained determines the subsequent behaviour of the metal towards the reagents used for producing the etch-figures. It is necessary to achieve not only a specular polish on a microscopic scale, but also on a macro-graphic scale. All macroscopic irregularities on the surface caused by too prolonged polishing must be avoided by constant agitation of the specimen during the electrolytic polishing process.

Micrographic etching must follow this polishing with the shortest possible delay, to avoid the disturbing action of the oxide film which forms very rapidly on electrolytically polished aluminium in contact with the air, as has been shown by Morize, Lacombe, and Chaudron.<sup>10</sup>

Finally, it is necessary to choose an appropriate reagent, since its chemical nature and the conditions under which it is used are able to impart to the surface of one and the same crystal, etch-figures of different form, though having a common symmetry. Thus, Mahl and Stranski <sup>11</sup> have recently shown, in the course of a study with the electron microscope, that hydrochloric acid in aqueous or alcoholic solution, or a mixture of hydrochloric and hydrofluoric acids, gives etch-figures on aluminium whose facets are parallel to the (100) cube faces. On the other hand, dry gaseous hydrogen chloride preferentially develops

figures with facets which are parallel to the (111) octahedral planes. Barrett and Levenson<sup>4</sup> further describe a reagent for zinc giving facets parallel to the (101*r*) planes (where *r* can have very different values). In such an exceptional case one might conceivably have an ambiguity in the determination of the crystalline orientation of the micrographic surfaces from the appearance of an etch-figure, and in the case of single crystals of copper we also observed that the facets were not parallel to the (100) planes. Indeed, attack with a warm 10% solution of ammonium persulphate on a copper surface parallel to a cube face, gives etched figures with the octahedral appearance shown in Fig. 2 (Plate I). In the course of what follows, it will be seen that there is a simple way of determining to which planes the faces of such figures are parallel, namely, by the relative orientation of etch-figures and slip-lines. In order that the determination of these orientations may be as simple as possible, it is best to choose a reagent giving etch-facets parallel to those crystallographic planes having the lowest Miller indices.

Thus, on aluminium, for example, the reagent fulfilling this condition is one containing three acids, namely Tucker's formula<sup>12</sup> which is :

15% fuming HNO<sub>3</sub>,  
45% pure HCl,  
25% distilled water,  
15% HF,

while Barrett's and Levenson's formula<sup>4</sup> is :

46% HCl,  
15% HNO<sub>3</sub>,  
10% HF,  
26% distilled water.

These reagents, however, are not suitable for the application proposed here, namely, for a microscopic observation of isolated and well formed figures. The above formulæ, in fact, give very numerous etch-figures but all having a small surface, and if one extends the period of attack, the etch-figures tend to overlap instead of being separate on grains having a particular orientation, thus preventing any subsequent interpretation.

A systematic study convinced us that the presence of intentionally added water must be excluded: progressive dilution of the reagent increases the number and diminishes the size of the figures produced. In the same way a study of the action of each one of the acids taken separately has shown that hydrochloric acid is the chief factor in causing localized attack, whilst hydrofluoric acid has a marked tendency towards

a uniform dissolving action. We thus finally adopted the following composition :

- 47% fuming nitric acid,
- 50% chemically pure hydrochloric acid,
- 3% pure hydrofluoric acid.

The purity of these reagents is necessary in order to avoid any secondary electrolytic action due to the presence of any metallic compounds (particularly traces of iron in hydrochloric acid). It is important to work at a low enough temperature (10° C.) to avoid a multiplication of the etch figures, and to allow their growth from isolated points of attack. The length of the attack depends mainly on the temperature and on the perfection of the polishing, and the time varies from 30 sec. to 1 min.

We have also confirmed the possibility of obtaining etch-figures by electrolytic means; C. H. Desch and S. Whyte<sup>13</sup> used a 5% NaCl solution for electrolytic attack on brass. Mahl and Stranski<sup>11</sup> have developed etch-figures on pure aluminium by an anodic dissolution in concentrated KCl. We have obtained them on both copper (Fig. 3, Plate I) and on aluminium (Fig. 4, Plate I) in the electrolytic polishing bath, by raising the current density towards the end of the polishing procedure. We have not so far been able to make use of this method of attack on aluminium, however, because the figures so obtained are extremely small and much more irregular than those formed by any chemical treatment (Fig. 4, Plate I).

### III.—APPLICATION TO THE STUDY OF THE CHEMICAL ANISOTROPY OF CRYSTALS AND THEIR INTERCRYSTALLINE BOUNDARIES.

Besides etch-figures, the etching agent reveals grain boundaries which are otherwise difficult to recognize on pure, electrolytically polished aluminium. The simultaneous observation of the relative orientation of etch-figures on two adjacent grains indicates that their common boundary is more attacked the greater the difference of orientation that exists between contiguous crystals. Thus, Fig. 5 (Plate II) shows a crystal having triangular etch-figures which seem to be situated in the middle of a single crystal having square figures. In reality, this single crystal consists of several grains of very nearly the same orientation, having boundaries which terminate at the angles of the perimeter of the central crystal. The importance of the intercrystalline region revealed by the reagent thus depends essentially on the respective orientation of adjacent grains. If one assumes with Rosenhain and Ewen<sup>14</sup> that an intergranular "amorphous cement"



is at the origin of the chemical attack on the boundaries, it is difficult to understand why its intensity should be so different according to the respective orientations of neighbouring grains. Either it must be supposed that the width of the amorphous layer is greater, the greater the difference between the orientation of neighbouring grains, or that there exists at their junction, a transitional structure which is more nearly that of the perfect lattice, the nearer the orientation of the two grains is to one another with the consequent continuation of the lattice planes owing to the approximate parallelism of the two crystals.

It could be argued that the particularly marked appearance of the grain boundaries separating two differently orientated crystals is caused by a change of level in the two neighbouring crystals due, in turn, to their unequal velocity of dissolution, either during electropolishing or in the reagent alone. This, however, cannot be the case because: (i) attack by the three mixed acids reagent is restricted (if not too prolonged) to isolated etch-figures and to the intergranular boundaries, and (ii) the microphotograph (Fig. 5), taken with slightly oblique illumination, does not show any relief comparable to that observed in the case when the electrolytic polishing was carried out at a very low current density, as it appears in Fig. 6 (Plate II). Here one sees a preferential dissolution of certain crystals, or a so-called "electrolytic engraving".

This particular resistance to chemical attack exhibited by certain grain boundaries has already been pointed out by a number of authors, and originally by Gough.<sup>15</sup> Etch-figures have enabled us to demonstrate that this is a function of the relative orientation of the crystals, and explains the great difficulty which one encounters when trying to determine their average grain-size in a sheet having such an orientated texture. It is known that cold working by rolling gives rise to a structure having preferential orientations which persist on subsequent recrystallization. The crystallographic elements of this orientated texture vary with the purity of the metal and with the degree of cold working. By a study of the etch-figures we have been able to confirm, in particular, Spillett's<sup>16</sup> X-ray observation that the greatest degree of working produces an orientated texture in the case of aluminium such that the (100) cube face is parallel to the plane of rolling. We have in fact observed a marked predominance of crystals showing square etch-figures in the case of extra pure aluminium which has undergone a degree of cold working of 5.000%\* ("cube texture (100) [001]").

\* Degree of working is expressed by the formula :

$$\frac{S_0 - S}{S} \times 100\%$$

where  $S_0$  = initial thickness  
 $S$  = final thickness.

The difference in behaviour of the grain boundaries as a function of the relative orientations of adjacent crystals, also provides an explanation of Snoek's<sup>17</sup> results on the intercrystalline corrosion of the solid solution FeNi. This alloy, when rolled, gives an orientated recrystallization texture analogous to that of pure aluminium, i.e. having the [001] edge in the direction of rolling, and a (100) face in the plane of rolling. In this "pseudo-single" state of granulation, the alloy completely resists intercrystalline attack at high temperatures by oxygen under reduced pressure. On the other hand, a random equi-axial texture in which the grain boundaries separate crystals having different orientations, undergoes a powerful intergranular attack.

Fig. 7 (Plate II) shows an even more characteristic example of chemical anisotropy observed at times both on crystal surfaces and at their grain boundaries. The difference in the speed of attack is characterized by the more or less numerous occurrences of etch-figures on the various crystalline faces. The enhanced resistance to attack of the surfaces of two grains parallel to the (100) planes (square etch-figures) should be noted, and also the very slight attack on the same boundary, becoming more marked where it separates the crystal parallel to a (100) plane from a pointed crystal containing triangular etch-figures. The difference in the speed of dissolution of the grains according to how they are orientated is even more clearly shown if one prolongs the time of the attack of the three-acids reagent. Fig. 8 (Plate II) shows the appearance of an aluminium sheet consisting of some large crystals which remained for 30 min. in this reagent. Before the attack, the sheet was of uniform thickness, but now presents a change in level from crystal to crystal of as much as 1 mm. By measuring these at known intervals during the attack, one can thus determine, quantitatively, the degree of anisotropy of the crystalline faces. By still further prolonging the attack, it is possible to isolate completely those crystals which have a surface parallel to the (100) cube face.

In conclusion, these anisotropic phenomena connected with the rate of solution explain the appearance of pinhole corrosion on aluminium in certain surroundings. For it is conceivable that a fine-grained sheet composed, for example, of a texture orientated according to the (100) planes would present a greater corrosion-resistance than a sheet having a random granulation. However, it should be remembered that an orientated texture is seldom perfect: certain isolated crystals such as those visible in Fig. 5 (Plate II) are embedded in this preferentially orientated structure, but have an entirely different orientation, and, in consequence, will be much more rapidly attacked than the rest. On



FIG. 1.—Influence of Local Cold Working on the Distribution of Etch-Figures on Refined Aluminium.  $\times 100$ ; reduced 25% in reproduction.



FIG. 2.—Octahedral Etch-Figures on a Single Crystal of Copper Having Its Surface Parallel to a (100) Face.  $\times 500$ ; reduced 25% in reproduction.



FIG. 3.—Octahedral Etch-Figures on the Same Crystal of Copper Obtained by Electrolytic Polishing.  $\times 750$ ; reduced 25% in reproduction.



FIG. 4.—Etch-Figures on Aluminium by Electrolytic Polishing.  $\times 1750$ ; reduced 25% in reproduction.





FIG. 5.—The Influence of the Relative Orientation of the Crystals on the Appearance of Their Intergranular Boundaries.  $\times 500$ ; reduced 25% in reproduction.

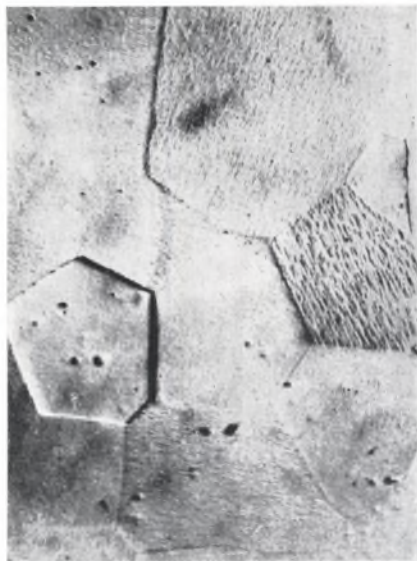


FIG. 6.—Anisotropy of Rate of Crystal Dissolution Appearing in the Course of Electrolytic Polishing.  $\times 220$ ; reduced 25% in reproduction.



FIG. 7.—Resistance to Chemical Attack of Two Grains Having a Surface Parallel to the (100) Cube Faces.  $\times 500$ ; reduced 25% in reproduction.

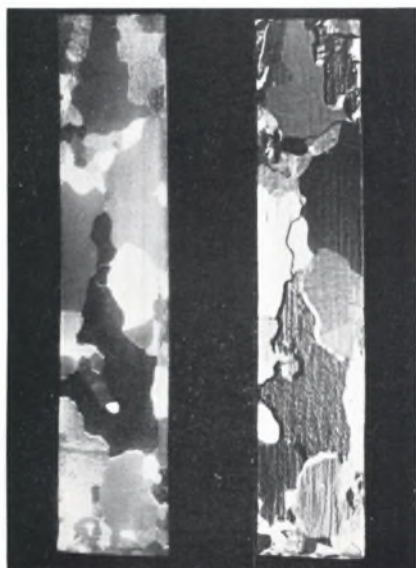


FIG. 8.—Extended Attack on a Coarse-Grained Specimen Confirming the Chemical Anisotropy of the Faces as Revealed by Etch-Figure Frequency.  $\times 1$ ; reduced 25% in reproduction.  
(a) 1-minute attack. (b) 30-minute attack.





FIG. 9.—Pitting Corrosion on a Sheet Having an Oriented Recrystallization Texture.  $\times 2$ ; reduced 25% in reproduction.



FIG. 10.—Appearance of a Complex Slip on Two Successive Octahedral Planes; Systematic Displacement of the Slip Lines at the Meeting Point with the First System.  $\times 750$ ; reduced 25% in reproduction.



FIG. 11.—Influence of Grain Boundary on the Slip-Deformation of Two Adjacent Boundaries.  $\times 750$ ; reduced 25% in reproduction.

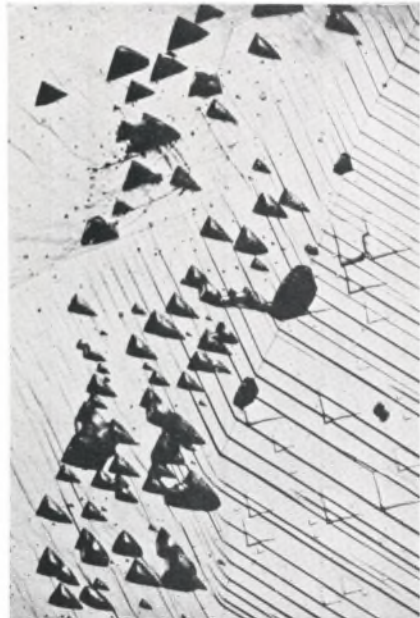


FIG. 12.—Propagation of the Deformation Across the Grain Boundary for Adjacent Grains Having Slightly Different Orientations.  $\times 750$ ; reduced 25% in reproduction.

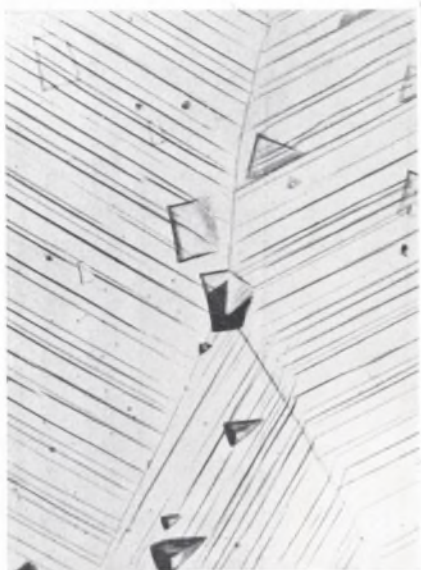


FIG. 13.—Absence of Propagation of Deformation Across Grain Boundaries for Crystals Having Very Different Orientation.  $\times 750$ ; reduced 25% in reproduction.

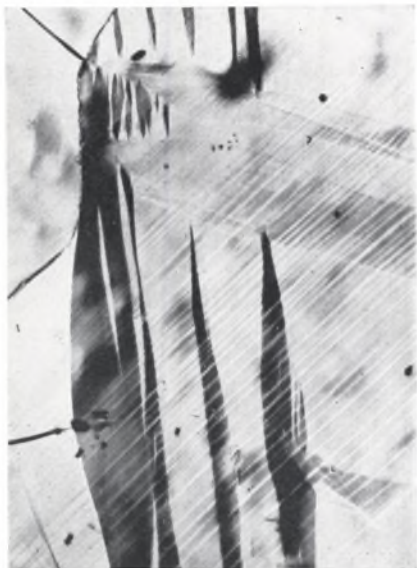


FIG. 14.—Continuity of the Slip Lines Across the Twinning Planes of a Single Crystal of Zinc.  $\times 220$ ; reduced 25% in reproduction.

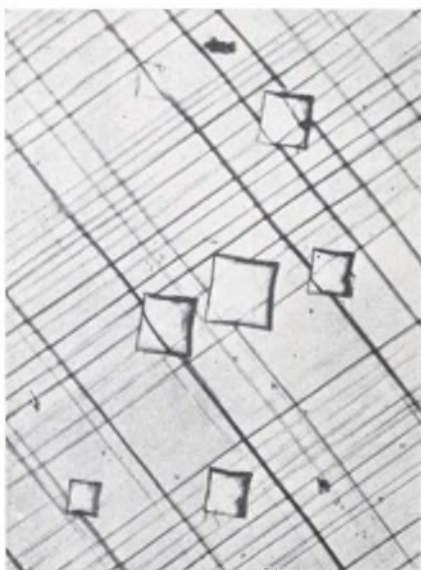


FIG. 15.—Relation Between the Orientation of the Slip Lines of a Crystal Having a Surface Parallel of a (100) Face and the Elements of Symmetry of the Etch-Figures.  $\times 650$ ; reduced 25% in reproduction.



FIG. 16.—Parallelism of the Three Slip Lines with the Sides of the Equilateral Etch-Figures Obtained on a Crystal Surface Parallel to a (111) Plane.  $\times 650$ ; reduced 25% in reproduction.



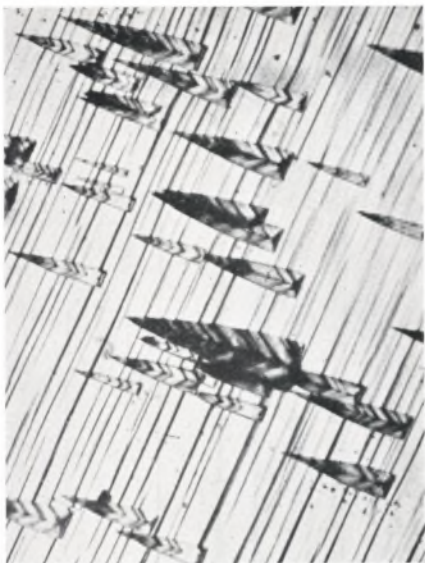


FIG. 17.—Appearance of the Spatial Orientation of the Glide-Planes from Their Tracks on the Etched Facets.  $\times 350$ ; reduced 25% in reproduction.

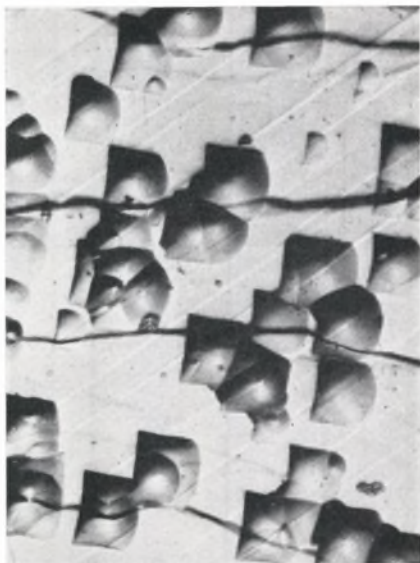


FIG. 18.—Changed the Orientation of the Slip Lines at a High Temperature; extension of a crystal parallel to a (100) face carried out at  $450^\circ$  after a weaker one at room temperature.  $\times 220$ ; reduced 25% in reproduction.

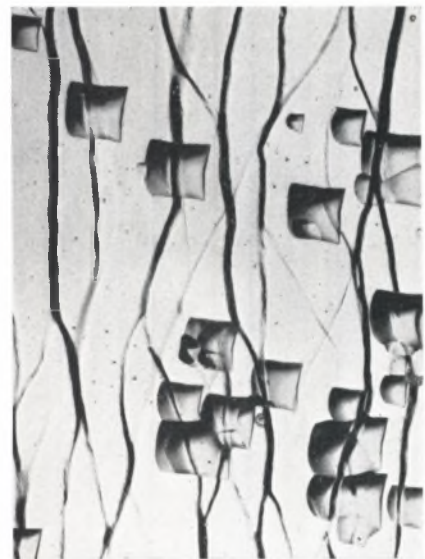
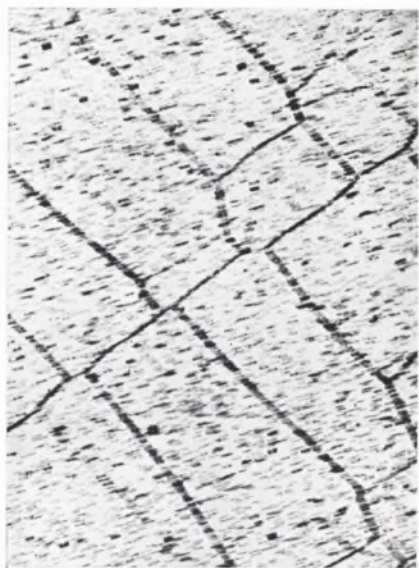


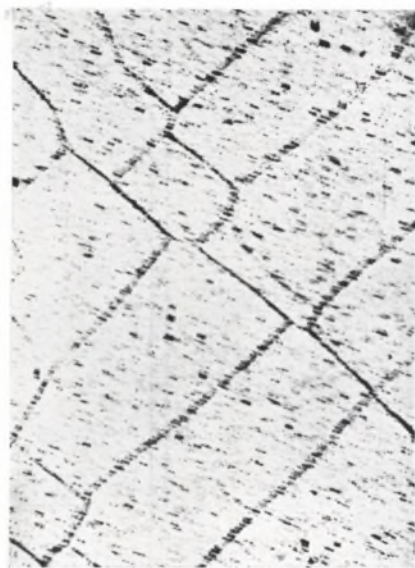
FIG. 19.—Extension of the Same Single Crystal at  $250^\circ$ .  $\times 220$ ; reduced 25% in reproduction.



FIG. 20.—Polyhedral Aspect of Alignments of the Etch-Figures on the Surface of the Same Single Crystal.  $\times 100$ ; reduced 25% in reproduction.



(a)



(b)

FIG. 21 (a) and (b).—Identity of the Network of Etch-Figures Aligned on the Front and Back of One and the Same Crystal.  $\times 100$ ; reduced 25% in reproduction.

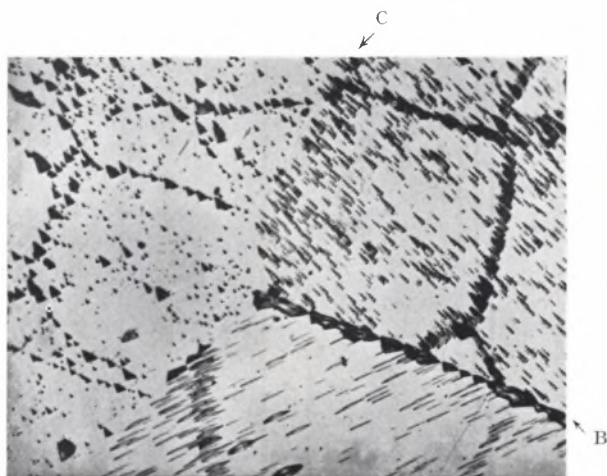


FIG. 22.—Alignments Observed on Three Adjacent Crystals Limited Strictly to the Actual Boundaries (A, B, and C).  $\times 300$ ; reduced 25% in reproduction.



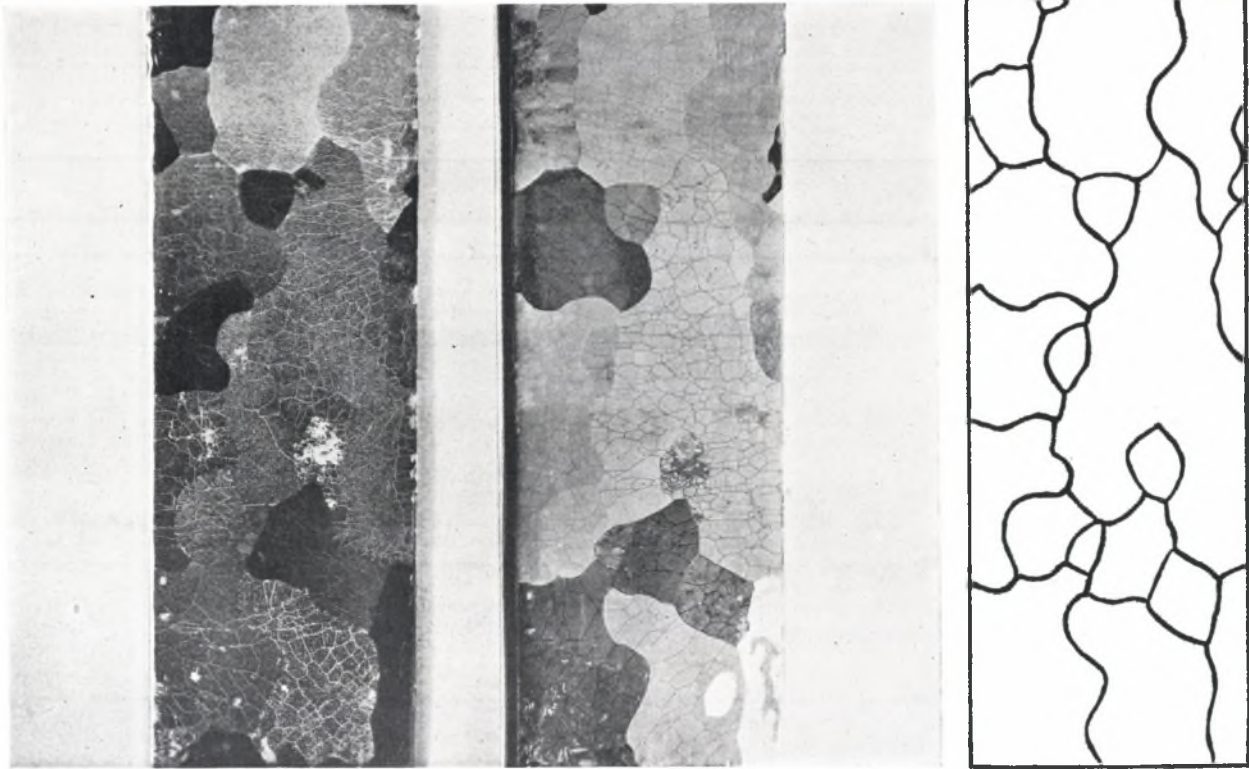


FIG. 23.—Variation of the Position of the Alignments by Prolonged Heating at 600° C. in a Polycrystalline Specimen. (a) Initial condition; alignments of etch-figures. (b) Same specimen after heating for 12 hr.: discontinuities in the oxide layer corresponding to the imperfections of crystals.  $\times 2$ . (c) True intercrystalline limits as they would appear after a classical macrographic attack on a rough specimen (without electrolytic polishing).

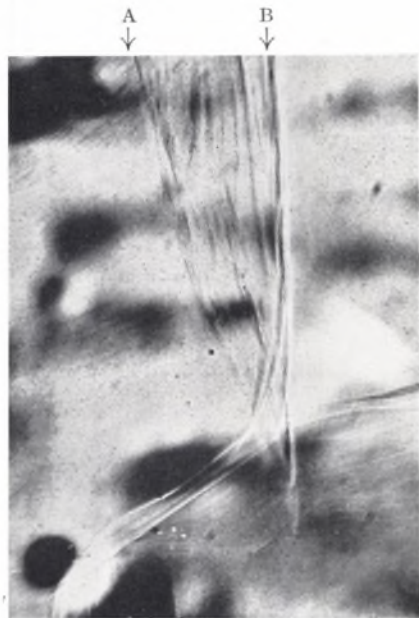


FIG. 24.—Displacement of the Alignments During the Course of Prolonged Heating at  $600^{\circ}\text{C}$ . as Revealed by Atmospheric Oxidation. (A) Original position, (B) final position.  $\times 350$ , oblique illumination; reduced 25% in reproduction.

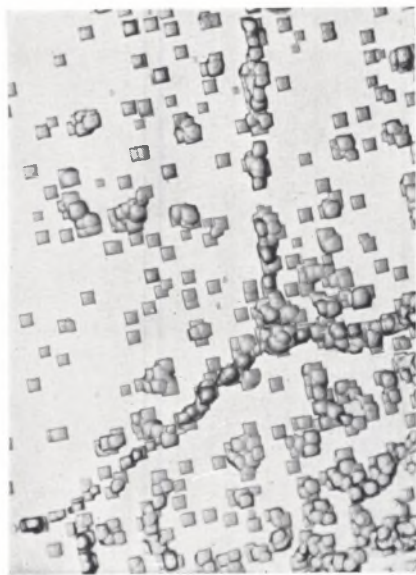


FIG. 25.—Same as Fig. 24 After a New Electrolytic Polishing and Etching; identity of the alignment position revealed by atmospheric oxidation and by the etch-figures.  $\times 350$ ; reduced 25% in reproduction.

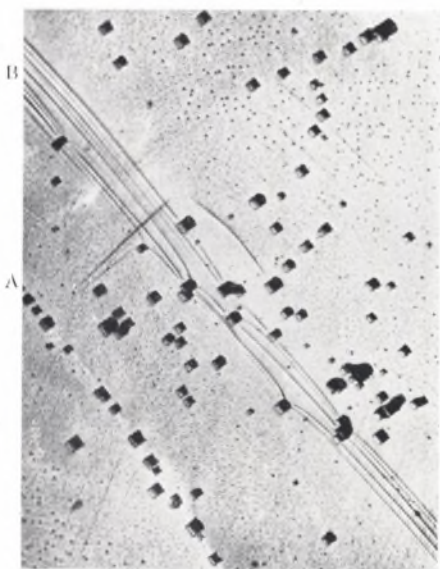


FIG. 26.—Comparison of the Appearance of the Displacement of the Alignments with Those of the Grain Boundaries During the Course of Recrystallization. (A) Original position, (B) final position after heating to  $600^{\circ}$ .  $\times 220$ ; reduced 25% in reproduction.



FIG. 27.—Successive Displacement of the Grain Boundaries by Recrystallization During the Course of One and the Same Heating at  $600^{\circ}\text{C}$ .  $\times 600$ ; reduced 25% in reproduction.

the macroscopic scale one would therefore have the impression that the sheet had sustained a strictly localized form of pinhole attack corresponding precisely to these particular crystals (Fig. 9, Plate III).

#### IV.—A STUDY OF PLASTIC DEFORMATION.

It is known that the simple tensile deformation of a metal generally follows one of two principal mechanisms, e.g. slip and twinning, and these are chiefly determined by the crystal symmetry of the metal. As was first shown by Ewing and Rosenhain,<sup>18</sup> those crystallizing in the cubic system are deformed by a relative sliding along certain crystallographic planes, analogous to cleavage planes, which are the octahedral (111) planes for face-centred metal crystals such as aluminium.<sup>19</sup> After tensile testing of a polycrystalline piece of aluminium, slip bands appear on the surface which are all parallel on any one particular grain, but whose direction varies from one crystal to another according to the different orientations of the crystals in the aggregate and to the general direction of the extension.

In certain crystals, when deformation is continued, new glide-bands appear, parallel amongst themselves but making an angle with the first set of bands which is variable from crystal to crystal. This effect is termed "multiple slipping". It is known that the cubic structure possesses 12 equivalent systems of slip; these are the planes and the directions of slip parallel to the faces and to the edges of the octahedron (111). At its commencement, deformation cannot simultaneously occur along all these possible glide systems, and the slip, in fact, starts in the plane and follows the direction where the shear stress is at a maximum, at the same time producing a rotation of the crystal with respect to the general direction of the tensile force. It would be therefore possible to have two simultaneous slips if the crystal happened initially to present two octahedral planes symmetrically disposed with respect to the direction of tension. This case is, however, rarely encountered, so that almost always there are two glides in succession. According to the relative positions of their elements of symmetry, etch-figures thus permit a rapid determination to be made of the direction of tension if there is a possibility of there having been two simultaneous glidings along the octahedral planes. Fig. 10 (Plate III) shows an example of multiple glide in which it is possible to recognize the order in which the slip-lines have appeared. Indeed, one of the systems of slip-lines is quite straight whilst the other suffers systematic displacements, always in the same direction at every intersection with a line of the former system.



It is also interesting to observe the influence of the grain boundaries upon the glide deformation of aluminium. We have shown, in fact, that those of two adjacent grains have not always been entirely independent of one another. Fig. 11 (Plate III) shows two adjacent grains greatly deformed by a tensile force. A change in the direction of the slip-lines is observable as one crosses the grain boundary due to the orientation of the two grains, but the most important point to note is that the slip-lines in each crystal seem to extend, apart from the slight changes in orientation, right across the grain boundary itself. The lines of deformation, in fact, end in every case on one and the same point in the grain boundary. This appears even more clearly if one observes the gliding due to a small deformation: the slip-lines then take on the strictly rectilinear appearance shown in Fig. 12 (Plate III).

Thus, the deformation of each one of the crystals does not appear to be completely independent of that of its neighbour. It seems difficult to assume, therefore, that the boundary of the grains constitutes a frontier, or rather a channel, filled up with amorphous metal, since any such genuine structural discontinuity would oppose a propagation of the deformation. The hypothesis of a "transition-structure" as first put forward by Gough<sup>15</sup> and Hargreaves,<sup>20</sup> and then by Chalmers,<sup>21</sup> in which it is maintained that the atoms are not scattered at random but are in definable positions in the structure appears very plausible. The importance of this transitional zone would be greater still, the greater the difference in the orientation of adjacent grains. One can conceive that here the propagation of a system of deformations across the crystal boundaries might be more or less continuous, but where the orientations of contiguous crystals are very different there is no longer any continuity of the slip-lines across the grain boundaries as is shown in Fig. 13 (Plate IV).

It should, however, be possible to compare these facts in the case of the deformation of single crystals of zinc, which show both the phenomena of slipping and twinning. In this case, we observed (Fig. 14, Plate IV), besides the change of orientation of the slip-lines on crossing the twins, the same continuity of the slip-lines crossing the twin boundaries, and there can be no question at all here of the presence of amorphous metal in the plane separating the two twins.

We had thought that the use of etch-figures would provide a definite picture of the crystallographic direction on which the deformation by slipping depended. At ordinary temperatures, according to Taylor and Elam,<sup>19</sup> this follows the octahedral (111) planes. This being so, it is sufficient to define the angle formed by the tracks of the (111)



planes on a simple crystallographic plane such as the (100) plane. The orthogonal appearance of the slip-lines in Fig. 15 (Plate IV) does not, however, alone suffice for attributing to the micrographic surface the direction of a (100) plane; there are, indeed, an infinite number of planes on which the tracks of the (111) planes are perpendicular. The use of etch-figures immediately allows us to settle the question. Thus, Fig. 15 shows a crystal where the two systems of slip-lines are more or less rectangular. The etching facets of quaternary symmetry indicate that the surface is indeed one of the (100) planes. It is important to observe that the relative orientation of the slip-lines and the elements of symmetry of the etch-figures is exactly determined. In the case of a single crystal having its surface parallel to a (111) plane (the orientation of which is verifiable by a Laue reflection pattern), one observes that the different systems of slip-lines are exactly parallel to the sides of the equilateral etch-figures (Fig. 16, Plate IV), and these facts provide a concrete confirmation of the octahedral slipping.

It is also possible to locate the position of the glide-planes in space, and to carry out the micrographic attack *before* the metal is deformed. After pulling a single crystal one observes the tracks of the glide-planes appearing like contour lines on the facets of the etch-figures (Fig. 17, Plate V). It is essential to carry out etching before deformation, or else the etch-figures would have entirely smooth facets. One knows that electrolytic polishing or any micrographic attack on a deformed metal, levels the surface and obliterates all traces of the slip-lines. On the other hand, the need to be able to focus simultaneously on the metal surface and bottom of the etch-pits demands that the latter should be large enough to be visible with a low power objective having a large depth of focus.

Finally, we have been able to produce evidence of a change in the direction of the slip-plane when a deformation is carried out at elevated temperature. Thus, Schmid and Boas<sup>22</sup> have shown by X-rays that for aluminium heated above 450° C. the slips follow the (100) faces of the cube. Our experiments on the high-temperature testing of monocrystalline test-pieces show that they do not behave in the same way as during cold deformation. In particular, the slip-lines are no longer geometrically parallel when hot. Thus, Fig. 18 (Plate V) shows a single crystal whose surface is perceptibly oriented along a (100) cube face, which was submitted to a slight extension in the cold, followed by another extension at 450° C. In this way one is most easily able to appreciate the difference between the cold and the hot slip-lines: the former are parallel to the diagonals of the square etch-figures, but the latter show a certain degree of deviation or scatter, although there is a

mean direction which is parallel to the sides of the square etch-figures, thus confirming Schmid and Boas's results.

If deformation takes place at temperatures intermediate between 450° C. and room temperature, the slip-lines take on a branched and irregular appearance (Fig. 19, Plate V). This can be explained by assuming that at these intermediate temperatures, the slipping can take place simultaneously on two sets of planes, namely, the (111) and (100) planes. This hypothesis would appear to be confirmed by the fact that one is able to produce a criss-cross pattern of slip-lines in two average directions, respectively parallel to the sides and to the diagonals of the square etch-figures (Fig. 19).

These observations accord with those established by the X-ray work of Andrade and his co-workers,<sup>23</sup> whose experiments led them to the conclusion that in the case of deformation by slipping it is the direction of slip which is the main and independent variable with rising temperature. The direction of slip is always along one of the planes which is perpendicular to the planes of maximum atomic density, while the slipping at higher temperatures is in the zone containing that direction.

#### V.—STUDY OF THE IMPERFECTIONS IN THE STRUCTURE OF LARGE CRYSTALS OF REFINED ALUMINIUM.

A controlled 30 sec. attack by the three-acid reagent on refined aluminium sheets of 2 mm. thickness, consisting of several large crystals obtained by Carpenter and Elam's method<sup>7</sup> of critical deformation, has enabled us to show a new fact having a direct relation to the internal crystal structure.

Thus, besides the purely chance arrangement of the etch-figures, the reagent produced alignments of figures having a polyhedral appearance on the surface of one and the same crystal (Fig. 20, Plate V). These are not to be attributed to a simple superficial defect in the crystal such as the persistence of local cold work due to a previous scratch, as described by Portevin.<sup>5</sup> Actually, it is possible after successive electrolytic polishings, taking off substantial thicknesses of metal, to reveal the same pattern of alignments. Furthermore, the back and front of one and the same individual crystal reproduce an identical pattern. This, therefore, demonstrates that these alignments are not due to a surface phenomenon, but show up the surfaces of separation existing throughout the whole thickness of a single crystal, and are of the same category as the grain boundaries in a polycrystalline aggregate. Thus, Fig. 21 (Plate VI) shows clearly that the appearances obtained on the

front and back of one and the same crystal are almost identical, the one photograph being naturally the mirror image of the other. The networks are not exactly similar because the surfaces of separation shown up by them are not necessarily perpendicular to the external surface of the crystal, as is the case also with the genuine grain boundaries.

From the polyhedral appearance of these alignments one might ask whether there is any *a priori* case for there being old grain boundaries from the first or even the secondary crystallization which are revealing their former traces by reaction with the three-acid etching agent. Such a persistence in the structure as originally cast, or of recrystallization even after the course of later thermal and mechanical treatment, is not rare. We described striking examples of it in the case of aluminium-magnesium alloys obtained by a new micrographic method of attack based on anodic oxidation.<sup>24</sup> This "heredity" of structure is more easily to be understood in the case of impure metals, where the impurities are thrown down and accumulated during the course of solidification even at the boundaries of dendritic crystals. This hypothesis cannot be upheld in the present case, however, for the following reasons:

(i) These alignments have been observed on aluminium refined to 99.99% whose extremely low impurity content is incompatible with the continuity of the alignment pattern.

(ii) Examination of several large joined crystals forming a single specimen shows that the alignments stop abruptly at the actual grain boundaries without crossing them (Fig. 22, Plate VI). This shows that they are an inherent property of the actual crystal itself and not the "memory" of an earlier crystallization.

(iii) Single crystals prepared by fusion and slow solidification give rise to an alignment of etch-figures having the same characters as those formed on large crystals prepared by starting from the solid state.

(iv) Whatever the length and kind of subsequent thermal treatments that the large crystals are made to undergo, the retention of a former state of granulation in the metal should remain unchanged. Actually, it is observed that a new heat-treatment completely modifies the alignment pattern as if the monocrystal had undergone a new recrystallization or a reorganization of its structure. Figs. 23 (a) and 23 (b) (Plate VII) show a strip of aluminium 2 mm. thick, 2 cm. broad, and 10 cm. long formed from such large crystals that they reveal macrographic attack. The network of lines is quite observably different after heating for some hours at 600° C.

A further means for showing the existence of these lines is available. If an aluminium specimen, which has previously been polished electrolytically, is heated at 600° C. in the presence of air saturated with water vapour, one observes that the surface of the metal is covered by an opalescent oxide film presenting discontinuities as if the oxide film had sustained shrinkage cracks from having been too violently cooled. This is not the case. We have verified, by renewed polishing and a later attack with the three-acid corrodant, that the apparent discontinuities of the oxide film correspond exactly with the lines on the etch-figures (Figs. 24 and 25, Plate VIII). Examination at a larger magnification shows that the opalescence of the oxide layer formed in the atmosphere is due to the accumulation of very small etch-figures which do not appear in the zone occupied by the main alignment. Thus, Fig. 26 (Plate VIII) shows at the same time an old grouping of etch-figures and the displacement of one by a further heating of the crystal. Apart from the large etch-figures formed by the chemical attack, an accumulation of little pits corresponding to the attack by atmospheric oxidation are observable.

The displacement of the alignments in the course of further heat-treatments on single crystals presents an appearance strikingly analogous to the displacement of the boundaries of genuine crystals in the course of corresponding heat-treatments. If an annealed aluminium specimen (which may be fine- or coarse-grained) is re-heated to 600° C., one may note a spontaneous movement of the boundaries of some crystals at the expense of their neighbours. This displacement of the grain boundaries has already been observed by Carpenter and Elam<sup>25</sup> and more recently by Gayler.<sup>26</sup> But we have observed a new effect. The displacement seems to be produced, in several stages during *one and the same heating*, if this is sufficiently prolonged, as in Fig. 27 (Plate VIII), where a multiplicity of grain boundaries is shown. This spontaneous displacement of the boundaries, without previous cold working, cannot be attributed to the growth of certain grains at the expense of others, because of the relative orientations of contiguous crystals. This hypothesis, favoured by certain authors, has not been verified by an examination of the etch-figures on adjacent grains.

All these experimental facts led us to think that the alignments were superficial marks of discontinuities separating the little "blocks" of very slight differences in orientation making up the crystal grain itself. We have actually been able, by the use of X-ray back-diffraction diagrams, to observe a difference of orientation of several minutes (15'-30'). Similar effects have already been announced by Bridgman<sup>27</sup> with zinc crystals obtained by melting and slow solidification. Likewise,



Buerger<sup>28</sup> has formulated a "lineage structure" hypothesis as the result of a very complete study of metal crystals formed by melting. The so-called single crystals as evidenced by macrographic attack would have a branched structure: that is to say, a progressive and continuous variation in orientation from the centre of the crystal to the outside.

The characteristics of dendritic crystal growth envisaged by Bridgman and Buerger are particularly favourable for the formation of imperfections in structure: for during the course of solidification the very slight traces of impurities in the metals are thrown down at the extremities of the dendrites. The cause of the existence of imperfections in crystals formed by recrystallization in the solid state, such as we find in the case of pure aluminium, is much more difficult to understand. The fact that even a prolonged heat-treatment neither makes these discontinuities in the structure disappear nor enlarges the average dimension of the meshes that limit them, leads one to suppose that these imperfections of structure constitute an intrinsic and necessary property of the metallic crystal.

#### VI.—CONCLUSION.

The systematic application of etch-figures has enabled us to show the very different behaviour of crystals and grain boundaries of aluminium according to their respective orientations.

We have further re-established by micrographic methods, the results earlier obtained by X-rays on the subject of deformation at high temperatures. We have shown the existence of discontinuities or imperfections in the structure of large crystals of aluminium obtained both by melting and by recrystallization. These large crystals are composed of an aggregate of little "blocks" whose orientations are very slightly different and whose dimensions are clearly larger than those of the submicroscopic mosaic structure. The possibility of a relation between these two structures is not excluded.

This study would not have been brought to a conclusion but for the use of extra pure, large aluminium crystals and electrolytic polishing prior to etching.

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# ON THE EQUILIBRIUM AND KINETICS OF ORDER-DISORDER TRANSFORMATIONS IN ALLOYS.\*

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## SYNOPSIS.

The equilibrium of order-disorder transformations in alloys is described, in the first approximation, by formulæ giving the structural parts of internal energy and entropy as functions of a suitable measure of the degree of disorder. In the second approximation, the influence of fluctuations is taken into account. The calculations are compared with experimental results of measurements on the internal energy of CuZn.

The kinetics of the transformations are described in conformity with simple diffusion phenomena in ranges of temperature sufficiently far below and above the critical point; but in a range on both sides of the critical point the rate of transformation is influenced by a thermodynamic potential barrier. This influence is theoretically described on the basis of the fluctuation theory, and the calculations are compared with results of X-ray measurements on AuCu.

## I.—INTRODUCTION.

THIS paper gives a concise survey of formulæ developed and used during experimental and theoretical investigations on order-disorder transformations carried out in this laboratory during some twenty years, as well as a comparison of these formulæ with experimental results. The aim of our theoretical work has differed in principle from that begun by Bethe and continued by many theoretical investigators. These investigators start from highly simplified assumptions on the interaction of neighbouring atoms and try to calculate as generally as possible the corresponding partition function and thermodynamic quantities, calculations which have proved to be very difficult. In our work we have avoided such simplifying assumptions on the interactions, which are doubtful from a physical point of view and have tried to deduce, necessarily in a simplified way, formulæ which can describe the phenomena by means of a few empirical constants. The formulæ obtained have been of great help for further experimental investigations.

Most of the formulæ presented here have been discussed in more detail in earlier papers. As, however, some of these papers were published during the war and in periodicals not often read by those

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specially interested in the physics of alloys, this survey might be appropriate.

## II.—GENERAL THERMODYNAMICS OF ORDER-DISORDER TRANSFORMATIONS.

The order-disorder transformations in alloys, the nature of which was established by the investigations of Johansson and Linde<sup>1</sup> in 1925-27, are, at least in the most important cases, characterized by the existence of a continuous series of conceivable, though not always realizable, intermediate states between the states of full order and full disorder.<sup>2</sup> The equilibrium state at a given temperature is the state which makes the thermodynamic potential a minimum; and, if there are no variations of concentration, the thermodynamic problem is solved if the thermodynamic potential is known as a function of the temperature and some measure of the degree of order. For a solid, the thermodynamic potential may with very good approximation be represented by the free energy :

$$F = U - TS \quad . \quad . \quad . \quad . \quad . \quad (1)$$

where  $U$  is the energy,  $S$  the entropy (e.g. of a gram atom of the alloy), and  $T$  the absolute temperature.  $U$  and  $S$  are the result partly of the vibrational motion and partly of the structural arrangement of the atoms. Now, it is a general experience that the vibrational and structural parts are to a great extent independent of each other, the vibrational part being dependent on the temperature only. As we are here only interested in variations of the structural arrangement, we may exclude the vibrational parts from our discussion.<sup>3</sup> Thus in (1)  $F$  is the structural part of the free energy, and  $U$  and  $S$  are functions of the degree of order but independent of  $T$ .

For a purely thermodynamic description of the transformations, the entropy itself may be chosen as a measure of the degree of disorder.<sup>4</sup> The condition that  $F$  has to be a minimum for the state of equilibrium at a given temperature may then be written :

$$\partial F / \partial S = 0,$$

which according to equation (1) gives :

$$\frac{\partial U}{\partial S} = T \quad . \quad . \quad . \quad . \quad . \quad (2)$$

Therefore, if  $U$  is known as a function of  $S$ , it is possible to determine the values of  $U$ ,  $S$ , and  $F$  for the equilibrium state of any temperature; and the transformation is fully described from a purely thermodynamic point of view by the relation  $U = f(S)$ .



As mentioned above, this simple treatment implies that the alloy remains homogeneous at all stages of the transformation. In the general case,<sup>4</sup> the free energy of an alloy system at a given temperature must be considered a function of two variables, the degree of order and the concentration; in addition to homogeneous transformations, cases where an alloy in the equilibrium state is split up into two phases of different concentrations and degrees of order are also to be expected. However, even in such cases the transformation process may appear homogeneous if the changes of concentration are slow in comparison with the changes of order. The present survey will be confined to the treatment of transformations without variations in the concentration of the alloy.

### III.—THERMODYNAMIC AND STATISTICAL TREATMENT OF ORDER-DISORDER TRANSFORMATIONS IN ALLOYS OF THE TYPE *AB*.

Though the thermodynamic method outlined above, with the entropy  $S$  as a measure of the degree of disorder, may be very convenient as a graphic description of the transformations, it is less useful for analytical treatment. All theoretical models show that the energy  $U$  must be a rather complicated function of  $S$ , which cannot be described by algebraic functions. For analysis, it is often convenient to express  $S$  and  $U$  as functions of a third variable. On the basis of a simple theoretical model, the author<sup>3</sup> derived the following formulæ for an alloy of composition  $AB$ :

$$S = -R [q \ln q + (1 - q) \ln (1 - q)] \dots \quad (3)$$

and

$$U = R [\alpha q(1 - q) + \beta q^2(1 - q)^2 + \gamma q^3(1 - q)^3 + \dots] \quad (4)$$

Here  $S$  and  $U$  are referred to one gram atom of the alloy (the sum of  $A$  and  $B$  atoms being equal to the number of atoms per gram atom).  $\alpha$ ,  $\beta$ ,  $\gamma$ , &c., are constants, different for different alloys.  $S$  and  $q$  are mutually connected by equation (3), and  $q$  is thus a thermodynamically fully defined measure of the degree of disorder as well as  $S$  itself. The advantage of  $q$  over  $S$  is that the energy  $U$  may be given in terms of  $q$  in a simple algebraic form.

Equations (3) and (4) were derived by statistical methods from a simple, approximate, theoretical model. This approximate model gives to  $q$  a simple physical meaning, and also gives a physical reason for the representation of the energy  $U$  by a broken series with the exclusion of higher terms as in equation (4). The atomic lattice of a fully ordered alloy of composition  $AB$  may be divided into interpenetrating lattices I and II, one occupied by  $A$ -atoms and the other by  $B$ -atoms. In a state of incomplete order, only a certain fraction,  $q$ , of

the possible positions of lattice I is occupied by *A*-atoms, the other part  $(1 - q)$  is occupied by *B*-atoms. In lattice II, the *A*- and *B*-atoms occupy the fractions  $(1 - q)$  and  $q$ , respectively. In order to facilitate a simple statistical deduction of entropy and energy, the model was simplified by the assumption that the arrangement of atoms in each lattice part is a statistically disordered one. With this assumption, equation (3), governing the entropy ( $S$ ), follows directly from Boltzmann statistics. Equation (4), on the energy, is based on the assumption that the energy of the lattice is built up by forces between neighbouring atoms. If only pairs of nearest neighbours were to be considered, there would be the three types *AA*, *BB*, and *AB* to account for, and all terms in equation (4) would be excluded except the first. The same would be the case even if groups of three atoms were considered. If groups of four or five neighbouring atoms make a contribution to the energy not already accounted for by considering the pairs and groups of three, the second term has also to be taken into consideration. Contributions from groups of six or seven neighbours give the third term and so on. As the forces between the atoms are not known, the constants  $\alpha, \beta, \gamma \dots$  have to be determined by measurement. The most direct way to do this ought to be by suitable calorimetric measurements.

Equations (3) and (4), together with the equilibrium condition (2), which may also be written

$$\frac{\partial U}{\partial q} - T \frac{\partial S}{\partial q} = 0 \quad . \quad . \quad . \quad . \quad . \quad (5)$$

are capable of describing various types of transformation by varying the values of the constants  $\alpha, \beta, \gamma \dots$  in equation (4). Fig. 1 shows some examples of these various types, using diagrams in which the structural energy ( $U$ ), a quantity determinable by experiment, is shown as a function of the absolute temperature,  $T$ .  $U$  is given in calories per gram atom.

Fig. 1 (a) shows how a positive value of  $\alpha$ , the other constants being zero, gives a transformation in which the order ends at a critical temperature with a discontinuity in the heat capacity,  $dU/dT$ , but without any discontinuity in  $U$  itself. A discontinuity of this kind is said to be of the second order, as distinct from discontinuities of the first order with a jump in  $U$ . The special case of a transformation with a discontinuity of the second order described by a single constant  $\alpha$ , corresponds to the approximation of Bragg and Williams, which is discussed in section VII. The transformations experimentally studied in the 50% alloys CuZn, AuCu, and CdMg are, however, all in some disagreement

with this approximation. They all require large negative values of  $\beta$  in addition to the positive value of  $\alpha$ .

Fig. 1 (b), for  $\alpha = 1850$  and  $\beta = -684$ , gives, as is shown in section IV, a first approximation to the case of CuZn. The discontinuity is here still of the second order.

Fig. 1 (c), for  $\alpha = 2325$  and  $\beta = -2250$ , corresponds, as is shown in section VI, to the case of AuCu with a discontinuity of the first order. The dotted continuation of the curves of equilibrium on both sides of the discontinuity correspond to relative but not absolute minima of the thermodynamic potential, i.e. to metastable states. The discontinuity appears at  $680^\circ\text{K}$ .

The term "discontinuities" of various orders is preferred to the term "transformations" of the first, second, or higher order, which has often been used by recent authors. One reason for this is that it is theoretically possible for the same order-disorder transformation phenomenon in an alloy system to have discontinuities of the first or second order in adjacent ranges of concentration passing continuously from one to the other type when the negative value of the constant  $\beta$  passes the limiting value  $\alpha/2$ . It does not seem practical to place one part of this transformation in the same class with changes of modifications and melting phenomena, and the other part in the same class with, for instance, ferromagnetic transformations.

As mentioned above, the theory of order-disorder transformations given by the three equations (3), (4), and (5), was derived statistically

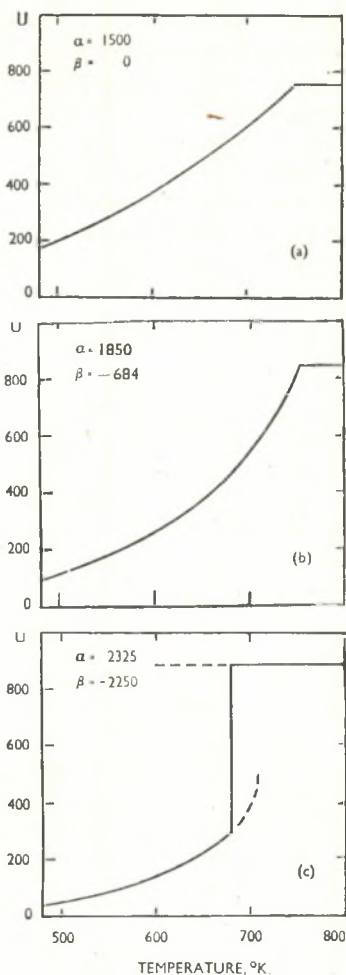


FIG. 1.—Structural Part,  $U$ , of the Internal Energy, in Calories per Mol., Versus Absolute Temperature,  $T$ . (a) Approximation of Bragg and Williams; (b) CuZn alloy, first approximation; (c) AuCu alloy.

on the basis of the simplifying assumption of full disorder within each of the two lattice parts. As a consequence of this simplification, we may not expect  $q$ , as defined by equation (3), to give exactly the fraction of the two kinds of atom in the lattice parts. Neither are the constants  $\alpha$ ,  $\beta$ ,  $\gamma$  in equation (4) exact measures of the contributions to the energy from groups of two or three, four or five, six or seven, &c., neighbouring atoms. Probably, however, this approximation will have very little influence on the possibility of describing various types of transformation by varied sets of constants  $\alpha$ ,  $\beta$ ,  $\gamma$ . . . .

Though the treatment here has been confined to alloys with the composition  $AB$ , there is no major difficulty in extending a similar treatment to other stoichiometric or non-stoichiometric compositions.

#### IV.—THE SMOOTHENING OF DISCONTINUITIES BY FLUCTUATIONS.

The equations (3), (4), and (5) are only able to describe transformations with sharp discontinuities of the first or second order. From certain investigations, especially the measurements of heat capacity of  $\beta$ -brass by Moser<sup>5</sup> and by Sykes and Wilkinson,<sup>6</sup> we know, however, that order-disorder transformations exist without any sharp discontinuity in the energy  $U$  or heat capacity  $dU/dT$ . Whether or not there are any discontinuities in higher derivatives is difficult to decide from the measurements available.

This discrepancy between theory and experiment does not mean that equations (3) and (4) should not be able to describe correctly the connection between energy  $U$  and entropy  $S$  for a given degree of disorder  $q$ . The probable cause of the discrepancy is that a fixed value of  $q$  has been ascribed to the state of equilibrium, the value making the free energy a minimum. This means that we have assumed the degree of disorder to be the same in all parts of the alloy, and have neglected fluctuations in  $q$ , which are apparently not negligible.

The problem of finding a theoretical expression for the energy  $U$  as a function of the temperature  $T$ , taking the fluctuations fully into account also, would, of course, be solved if it were possible to derive a general expression for the partition function from assumptions about the interatomic forces. As is pointed out in section VII, we are at present very far from a solution of this kind.

There is, however, a certain chance of approaching the experimental  $U$ - $T$  curves in two steps, neglecting the fluctuations in the first approximation and taking them into account in the second. The first approximation for an alloy of the type  $AB$  is obtained, as described in section III, by choosing the best possible values for the constants  $\alpha$ ,  $\beta$ , &c., in equation (4).



In order to obtain the second approximation, we first consider the fluctuations in a group of  $l$  atoms,  $l$  being a number large enough to allow the relations (1), (3), and (4) between  $U$ ,  $S$ ,  $F$ , and  $q$  to be valid within the group. The degree of disorder,  $q$ , will fluctuate with the time, giving a distribution function such that the probability of the occurrence of a certain value of  $q$  is greater the smaller the corresponding value of the free energy,  $F$ .

As the energy,  $U$ , of the group also varies with  $q$ ,  $U$  will attain a certain mean value  $\bar{U}$  dependent on the distribution function. The theory of fluctuations <sup>7</sup> gives for this mean value :

$$\bar{U} = \frac{\int_0^{0.5} U e^{-\frac{lF}{RT}} dq}{\int_0^{0.5} e^{-\frac{lF}{RT}} dq} \quad \dots \dots \dots (6)$$

If  $l$  is very large, only the ranges near the minima for  $F$  give any appreciable contribution to the integrals, and for the limit  $l = \infty$  we obtain  $\bar{U} = U$ , corresponding to our first approximation. If, on the other hand,  $l$  is small, the group containing only some hundreds or thousands of atoms; and, if in addition, the minimum of  $F$  is rather flat, or if there are two minima of nearly the same height (cf. Fig. 4); the conditions which are fulfilled near the critical points, there might be an appreciable difference between  $\bar{U}$  and  $U$ . As we know  $U$  and  $F$  as functions of  $q$  and  $T$  from the first approximation, we are, in principle, able to compute the mean value  $\bar{U}$  for an assumed value of  $l$ .

From equation (6) we learn that to obtain the effect of the fluctuations indicated by experimental results, we must calculate with fluctuating groups with rather limited numbers of atoms. As we have no basis for predicting these numbers, the best thing to do is to compute  $\bar{U}-T$  curves according to equation (6) for different values of  $l$  and then get a rough idea of  $l$  by comparison with experiment.

In this way we have found that the results of Sykes and Wilkinson <sup>6</sup> on  $\beta$ -brass (CuZn) may be tolerably satisfied by a constant value of  $l$  of about 1200. Fig. 2 shows a collation of theoretical and experimental results. The dotted line gives the  $U-T$  curve in the first approximation calculated from equations (3), (4), and (5) with  $\alpha = 1850$  and  $\beta = -684$ . The full line gives the  $\bar{U}-T$  curve in the second approximation, calculated from equation (6) with  $l = 1200$ . The circles give values computed from the heat-capacity curves published by Sykes and Wilkinson, which were measured with a co-ordinate microscope and integrated graphically. The agreement between theory and experiment is fairly good in the critical range. The discrepancy at low

temperatures is most probably chiefly due to the specimen here not having been in the equilibrium state, which is reached more and more slowly with decreasing temperature. The fact that the alloy of Sykes and Wilkinson did not have the exact stoichiometric composition influences the result in the same direction.

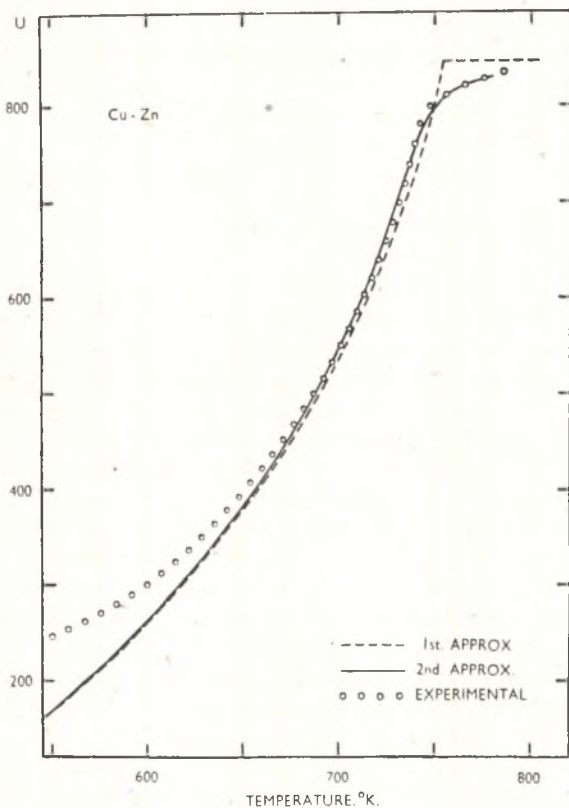


FIG. 2.—Structural Energy,  $U$ , for CuZn, in Calories per Mol., Versus Temperature. Comparison of calculated values with experimental results of Sykes and Wilkinson.

The interpretation of this approximate agreement between experimental results and our theoretical formulæ is difficult, and has to be done with great caution. We are not allowed to conclude that the crystal is divided into domains of about 1200 atoms fluctuating independently. Neither may we look upon the value of  $l$  as a fixed number. It may well be that  $l$  is only an effective mean for a widespread distribu-

tion function. Furthermore, a considerable variation of  $l$  with temperature is not excluded. A conceivable interpretation of  $l$ , worthy of theoretical investigation, is that  $l$  should simply be a lower limit for the number of atoms in groups for which the thermodynamic functions may be defined statistically.

It ought to be mentioned that a theoretical treatment of the fluctuations, somewhat similar to that given here, has been applied by Frenkel,<sup>8</sup> Band,<sup>9</sup> and Mayer and Streeter<sup>10</sup> to melting or condensation phenomena.

Since it has been possible to describe, within, or near to, the limits of experimental error, with only three empirical constants, the rather complicated temperature dependence of the energy content of CuZn over the range where equilibrium states are obtainable, it may finally be emphasized that our approximations appear to be physically sound.

#### V.—KINETICS OF ORDER-DISORDER TRANSFORMATIONS.

The proper way to obtain knowledge of the kinetics of the transformations has proved to be the study of the ordering process of undercooled disordered alloys (or the disordering of overheated ordered alloys) as a function of time at constant temperature. The study may concern the evolution of heat, the changes of some line on an X-ray photograph, or the variation of electrical resistance or some other physical property. For a comparison of the rate of transformation at different temperatures, one may use either the velocity of change or the inverse function, the time needed for some part of the transformation. In the following short survey, we shall use the half-value time,  $\tau$ , which is the time necessary for the first half of the change considered.

If the ordering process is an uncomplicated wandering of the atoms to their new positions on the lattice points, i.e. a pure diffusion phenomenon, we may expect the same dependence of temperature as for other diffusion phenomena, and the half-value time  $\tau$  should approximately satisfy the equation :

$$\tau = \tau_0 e^{\frac{W}{RT}} \dots \dots \dots (7)$$

Here  $W$  divided by the number of atoms in a gram atom is the energy necessary for two atoms to change places,  $R$  is the gas constant, and  $T$  the absolute temperature.  $\tau_0$  is the limiting value of  $\tau$  at infinitely increasing temperature. Its physical meaning is illustrated by results from AuCu, reported in section VI.

In a range of temperature on both sides of a discontinuity of the first order, there are, however, certain complications which were first

observed<sup>2</sup> as a hysteresis difference between resistance-temperature curves taken at increasing and decreasing temperatures. In connection with the publication of these observations, they were already tentatively interpreted as a result of thermodynamic potential barriers. This idea has been further developed on the basis of a discussion of the fluctuations of order necessary to form stable nuclei of the new state. Although it was suggested that the existence of thermodynamic potential barriers must have a great influence on the fluctuations, it has not yet been possible to establish the condition for a stable nucleus from purely theoretical considerations. It has been necessary to search for this condition in a half-empirical way. For this purpose, investigations on order-disorder transformations have proceeded parallel with investigations on the kinetics of precipitation.<sup>11, 12</sup> For the present, the most promising formulation of a condition is that a stable nucleus is received by a fluctuation which makes a group of  $n$  neighbouring atoms surmount the top of the potential barrier. The number  $n$  has to be determined empirically. With this formulation, one obtains from the fluctuation theory, according to an analysis of Einstein,<sup>7</sup> an additional factor to equation (7) for the half-value time giving the extended expression :

$$\tau = \tau_0 e^{\frac{W}{RT}} e^{\frac{n\Delta F}{RT}} \dots \dots \dots (8)$$

where  $n$  is the number of atoms in a fluctuating group and  $\Delta F$  the height of the potential barrier in terms of free energy per gram atom. In ranges of temperature where there is no barrier, the last factor is unity. Taking the natural logarithms of both sides of equation (8) we obtain :

$$\ln \tau = \ln \tau_0 + \frac{W}{RT} + \frac{n\Delta F}{RT} \dots \dots \dots (9)$$

The connection between  $\tau$  and  $T$  is conveniently shown by diagrams with  $\ln \tau$  (or  $\log \tau$ ) and  $1/T$  as variables.

#### VI.—TEST OF THE THEORY ON RESULTS OF X-RAY MEASUREMENTS ON THE KINETICS OF TRANSFORMATION IN AuCu.

The kinetics of the order-disorder transformation in an alloy with the composition AuCu was studied fairly thoroughly by X-ray investigations by Källbäck, Nyström, and Borelius.<sup>13</sup> From the change of certain lines in Debye-Scherrer diagrams, the half-value time  $\tau$  was derived for different temperatures and compared with the predictions of the fluctuation theory. In Fig. 3 the experimental results are plotted as crosses in a  $\log \tau - 1/T$  diagram. The circles on the figure give the results of a renewed calculation according to the formulæ given in the



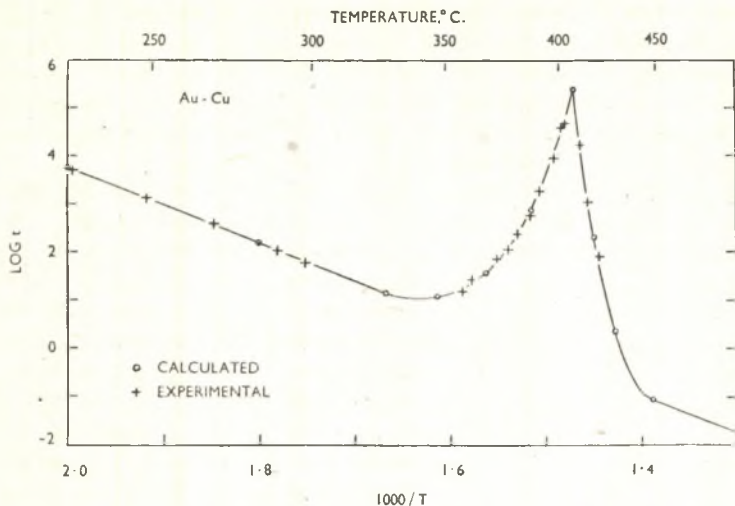


FIG. 3.—Dependence on Temperature of Half-Value Time for the Transitions in AuCu.

foregoing sections, using suitable values of the empirical constants. In order to fit the experiments, equation (9) has to be written :

$$\log \tau = \log 1.1 \times 10^{-12} + \frac{18000 \log e}{T} + \frac{1740 \Delta F \log e}{RT} \quad (10)$$

where the potential barriers  $\Delta F$  are obtained graphically from an expression of the free energy :

$$F = R[2325q(1 - q) - 2250q^2(1 - q)^2 + T(q \ln q + (1 - q) \ln(1 - q))] \quad (11)$$

formed in agreement with the equations (1), (3), and (4), where only the first two terms in equation (4) for the energy are taken into account.

The experimental points in the low-temperature range (200°–300° C.) are located in the diagram on a straight line, determined by the two first terms of equation (10). The slope of this line is  $W/R = 18,000$ , a value that is of the same order as the constants obtained from diffusion in copper, gold, and their alloys. The extension of this line cuts the  $1/T$  axis, giving  $\tau_0 = 1.1 \times 10^{-12}$ . The order of magnitude of this constant is also understandable from a physical point of view. As appears from equation (7),  $\tau_0$  is the half-value time of a transformation when the Boltzmann factor is unity, i.e. when there is no energy hindrance for the atoms to change places. In such a case, one could expect about one exchange per atom and period of thermal vibration. This period is

obtained from the Debye characteristic temperature to about  $2 \times 10^{-13}$  sec. The empirical value of the half-value time  $\tau_0$  is some five times the period of vibration, which, although there are as yet no detailed theoretical investigations on this subject, seems to be a reasonable result. An ordered arrangement may be obtained from a disordered one with a mean displacement of a few steps per atom.

The deviation of the curve from the straight line between  $330^\circ$  and  $440^\circ$  C. is due to the third term on the right in equation (10). The position, extension, and shape of this deviation are determined by the

two constants  $\alpha = 2325$  and  $\beta = -2250$  of equation (11). These constants are obtained by trial. A diagram with  $F$  as a function of  $q$ , used for graphical determination of the potential barrier  $\Delta F$ , is given in Fig. 4. The height of the deviation gives the value of the number,  $n = 1740$ , of atoms in a fluctuating group producing a stable nucleus. Although we have no theoretical ground for dealing with  $n$  as a constant, the result so far gives no reason to assume a dependence of  $n$  on temperature. The order of magnitude of  $n$  seems reasonable, and  $n$  is great enough to justify the statistical background of the last factor in equation (8).

The results of the X-ray investigations on AuCu have also given support in another way to the idea of the influence of thermodynamic potential barriers on transformations. The change of the Debye-Scherrer diagram during the transformation from disorder to order is different in the low-temperature range, where there is no barrier, and in the range of a barrier. The difference is exemplified in Fig. 5 by microphotometer curves for the line (311) for various times of heat-treatment at  $290^\circ$  and  $395^\circ$  C.

At  $290^\circ$  C., where according to Fig. 3 there is no barrier, the transformation passes through a continuous series of intermediate states. At  $395^\circ$  C., where the barrier is well developed, the intermediate states, which are statistically improbable, have a relatively short lifetime and are very evanescent in the photometer diagram.

A further test of the theory should be obtained if the energy constants  $\alpha, \beta, \dots$ , which are here calculated for AuCu from kinetic data, could

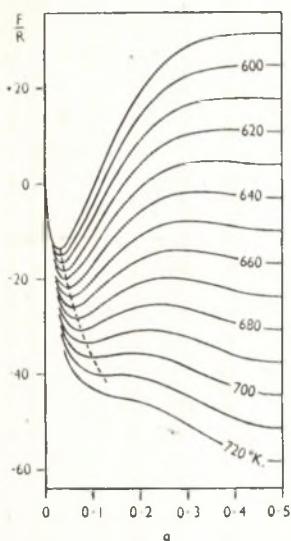


FIG. 4.—Thermodynamic Potential Versus Degree of Disorder for AuCu.

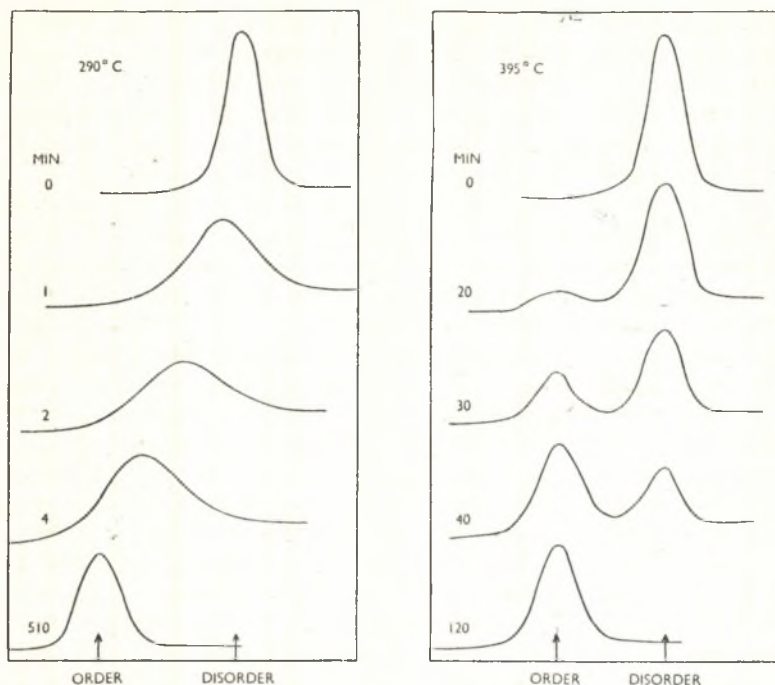


FIG. 5.—Microphotometer Curves for Various Times of Heat-Treatment of AuCu at 290° and 395° C.

be determined independently by calorimetric measurements. The slowness of the transformation near the point of discontinuity, however, prevents usual calorimetric methods from being applied. It is possible that the determination will succeed by special methods now being tried out in our laboratory.\*

#### VII.—COMPARISON WITH APPROXIMATIONS OF BRAGG AND WILLIAMS, BETHE, AND OTHERS.

To the survey of our own investigation on the theory of equilibrium of the kinetics of order-disorder transformations, a few words may be added on the relation of our results to those of other investigators.

At about the same time as the paper describing the theory of equilibrium of transformations, summarized in equations (3), (4), and

\* At the time of passing the proof of this paper in August 1947 preliminary results were obtained on the energy of transformation at 666° K. These results agree with the diagram of Fig. 1 (c).

(5), was published, there appeared an investigation on the same subject by Bragg and Williams.<sup>14</sup> The basic assumption as to the atomic interactions was more specialized than ours and, although formulated in another way, it has been shown by Bethe<sup>15</sup> to be consistent with the assumption that the energy is given exclusively by the number of pairs of nearest atoms of the types *AA*, *BB*, and *AB*. We, on the other hand, had left it to be decided by comparison with experimental results whether interactions between two, four, six, or more atoms were to be considered. Though the methods of discussion in our paper and in that of Bragg and Williams are at first sight rather different, a further analysis shows that all other essential assumptions are the same. Thus our conception of two interpenetrating lattice parts, to which the *A*- and *B*-atoms are referred in the ordered state, and the use of Boltzmann statistics on the assumption that there is a random distribution of *A*- and *B*-atoms within each lattice part, are tacitly understood by Bragg and Williams. They are more directly expressed in the paper of Bethe and in later works from this group of investigators. A difference between these investigators and us in the definition of degree of order is purely formal. The degree of order, *S*, used by Bragg and Williams, Bethe, and others is connected with the degree of disorder, *q*, used in the present paper by the formula  $S = 1 - 2q$ ,  $S = +1$ , and  $S = -1$ , corresponding to  $q = 0$  and  $q = 1$ , means full order;  $S = 0$ , corresponding to  $q = 0.5$ , means full disorder. Thus the approximation of Bragg and Williams is a special case of our theory, obtained by making  $\beta$ ,  $\gamma$ , &c., in the energy expression, equation (4), equal to zero. The *U-T* curve according to Bragg and Williams was shown in Fig. 1 (*a*). It is rather different from all observed curves. A fairly large negative value of  $\beta$  seems to be essential for all experimental curves observed until now.

The possible influence of fluctuations on the state of order of small groups of atoms (order of neighbours) was first pointed out by Bethe,<sup>15</sup> who tried to obtain approximations to the partition function including this influence. This line of investigation has been continued by Kirkwood<sup>16</sup> and others. The statistical problem of computing the partition function has been found very difficult, even with the utmost simplification of the basic assumptions, and only considering interactions between pairs of neighbouring atoms. For the related problem of ferromagnetism, Onsager<sup>17</sup> seems to have succeeded in finding the general solution for a quadratic two-dimensional lattice. It must be emphasized, however, that even if solutions could be found for three-dimensional lattices on the basis of interactions of pairs, these solutions would probably not be able to describe real transformations in alloys.



The mathematical simplification of interactions in pairs, which was considered at an early stage in this discussion,<sup>2</sup> has probably no physical meaning. There are arguments against it from the observed types of two-phase boundaries in alloy systems<sup>11</sup>; and the great empirical values of the constant  $\beta$  in equation (4) indicate the necessity for taking into account the interactions in different groups of at least four atoms. For instance, in a binary alloy  $AB$  with close-packed structure one could expect the small tetrahedral elements of volume, which have the centres of four neighbouring atoms at their corners, to give different contributions to the energy for the different configurations  $A_4$ ,  $A_3B$ ,  $A_2B_2$ ,  $AB_3$ , and  $B_4$ .

The theory of the kinetics of order-disorder transformations was discussed to some extent by Bragg and Williams. They discussed the changes of place of individual atoms, and their results are confined to the first two factors  $\tau_0$  and  $\exp. (W/RT)$  of equation (8). The third factor, which is dependent on the fluctuation of large groups of atoms, was not included in this discussion. Siegel<sup>18</sup> has carried out an interesting experimental investigation on the kinetics of transformation in  $Cu_3Au$ , using variations of the elastic moduli. He found a decrease in the rate of transformation slightly below the critical point, similar to that found by us in the case of  $AuCu$ . The phenomenon was qualitatively described by adding to the formula for the time of relaxation an empirical factor  $\exp. (L/R(T_c - T))$ , where  $L$  is a constant and  $T_c$  the critical temperature. The results have not yet been analysed from the point of view of the fluctuation theory reported in section V.

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# THE CONSTITUTION OF ALLOYS OF ALUMINIUM WITH COPPER AND MANGANESE.\*

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## SYNOPSIS.

The constitution of the ternary alloys aluminium-copper-manganese, as slowly cooled, has been studied in detail by thermal and microscopic methods over the range copper 0-18, manganese 0-8%. The constituents present were found to be  $MnAl_4$ ,  $MnAl_6$ ,  $\alpha(Cu-Mn)$ ,  $CuAl_2$ , and aluminium. Depending on the quantity of copper present,  $MnAl_4$  reacts peritectically with the liquid to form either  $MnAl_6$  or  $\alpha(Cu-Mn)$ ;  $MnAl_6$  reacts peritectically with the liquid to form  $\alpha(Cu-Mn)$ . The invariant points were found to be :

Point.	Phases Present.	Composition, %.		Temperature, °C.
		Cu.	Mn.	
Peritectic. } Ternary } eutectic. }	$MnAl_4$ , $MnAl_6$ , $\alpha(Cu-Mn)$ , Liq.	15.75	2.20	625
	Al, $MnAl_6$ , $\alpha(Cu-Mn)$ , Liq.	14.85	0.90	616
	Al, $\alpha(Cu-Mn)$ , $CuAl_2$ , Liq.	32.5	0.60	547.5

In the primary aluminium field the liquidus temperature is extremely sensitive to changes in copper content and is practically unaffected by changes in manganese content; in the primary  $MnAl_4$  field the converse is true; and in the primary  $MnAl_6$  field the liquidus temperature is affected equally by changes in copper and manganese content.

When copper is added to 1.25% manganese alloys the solidus temperature is lowered:  $\alpha(Cu-Mn)$  appears at 0.3% copper, at 616°C., and  $CuAl_2$  at 0.4% and 547.5°C. This may involve risk of hot-shortness.

Additions of manganese have very little effect on the structure of 4.0% copper alloys until the  $MnAl_6$  field is entered at 1.6%, when there is risk of segregation.

## I.—INTRODUCTION.

THIS investigation is one of a series<sup>1</sup> on the constitution of aluminium alloys under conditions of metastable equilibrium such as are likely to be met with in commercial practice.

There are only a few aluminium-rich alloys of this system which are important commercially. The addition of small quantities of manganese to binary aluminium-copper alloys as a means of improving their strength at high temperature has been suggested by Corson,<sup>2</sup>

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although he points out that these additions will impair the heat conductivity of the alloys.<sup>3</sup> However, little or no commercial application seems to have been made on these lines, and the alloys now in use fall into two main categories. One containing copper 3·9–5·0 with manganese 0·5–1·2% is used for forgings,<sup>4</sup> also in sheet form as a general-purpose alloy of medium strength; and the other, containing copper 1·0–1·5 and manganese 0·7–1·2%, is used for sand castings.<sup>5</sup>

Quaternary and more complex alloys containing aluminium, copper, and manganese are, of course, in widespread use—Duralumin might be mentioned as an important example—and in investigating the structure of these complex alloys it is necessary to know something of the constitution of the ternary systems involved, since binary and ternary constituents of the simpler systems may persist in the presence of other alloying additions.

Manganese may be present as a contaminant in cast and wrought aluminium-copper alloys, and similarly copper may occur in binary aluminium-manganese alloys: a knowledge of the ternary system is therefore helpful in fixing the limits within which such contaminants may be permissible commercially.

## II.—SCOPE OF THE INVESTIGATION.

The work completed in this laboratory<sup>6</sup> some years ago on the aluminium-rich end of the aluminium-copper binary system agrees substantially with that published by Raynor,<sup>7</sup> and the binary system aluminium-manganese is now well established;<sup>8</sup> so no further work was undertaken on the binary systems.

It was decided to study the range copper 0–18, manganese 0–8% in detail, though a few cooling curves were carried out in the range copper 20, manganese 0–8%, and some alloys were studied in the neighbourhood of the ternary eutectic covering a range copper 30–35, manganese 0–2·0%.

The alloys were made up from super-purity aluminium (99·992%). The copper and manganese were added in the form of hardeners containing approximately 50 and 20%, respectively, of the alloying element. The detailed analysis of the hardeners was as shown in Table I.

TABLE I.

	Mn.	Cu.	Si.	Fe.
Manganese hardener, % . . .	19·6	0·0015	0·0045	0·0020
Copper hardener, % . . .	<0·01	50·0	0·003	0·0025



The detailed analysis of some of the alloys given in Table II indicates the order of the quantities of impurities actually present in the alloys studied.

TABLE II.

Nominal Composition, %.		Detailed Analysis, %.			
Cu.	Mn.	Cu.	Mn.	Si.	Fe.
8	5	7.85	5.00	0.0025	0.0020
10	5	15.7	5.1	0.0020	0.0020
16	8	15.9	7.8	0.0015	0.0025

Cooling curves were taken on samples weighing 200 g., and the ingots were afterwards sectioned for microscopic examination. The rate of cooling was in general about 4°–7° C. per minute, though slower and faster rates were employed on occasion. Temperatures were measured by a platinum/platinum–rhodium thermocouple used in conjunction with a Carpenter–Stansfield potentiometer. In the cases where ingots were to be analysed they were remelted and chill cast before analysis to minimize errors due to segregation of manganese-rich constituents.

Copper, if present in amounts of less than 18%, was determined volumetrically by means of thiosulphate.<sup>9</sup> For alloys in the neighbourhood of the ternary eutectic, containing amounts of copper of the order of 32%, the electrolytic method was used.<sup>10</sup> Where copper was present only as a minor impurity, as in super-purity aluminium and certain of the hardener alloys, the estimation was made by the colorimetric method based on the use of sodium diethyldithiocarbamate.<sup>11</sup> Manganese, in general, was estimated photometrically by the periodate process,<sup>12</sup> the figures being confirmed by the volumetric process involving sodium bismuthate as oxidant.<sup>13</sup> If present only as a minor impurity, manganese was determined colorimetrically, potassium periodate being used as the oxidant and the colour matched visually against appropriate standards.<sup>14</sup> Silicon and iron were present only as incidental impurities. Both were estimated colorimetrically, the former by matching the yellow colour developed by ammonium molybdate in acid solution against standard picric acid,<sup>15</sup> and the latter, after removal of the copper, by the thiocyanate process.<sup>16</sup>

### III.—HISTORICAL SURVEY.

The constitution of alloys belonging to the aluminium-rich corner of this system was first studied by Rosenhain and Lantsberry<sup>17</sup> who

identified an aluminium-rich compound, which they called  $MnAl_3$ , but gave no details of the ternary structure. Krings and Ostmann<sup>18</sup> used thermal and microscopic methods to investigate the constitution of the system; they identified no ternary compound, and concluded that the phases  $MnAl_4$ ,  $CuAl_2$ , and aluminium formed a ternary eutectic of composition copper 29.6, manganese 3%, freezing at 536° C.

Sawamoto<sup>19</sup> investigated the system over the range copper 0-40, manganese 0-3%, employing differential thermal analysis and microscopic methods. He reported the occurrence of a peritectic reaction resulting in the formation of  $MnAl_3$  and aluminium at a temperature of 630° C. and a composition of copper 8.3, manganese 1.75%. The ternary eutectic of copper 30.5, manganese 0.65% froze at 544° C., and contained the phases  $MnAl_3$ ,  $CuAl_2$ , and aluminium.

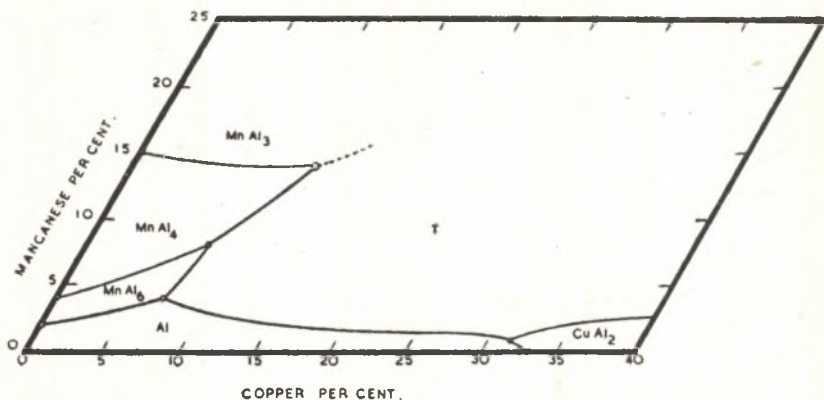


FIG. 1.—The Liquidus Surface (Petri).

Petri<sup>20</sup> used thermal and microscopic methods to make a study of the alloys over the range copper 0-45, manganese 0-17%. The alloys were prepared from aluminium of 99.996% purity, electrolytic copper, and anhydrous manganese chloride, and were studied as slowly cooled and also after prolonged annealing. His liquidus surface is shown in Fig. 1; and his 500° C. isotherm indicating the phases present in alloys which had been annealed in a vacuum for 14 days at 500° C. and then quenched in water, is shown in Fig. 2. The phases present were aluminium containing copper and manganese in solid solution (Al),  $MnAl_6$ ,  $MnAl_4$ ,  $MnAl_3$ , and the ternary phases "T" and "Y"; he did not investigate the latter ternary phase in detail. "T" was a phase of variable composition; its homogeneity limits, under conditions of equilibrium at 500° C., extending between the compositions indicated by the points "T<sub>1</sub>" and "T<sub>2</sub>" in Fig. 2. As Raynor<sup>21</sup> has

pointed out, "T" appears to be based on the composition  $\text{Cu}_2\text{Mn}_3\text{Al}_{20}$ . The point *P* corresponding to this composition has been inserted in Fig. 2, and, as will be seen, lies in close proximity to Petri's point "T<sub>2</sub>". The phases Al,  $\text{CuAl}_2$ , and "T" formed a ternary eutectic containing copper 31.5 and manganese 0.8%, and freezing at 547.3° C. Three peritectic invariant reactions occurred, but owing to undercooling and segregation their temperatures could not be determined accurately.

Liq. +  $\text{MnAl}_6 \rightleftharpoons \text{Al} + \text{"T"}$ , 628.5° C. . . . . Approx. 7% Cu, 4% Mn  
 Liq. +  $\text{MnAl}_4 \rightleftharpoons \text{MnAl}_6 + \text{"T"}$ , approx. 700° C. . . . . " 8% Cu, 8% Mn  
 Liq. +  $\text{MnAl}_3 \rightleftharpoons \text{MnAl}_4 + \text{"T"}$  . . . . . " 12% Cu, 14% Mn

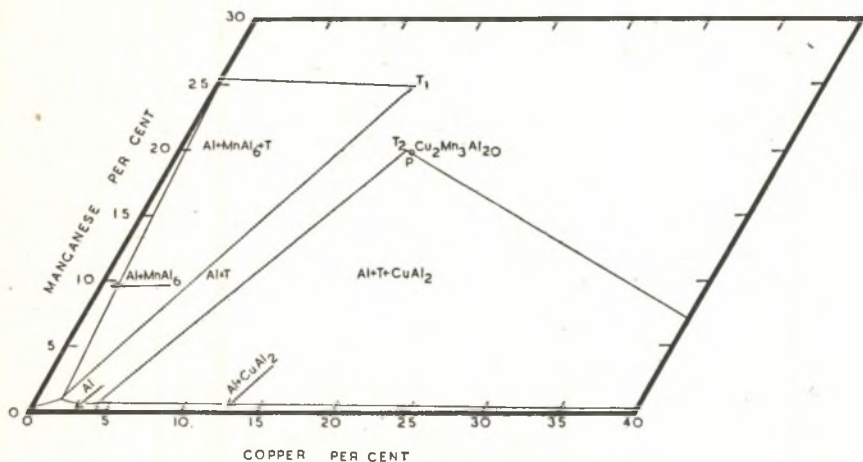


FIG. 2.—The 500° C. Isotherm (Petri).

The constituent "T," extracted from an alloy in which it was primary, was examined by X-rays and found to crystallize in the orthorhombic system ( $a = 7.69$ ,  $b = 24.06$ ,  $c = 12.48$  Å), the particular crystal examined having the composition copper 19 and manganese 24%. The constituent "Y" occurred in alloys rich in copper and manganese, and was also found to be orthorhombic ( $a = 14.79$ ,  $b = 12.60$ ,  $c = 12.43$  Å).

Mondolfo<sup>22</sup> has also studied the system. His liquidus surface showed a continuous phase field,  $(\text{Cu,Mn})\text{Al}_4$ , extending from the  $\text{MnAl}_4$  field on the manganese axis across to the eutectic. He found that the ternary eutectic point had the same composition and melting point as reported by Petri, but concluded that the phases present were  $\text{Al}-(\text{CuMn})\text{Al}_4-\text{CuAl}_2$ . Since he did not identify any distinct ternary body the only peritectic reaction between  $\text{MnAl}_4$  and the liquid which

he reported was that which results in the formation of  $MnAl_6$ : this was limited to alloys in which the copper content did not exceed 19%.

Guertler and Rassmann<sup>23</sup> employed X-rays to study the constitution of alloys of the system under equilibrium conditions and confirmed Petri's<sup>20</sup> diagram as regards phase fields. They reported that the sections  $MnAl_6$ -“T”, Al-“T”, and  $CuAl_2$ -“T” were quasi-binary.

#### IV.—MICROGRAPHY OF THE ALLOYS.

Within the range of compositions studied, copper 0-18 and manganese 0-8%, five phases were identified. They may be listed as follows:

(a) *Aluminium*. This forms the continuous phase in all the alloys investigated. It contains some copper and manganese in solid solution, the amount depending on the conditions of cooling during solidification and subsequently. Variation in the copper content from the centre to the exterior of the aluminium crystallites was sufficient to give rise to visible coring on etching with dilute sodium hydroxide solution or hydrofluoric acid. It would be expected that both copper and manganese would be rejected from solid solution during cooling below the solidus, and that on examination under the highest powers of the microscope it would be possible to detect fine particles of a highly dispersed phase in the aluminium crystallites; but no such particles could be detected.

(b)  $MnAl_6$ . This constituent retains, in the ternary alloys, the characteristic crystal habit which it exhibits in the binary alloys of aluminium and manganese. Hoffmann<sup>24</sup> examined the crystals by X-rays, and found that they crystallized in the orthorhombic system ( $a = 6.51$ ,  $b = 7.57$ ,  $c = 8.87$  Å). The crystals are regular in outline, dendritic in habit, and frequently hollow. They are purplish-pink in colour, and are only lightly attacked by dilute sodium hydroxide solution or hydrofluoric acid. They are frequently surrounded by a sheath of the reaction product  $\alpha(Cu-Mn)$ : where the reaction has taken place in the presence of the liquid phase, the  $MnAl_6$  shows the usual indented appearance characteristic of peritectic attack, but where it has taken place by diffusion in the solid the outlines are smoother, the  $\alpha(Cu-Mn)$  being pseudo-morphic after the  $MnAl_6$ . The boundary between  $MnAl_6$  and  $\alpha(Cu-Mn)$  is sometimes difficult to distinguish in the unetched state, but can readily be rendered visible by etching for  $1\frac{1}{2}$  minutes in 20% nitric acid at 70° C., the etch giving the reaction product a distinct brownish tinge, shown in half-tone in Fig. 3 (Plate



IX). According to Raynor<sup>21</sup>  $MnAl_6$  is practically incapable of taking any copper into solid solution.

(c)  $MnAl_4$ . This constituent occurs as cores in duplex crystals, the sheath being either  $MnAl_6$  or  $\alpha(Cu-Mn)$ , or as the inner core in triplex crystals  $MnAl_4-MnAl_6-\alpha(Cu-Mn)$ . It is purplish-pink in colour, though the colour is less pronounced than that of  $MnAl_6$ . The  $MnAl_4$  cores are roughly hexagonal in outline, showing marked signs of peritectic attack, and often contain central cavities in the form of 6-rayed stars. The boundaries between  $MnAl_4$  and its sheath are extremely difficult to distinguish in unetched specimens, whatever the nature of the sheath, particularly if relief effects have not developed during the final polishing. Etching with 20% nitric acid at 70° C. serves to render visible the boundary between  $MnAl_4$  and  $MnAl_6$  or  $\alpha(Cu-Mn)$  in duplex crystals; and that between  $MnAl_4$  and  $\alpha(Cu-Mn)$ , but, strangely enough, not that between  $MnAl_4$  and  $MnAl_6$ , in triplex crystals. For these crystals, a second etch is necessary: with a few seconds' immersion in 12.5% sodium hydroxide solution the inner boundary  $MnAl_4-MnAl_6$  is well developed, and the  $MnAl_4$  darkened (Fig. 4, Plate IX). An example of a duplex crystal  $MnAl_4-\alpha(Cu-Mn)$  is shown in Fig. 5 (Plate X).

Hoffmann<sup>24</sup> has shown that  $MnAl_4$  crystallizes in the hexagonal system ( $a = 28.35$ ,  $c = 12.36$  Å).

(d)  $\alpha(Cu-Mn)$ . This constituent, termed "T" by Petri,<sup>20</sup> is a phase of variable composition. According to Petri two of the apices of the concentration area representing its homogeneity range are the points "T<sub>1</sub>" (Cu 13, Mn 24.9%) and "T<sub>2</sub>" (Cu 15, Mn 20%), indicated in Fig. 2. In the present investigation the composition of  $\alpha(Cu-Mn)$  which fitted the experimental results best was found to be copper 19, manganese 24%: this agrees with the composition of the sample of "T" which Petri extracted and on which he made his X-ray measurements. Over a certain range of compositions  $\alpha(Cu-Mn)$  separates directly from the melt: elsewhere it occurs as the product of a peritectic reaction between  $MnAl_4$  or  $MnAl_6$  and the liquid. It is more rounded in outline than either  $MnAl_4$  or  $MnAl_6$ , and is rather darker in colour in the unetched state. Its characteristic crystal form is shown in Fig. 6 (Plate X). It is isomorphous with the ternary phases  $\alpha(Mn-Si)$  and  $\alpha(Fe-Si)$ .<sup>25, 26</sup>

(e)  $CuAl_2$ . This designation has become established by long usage and will therefore be retained, although, as Stockdale<sup>27</sup> has shown, an alloy of which the composition corresponds with the formula lies just outside the region of homogeneity of the phase. It has been found from X-ray investigation that  $CuAl_2$  crystallizes in a body-

centred tetragonal structure with 8 Al and 4 Cu atoms per unit cell ( $a = 6.05$ ,  $c = 4.86$  Å).<sup>28, 29, 30</sup> Over the range of compositions studied,  $\text{CuAl}_2$  appears only as one of the constituents of the ternary eutectic. In the unetched state it is pale pink in colour, and it is readily identified by its reaction with the hot nitric acid etch, which blackens it. Raynor<sup>21</sup> has shown that it is practically incapable of dissolving any manganese.

#### V.—THE LIQUIDUS SURFACE.

The liquidus surface is shown in plan in Fig. 7. It is composed of four primary phase fields:

Aluminium	.	.	.	.	.	.	ABCKH
$\text{MnAl}_6$	.	.	.	.	.	.	HKLG
$\alpha(\text{Cu-Mn})$	.	.	.	.	.	.	KCDL
$\text{MnAl}_4$	.	.	.	.	.	.	GLDEF

The primary aluminium field is bounded by the  $\text{MnAl}_6$ -aluminium eutectic valley  $HK$  and the  $\alpha(\text{Cu-Mn})$ -aluminium eutectic valley  $KC$ ; beyond the range of composition studied the valley  $KC$  runs down to the ternary eutectic point  $T$ , and the remaining side of the field is closed by the  $\text{CuAl}_2$ -aluminium eutectic valley.

The temperature and composition corresponding with the ternary eutectic point  $T$  were deduced from cooling-curve arrests and the examination of a number of micro-sections of slowly cooled alloys of compositions within the range copper 30–35 and manganese 0.25–2.0%, the best estimate being copper 32.50 and manganese 0.60% at a temperature of 547.5° C. It was found that the eutectic was so segregated that analysis of an apparently all-eutectic area would have been misleading.

The primary  $\text{MnAl}_6$  field is separated from the  $\text{MnAl}_4$  and  $\alpha(\text{Cu-Mn})$  fields by the lines  $GL$  and  $LK$ , respectively, each of which is associated with a peritectic reaction.  $GL$  is associated with the peritectic reaction between  $\text{MnAl}_4$  and the liquid to form  $\text{MnAl}_6$ , and  $LK$  that between  $\text{MnAl}_6$  and the liquid to form  $\alpha(\text{Cu-Mn})$ .

The compositions and temperatures corresponding with the invariant points  $K$  and  $L$  were determined by interpolation from the phase boundaries and from the liquidus isothermals, and are shown in Table III.

The only boundary of the  $\alpha(\text{Cu-Mn})$  field which has not yet been mentioned is  $DL$ : this is associated with the peritectic reaction between  $\text{MnAl}_4$  and the liquid to form  $\alpha(\text{Cu-Mn})$ .

It is interesting to note that the atomic percentages of points lying on the isothermals of the  $\text{MnAl}_4$  field, when plotted on a log-log



FIG. 3.— $MnAl_6$  Partly Surrounded by  $\alpha(Cu-Mn)$  in Slowly Cooled Alloy Containing Copper 8.0 and Manganese 3.0%. (No  $MnAl_4$  present, due to undercooling.) Specimen etched  $1\frac{1}{2}$  min. in 20%  $HNO_3$  at  $70^\circ C.$   $\times 150$ .

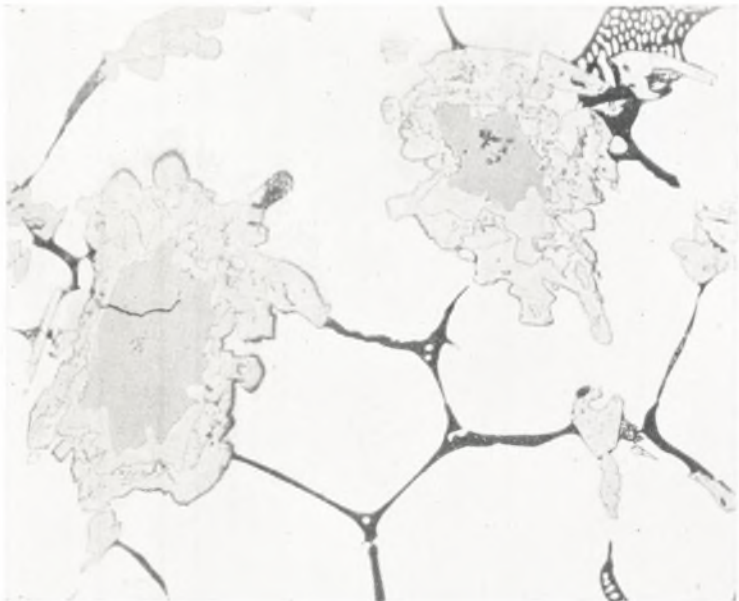


FIG. 4.—Triplex Crystals of  $MnAl_4$  Core in  $MnAl_6$  Surrounded by  $\alpha(Cu-Mn)$  in Slowly Cooled Alloy Containing Copper 8.0 and Manganese 4.0%. Specimen etched  $1\frac{1}{2}$  min. in 20%  $HNO_3$  at  $70^\circ C.$ , followed by dip in cold 12.5%  $NaOH$ .  $\times 175$ .

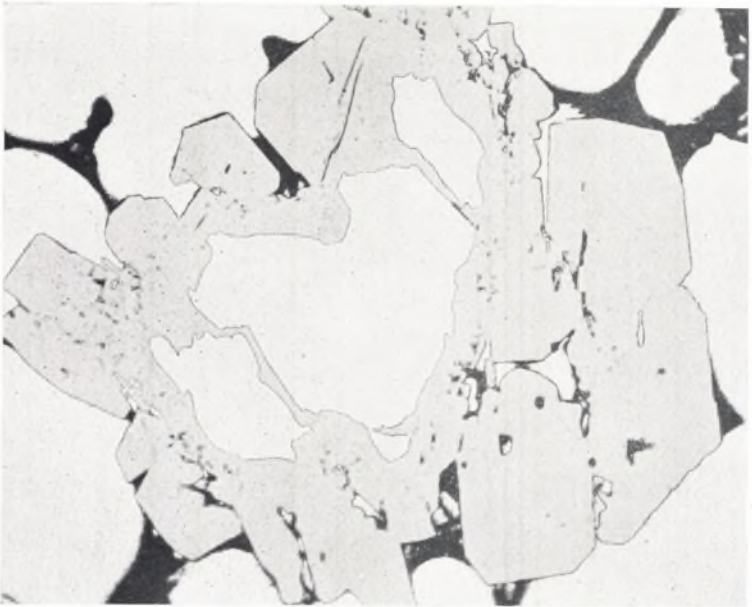


FIG. 5.— $MnAl_4$  Surrounded by  $\alpha(Cu-Mn)$  in Slowly Cooled Alloy Containing Copper 14 and Manganese 8.0%. Specimen etched  $1\frac{1}{2}$  min. in 20%  $HNO_3$  at  $70^\circ C.$   $\times 150$ .

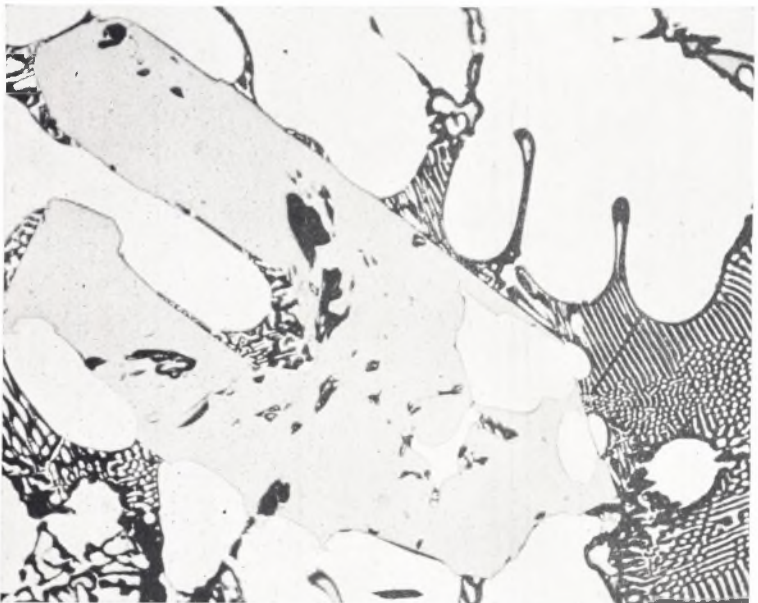


FIG. 6.—Primary Crystals of  $\alpha(Cu-Mn)$  and Eutectics Area in Slowly Cooled Alloy Containing Copper 20 and Manganese 2.0%. Specimen etched 20 sec. in 20%  $HNO_3$  at  $70^\circ C.$   $\times 150$ .



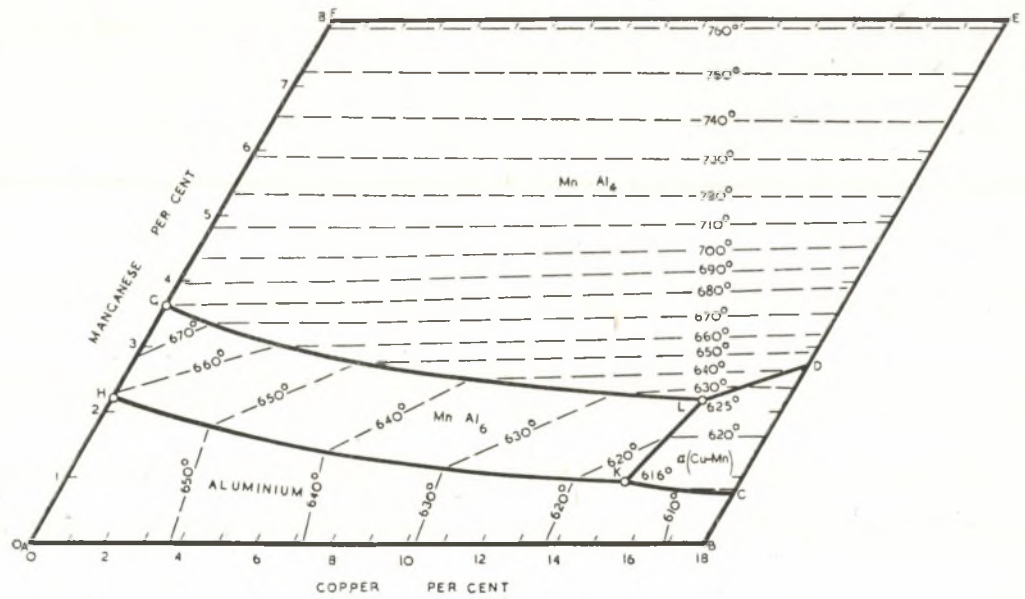


FIG. 7.—The Liquidus Surface.

scale, do not conform with a gradient of 1:4 as might have been expected from the Law of Mass Action. The 750° C. isothermal has been plotted in this manner in Fig. 8 and might lead one to suppose, from its slope, that the compound separating contains manganese and aluminium in equal atomic proportions.

TABLE III.—*Ternary Invariant Points: Liquid Surface.*

Point.	Phases Present.	Composition, %.		Temperature, ° C.
		Copper.	Manganese.	
<i>K</i>	Al, MnAl <sub>6</sub> , α(Cu-Mn), Liq.	14.85	0.90	616
<i>L</i>	MnAl <sub>4</sub> , MnAl <sub>6</sub> , α(Cu-Mn), Liq.	15.75	2.20	625
<i>T</i>	Al, α(Cu-Mn), CuAl <sub>2</sub> , Liq.	32.50	0.60	547.5

The Le Chatelier<sup>31</sup> form of the equation connecting the apparent solubility product *K* of an intermetallic phase with temperature is :

$$\text{Log } K = \frac{\Delta H}{R} \left( \frac{1}{T_0} - \frac{1}{T} \right)$$

where  $\Delta H$  is the difference in heat content between reactant and resultant, and includes the heat of formation and the latent heat of

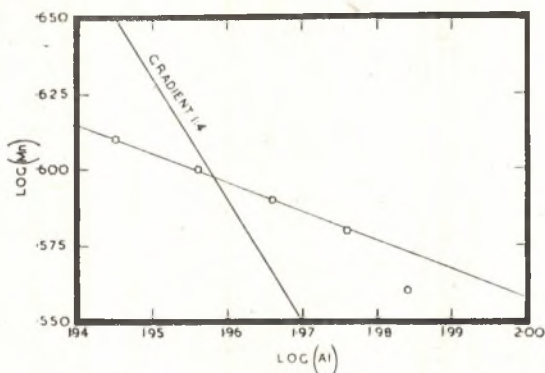
750° C. Isothermal of the Primary MnAl<sub>4</sub> Field.

FIG. 8.

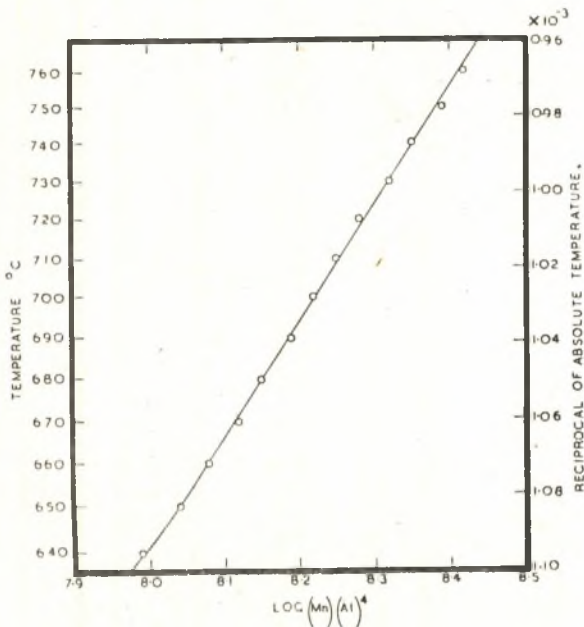
fusion of the compound concerned;  $T_0$  is the freezing point of the pure substance,  $T$  is the operating temperature, and  $R$  the gas constant.

Fig. 9 shows the application of this equation to the liquidus isothermals of the primary MnAl<sub>4</sub> field. It will be seen that the graph is linear over a wide range of temperature; the value of  $\Delta H$  calculated

from the slope of the graph was found to be  $1.49 \times 10^4$  cal./g. mol, which is equal to 91.7 cal./g.

The intersections of the liquidus isothermals with the univariant lines comply in all cases with the Lipson-Wilson<sup>32</sup> rules for ternary equilibria.

It may be noted that in the primary aluminium field the liquidus



Variation of the Apparent Solubility Product with Temperature.

FIG. 9.

isothermals run practically parallel to the manganese axis, so that additions of manganese have no appreciable effect on the liquidus temperature, whereas this temperature is extremely sensitive to changes in copper content. In the primary  $MnAl_6$  field, variations in the copper and manganese content have equal effects on the liquidus temperature, and in the primary  $MnAl_4$  field, the isothermals run practically parallel to the copper axis so that the liquidus temperature is very sensitive to changes in the manganese content and is unaffected by changes in the copper content.

The above-mentioned points are important when considering the possible compositions of hardener alloys; it will be seen that in the  $MnAl_4$  field the liquidus temperature rises very rapidly with small

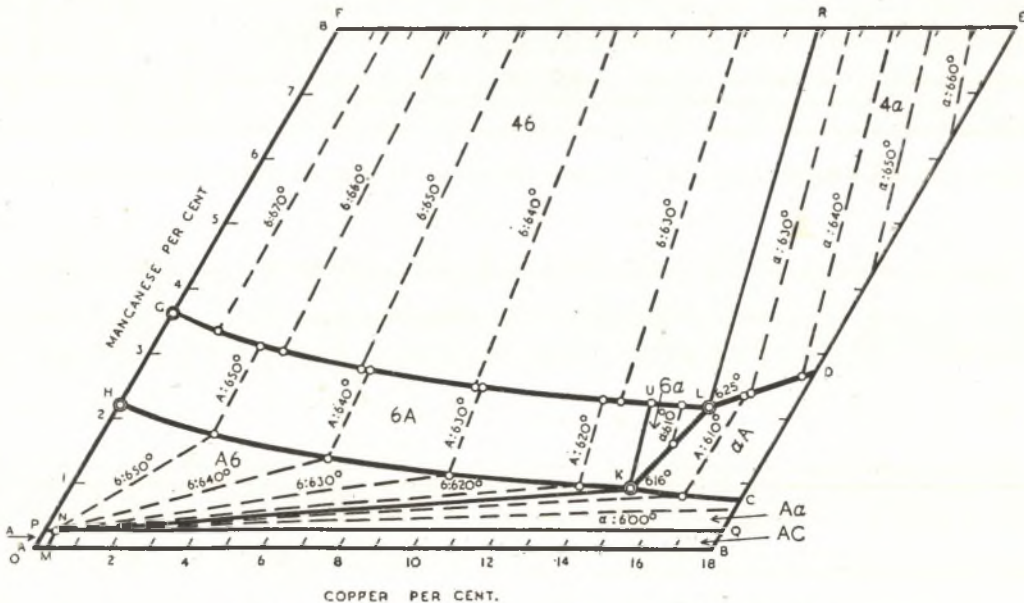


FIG. 10.—Secondary Separations. Alloys as slowly solidified.



increases of manganese, so that close analytical control of this element would be essential.

#### VI.—REACTIONS BELOW THE LIQUIDUS.

The surfaces of the secondary and later separations are shown in plan in Figs. 10 and 10 (*a*). The key to the symbols used is given in Table IV.

TABLE IV.—*Symbols Used in Figs. 10-16.*

<i>A</i>	Aluminium	dashed isothermals; secondary separation.
<i>4</i>	MnAl <sub>4</sub>	dotted isothermals; ternary separation.
<i>6</i>	MnAl <sub>6</sub>	
<i>α</i>	α(Cu-Mn)	
<i>C</i>	CuAl <sub>2</sub>	
<i>L</i>	Liquid	
The order of crystallization of phases is indicated by the order in which the symbols are written.		The first symbol written along the isotherma represents the constituent separating, the second the temperature.

There are three main secondary fields associated with the primary aluminium field *ABCKH*, the dividing lines being *NK*, *NM*, and *NQ*, the last of which runs down to the ternary eutectic point *T*. In addition, there is the area *AMNP*, which represents the limits within which the solid solution of copper and manganese in aluminium is the only constituent present under the conditions of cooling employed in the present investigation. In the field *PNKH*, the secondary separation is that of the aluminium-MnAl<sub>6</sub> binary complex. This may be sub-divided into three subsidiary fields. If the copper content of the alloy is very small—as in alloys lying to the left of the line *NZ*—solidification will be completed during this stage; if the copper content is higher the secondary separation of aluminium and MnAl<sub>6</sub> will continue until the temperature has fallen to 616° C. At this point the peritectic reaction which results in the formation of α(Cu-Mn) takes place; this reaction does not usually proceed to completion, and the alloys often contain a fair amount of residual MnAl<sub>6</sub>. Freezing continues with the separation of the aluminium-α(Cu-Mn) complex; in the case of alloys of compositions lying in the field marked *A6α* in the figure, solidification is completed during this stage; if solidification is not completed the separation will continue until the temperature has fallen to that of the ternary eutectic (547.5° C.).

In the field *NQCK*, the secondary separation is that of the aluminium-α(Cu-Mn) complex, and this will continue until the temperature has fallen to that of the ternary eutectic. In the field *MBQN*, the aluminium-CuAl<sub>2</sub> binary complex is secondary; if the manganese

content of the alloy is very small—as in alloys lying below the line *NX*—solidification will be completed during this stage, but if the manganese content is greater than 0.2% the secondary separation will continue until the temperature has dropped to that of the ternary eutectic.

The primary  $MnAl_6$  field has associated with it two secondary fields separated by the line *KU*. The tie lines in these fields are consistent with the calculated composition of  $MnAl_6$ , i.e. 25.35% manganese. In the concentration area *HKUG* the secondary separation is that of aluminium; if the copper content of the alloy is very small solidification may be completed at this stage, otherwise the separation will proceed along the binary valley *HKCT*, and the course of solidification will be as described for alloys in the field *PNKH*. In the concentration area *KLU* the secondary separation is that of  $\alpha(Cu-Mn)$ , formed as the result of the peritectic reaction between  $MnAl_6$  and the liquid. As is general in aluminium alloy systems, the peritectic reaction does not proceed to completion, but ceases as soon as the primary constituent ( $MnAl_6$ ) has become completely enveloped in a sheath of reaction product. When this happens, the point on the diagram representing the composition of the liquid phase leaves the phase boundary *KL*, and moves across the field of the  $\alpha(Cu-Mn)$  phase, reaching the eutectic valley at some point to the right of the point *K*. The tie lines in this field are consistent with the findings of Petri<sup>20</sup> that  $\alpha(Cu-Mn)$  can have the approximate composition copper 19 and manganese 24%. When the eutectic valley is reached, aluminium commences to separate in its binary complex with  $\alpha(Cu-Mn)$ , and solidification proceeds until the ternary eutectic point *T* is reached.

Alloys lying in the concentration area *KCDL* have  $\alpha(Cu-Mn)$  as their primary, aluminium as their secondary, and  $CuAl_2$  as their ternary constituent.

There are two secondary-phase fields associated with the primary  $MnAl_4$  field. The tie lines in these fields are consistent with the calculated values of the composition of  $MnAl_4$  and  $MnAl_6$ , i.e. 33.72 and 25.35% manganese, respectively. In the area *GLRF* the secondary separation is that of  $MnAl_6$  which is formed as a result of the peritectic reaction between  $MnAl_4$  and the liquid: as this reaction rarely proceeds to completion, the alloys almost invariably contain some residual  $MnAl_4$ . Later separations proceed in the manner described for the  $MnAl_6$  primary field; in the area *GUSF* aluminium is the ternary constituent, and in the area *ULRS*  $\alpha(Cu-Mn)$  is ternary. In the field *LDER* the secondary separation is  $\alpha(Cu-Mn)$  formed by the peritectic

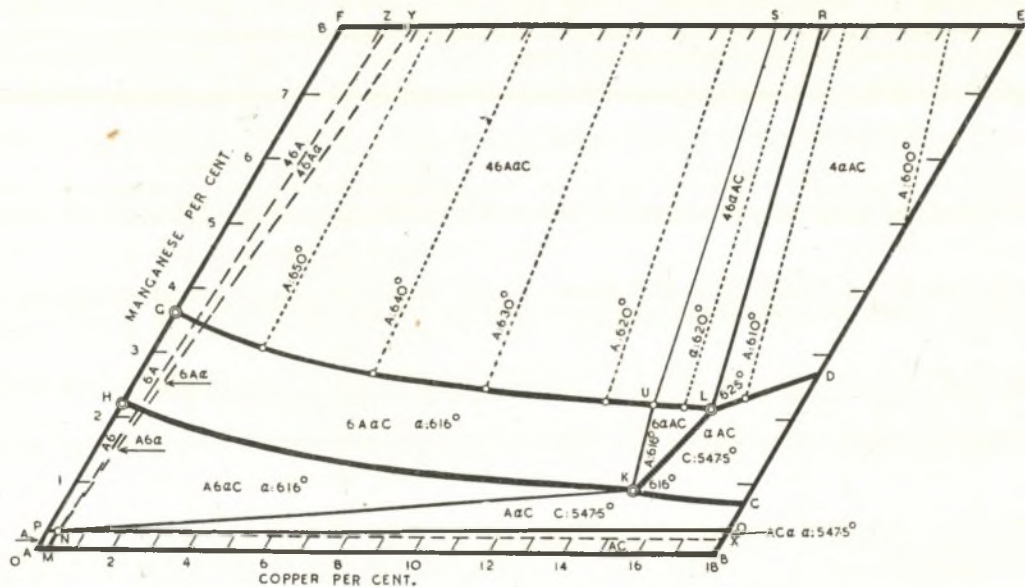


FIG. 10 (a).—Ternary and Later Separations. Alloys as slowly solidified.

reaction between  $\text{MnAl}_4$  and the liquid, again the reaction rarely proceeds to completion and the alloys often contain some residual  $\text{MnAl}_4$ ; the ternary constituent is aluminium, and  $\text{CuAl}_2$  the quaternary one.

#### VII.—CONSTITUENTS PRESENT IN THE SOLID STATE.

The limits of occurrence of the various phases under the conditions of cooling employed in the investigation are shown in Fig. 10 (a).

The area  $AMNP$  represents the limits of occurrence of single-phase alloys, the aluminium-rich solid solution alone being present.

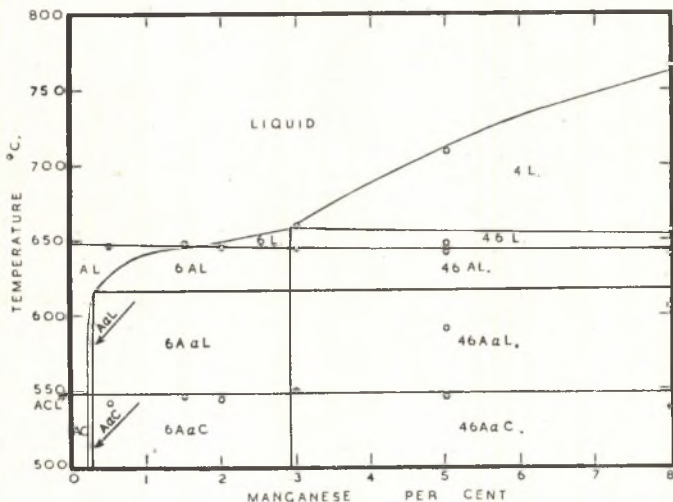


FIG. 11.—Copper 4%. Alloys as slowly solidified.

$\alpha(\text{Cu-Mn})$  is to be found in all alloys lying to the right of the dotted line  $MNZ$  and above the dotted line  $PNX$ .  $\text{CuAl}_2$  occurs in all alloys other than those in the area lying to the left of the line  $MNY$ . The peritectic line  $GLD$  marks the lower limit of occurrence of  $\text{MnAl}_4$ , while  $\text{MnAl}_6$  occurs in all alloys lying within the area  $PNKLRP$ .

#### VIII.—SECTIONAL DIAGRAMS.

Six diagrams, Figs. 11–16, have been included. They show the constitution of series of alloys containing 4, 8, and 16% copper, and 5, 8, and 1.25% manganese, respectively. The only sections which call for detailed comment are those of the series of alloys containing 4% copper and 1.25% manganese.

When a small quantity of manganese is present as an impurity in



a 4% copper alloy, no manganese-bearing phase appears until the amount of manganese has reached 0.2%, and there is no appreciable change in the temperature of the liquidus and solidus of the alloy until manganese 1.6% is present. At the latter composition, the  $MnAl_6$  field is entered, and risk of segregation becomes pronounced.

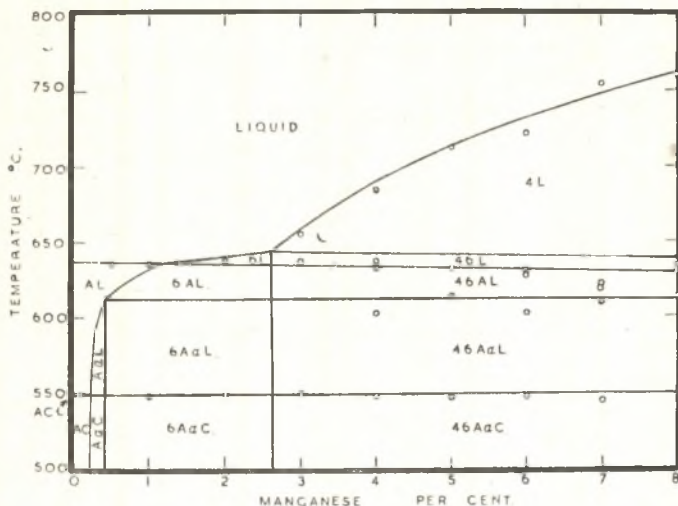


FIG. 12.—Copper 8%. Alloys as slowly solidified.

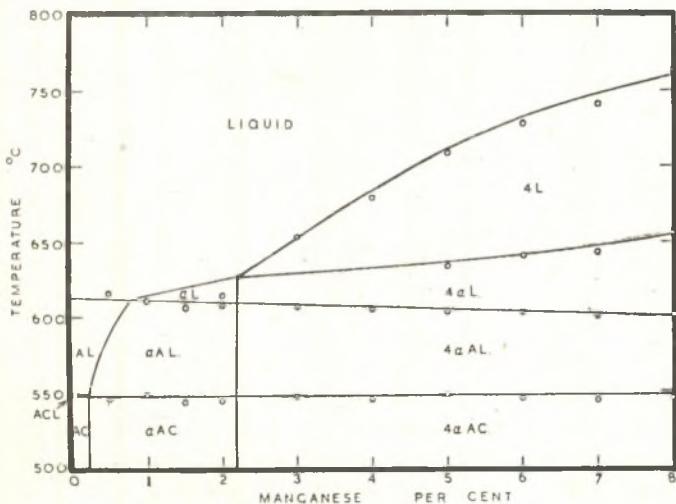


FIG. 13.—Copper 16%. Alloys as slowly solidified.



In the case of 1.25%-manganese commercial alloys, the effect of adding copper is to lower the solidus temperature: the solidus slopes down steeply to the  $\alpha(\text{Cu-Mn})$  arrest, and still more steeply to the  $\text{CuAl}_2$  arrest, reaching the ternary eutectic temperature in the latter case. This may involve risk of hot-shortness. The first appearance of a copper-bearing phase  $\alpha(\text{Cu-Mn})$  occurs at copper 0.3%, and  $\text{CuAl}_2$

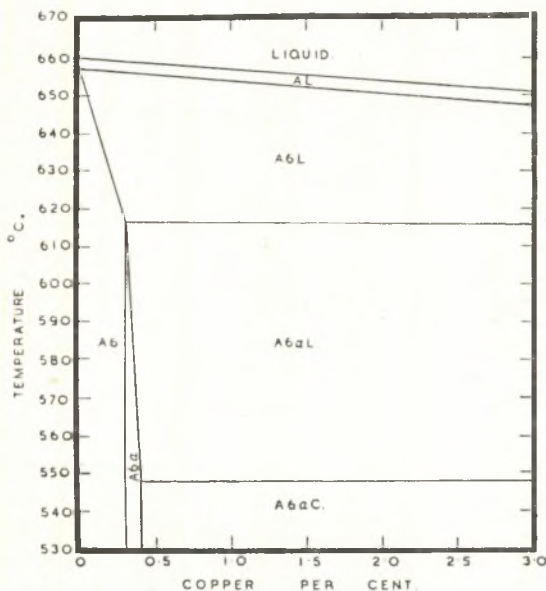


FIG. 16.—Manganese 1.25%. Alloys as slowly solidified.

itself appears at copper 0.4%. Annealing at a high temperature would cause copper to be taken into solution, thereby raising the solidus temperature and reducing the tendency to hot-shortness.

#### IX.—STRUCTURE OF ALLOYS AS ANNEALED.

A preliminary investigation of alloys of which the composition lay within the range copper 0.5-0 and manganese 0-0.75%, annealed at 500° C. and quenched, showed that very prolonged annealing times would be needed to ensure equilibrium even at relatively high temperatures, and that it would be a matter of considerable difficulty to determine accurately the positions of the various phase boundaries. The new aluminium-rich phase which Little, Raynor, and Hume-Rothery<sup>33</sup> have called "G" was identified in a number of micro-

sections, but was difficult to distinguish from  $\alpha(\text{Cu-Mn})$ . No chemical etch has yet been found which will satisfactorily discriminate between these two constituents, and electrolytic etching, whilst very suitable for revealing minute quantities of a precipitated phase, tends to give a variety of colours on any of the manganese-bearing constituents, and so is unreliable as an aid to their identification. Little, Raynor, and Hume-Rothery showed that a limited quantity of "G" was present in binary aluminium-manganese alloys even after a prolonged period of annealing (40 days at  $550^\circ \text{C}$ .), and that it appears to be a metastable phase. Work on the ternary alloys, which is still proceeding, suggests very strongly that similar conditions of metastability persist in the presence of copper.

#### SUMMARY.

The constitution of the ternary alloys of aluminium with copper and manganese as slowly cooled was studied in detail over the range copper 0-18 and manganese 0-8%, by thermal and microscopic methods, but a few additional alloys were examined in the neighbourhood of the ternary eutectic.

The constituents present were found to be  $\text{MnAl}_4$ ,  $\text{MnAl}_6$ , a ternary complex called  $\alpha(\text{Cu-Mn})$ ,  $\text{CuAl}_2$ , and aluminium. The last three form a ternary eutectic containing copper 32.5 and manganese 0.6%, and freezing at  $547.5^\circ \text{C}$ . Depending on the quantity of copper present,  $\text{MnAl}_4$  reacts peritectically with the liquid to form either  $\text{MnAl}_6$  or  $\alpha(\text{Cu-Mn})$ , and  $\text{MnAl}_6$  also reacts peritectically with the liquid to form  $\alpha(\text{Cu-Mn})$ ; these peritectic reactions rarely proceed to completion, and in the solid alloys of suitable composition, residual cores of  $\text{MnAl}_4$  or  $\text{MnAl}_6$ , or both, can be detected in the crystals of  $\text{MnAl}_6$  and  $\alpha(\text{Cu-Mn})$ . The invariant point at which  $\text{MnAl}_4$ ,  $\text{MnAl}_6$ , and  $\alpha(\text{Cu-Mn})$  are in equilibrium with the liquid lies at copper 15.75 and manganese 2.20% at a temperature of  $625^\circ \text{C}$ ., and that at which aluminium,  $\text{MnAl}_6$ ,  $\alpha(\text{Cu-Mn})$ , and the liquid are in equilibrium lies at copper 14.85 and manganese 0.90% and  $616^\circ \text{C}$ .

In the primary aluminium field the liquidus isothermals run practically parallel to the manganese axis; in the  $\text{MnAl}_6$  field variations in the copper and manganese content have equal effects on the liquidus temperature, and in the  $\text{MnAl}_4$  field the liquidus isothermals run parallel to the copper axis. There is no metallurgical objection to the use of ternary alloys as hardeners, but owing to this relationship between manganese content and liquidus temperatures, critical analytical control will be needed. The intersections of the liquidus isothermals



with the univariant lines comply with the Lipson-Wilson rules for ternary equilibria. The atomic percentages of points lying on the isothermals of the  $\text{MnAl}_4$  field, when plotted on a log-log scale, do not conform with a gradient of 1:4 as might have been expected from the Law of Mass Action. When the Le Chatelier form of the equation connecting the apparent solubility product of an intermetallic phase with temperature is applied to the liquidus isothermals of the primary  $\text{MnAl}_4$  field the graph obtained is linear over a wide range of temperature.

The tie lines of the various phase fields plotted are consistent with the calculated values of the composition of  $\text{MnAl}_4$  and  $\text{MnAl}_6$ , i.e. 33.72 and 25.35% manganese, respectively, and with a value of the composition of  $\alpha(\text{Cu-Mn})$  of copper 19 and manganese 24%.

The boundary between the primary aluminium and  $\text{MnAl}_6$  fields slopes towards the aluminium-copper axis; primary  $\text{MnAl}_6$  appears at 1.6% manganese in commercial 4.0%-copper alloys: this point is important, since alloys in the primary  $\text{MnAl}_6$  field are liable to segregation. In 1.25%-manganese commercial alloys small additions of copper lower the solidus temperatures of the alloys considerably, and this may involve the risk of hot-shortness: an addition of as little as 0.4% is sufficient to bring the temperature of final solidification down to that of the ternary eutectic, 547.5° C.

Very little work has yet been done on the structure of the alloys as annealed as, owing to the presence of a metastable phase "G", it was found to be impossible to reach equilibrium within a reasonable period of time, and difficulty has also been experienced in identifying the phases.

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# METALLURGICAL ABSTRACTS

	PAGE
I. Properties of Metals . . . . .	1
II. Properties of Alloys . . . . .	3
III. Structure (Metallography; Macrography; Crystal Structure).	7
IV. Dental Metallurgy . . . . .	—
V. Powder Metallurgy . . . . .	9
VI. Corrosion and Related Phenomena . . . . .	9
VII. Protection (Other than by Electrodeposition) . . . . .	11
VIII. Electrodeposition . . . . .	13
IX. Electrometallurgy and Electrochemistry (Other than Electrodeposition) . . . . .	—
X. Refining . . . . .	18
XI. Analysis . . . . .	18
XII. Laboratory Apparatus, Instruments, &c. . . . .	22
XIII. Physical and Mechanical Testing, Inspection, and Radiology.	25
XIV. Temperature Measurement and Control . . . . .	26
XV. Foundry Practice and Appliances . . . . .	—
XVI. Secondary Metals: Scrap, Residues, &c. . . . .	—
XVII. Furnaces, Fuels, and Refractory Materials . . . . .	—
XVIII. Heat-Treatment . . . . .	—
XIX. Working . . . . .	—
XX. Cleaning and Finishing . . . . .	—
XXI. Joining . . . . .	—
XXII. Industrial Uses and Applications . . . . .	—
XXIII. Miscellaneous . . . . .	—
XXIV. Bibliography . . . . .	28
XXV. Book Review . . . . .	32

# AUTHOR INDEX TO ABSTRACTS

- ADDISON, E. J., Jr., 16.  
 Agruss, B., 14.  
 Anderson, E. A., 15.  
 Andrew, J. F., 16.  
 Ashby, D., 17.
- Bach, B. B., 18.  
 Baer, W. H., 26.  
 Ball, C. J. P., 28.  
 Ballay, M., 15.  
 Bandes, H., 17.  
 Barber, C. R., 23.  
 Baret, E., 14, 16.  
 Barrett, S. J., 3.  
 Bastien, P., 3.  
 Beaumont, R. A., 28.  
 Beck, G., 20.  
 Belaieff, N., 10.  
 Besson, J., 20.  
 Bézier, D., 18.  
 Black, G., 12.  
 Blazy, A., 17.  
 Bogroff, S., 30.  
 Bregman, A., 17.  
 Bresson, Y., 3.  
 Brick, R. M., 3.  
 Broomfield, J., 22.  
 Bruck, H., 24.  
 Buc, G. L., 8.  
 Burns, G., 7.
- Cambon, T., 4.  
 Carrington, E., 32.  
 Cartwright, B., 18.  
 Chamagne, L., 8.  
 Chamie, C., 5.  
 Charlot, G., 18.  
 Chaston, J. C., 10.  
 Clarke, S. G., 16.  
 Combs, E. L., 14.  
 Cornu, A., 22.  
 Cosslett, V. E., 23.
- Dauvillier, A., 24.  
 Davis, W. O., 22.  
 Daymude, J. F., 14.  
 Deribéré, M., 8, 30.  
 Dice, J. W., 26.  
 Dobson, J. G., 17.  
 Van Dorsten, A. C., 23.  
 Draper, C. R., 11.  
 Drummond, D. G., 24.  
 Duckett, R., 5.  
 Ducret, L., 19.
- Dupouy, G., 23, 24.
- Ebert, L. J., 25.  
 Ehrhardt, R. A., 13.  
 Elliott, E. S., 5.
- Falk, J., 15.  
 Farran, J., 1.  
 Farrant, J. L., 24.  
 Faust, C. L., 14.  
 Feigl, F., 30.  
 Fitzgerald-Lee, G., 4.  
 Fontana, M. G., 11.  
 Forrester, P. G., 5.  
 Fox, J. C., 15.  
 Fred, M., 21.  
 Freid, M. L., 25.
- Gabor, D., 24.  
 Gant, M. E., 4.  
 Gardam, G. E., 14.  
 Garrod, R., 24.  
 Gensamer, M., 7.  
 George, H. P., 4.  
 Gillis, J., 22.  
 Girschig, R., 25.  
 Glayman, J., 12.  
 Glover, K. E., 26.  
 Goldschmidt, H. J., 27.  
 Gordet, A., 12.  
 Granjon, H., 8.  
 Grant, H. J., 6.  
 Greenfield, L. T., 5.  
 Greenland, K. M., 23.  
 Greenwood, H. W., 1.  
 Griswood, J., 30.  
 Guthrie, J. M., 13.
- Haine, M. E., 23.  
 Hanna, R. K., 9.  
 Harkins, L. B., 30.  
 Harris, W. J., 24.  
 Harvey, C. E., 21.  
 Haughton, J. L., 22.  
 Hauser, H. H., 30.  
 Head, H. E., 13.  
 Heggen, G. E., 21.  
 Hérenghuel, J., 7, 10, 12.  
 Herr, F. A., 17.  
 Herzog, E., 11.  
 Heyrovsky, J., 20.  
 Holm, R., 30.  
 Howard, A., 27.  
 Hume-Rothery, W., 2.
- Jack, D. E., 10.  
 Jones, W. M., 24.  
 Jupnik, H., 8.
- Keeling, K. G., 7.  
 Khokhlov, A. S., 2.  
 Kienle, R. H., 8.  
 Krause, H., 31.
- Lacroix, S., 18.  
 Land, T., 26, 27.  
 Landau, R., 10.  
 Laurent, P., 7, 30.  
 Le Poole, J. B., 23, 24.  
 Leroy, A., 8.  
 Levine, W. S., 19.  
 Liger, J., 8, 16.  
 Lipson, O., 31.
- McGregor, D. S., 1.  
 Malcuit, S. V., 31.  
 Marsh, J. K., 1.  
 Mason, J. F., 11.  
 Melsheimer, L. A., 8.  
 Mezger, E., 2.  
 Michels, L. R., 18.  
 De Mille, J. B., 31.  
 Mohler, J. B., 17.  
 Morris, G., 9.  
 Moseley, D. L., 3.  
 Mudge, W. A., 5.  
 Murray, W. M., 31.
- Nachtrieb, N. H., 21.  
 New, J. C., 25.  
 Nixon, C. F., 15.  
 North, H. E., 3.  
 North, S., 3.
- Oliensis, G. L., 13.  
 Oosterkamp, W. J., 23.  
 Osterberg, H., 8.  
 Ostertag, H., 19.
- Parker, E. A., 19.  
 Pellowe, E. F., 9.  
 Petrie, W., 21.  
 Phillips, F. C., 31.  
 Pocock, B. W., 17.  
 Pollak, F. F., 9.  
 Preston, G. D., 23.  
 Price, J. W., 16.  
 Pride, G. E., 8.  
 Proell, W. A., 14.
- Reeve, L., 27.  
 Reinken, L. W., 17.  
 Rhines, F. N., 10.  
 Ribaud, G., 2.  
 Ricard, R., 21.  
 Rinck, E., 19.  
 Rivitz, S. F., 18.  
 Robinson, K. G., 25.  
 Rose, A.-J., 8.  
 Rosen, R., 10.  
 Rymer, T. B., 23.
- Salt, F. W., 19, 20.  
 Sanford, R. L., 26.  
 Santini, F., 7.  
 Schapiro, L., 3.  
 Scherzer, O., 24.  
 Scott, N. W., 22.  
 Scott, R. V., 24.  
 Seemann, H. E., 26.  
 Segond, R., 12.  
 Serfass, E. J., 19.  
 Shur, Ya. S., 2.  
 Siegfried, W., 25.  
 Silverman, L., 16.  
 Singh, N. L., 21.  
 Snutch, A. D., 22.  
 Spalenka, M., 21.  
 Stearns, E. I., 8.  
 Stevenson, C. H., 3.  
 Stewart, K. H., 24.  
 Strock, L. W., 21.  
 Stross, W., 18.
- Talbot, A. M., 5.  
 Thienchi, N., 9.  
 Thon, N., 16.  
 Tomashov, N. D., 31.  
 Tomkins, F. S., 21.  
 Toole, A. R., 25.  
 Trillat, J. J., 8.  
 Turner, A. F., 7.
- Valeur, J., 30.  
 Vernon, A. A., 22.  
 Victor, M. T., 9.  
 Vogt, P., 15.  
 Vonsovsky, S. V., 2.
- Walton, W. H., 24.  
 Wernick, S., 17.  
 Wheelon, O. A., 3.  
 Whipple, G. A., 22.  
 Wood, R. T., 13.  
 Worner, H. W., 9.



# METALLURGICAL ABSTRACTS

(GENERAL AND NON-FERROUS)

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Part 1

## I.—PROPERTIES OF METALS

\*The Place of Yttrium and the Application of Ferricyanide Precipitation in the Rare-Earth Group. The Purification of Erbium. Joseph K. Marsh (*J. Chem. Soc.*, 1947, (Jan.), 118–122).—The position of yttrium (atomic number 39) is discussed in relation to its atomic and ionic radii. On the former, yttrium interpolates between the rare-earth elements neodymium (atomic number 60) and samarium (atomic number 62), and on the latter between holmium (atomic number 67) and dysprosium (atomic number 66). This provides a method of eliminating and purifying yttrium. By means of ferricyanide precipitation, yttrium may be separated from holmium and erbium. The reduction of yttrium in erbium from 7 to 0.32 at.-% was accomplished by six precipitations with 50% yield. Erbium may be purified by ferricyanide precipitation and hydrolysis. Six precipitations of 93% erbium gave a 50% yield of 99.7 at.-% material.—D. P. M.

Titanium—Some Properties and Applications. H. W. Greenwood (*Metalurgia*, 1947, 36, (211), 44).—The properties of titanium and some of its alloys are briefly summarized. Applications in non-ferrous metallurgy include age-hardening copper and nickel alloys and grain-refining light-metal alloys.

—M. A. V.

\*Contamination and Failure of Rare-Metal Thermocouples. (Manterfield). See p. 28.

Relations Between the Elastic Constants. D. S. McGregor (*Engineering*, 1947, 164, (4252), 76).—A method is described of deriving the relation between the modulus of elasticity, the modulus of rigidity, and Poisson's ratio for a material. The method is claimed to be simple and to avoid the approximation difficulty associated with another method based on the distortion of a square section of a piece of a material. The method is derived from the facts that equal complementary shear stresses exist on planes at right angles, and that on planes at 45° to the pure shear planes there are mutually perpendicular pure tensile and compressive stresses, each of magnitude equal to the pure shear stress.—R. GR.

\*On the Surface Optical Anisotropy of Striated Reflecting Surfaces. Jacques Farran (*Compt. rend.*, 1947, 224, (15), 1103–1105).—The surface anisotropy of reflecting surfaces scratched by a fine abrasive has already been described and explained by Capdecombe (*Bull. Soc. franç. Minéral.*, 1940, 63, 57–63). F. has continued this study to determine if abrasion gives rise to definite, reproducible, optical phenomena, capable of being related to the physical constants. Aluminium, copper, cadmium, and V<sub>2</sub>A steel specimens were first electrolytically polished, a chromium specimen being given a simple mechanical polish. The pressure exerted had little effect on the anisotropy. Apart from copper, with a difference of up to 15 or 20%, all the metals gave concordant values for anisotropy, within about 2%. The anisotropy increased with the grain-size of the abrasive, tending, for the plastic metals, to an asymptotic value with large grains, as follows: aluminium 2.05, cadmium 1.9, copper 1.7, V<sub>2</sub>A steel 1.55; chromium has a lower value and behaves somewhat differently. The optical significance of these results is discussed.—J. H. W.

\* Denotes a paper describing the results of original research.

† Denotes a first-class critical review.

†Electrons and Metals. I.—The Nature of an Atom. II.—The Nature of a Metal. III.—The Nature of an Alloy. William Hume-Rothery (*Metal Ind.*, 1946, **68**, (4), 70–72; (5), 89–91; (6), 109–110; (7), 129–132; (8), 149–151; (9), 169–170; (10), 189–191; (11), 209–212; (12), 229–232; (13), 249–251; (14), 269–272; (15), 289–292; (16), 309–311; (17), 329–332; (18), 349–350; (19), 369–370; 1946, **69**, (9), 178–180; (10), 199–202; (11), 217–219; (12), 241–245; (13), 259–262; (14), 283–287; (15), 305–307; (16), 325–328; (17), 343–346; (18), 367–372; (19), 387–391; (20), 409–412; (21), 427–431; (22), 451–455; (23), 471–475; (24), 491–496; 1947, **70**, (6), 107–110; (7), 131–133; (8/9/10), 149–152; (11), 167–170; (12), 187–191; (13), 209–212; (14), 229–234; (15), 249–252; (23), 421–422; (24), 441–443; (25), 459–463; (26), 481–483). An interpretation of the present state of knowledge, designed for the older metallurgist.—W. G. A.

**Energy Dissipated by Foucault Currents in a Thin Ferromagnetic Disc Normal to the Field.** Gustav Ribaud (*Compt. rend.*, 1946, **222**, (13), 726–727).—In a previous note (*Compt. rend.*, 1943, **216**, 377), R. discusses the case of a thin disc heated by induction in an alternating field normal to its plane. For a sufficiently high frequency, the current is confined to a layer of thickness

$\epsilon' = \sqrt{\frac{\rho}{2\pi\mu\omega}}$ , in the vicinity of the two plane faces of the disc. For a ferromagnetic disc, the magnetic charges on its faces produce a uniform demagnetizing field which must be added to the field due to the Foucault currents. The energy  $W'$  dissipated by induction in the disc is shown to be

$W' = H_{\text{ext}}^2 \cdot \frac{\rho r^2}{8\pi\epsilon'} \cdot \frac{1}{(1 + \mu \cdot \epsilon'/r)^2}$ , and its ratio to the energy  $W$  dissipated in a

disc of the same resistance but non-magnetic is  $\frac{W'}{W} = \frac{\sqrt{\mu}}{(1 + \epsilon'/r \cdot \sqrt{\mu})^2}$ , where  $\epsilon$  is the thickness of the current layer. This factor, the magnetic factor, has a max. value of  $r/4\epsilon$ , when  $\mu = r^2/\epsilon^2$ , and tends to zero for large values of  $\mu$ . The equations are not valid unless the thickness of the disc is greater than 2 or 3 times the layer thickness  $\epsilon'$ .—D. P. M.

**On the Exchange Interaction of the Valence and Inner Electrons in Ferromagnetic (Transition) Metals.** S. V. Vonsovsky (*Zhur. Eksper. i Teoret. Fiziki*, 1946, **16**, (11), 981–989).—[In Russian]. For English version, see *J. Physics (U.S.S.R.)*, 1946, **10**, 468–475; *Met. Abs.*, 1947, **14**, 362.—N. A.

\***The Dependence of the Magnetostriction of Nickel Upon Initial Magnetic Texture and Sequence of Applying Magnetic Field and Unidirectional Elastic Tension.** Ya. S. Shur and A. S. Khokhlov (*Zhur. Eksper. i Teoret. Fiziki*, 1946, **16**, (11), 1011–1020).—[In Russian]. For English version, see *J. Physics (U.S.S.R.)*, 1947, **11**, (1), 77–84; *Met. Abs.*, 1947, **14**, 356.—N. A.

**The Law of Variation of Surface Tension with Temperature.** Eddy Mezger (*Compt. rend.*, 1946, **222**, (16), 948–950).—Developing the internal energy,  $U$ , of a liquid molecule in a series  $U = U_0 + \alpha T + \beta T^2 + \gamma T^3 + \dots$ , and integrating Helmholtz's formula  $\frac{A}{T} = - \int \frac{U}{T^2} \cdot dT + \text{constant}$ , where  $A$  is the free energy, M. finds that  $\gamma s = A = U_0 - \beta T^2$ , where  $\gamma$  is the surface tension and  $s$  the surface area occupied by a molecule on the surface of a liquid. If  $v =$  volume of a gram-molecule and  $N =$  Avogadro's Number,  $s = v^{2/3}/N^{2/3}$ , if the molecules have the same distribution at the surface as in the interior, but if a different distribution be assumed,  $s = \frac{(v^{1/3} - l)^2}{N^{2/3}}$ . Hence

$\gamma = \frac{(U_0 - \beta T^2)N^{2/3}}{(v^{1/3} - l)^2}$ . At the critical temp.,  $\gamma = 0$  and  $\beta = U_0/T_c^2$ , and by

applying the formula at two different temp.,  $l = \frac{v_1^{1/3} - Bv_2^{1/3}}{1 - B}$ , where

$B = \sqrt{\frac{T_c^2 - T_1^2}{T_c^2 - T_2^2} \cdot \frac{\gamma_2}{\gamma_1}}$ . Application of the formula to various substances gave satisfactory agreement, and a table of results for water and benzene is given. A correction due to the presence of gaseous molecules above the liquid surface is quoted.—D. P. M.

## II.—PROPERTIES OF ALLOYS

**Residual Stresses in Aluminium Alloys.** R. M. Brick (*Aluminium and Magnesium*, 1947, 3, (4), 6-7, 24).—A general descriptive account of residual stresses arising from quenching and mechanical working; stress relief is briefly considered.—F. A. F.

**A Comparison Between the French "Alpax" and the German "Silumin" (13% Silicon and 0.3-0.5% Magnesium).** — (*Rev. Aluminium*, 1947, (130), 68).—The alloy can be obtained (1) by mixing aluminium (99.5%) and silicon (98-99%); (2) by electrolysis, adding quartz or silicon to the usual bath (French process); (3) by mixing aluminium and an alloy of silicon obtained by reducing quartz with carbon (German process). In the French process, iron is eliminated by cobalt, which is to be preferred to manganese.—M. E.

**\*Influence of the Crystallization of Constituents Out of Solid Solution, and of Impurities, on the Forgeability of Light Alloys.** Paul Bastien (*Métaux et Corrosion*, 1946, 21, (252/253), 105-119).—The pressure necessary for plastic flow can be estimated by static bending tests. The solid solution magnesium-6% aluminium flows under a higher pressure than pure magnesium. This is also the case for aluminium-copper and aluminium-magnesium alloys and, according to Kurnakow, is a general property with metals and alloys. When the percentage of the added metal is higher than the limit of solid solubility, the forgeability depends on the precipitated constituent: on its form (globular or acicular), its distribution, and probably its own forgeability. Aluminium-copper and magnesium-copper alloys can be forged easily in the Al + Al<sub>2</sub>Cu and Mg + Mg<sub>2</sub>Cu regions of the equilibrium diagram, and with difficulty in the Al + Al<sub>3</sub>Mg<sub>2</sub> and Mg + Mg<sub>3</sub>Al<sub>2</sub> regions. Even aluminium-6.5% silicon and aluminium-13% silicon alloys are difficult to forge, owing to the precipitated crystals of the eutectic.—M. E.

**Notch Effects in High-Strength Aluminium Alloys.** L. Schapiro (*Iron Age*, 1947, 159, (24), 52-54).—An abstract of several papers by S. and H. E. North, C. H. Stevenson, D. L. Moseley, O. A. Wheelon, and S. J. Barrett, prepared by S. The relative notch sensitivity of the aluminium alloys 14S-T, 24S-T, and 75S-T was evaluated on the assumption that the pressure of a notch changes the ultimate-strength and limiting-strain characteristics of the material. The effect of holes in large structural members was also investigated. The validity of the equation:  $\log(1-x)S_u/S_n = -Nx$  was tested,  $S_n$  being the notched ultimate strength,  $S_u$  the un-notched ultimate strength,  $x$  the fraction of the original area removed by the notch, and  $N$  the index of stress-notch sensitivity, being zero for an ideal notch-insensitive material.—J. H. W.

**A Better Duralumin—Ageing Slowly, for Rivets.** Yves Bresson (*Rev. Aluminium*, 1946, (128), 384-390).—Duralumin used for rivets must age slowly. Research has been carried out on the influence of its three addition elements on the rate of ageing. Copper and silicon increase the rate of ageing; magnesium has little influence on the rate, but increases the ultimate stress after ageing. The best alloy contains copper 2, silicon 0.15, and magnesium 0.6%; its tensile characteristics are: yield point 16.5, ultimate stress 33, and shear stress 1 kg./mm.<sup>2</sup>.—M. E.



**\*On the Optical Anisotropy of the Silicides of Iron, Manganese, and Nickel.** Théophile Cambon (*Compt. rend.*, 1947, 224, (15), 1112-1114).—C. describes the metallographic and molecular structures obtained and the optical anisotropy observed with solid solutions and compounds of iron, manganese, and nickel with silicon.—J. H. W.

**\*Lead-Base Babbitt Alloys. I.—Physical and Corrosion Properties. II.—Fatigue and Wear Properties.** Henry P. George (*Product Eng.*, 1947, 18, (5), 118-121; (6), 138-141).—(I.—) Ten lead-base alloys were tested and compared with a tin-base alloy (antimony 7.5, copper 3.5%, tin balance) used as a reference. The composition of the lead alloys varied from 0.75-11 tin, 8-15 antimony, 0.2-2 copper, and 0.2-3% arsenic, the balance being lead. Typical microstructures of the alloys show a duplex structure with hard intermetallic compounds and softer phases, except for a straight lead-tin alloy (tin 3.5%). A comprehensive table gives some of the most important physical properties, and it appears that the liquidus temp. of the lead alloys is generally lower than that of the reference tin alloy, while the solidus temp. is higher. There is no significant difference in the coeff. of friction, and no significant change in weight or visible evidence of corrosion was noted in any of the alloys after immersion at 300° F. (150° C.) for 6 weeks in unagitated oil. Several of the lead-base alloys are softer at room temp. than the tin-base reference alloy, although at 250° F. (122° C.) the difference tends to disappear. Other properties such as yield strength, ultimate compressive strength, dynamic compression, distortion of Babbitt lining under impact, creep under compression, and rate of contraction under creep compressive tests are tabulated. (II.—) The load-carrying ability, as bearings, of the lead-base alloys were determined in a rotational wear test. Loads were applied to the bushing in increasing increments through a collar by a calibrated helical spring. Each increment of load was carried for 24 hr. before the next increase was made. Under this test, none of the lead-base alloys supported as much load as the tin-base reference alloy. All the lead-base alloys showed better resistance to the development of cracks than the tin-base reference alloy. Wear-fatigue tests were performed in a device that simulated in accelerated form the conditions that lead to failure of automotive bearings. The specimens were placed in a centrifugal bearing fatigue-testing machine, in which a rotating load simulated the loading of a main bearing. In the majority of the runs, the rod (upper) bearing was damaged more than the rod cap (lower bearing), and in some instances small areas were surrounded completely by fatigue cracks.—H. V.

**\*Magnesium Alloys : [Effect of] Small Calcium Additions.** Marvin E. Gantz (*Amer. Foundryman*, 1947, 11, (3), 33-38).—G. reports that when calcium is employed in contents of 0.1-0.2%, the hot-rolling characteristics of such alloys as AZ31X and M1 are improved to such a degree that it is possible to conduct the direct rolling of sheet from sheet ingots. In addition, the mechanical properties of the sheet are enhanced. Microporosity in castings in such alloys as AZ92 and AZ63 is reduced by the addition of 0.5% of calcium. This calcium content also decreases the time required for satisfactory solution treatment, and eliminates the necessity for preheating cycles in the solution treatment of castings of the magnesium-aluminium-zinc type. Calcium additions reduce the oxidation rate of molten alloys, possibly by virtue of the formation of a partially protective film on the exposed surface of the molten alloy. The incidence of hot cracks in fine-grained castings in certain magnesium alloys (e.g. AZ92) is reduced by calcium additions. One of the most serious objections to the use of calcium additions is its apparent tendency to form skins in castings. This defect, however, may be partially overcome by modification of the method of gating, so as to reduce turbulence.—J. E. G.

**Magnesium : Wrought Alloys.** G. Fitzgerald-Lee (*Machine Shop Mag.*, 1947, 8, (6), 93-96).—A summary of the mechanical properties of magnesium



alloys in general and of the Elektron and Magnaminium series in particular. Nothing novel is disclosed.—H. V.

**Ni-Span [Nickel-Iron-Titanium Alloys].** W. A. Mudge and A. M. Talbot (*Iron Age*, 1946, 157, (17), 66-70).—The authors describe the properties of 5 new nickel-iron-titanium alloys of the Invar type, which are age-hardened after cold working to 35-50% reduction. The alloys may be divided into 3 groups: (1) low-expansion alloys: Ni-Span "Lo42" (nickel 41.5, titanium 2.4%), Ni-Span "Lo45" (nickel 45.5, titanium 2.4%), and Ni-Span "Lo52" (nickel 52, titanium 2.4%); (2) a high-expansivity alloy: Ni-Span "Hi" (nickel 29, titanium 2.4, chromium 8.5%); and (3) a const.-modulus alloy: Ni-Span "C" (nickel 42, titanium 2.4, chromium 5.4%). All the alloys contain about 2% of the minor additions and impurities carbon, manganese, silicon, aluminium, phosphorus, and sulphur, the balance being iron. The influence of the alloying elements on the basic iron-nickel system and on the mechanical properties of the alloys is discussed in some detail. By compound formation, carbon neutralizes four times its weight of titanium, while titanium neutralizes more than twice its weight of nickel. Increase of titanium content lowers the temp. at which the inflection in the coeff. of expansion/temp. curve occurs in the "Lo" alloys, and moves the thermo-elastic coeff. towards a negative value in the "C" alloy. A similar effect is produced by chromium, but the response to ageing is lowered and the rate of work-hardening increased. Full details are given of the recommended heat-treatments. Softening is accomplished by heating at 1700°-1850° F. (926°-1010° C.) for 20-90 min., and hardening by heating at 1100°-1350° F. (593°-732° C.) for 3-24 hr.; forging and hot rolling are carried out at 2100°-2200° F. (1149°-1204° C.), with a finishing temp. of 1600°-1800° F. (871°-982° C.) to refine the grain. The alloys are capable of developing ultimate strengths of up to 200,000 lb./in.<sup>2</sup>. The elastic moduli vary between 21-27 × 10<sup>6</sup> lb./in.<sup>2</sup>. The "Lo" and "C" alloys are ferromagnetic at room temp., while the austenitic "Hi" alloy is non-magnetic at all temp. Some information is given with regard to corrosion-resistance.—R. W. R.

**\*On the Crystals of the Amalgam of Polonium and Silver.** (Mlle.) Catherine Chamié (*Compt. rend.*, 1947, 224, (18), 1282-1283).—When mercury that has absorbed polonium, by contact with an acid solution of polonium, is turned out on the gelatine of a photographic plate, the polonium gives star-like images in discontinuous groups, the points of the star representing the dispositions of the  $\alpha$ -particles in the gelatine. To explain how the polonium ions produce these photographic impressions, C. made experiments: (1) on the diffusion of the polonium across the mercury, (2) on the fixation of the polonium on the gelatine, (3) on the activation of the mercury by the polonium disposed on the silver, and (4) in an examination of the polonium-silver amalgam. She concluded that: (1) with mercury the polonium forms an amalgam that can separate itself from the mercury in (probably crystalline) grains, and (2) for large amounts of polonium these radio-active grains can serve as centres of crystallization for the silver amalgam; without silver these crystals are never obtained.—J. H. W.

**The Effect of Crystal (Piezo-Electric) Vibrations at Radio and Higher Frequencies on Metals (Primarily Steel).** E. S. Elliott (*J. Record Trans. Junior Inst. Eng.*, 1945-46, 56, 131-136).—E. describes apparatus for the production of high-frequency mechanical vibrations by using the piezo-electric properties of quartz, and suggests possible lines of research into the effect of such vibrations during the melting, welding, or nitriding of steel.—H. J. A.

**\*The Fatigue Strength of Some Tin-Antimony-Copper and Other Tin-Base Alloys.** P. G. Forrester, L. T. Greenfield, and R. Duckett (*Metallurgia*, 1947, 36, (213), 113-117).—A range of commercially used tin-base alloys was fatigue tested in rotating cantilever machines at 100° C., and test data are presented in tabular form. In general, increasing antimony and copper in

tin-antimony-copper alloys led to increased fatigue strength, and antimony in solid solution and copper in eutectic were more effective than either element in the form of massive compounds. An alloy containing antimony 7 and copper 1% showed a fatigue-strength maximum, thought to correspond to a ternary peritectic complex. Continuous stressing of tin-antimony alloys led to a fine precipitate of SbSn. Casting conditions had little effect on the fatigue strength of tin-antimony-copper alloys. Fatigue strengths of some alloys could be increased by additions of 1% bismuth or 0.1% tellurium.

—M. A. V.

\***An Investigation of the Embrittlement of Platinum-Rhodium Wire in the Heads of Liquid-Steel Pyrometers.** (Land). See p. 26.

\***Fracture of Platinum and Platinum-13% Rhodium Wires Used in the Immersion Thermocouple.** (Reeve and Howard). See p. 27.

\***Embrittlement of Platinum/Platinum-Rhodium Thermocouples.** (Chaston, Edwards, and Lever). See p. 28.

\***High-Temperature Alloys [for Use in Gas Turbines].** H. J. Grant (*Iron Age*, 1946, 157, (21), 42-48; (22), 50-56; (23), 77-80; (25), 60-63).—A summary of research carried out at the Massachusetts Institute of Technology on high-temperature, high-strength alloys for use in gas turbines. The alloys investigated were: (1) 37 low-carbon forging alloys of the nickel-chromium-cobalt-iron and nickel-chromium-iron types, (2) 80 casting alloys of similar composition but with higher carbon contents, and (3) 37 cast alloys of the Vitallium type (approx. cobalt 69, chromium 23, and molybdenum 6%). Creep tests were carried out on all these alloys at 1500° and 1600° F. (816° and 871° C.), and in some instances room-temp. tensile tests were performed. A metallographic and X-ray study was made of the effect of heat-treatment on the alloys, and the effect of such treatment on rupture life and ductility was investigated. Tests of the forgeability of alloys in group (1) were made, and the effect of variations of mould temp. in the casting of the alloys of groups (2) and (3) was studied. The effect of added elements (nitrogen, tantalum, zirconium, and tungsten) was also investigated. The optimum carbon content for the cast nickel-chromium-cobalt-iron alloys lay in the range 1.00-1.20%. High-temperature strength was increased by the addition of carbon in all instances, but with the nickel-chromium-cobalt-iron alloys was accompanied by loss of ductility; no such loss occurred with the cobalt-chromium-molybdenum materials. At 1500° F. (816° C.), the best alloy of the cast nickel-chromium-cobalt-iron type contained carbon 1, nickel 30, chromium 20, cobalt 21, molybdenum 3, tantalum 2.0, and tungsten 2.2%, and broke after 109.5 hr. (30,000 lb./in.<sup>2</sup> load). This material had a life of 542.4 hr. at 25,000 lb./in.<sup>2</sup> load. At the same temp., a similar alloy containing 1.21% carbon, and with 1% columbium replacing the tantalum, gave the extremely low creep rate of 0.00001% per hr. (15,000 lb./in.<sup>2</sup> load). Of the Vitallium alloys, one containing 2% tantalum showed the best properties, having a life at 1500° F. (816° C.) of 297.3 hr. under 30,000 lb./in.<sup>2</sup> load; its creep properties were, however, inferior to the best of the nickel-chromium-cobalt-iron group, a creep rate of 0.000034% per hr. (13,500 lb./in.<sup>2</sup>) being found. At 1600° F. (871° C.), this alloy gave a life of 122.2 hr. at 25,000 lb./in.<sup>2</sup>; while a nickel-chromium-cobalt-iron alloy (carbon 1.02, tantalum 2.0%) had a life of 148 hr. under the same stress. The best heat-treatment for the cast nickel-chromium-cobalt-iron alloys is 2260° F. (1238° C.) followed by water quenching. In general, a nickel content of 30% was better than one of 20%, and tantalum is more effective than columbium. The presence of nitrogen was also beneficial. Polishing the test specimens considerably increased their strength. The optimum mould temp. for the cast alloys was about 1850° F. (1010° C.).—R. W. R.

**Creep and Some Creep-Resisting Alloys.** G. Burns (*Papers on Engineering Subjects* (a restricted Admiralty publication), 1946, (22); also (abridged) *Metallurgia*, 1947, 36, (212), 63–65; and *Mech. World*, 1947, 122, (3155), 17–19).—A brief review of the present-day conception of creep. The creep characteristics of typical alloys are discussed, reference being made to the non-ferrous nickel–chromium alloys (Nimonic).—J. E. G.

**\*On the State of Equilibrium of Solid Solutions.** Pierre Laurent (*Compt. rend.*, 1947, 224, (20), 1431–1433).—Studies of the mechanism of precipitation in supersaturated solid solutions show that this results from the superposition of these two processes: (1) collection of the atoms in excess in certain regions without alteration of the crystalline system (maturation), and (2) transformation of these regions with alteration of the crystalline system (actual precipitation). L. studied the conditions of stability of the heterogeneous solid solutions resulting from maturation from a mathematical point of view. He concluded that below a certain temp. a homogeneous solid solution is not stable, and that the degree of heterogeneity increases when the temp. diminishes.—J. H. W.

**The Fundamentals of Metallurgy.** Kenneth G. Keeling (*Assoc. Eng. Ship Draughtsmen [Publ.]*, 1946–47, 31 pp.).—The significance and determination of metallurgical equilibrium diagrams are described. The composition, heat-treatment, and mechanical properties of some plain carbon- and alloy steels are recorded, and the fields of application of some copper-, aluminium-, magnesium-, and tin-base alloys are briefly reviewed.—H. J. A.

**Present Trends in Alloys.** — (*Canad. Metals*, 1947, 10, (3), 25–26).—A brief review of some modern alloys, particularly triple alloy steels containing controlled amounts of nickel, chromium, and molybdenum; stainless steels; cast bronzes; high-nickel irons; and high-nickel alloys of the Monel and Inconel type.—H. J. A.

**Strength and Ductility.** Maxwell Gensamer (*Engineer*, 1947, 183, (4754), 195; and *Iron Age*, 1946, 157, (7), 66–70).—*Sec. Met. Abs.*, 1946, 13, 314.

—W. G. A.

### III.—STRUCTURE

#### (Metallography; Macrography; Crystal Structure.)

[For all abstracts on the constitution of alloy systems, including X-ray studies, see II.—Properties of Alloys.]

**\*Notes on the Macrographic Study of Aluminium and Its Alloys.** J. Hérenghuel and F. Santini (*Rev. Mét.*, 1946, 43, (5/6), 132–136).—There are two types of macrographic technique; one reveals the grain boundaries and the other the relative orientation of the grains. In the technique suggested, impurities and heterogeneity are revealed. Samples are immersed in a hot solution of hydrochloric acid (10–30%) for 10–40 sec. By this method, the authors have studied the distribution of impurities in ingots cooled at different rates. Except when high corrosion-resistance is required, it is best to cool quickly in order to obtain the most complete dispersion of the constituents.—M. E.

**Increasing the Reflectance of Metals [Aluminium] with Multiple Films.** A. F. Turner (*J. Opt. Soc. Amer.*, 1946, 36, (12), 711).—A summary of a paper on the application to metal reflectors of a process similar to the “blooming” of lenses, but with opposite effect. The reflectivity of a polished aluminium surface can be increased from 91 to 96% by a pair of surface films, one of  $MgF_2$  and one of  $ZnS$ . A second pair of similar films improves the factor to 98%. The improvement, which is valid for a wide range of wave-lengths of reflected light, is in contrast to the reflection-reducing effect on glass.

—E. VAN S.



**Phenomenon of Bronze in Surface Coatings.** G. L. Buc, R. H. Kienle, L. A. Melsheimer, and E. I. Stearns (*Indust. and Eng. Chem.*, 1947, **39**, (2), 147-154).—Read before the American Chemical Society. "Bronze" is defined as the appearance of colour which originates in selective reflection at one interface, or interference of white light reflected at closely adjacent structures. These two types are known as "interface bronze" and "interference bronze". For the latter, the colour varies widely with varying angle of view, but is fairly const. for the former. Methods of measuring bronze and some of the unusual colours exhibited are described, and a theoretical calculation predicts the general shape of the spectrophotometric curve of interface bronze.—D. P. M.

**\*On the Examination by Electronic Diffraction of Surfaces Obtained by Electrolytic Polishing [Iron, Aluminium, Copper].** Jean Jacques Trillat (*Compt. rend.*, 1947, **224**, (15), 1102-1103).—T. examined, by electronic diffraction, specimens of pure iron, aluminium, and copper, electrolytically polished in either a perchloric-acetic bath (iron and aluminium) or a phosphoric bath (all). Electrolytic polishing causes the Beilby layer to disappear more or less completely, according to the duration of the operation. The subjacent metal re-appears with its particular structure, as well as any oxides that arise. The nature of the bath appears to be of importance in the case of the readily oxidizable aluminium. Heterogeneity of structure appears to be the general rule.—J. H. W.

**\*An X-Ray Investigation of the Embrittlement of Platinum and Platinum-Rhodium Wires.** (Goldschmidt and Land). See p. 27.

**\*Examination of the Microstructure of Contaminated and Embrittled Platinum and Platinum-Rhodium Wires.** (Jewell). See p. 28.

**An Accelerated Method of Electrolytic Polishing.** L. Chamagne, H. Granjon, and A. Leroy (*Métaux et Corrosion*, 1946, **21**, (250), 73-80).—With the usual polishing method, using Jacquet's electrolyte, polishing does not begin until the potential reaches the required level of the potential-intensity curve. This results in a loss of time. In the new process, the sample is introduced into the electrolyte with the potential already at the required level, thereby reducing the time of polishing from 8 min. to 15 sec.—M. E.

**Metallography for the Electroplater.** (Blazy and Mohler). See p. 17.

**Structure of Metallic Surfaces.** J. Liger (*Galvano*, 1947, **16**, (124), 7-9).—An elementary survey.—M. E.

**The Control and the Study of Surfaces with an Electron Microscope.** Maurice Deribéré (*Machines et Métaux*, 1947, **31**, (344), 125-128).—M. E.

**Summarized Proceedings of Conference on the Electron Microscope, Oxford, 1946.** (Cosslett). See p. 23.

**\*Phase Microscopy with Vertical Illumination.** Helen Junnik, Harold Osterberg, and G. E. Pride (*J. Opt. Soc. Amer.*, 1946, **36**, (12), 710).—A summary of a pioneer paper on the application of phase-contrast microscopy to metal surfaces, which must be highly polished. The vertical illuminator is modified by the addition of an annular diaphragm, which is imaged on a diffraction plate near the objective. Depressions of the surface of the order of a fraction of a wave-length of light in thickness, or transparent surface films of similar thickness, cause local retardations of the light waves, which are visible in the field of the diffraction plate and sometimes have better visibility than could be produced by any other method of illumination.—E. VAN S.

**\*X-Ray Spectra with Monochromatic Radiation.** Adolphe-Jean Rose (*Compt. rend.*, 1946, **222**, (14), 805-806).—R. describes the use of strictly monochromatic radiation with a Debye-Scherrer camera to obtain very fine spectrum lines, free from diffuseness due to the continuous background. The monochromator consists of a piece of quartz bent to a radius of curvature of



600 mm., which focuses the  $K\alpha_1$  and  $K\alpha_2$  rays on the camera entrance, a slit of calcite isolating the  $K\alpha_1$  beam. It is claimed that exposure times are not notably increased. The method of measurement of the films is described and corrections are applied for eccentricity of the camera, vertical divergence of the beam, height of the beam, size of specimen, and for refraction. Experimental values are given for a copper specimen, the extrapolated value of the parameter at 21° C. being  $3.6073 \pm 0.0003 \text{ \AA}$ .—D. P. M.

## V.—POWDER METALLURGY

**Powder Metallurgy: Advantages of Self-Lubricating Bearings Made by Powder Metallurgy.** M. T. Victor (*Canad. Metals*, 1947, 10, (4), 18–21, 38).—The theory of porous self-lubricating bearings is discussed, and their design is considered in terms of (1) the metallurgy of manufacture, and (2) the type of oil with which the bearings are impregnated. The selection of lubricants, corrosive action of certain oils, and failure of porous bearings are briefly discussed.—H. J. A.

**\*Metallurgy of Powders—Study of Sintered Kovar.** Nguyen Thienchi (*Compt. rend.*, 1946, 222, (18), 1046–1047).—Preliminary results are given on the preparation and properties of Kovar (nickel 29, cobalt 17, iron 54%, no manganese). The pure metals were obtained by reduction of the oxides, and the powders (nickel and iron 200 sieve, cobalt 300) moulded at 3 tons/cm.<sup>2</sup>. Sintering was carried out in hydrogen, three samples being prepared: (1) in a resistance furnace at 1050° C. for 10 hr., density 6, (2) at 1050° C. for 20 hr., density 6.5, (3) by induction heating at 1250° C. for 1 hr., density 7.8. The density of solid Kovar is 8.3. All three samples sealed perfectly to the same glass, with no blisters. Debye–Scherrer photographs are shown.—D. P. M.

**\*Preparation of Titanium Powder.** R. K. Hanna and H. W. Worner (*J. Council Sci. Indust. Research, Australia*, 1946, 19, (4), 449–454).—Several methods of preparing titanium were critically studied with a view to selecting one for producing, in Australia, the titanium needed in compound gettering mixtures. The method selected was a modification of one developed by Kroll, and consisted in the reduction of titanium tetrachloride with magnesium at 800°–900° C. The product, containing not less than 98% titanium, proved satisfactory as a constituent of certain gettering mixtures.—H. J. A.

**The Static Hazard in [the Metallic Powder] Industry.** George Morris (*Engineering*, 1947, 164, (4251), 49–51; (4252), 73–75).—M. reviews the conditions necessary for the generation of electrostatic charges in various industries, including the metallic powder industry, and suggests methods of test for dangerous conditions. The gold-leaf electroscope is quite unreliable for testing conditions. Metallic dust clouds are much more inflammable than clouds of organic dusts. Dow metal, iron dust, and magnesium powders can be ignited with less than 0.1 joule.—R. GR.

## VI.—CORROSION AND RELATED PHENOMENA

**\*Contact-Corrosion Problems in the [Aluminium] Metal-Window Industry: An Assessment of Their Relative Importance.** E. F. Pellowe and F. F. Pollak (*Metallurgia*, 1947, 36, (212), 67–70).—Cast aluminium alloy window frames may be in contact with steel, galvanized steel, brass, pure aluminium, or stainless steel, and experiments were carried out to determine the effect of the couples so set up on corrosion. The pairs of metals were immersed for 1 month in 3% NaCl solution, followed by 1 month in tap water (pH = 8.0). Each system was studied in the painted and unpainted form. Results were in

agreement with the A.S.T.M. 3-year natural corrosion test. Corrosion of aluminium in contact with bare steel was unexpectedly heavy, due possibly to the effect of iron corrosion products. The stoved paint film was more effective than had been expected. In the case of the aluminium-zinc couple, the aluminium was protected by the zinc, and the zinc corrosion not unduly high. Aluminium in contact with brass showed pitting, especially in the contact area.

—M. A. V.

†**The Causes and Prevention of Stress Corrosion in Brass.** J. C. Chaston (*Sheet Metal Ind.*, 1947, 24, (243), 1395-1401, 1404).—C. discusses the present state of knowledge of season cracking. Low-temperature annealing to relieve stress is only satisfactory if the brass is not subsequently stressed beyond the yield point. Too much reliance cannot be placed on the mercurous nitrate test, since service failures may occur at stress levels at which mercury will not cause cracking. Ammonia cracking occurs at very much lower stresses. C. considers that the provision of a brass of suitable composition which will be free from season cracking is the best solution of the problem; the addition of certain elements, particularly silicon, having shown the possibilities of this method.—R. Gr.

**Corrosion-Resistance of Spot-Welded Aluminium Assemblies [in Salt Water].** Nicolas Beliaeff (*Rev. Aluminium*, 1946, (129), 3-9).—A welded joint is not attacked more vigorously at the weld than elsewhere. The French alloy "Duralinox" gives the best corrosion-resistance. Anodizing, and metallizing with pure aluminium, increase corrosion-resistance. Tempering spot-welded Duralumin improves the tensile properties of the joints, but lowers resistance to corrosion.—M. E.

**The Use of Glass Wool for Thermal Insulation of Apparatus in Light Alloys.** Jean Hérenguel (*Rev. Aluminium*, 1946, (129), 10-11).—Hot water or steam passing through glass wool becomes alkaline and attacks the aluminium. However, wool made of scorias can be used because it is only very slightly alkaline.—M. E.

\***Lead-Base Babbitt Alloys. I.—Physical and Corrosion Properties. II.—Fatigue and Wear Properties.** (George). See p. 4.

\***Internal Oxidation.** F. N. Rhines (*Corrosion and Material Protection*, 1947, 4, (2), 15-20).—A review of the present state of knowledge of internal oxidation or sub-scale formation. The mechanism of the process is discussed, and the role of diffusion stressed. The importance of the manner of nucleation of the oxide in determining the structure of the sub-scale is pointed out, and is shown to depend on the temp. of reaction and the nature and concentration of the alloying element. The rate of advance of the oxidation is controlled by the diffusion rate of oxygen in the metal and of the reacting element. There is no evidence that the precipitated oxides cause a hardening effect similar to age-hardening, but they do appear to influence recrystallization and grain growth. The text is well illustrated with photomicrographs, and 14 literature references are given.—G. T. C.

**Is there Poison in Your Corrosion-Resisting Alloy?** D. E. Jack (*Corrosion and Material Protection*, 1947, 4, (2), 12).—J. discusses in general terms the possibility of traces of copper dissolved from various corrosion-resisting alloys being sufficient to cause serious contamination of liquids coming in contact with the alloys.—G. T. C.

**Industrial Handling of Fluorine.** Ralph Landau and R. Rosen (*Indust. and Eng. Chem.*, 1947, 39, (3), 281-288).—The unusual physical, chemical, corrosive, and physiological properties of fluorine are summarized, and its comparative heats of reaction and its effect on various metals and non-metallic substances tabulated. A schematic flow diagram used to study the latter at various pressures is shown. Dilute and pure fluorine have substantially different properties, while nickel, Monel metal, aluminium, magnesium, and

metal fluorides in their highest valence states have excellent corrosion properties. Unexplained inhibited reactions sometimes occur. Standard or extra-strong iron pipes for atmospheric, and nickel and Monel-metal pipes for greater pressures are recommended for conveying, using welded joints. Flanged joints, with soft copper, aluminium, or Teflon (tetrafluoroethylene) gaskets may be used for low pressures, while valves with Teflon as seating element and Monel metal or nickel as mating element are recommended, welded or silver- (not soft-) soldered into the line. Instruments, compressors, &c., must be selected with great care. Storage, which is hazardous, requires thick Monel-metal or nickel containers. Safety precautions, an efficient treatment for burns, and successful disposal methods are described. Fire-proof neoprene is a successful protective fabric.—D. P. M.

**The Prevention of Corrosion by Fluorine and by Hydrofluoric Acid Requires Quite Special Apparatus.** Mars G. Fontana (*Indust. and Eng. Chem.*, 1947, **39**, (3), 91A-92A).—A brief summary of the corrosive properties of fluorine and hydrofluoric acid, and materials suitable for handling them.—D. P. M.

**Corrosion.** Mars G. Fontana (*Indust. and Eng. Chem.*, 1947, **39**, (4), 81A-82A).—On the basis of the appearance of corroded metal, corrosion is classified arbitrarily into eight forms: uniform attack or general corrosion, intergranular corrosion, galvanic or two-metal corrosion, erosion-corrosion, dezincification, pitting, stress corrosion, and concentration-cell corrosion. A brief summary, with examples, of the first two is given.—D. P. M.

**Studies on Corrosion in Great Britain and the U.S.A. During the War (1940-1945).** E. Herzog (*Métaux et Corrosion*, 1946, **21**, (251), 92-100).—H. summarizes papers by U. R. Evans and G. D. Bengough, on theories of corrosion and protection, read at the conference of the Centre de la Lutte Contre la Corrosion in Paris in April 1946.—M. E.

**Corrosion Inhibitors.** — (*Metallurgia*, 1946, **34**, (203), 270-273).—A report of a symposium on "Inhibitors for Control of Scale and Corrosion in Water" held at a joint session of the Division of Industrial and Engineering Chemistry and of Water, Sewage, and Sanitation Chemistry of the American Chemical Society.—W. G. A.

## VII.—PROTECTION

(Other than by Electrodeposition.)

**Corrosion Protection Lengthens Life of Aluminium Transportation Units.** J. F. Mason (*Aluminium and Magnesium*, 1947, **3**, (5), 14-15, 17, 21).—A general discussion of the corrosion of aluminium alloys used in the transport industry and of the types of finishes and paints which may best be used to protect them. The use of zinc chromate is considered.—F. A. F.

**Metallization with Aluminium.** C. R. Draper (*Light Metals*, 1947, **10**, (110), 124-160; correspondence, (112), 254).—A lengthy discussion of current techniques and apparatus for the coating of metallic and non-metallic articles with aluminium. Mechanical, dipping, and calorizing methods are dealt with, and spraying methods are described at length; uses of sprayed coats are also discussed. Corrosion findings are considered, as well as resistance to high-temp. oxidation. The use of aluminium in paint, electrodeposition, evaporation, and sputtering is then reviewed, together with its application to mirrors.  
—F. A. F.

**Surface Preparation for Metal Spraying [Zinc and Aluminium].** — (*Machinist (Eur. Edn.)*, 1947, **91**, (8), 235-238).—The necessity for proper surface preparation to secure adhesion in metal spraying, the metallurgical considerations involved, and the methods of preparation that can be used are



described. The metals primarily concerned are zinc and aluminium; cadmium has somewhat limited applications on account of its high cost.

—J. H. W.

**Shipbuilding at Home and Abroad [Use of Sprayed Zinc Coatings].** — (*Syren and Shipping*, 1947, 202, (2639), 480).—In the U.S., widespread use is made of sprayed zinc coatings on the welds of ships' plating and on a variety of auxiliary parts. The method of applying the zinc coating is by means of the Schori metal-powder pistol and the Schori metallizing process. The adhesion between paint and galvanized iron is poor, and once a repainted film reaches any appreciable thickness the stresses of cold and heat cause it to blister and peel off. Paint adheres well to sprayed zinc, regardless of the thickness of the film, and, although the zinc spraying costs more initially, the total cost over a period of 10–20 years is substantially less.—H. V.

**Equipment for Hot Galvanizing.** A. Gordet (*Galvano*, 1947, 16, (121), 18–19).—General advice is given on equipment to be used.—M. E.

**The Term "Deoxidation" in Hot Galvanizing.** A. Gordet (*Galvano*, 1947, 16, (124), 17–19).—Answers to letters received by G.—M. E.

**Theory of the Anodic Oxidation of Aluminium in a Bath of Sulphuric [Acid].—I, —II, —III, —IV.** J. Glayman (*Galvano*, 1947, 16, (121), 13–17; (122), 13–17; (123), 12–14; (124), 13–16).—(I.—) A discussion of the application of Nernst's Law in the anodizing process. (II.—) The best potential for anodizing is 50 V., and the potential at the electrodes increases according to the logarithm of the c.d. Colloidal additions lower the surface tension. Aluminium sulphate increases the resistance, but the addition of nitric acid gives aluminium nitrate, which decreases the resistance of the solution. Copper sulphate impedes the anodizing process, but if tartaric acid is added a complex copper ion is formed which is not harmful. (III.—) The colour of the hydroxide is a function of its concentration. If colloidal impurities are present in the bath, the intensity of the colour is diminished. (IV.—) The colour of the hydroxide is also due to adsorption. Fixing is effected by the polymerization of the alumina after dehydration of the hydroxide.—M. E.

**Grey- or Black-Coloured Anodic Films on Industrial Aluminium-Magnesium Alloys.** J. Hérenguel and R. Segond (*Métaux et Corrosion*, 1946, 21, (252/253), 101–104).—This effect is generally due to silicon, precipitating out of solid solution. The solubility of silicon decreases with increasing percentage of magnesium. Incomplete oxidation of the silicon causes a precipitation of  $Al_3Mg_2$ , which gives the same result. Tempering at 450° C. prevents this precipitation. It is advisable to use pure metals for making the alloys and to cool the ingots quickly.—M. E.

**The Dichromate Treatment for Magnesium.** — (*Products Finishing*, 1947, 11, (7), 96, 98).—A brief outline of a dichromate treatment for magnesium prior to painting. Operating details are given.—G. T. C.

**Anodizing of Magnesium Alloys for Protection and Appearance.** George Black (*Product Eng.*, 1947, 18, (5), 122–124).—The process produces a protective and decorative magnesium oxide-silicate film on the alloys of magnesium. The coating is applied in a strong alkaline solution at elevated temp. (180° F. (88° C.)) at 4–6 V. The decorative properties can be further enhanced by dyeing to almost any colour desired. The type, thickness, and shape of the material being treated, and the desired end product, determine whether A.C. or D.C. should be used. In general, the A.C. process is best for narrow-gauge wrought alloys and the D.C. for heavier gauges, castings, and intricate shapes. The coating and bath characteristics for A.C. are: light- to dark-grey colour, 20–30 amp./ft.<sup>2</sup>, good throwing power, 0.0002–0.0005 in. thickness, and extremely dense and compact film. The D.C. characteristics are: light-green colour, 10–20 amp./ft.<sup>2</sup>, excellent throwing power, 0.0004–0.001 in.



thickness, and some porosity noticeable at 250 magnifications. The cycle of operations is as follows: degrease, alkaline cleaner, water rinse, water rinse, acid cleaner, water rinse, anodize, water rinse, water rinse, neutralizer.

—H. V.

**Finishes for Magnesium.** R. T. Wood (*Aluminum and Magnesium*, 1947, 3, (4), 12–14, 17, 22).—An address to the Magnesium Association. A general review of the subject, covering surface preparation and cleaning, treatments used as preparation for painting (including anodic methods), paint systems, and chemical coatings.—F. A. F.

**Aluminium-Coated Steel May Offer Advantages for Many Products.** — (*Products Finishing*, 1947, 11, (5), 58, 60, 62).—Aluminium-coated steel is now being produced by a hot-dipping process by the American Rolling Mill Co. The finish is soft and satiny, but a brighter "extra-smooth" grade is also available. The weight of coating is 0.50 oz./ft.<sup>2</sup> on both sides, equivalent to a thickness of 0.001 in. per side. The material withstands up to 2000 hr. in 20% salt spray at 95° F. (35° C.). It shows no discoloration below about 900° F. (482° C.), and no destructive scaling below about 1600° F. (871° C.). Tests have been carried out on car exhaust pipes, domestic-range oven liners, circulating boiler parts, &c. When the sheet is to be painted, surfaces should be "mill-bonderized", which is carried out at the works before despatch.

—G. T. C.

**\*Ten Years' Outdoor Exposure of Filled Asphalt Coatings on Saturated Felts.** G. L. Oliensis (*Bull. Amer. Soc. Test. Mat.*, 1947, (144), 32–41; discussion, 41–48).—Past exposure tests on filled coatings have been made on small rigid aluminium panels for relatively short periods under artificially produced weathering conditions. These tests have indicated that nearly all fillers improve the weatherability of asphalt. In the present paper, O. presents test data for asphalts coated on asphalt-saturated felt. The specimens were exposed out of doors for approx. 10 years, and the results indicate that fillers do stabilize the flow and thereby improve the life of roofing cements which have a relatively low softening point; but fillers do not improve the crack resistance of coatings which have a high softening point and which do not flow when unfilled.—H. J. A.

## VIII.—ELECTRODEPOSITION

**\*Plating on Aluminium.** R. A. Ehrhardt and J. M. Guthrie (*Monthly Rev. Amer. Electroplaters' Soc.*, 1947, 34, (4), 421–428).—E. and G. discuss commercial applications of plating on aluminium, and review early developments. They describe in detail a soldering test for the adhesion of electroplate to aluminium and its alloys. Two specimens,  $1 \times \frac{1}{8} \times \frac{1}{8}$  in., are soldered end to end in a suitable jig, using 50:50 lead-tin solder. They are then pulled in a tensile machine. After breaking, an attempt is made to resolder in order to determine if the break has occurred in the solder or at the bond between the plate and the aluminium. Four materials were tested: commercial-purity aluminium; aluminium with 4.5% copper and 1.25% magnesium; aluminium with 5.0% silicon; and aluminium with 2.5% magnesium and 0.25% chromium. Zincate solutions were used as a pre-treatment, and the optimum conditions established in each case by means of the adhesion test described. These were: sp. gr. 1.38–1.41, copper content 3–7 g./l., zinc content 65–78 g./l. The composition was most critical with the alloy containing chromium. A hydrofluoric acid dip was used in the case of the silicon-containing alloy. The optimum time of immersion in this was found to be 15–45 sec.—G. T. C.

**Improved Barrel-Plating Technique [Cadmium Plating].** Herberth E. Head (*Metal Finishing*, 1947, 45, (3), 55–57).—In order to avoid the necessity of

removing work from barrels to baskets and *vice versa* during the various stages of the cadmium-plating cycle, a cylindrical barrel was designed in which cleaning, rinsing, acid dipping, and plating were carried out in succession without removing the work. The cylinder material was melamine-canvas laminate. There was negligible contamination of solutions due to drag-out, and the materials used in the construction withstood the conditions well. A 50% increase in production is claimed.—G. T. C.

**Practical Advice on Chromium-Plating Baths.** — (*Galvano*, 1947, 16, (121), 10–11; (122), 7–8).—If iron impurities are present in the bath, it is necessary to add sulphuric acid. When this is present in excess, gas is evolved; too little acid results in brown spots on the plating.—M. E.

**\*Copper Plating in Alkane Sulphonic Acid Baths.** C. L. Faust, B. Agruss, E. L. Combs, and Wayne A. Proell (*Monthly Rev. Amer. Electroplaters' Soc.*, 1947, 34, (5), 541–549).—A description of the deposition of copper at c.d. of from 60 to 1000 amp./ft.<sup>2</sup> from baths based on a new class of strong acids, viz. the alkane sulphonic acids of methane, ethane, propane, and butane. The plate obtained is semi-bright without the addition of brightening agents to the bath. Bright nickel directly over this gives a plating of good colour. The effect of varying the operating conditions for four different plating baths is discussed. These are: a dilute bath operating at about 60 amp./ft.<sup>2</sup>, a similar bath at 200 amp./ft.<sup>2</sup>, a bath giving a semi-bright plate with a lustre, operating at 150 amp./ft.<sup>2</sup>, and one operating at 1000 amp./ft.<sup>2</sup> with simple agitation. The work has not been applied on a commercial scale.—G. T. C.

**Maintenance of a Bath [for Copper Plating] by Correct Additions.** E. Baret (*Galvano*, 1947, 16, (121), 11–12).—The cyanide concentration must be kept const., and addition should be made in the form of solutions, not in the solid state.—M. E.

**\*Effect of Small Amounts of Zinc in Watts-Type Nickel Depositing Solutions.** G. E. Gardam (*J. Electrodepositors' Tech. Soc.*, 1947, 22, 8–13).—G. discusses various aspects of the presence of small quantities of zinc in a Watts-type bath, and gives quantitative data. The effect of addition of amounts of zinc up to 0.40 g./l. on the Brinell hardness of the coating is given for two solution compositions. There is in both cases an initial decrease in hardness, believed to be due to the "ageing" of the solution, followed by an increase. The effect on the appearance of the coating obtained by varying the pH of the solution between 1.8 and 6.3 and with a zinc content of 0.25 g./l., is given. The cathode potential was not found to vary significantly with variations in zinc content. The proportion of zinc in the coating was found to be much higher than in the solution. Results are given briefly of tests on eight methods of removing zinc from the solution. Only one, electrolysis at 10 amp./hr./l. at 15 amp./ft.<sup>2</sup> over 20 hr., was entirely successful.—G. T. C.

**\*Note on the Effect of Copper on Nickel Deposition.** G. E. Gardam (*J. Electrodepositors' Tech. Soc.*, 1947, 22, 14).—A dilute solution of copper sulphate was allowed to drip at various rates into an air-agitated Watts solution while electrolysis was being carried out with a nickel-foil cathode. The rate of addition at which "growthy" deposits formed on the cathode was found to correspond with a content of 0.13–0.17% of copper in anodes.

—G. T. C.

**Tin Plating and Control of Tin Solution by Anode Colour.** John Franklin Daymude (*Products Finishing*, 1947, 11, (6), 42–44, 46, 48, 50, 52, 54, 56).—Solutions for still, automatic, and barrel plating of tin are described, applicable to a wide range of base metals and having excellent throwing power. The method of making up the solution is given, and control of its operation by visual observation is described, primarily for the layman without a technical education. There are sections dealing with cleaners, filtering, simple analysis, and replating on tinplate.—G. T. C.

**Fusing of Electrodeposited Tin Coatings.** J. Falk (*Metal Finishing*, 1947, 45, (4), 63–65, 71).—A detailed description of a simplified method of fusing tin coatings to improve their appearance and corrosion-resistance. The fusing medium may be either hydrogenated oil, tallow, or petroleum product with a flash point above 500° F. (260° C.). The container is an electrically heated steel tank. Temp. control is important, the optimum range being 475°–490° F. (246°–254° C.). An exhaust system is essential to remove oil fumes. Time of immersion is 3–10 sec., and may be controlled visually. For small parts, a basket is used to immerse them in the oil. To prevent small parts fusing together they are placed in a breaker immediately after removal from the bath. This rotates at 200–300 r.p.m., and prevents sticking of the parts together. Trichlorethylene is used as a quench and a degreaser.

—G. T. C.

**Manufacture of Zinc-Base Alloy Die-Castings.** J. C. Fox (*Monthly Rev. Amer. Electroplaters' Soc.*, 1947, 34, (3), 288–297).—The first of three papers (the other two are abstracted below) of a symposium on the plating of zinc-base alloy die-castings held at the Annual Educational Session of the Detroit Branch of the American Electroplaters' Society. F. outlines briefly the development and composition of the alloys used, and discusses casting practice, casting machines, dies, die steels, the importance of design, &c.—G. T. C.

**Plating on Zinc-Base Alloy Die-Castings, Including Buffing, Cleaning, Stripping.** C. F. Nixon (*Monthly Rev. Amer. Electroplaters' Soc.*, 1947, 34, (3), 298–309).—N. describes the plating of zinc-base alloy die-castings at the Fisher Body–Ternstedt division of the General Motors Corporation. Details of degreasing, cleaning, the copper "strike" solution, and the plating solutions are given. The use of a bright copper or nickel solution followed by chromium plating is advocated as a means of reducing buffing costs. Stripping of composite copper–nickel coatings is carried out in 50–55% H<sub>2</sub>SO<sub>4</sub>, which is rapid and does not attack the zinc alloy excessively. No addition agent is used. The bath operates at 150° F. (66° C.) and with a voltage of 9 V. Several good photographs are given, illustrating such defects in die-castings as chills, swirls, cold laps, splashing, gate holes, and "soldering", and their effects on subsequent plating.—G. T. C.

**\*Testing of Plated Coatings on Zinc-Base Alloy Die-Castings.** E. A. Anderson (*Monthly Rev. Amer. Electroplaters' Soc.*, 1947, 34, (3), 313–318).—A. describes the study of plated zinc-base alloy parts taken from cars after five years in service. Corrosion was judged visually, thickness by microscopic measurement, which A. considers inadequate, and adhesion by testing with a sharp knife. The data obtained are plotted on a graph. The importance of ensuring good adhesion of the deposited metal to the zinc alloy is stressed. Some failures of the plating are ascribed to intercrystalline corrosion of the alloy itself, resulting in an expansion, with consequent rupture of the plating. Chemical analysis confirmed unsuitable composition of the alloys in some cases. Several photomicrographs are given of failed coatings.—G. T. C.

**Electroplating Zinc-Base Die-Castings.** C. F. Nixon (*Metal Finishing*, 1947, 45, (3), 58–60).—A general review of polishing, buffing, cleaning, plating, and stripping of zinc-base die-castings. The influence of casting technique on subsequent plating is mentioned. 50–55% H<sub>2</sub>SO<sub>4</sub> is recommended for stripping nickel and copper plate from die-castings. The process is an electrolytic one, operated at 150° F. (66° C.) and with a voltage of 9 V.

—G. T. C.

**\*Bright Zinc Plating Using a Cyanide Bath Containing Sodium Sulphide.** Marcel Ballay and Pierre Vogt (*Métaux et Corrosion*, 1946, 21, (251), 89–91).—The brightness is not due to the elimination of the heavy metals by precipitation as sulphides, but to the formation of a colloidal suspension by the action of the sodium sulphide. The best electrolyte tested was: zinc 45 g./l. (Zn(CN)<sub>2</sub>),



NaCN 110 g./l., NaOH 70 g./l., and 20 c.c. of a solution of sodium sulphide (100 g./l.).—M. E.

**Porosity of Electrodeposited Metals (A.E.S. Research Project No. 6). I.—Bibliography. II.—Critical Literature Review.** N. Thon and E. J. Addison, Jr. (*Monthly Rev. Amer. Electroplaters' Soc.*, 1947, **34**, (4), 445-453; (5), 568-576).—(I.—) The bibliography covers the period from 1887 to early 1946. It contains 226 references, mostly to published work, but a few to unpublished private communications. It represents the preliminary step in the work of A.E.S. Research Project No. 6, and is confined to sources which make explicit contributions to the specific study of the nature and causes of the porosity which seems to be a systematic occurrence in metal electrodeposits. (II.—) A preliminary part of A.E.S. Research Project No. 6, preparatory to experimental and laboratory work. The early history of the problem of porosity in electrodeposited coatings is given briefly. This is followed by a discussion of the ferroxyl test.—G. T. C.

**Conductivity of Electrolytic Baths.** J. Liger (*Galvano*, 1947, **16**, (121), 8-10).—A general survey of the conductivity of ions.—M. E.

**Anodes and Electrolytes.** E. Baret (*Galvano*, 1947, **16**, (123), 11).—Elementary advice about copper-plating baths.—M. E.

**\*Determination of Free Sodium Hydroxide and Sodium Carbonate in Plating Solutions.** Louis Silverman (*Metal Finishing*, 1947, **45**, (4), 72-73).—S. describes a volumetric method in which, to obtain the sodium hydroxide content of the solution, the carbonate is precipitated with barium chloride and any cyanide present is prevented from hydrolysing by the addition of excess alcohol. A direct titration with HCl gives the NaOH content. The carbonate content is obtained by precipitating the carbonate from a second sample, filtering it off, and titrating.—G. T. C.

**\*A New Time-of-Gassing Test of Thickness of Cadmium Coatings, Particularly for Small Parts.** S. G. Clarke and J. F. Andrew (*J. Electrodepositors' Tech. Soc.*, 1947, **22**, 1-7).—The solution used is 10% nickel sulphate crystals in hydrochloric acid (sp. gr. 1.16); with it the rates of solution of cadmium coatings formed in different types of plating bath differ from each other by a comparatively small amount. The test is carried out by immersing the degreased part in the solution and noting the time with a stop watch from the commencement of gassing until there is a rapid falling off of the rate of gassing. A temp. correction of 2% of the apparent thickness for each 1° C. above or below 20° C. is added or subtracted. Curves are given for converting gassing times to thickness of coating. The accuracy of the method is considered to be within  $\pm 25\%$ . The method is applicable to cadmium coatings on steel and copper, and probably on brass, though this was not tested.—G. T. C.

**\*Apparatus for Anodically Stripping Electrodeposits from One Side of a Sheet for Analytical Purposes.** J. W. Price (*J. Electrodepositors' Tech. Soc.*, 1947, **22**, 22-23).—Owing to the difficulty of finding a stopping-off compound that would resist hot caustic soda-cyanide solution used in stripping speculum, an apparatus was devised of alkali-resistant plastic which allowed electrolytic stripping to be carried out on one side only of a plate sheet specimen. The circular specimen was placed in a threaded recess of its own dia. in a plastic block. A rubber ring gasket was placed on top, and a plastic cylinder screwed down tightly. Contact was made with the back of the specimen by means of a terminal screw. The cylinder was filled with stripping solution and a tin foil anode was suspended in it. The solution was heated by immersion of the apparatus in water near its boiling point.—G. T. C.

**\*Methods of Analysis of Chromium-Plating Solutions.** (Salt). See p. 19.

**\*Analysis of Gold and Gold Alloy [Plating] Solutions.** (Parker). See p. 19.

**\*Estimation of Nickel, Cobalt, Chloride, Formate, and Formaldehyde in Nickel-Cobalt Plating Solutions.** (Salt). See p. 20.



\*Determination of Impurities in Electroplating Solutions. (A.E.S. Research Project No. 2.) V.—Manganese in Nickel-Plating Baths. VI.—Traces of Copper in Nickel-Plating Baths. (Serfass and Levine). See p. 19.

**Metallography for the Electroplater.** Alex Blazy and J. B. Mohler (*Metal Finishing*, 1947, 45, (4), 54–57).—A general review of metallographic techniques for examining electrodeposited coatings. Types of microscope for low- and high-power examination, and methods of mounting, polishing, and lighting are discussed.—G. T. C.

**The Significance of Polarization in Electroplating.** H. Bandes (*Metal Finishing*, 1946, 44, (12), 516–521).—A talk given before the Newark Branch of the American Electroplaters' Society.—W. G. A.

**Control Apparatus for the Production of Uniform Electrodeposits from a Rectified A.C. Supply.** D. Ashby and S. Wernick (*Monthly Rev. Amer. Electroplaters' Soc.*, 1947, 34, (1), 42–49).—A. and W. discuss the difficulty of obtaining electrodeposits of uniform thickness. The minimum thickness required is usually specified, and any metal deposited in excess of this is uneconomical. Correct choice of solution, anode arrangement, and c.d. are important. An electrical unit is described which applies and maintains the predetermined current and rectifies fluctuations that occur. It also cuts off the current at the end of the plating cycle. In this way, the deposition of more metal than is required is guarded against, and substantial economies are achieved. The machine is most suitable for automatic and semi-automatic plants. During loading of the work, it can be arranged to provide a polarizing current for work already in the bath. Similarly, it can be arranged to give an initial high-c.d. "strike", prior to the main plating operation. A stand-by power supply (large-capacity battery) is also incorporated.—G. T. C.

**Rectifiers for Electroplating.—II.** Louis W. Reinken (*Metal Finishing*, 1947, 45, (4), 58–62).—R. discusses variable auto-transformers, their advantages and disadvantages, and rectifier construction. In connection with the latter, the importance of ventilation is particularly stressed.—G. T. C.

**Automatic Conveyor for Electroplating.** Adolph Bregman (*Iron Age*, 1947, 159, (25), 68–74; (26), 58–64).—B. reviews the development, advantages, and limitations of automatic plating equipment, describes the principal types of machines suitable, and indicates where each part may be most effectively used. He gives detailed descriptions of the operating characteristics of a number of automatic conveyors available for plating work, and discusses their costs.

—J. H. W.

**Electroplating at Flint's A.C. Spark Plug Division.** Bryant W. Pooock. (*Products Finishing*, 1947, 11, (5), 30–32, 34, 36, 38, 40, 42, 44, 46).—A description of a large-scale plating equipment. Operating details are given of cleaning procedure, bright zinc plating with automatic plant, zinc barrel plating, and deposition of copper, nickel, and chromium on zinc-base die-castings.—G. T. C.

**Disposal of Cyanide Wastes.** John G. Dobson (*Metal Finishing*, 1947, 45, (3), 68–71).—The use of chlorine to destroy cyanide in waste waters is discussed. The exact chemistry of the process is not fully understood, but it is suggested that the end-products of the reaction are ammonium carbonate, nitrogen, and nitrous oxide. Factors to be considered in designing a chlorination plant are discussed. The relative merits of continuous, automatically controlled, and intermittent types of treatment are mentioned. The process is apparently very successful, as in no operating plant has any failure occurred.—G. T. C.

**Los Angeles Plating-Plant Explosion.** Fred A. Herr (*Metal Finishing*, 1947, 45, (3), 72–73, 107).—A serious explosion in a plating plant was ascribed to a vat of 900 gal. of perchloric acid-acetic anhydride being allowed to heat up above 80° F. (27° C.)—considered to be the max. safe temp.—owing to the

failure of refrigerating equipment. The mixture was used in the electrolytic polishing of aluminium.—G. T. C.

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### X.—REFINING

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**\*Reduction of Magnesium Chloride by Calcium Carbide.** Lloyd R. Michels, Burke Cartwright, and S. F. Rivitz (*U.S. Bur. Mines Rep. Invest. No. 4059*, 1947, 8 pp.).—An examination of the reaction  $\text{CaC}_2 + \text{MgCl}_2 = \text{Mg} + \text{CaCl}_2 + 2\text{C}$ , from the point of view of its possible utility in the commercial production of magnesium. Thermodynamic data are given, and experimental work on the reaction, using welded-steel bombs, is reported. The magnesium formed by the reduction was present as minute particles, so intimately mixed with the other reaction products that it could not readily be separated; and it was concluded that the reaction does not afford a promising method for the commercial production of magnesium.—F. A. F.

**\*The Place of Yttrium and the Application of Ferricyanide Precipitation in the Rare-Earth Group. The Purification of Erbium.** (Marsh). See p. 1.

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### XI.—ANALYSIS

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**Polarographic Determination of Zinc in Aluminium Alloys.** W. Stross (*Metallurgia*, 1947, 36, (213), 163–166).—The difficulties of devising a method of polarographic analysis of wide application are discussed, and a method described which is claimed to be generally applicable to the determination of Zn in Al alloys. This utilizes the fact that Zn gives a good wave in NaOH solution; as Al is reduced at a more negative potential than Zn, its presence does not interfere. The method is usable on a semi-micro or micro scale.

—M. A. V.

**\*Acid Titration of the Aluminium Ion.** S. Lacroix (*Anal. Chim. Acta*, 1947, 1, (1), 3–12).—[In French]. A method is described of determining Al and free acid in an acid solution containing  $[\text{Al}^{3+}]$  ions. An excess of oxalate or hydrofluoride ions is added to the solution, which retards the precipitation of  $\text{Al}(\text{OH})_3$  during titration of the free acid with NaOH, using bromothymol blue as indicator. Al is determined by simultaneous titration of the  $[\text{Al}^{3+}]$  ions and the free acid, the Al content being determined by difference. In this titration, an excess of NaOH is added and a back titration with HCl is made at the boiling point, again using bromothymol blue as indicator. These two titrations enable the free acid and  $[\text{Al}^{3+}]$  to be determined to within 0.3%. An extension of the method, for determining  $[\text{Al}^{3+}]$  and  $[\text{Fe}^{3+}]$  in the presence of free acid, is described.—R. W. R.

**\*Method of Qualitative Analysis of Cations Without Systematic Separations. I.—Reactions of Arsenic, Tin, Antimony, Bismuth.** G. Charlot and D. Bézier (*Anal. Chim. Acta*, 1947, 1, (2), 113–123).—[In French]. C. and B. have previously advanced a method for the qualitative analysis of cations in which separations are reduced to a minimum, each metal being detected by an individual specific test (see *Ann. Chim. Anal. Appl.*, 1943, 25, 90, 114), and are now evolving refined methods for the detection of small quantities of an element in the presence of large quantities of other cations. In this paper, B. presents such methods for As, Sn, Sb, and Bi. As is detected with a molybdate reagent or by reaction with Al in alkaline solution, Sn by reaction with  $\text{I}_2\text{-KI}$  solution, Sb with Rhodamine B, and Bi by reaction with  $\text{SnCl}_4$ .—R. W. R.

**The Determination of Tin and Copper in Phosphor Bronze.** Brian B. Bach (*Metallurgia*, 1947, 36, (212), 65–66).—Methods of determining Sn and Cu in

tin bronze by electrodeposition are described. These were specially designed to overcome difficulties due to the presence of P, and are accurate without being unduly slow.—M. A. V.

**\*Colorimetric Determination of Calcium in the Presence of a Large Excess of Magnesium.** Emile Rinck and (Mlle.) Hélène Ostertag (*Compt. rend.*, 1947, **224**, (15), 1108–1110).—H. Beutelspracher (*Z. anal. Chem.*, 1934, **96**, 161) has described a method of micro-analysis of Ca by precipitation with  $\text{Na}_2\text{WO}_4$ . R. and O. found that this method was not very accurate, especially in the presence of large amounts of Mg, owing to the formation of double parangstates of Na and Mg, insoluble in water. They showed that the formation of these compounds could be prevented by not evaporating the precipitating solution to dryness as recommended by B. They describe their method in detail.—J. H. W.

**\*Rapid Titration of Chromium and Vanadium.** L. Ducret (*Anal. Chim. Acta*, 1947, **1**, (2), 135–139).—[In French]. Cr and V, present together in solution as  $\text{Cr}^{3+}$  and  $\text{V}^{4+}$  in concentrations between  $N/10$  and  $N/100$ , may be determined as follows: The total content of both elements is first determined by titration with  $N/10\text{-Fe}^{2+}$  solution, in  $2\text{-}3N\text{-H}_2\text{SO}_4$  solution, using sulphonated diphenylamine as an oxydo-reduction indicator. The  $\text{V}^{3+}$  in the solution is then oxidized to  $\text{V}^{4+}$  by an excess of  $\text{KMnO}_4$  ( $N/10$ ) and this excess of  $\text{KMnO}_4$  is destroyed by the addition of 2 c.c. of a 1% solution of sodium azide. The titration with  $N/10\text{-Fe}^{2+}$  is then repeated as before, and the V content thus obtained. About 5 c.c. of 85%  $\text{H}_3\text{PO}_4$  must be added before each titration. The accuracy of the estimation is about 0.2%.—R. W. R.

**\*Methods of Analysis of Chromium-Plating Solutions.** F. W. Salt (*J. Electrodepositors' Tech. Soc.*, 1947, **22**, 15–18).—Methods are described for the complete analysis of a chromium-plating solution in  $1\frac{1}{2}$  hr., as against at least 4 hr. by other methods. Chromic acid and trivalent Cr (after oxidation) are determined by titration with ferrous ammonium sulphate using  $N$ -phenylanthranilic acid as indicator. Fe is estimated by reduction with stannous chloride and titration with potassium dichromate, using barium diphenylamine sulphonate as an indicator. Alternative methods are given for trivalent Cr and Fe when the former is present in only small quantities. Sulphate is determined by precipitation in the presence of picric acid, which increases the particle size of the precipitate and thus enables it to be filtered immediately.—G. T. C.

**\*Analysis of Gold and Gold Alloy [Plating] Solutions.** Edward A. Parker (*Monthly Rev. Amer. Electroplaters' Soc.*, 1947, **34**, (1), 33–40).—P. describes methods of estimating Au, Ni, and Cu in plating solutions used for the deposition of pure Au and coloured alloy Au. For the estimation of Au, a modification of Herschlag's method is developed. As finally modified, this involves the elimination of cyanides with HCl and  $\text{H}_2\text{SO}_4$ , the removal of excess nitrites with hypochlorite, followed by neutralization with bicarbonate. KI is added, and titration carried out with arsenious oxide. At pH 7–8,  $\text{Fe}''$ ,  $\text{Fe}'''$ ,  $\text{Zn}''$ ,  $\text{Sn}''$ ,  $\text{SnO}_3^-$ ,  $\text{Ni}''$ ,  $\text{Ag}'$ ,  $\text{Cu}'$ , and  $\text{Cu}''$  have no effect unless their basic salts are coloured, and even then it is not serious. For Ni, a colorimetric method based on the use of dimethylglyoxime is used. For Cu, Mehlig's Cu-ammonia-complex method is modified. It is a colorimetric method, and suffers from interference in presence of Ni and such ions as chromates. These are unlikely to be present in sufficient quantity to be serious. Full analytical details are given for the three determinations.—G. T. C.

**\*Determination of Impurities in Electroplating Solutions.** (A.E.S. Research Project No. 2.) V.—Manganese in Nickel-Plating Baths. VI.—Traces of Copper in Nickel-Plating Baths. Earl J. Serfass and W. S. Levine (*Monthly Rev. Amer. Electroplaters' Soc.*, 1947, **34**, (3), 320–327; (4), 454–462).—Cf. *Met. Abs.*, 1947, **14**, 122. (V.—) After reviewing the possible methods, the



authors describe in detail one in which the Mn is oxidized to permanganate with sodium periodate in an acid mixture of phosphoric and perchloric acids. The colour of the resulting solution, after suitable dilution, is measured with a colorimeter. Most ions do not interfere with the determination except when coloured. Cu, Ni, and chromates are compensated for by adding a similar quantity to a blank. Ag, Pb, and Hg give no precipitate if enough acid is present. However, Bi and Sn do give precipitates, even in the presence of acid. Full experimental details of the investigation and the method used are given. The method was found suitable for all-chloride, 50 : 50 sulphate-chloride, high-sulphate, and Watts types of Ni bath. (VI.—) After reviewing past work on the determination of small amounts of Cu in Ni-plating solutions, the authors describe a method in which the Cu is precipitated from the solution by means of 2-mercaptobenzothiazole. The precipitate is extracted with amyl acetate, and the colour developed by adding dibutyl amine and carbon disulphide. The colour is measured with a colorimeter after dilution with ethyl alcohol. The method is applicable in the presence of chromates, silicates, Ca, Al, Cd, Pb, Fe<sup>II</sup>, Fe<sup>III</sup>, and Mn. The precision of the method is  $\pm 2\%$ . Full details of the development work and of the method itself are given. It is applicable to all-chloride, 50 : 50 sulphate-chloride, high-sulphate, and Watts types of Ni solution.—G. T. C.

**\*Estimation of Nickel, Cobalt, Chloride, Formate, and Formaldehyde in Nickel-Cobalt Plating Solutions.** F. W. Salt (*J. Electrodepositors' Tech. Soc.*, 1947, 22, 19-21).—All methods given are volumetric and applicable in the presence of boric acid. Titration with silver nitrate is used for Ni, Co, and chloride. Formaldehyde is estimated by heating with I<sub>2</sub> and titrating the excess with sodium thiosulphate. Sodium formate is determined, after evaporation nearly to dryness, by addition of potassium permanganate, sodium carbonate, and potassium iodide, followed by titration with sodium thiosulphate.—G. T. C.

**\*Apparatus for Anodically Stripping Electrodeposits from One Side of a Sheet for Analytical Purposes.** (Price). See p. 16.

**\*Determination of Free Sodium Hydroxide and Sodium Carbonate in Plating Solutions.** (Silverman). See p. 16.

**\*Detection of Scandium, Rare Earths, Zirconium, and Thorium with Murexid.** G. Beck (*Anal. Chim. Acta*, 1947, 1, (1), 69-71).—[In German]. The reactions with murexid of Sc, Zr, Th, the rare earths, and other elements are briefly described, and qualitative tests for these elements using murexid reagent are given. The presence of Al or Be does not interfere. Further tests for discrimination between the elements are suggested. A titrimetric method for the estimation of Sc, using murexid and nitrilotriacetate, is proposed.

—R. W. R.

**\*The Electrolytic Determination of Thallium.** Jean Besson (*Compt. rend.*, 1947, 224, (17), 1226-1227).—B. briefly points out the disadvantages of each of the many methods previously proposed for the electrolytic determination of Tl. He investigated the nature of the oxide of Tl deposited at the anode in the electrolysis of a Tl salt in an acid solution, and shows that it is neither the dioxide, TlO<sub>2</sub>, nor the intermediate oxide, Tl<sub>3</sub>O<sub>5</sub>, but the ordinary sesquioxide, Tl<sub>2</sub>O<sub>3</sub>. The electrometric reduction of this oxide is not suitable as a method of determining Tl, as it is difficult to effect this quantitatively.—J. H. W.

**Applications of Polarography to Metallurgical Analysis.** J. Heyrovsky (*J. Proc. Roy. Inst. Chem.*, 1947, (II), 82-83).—A lecture to the Sheffield, S. Yorkshire, and N. Midlands Section of the Royal Institute of Chemistry. H. reviews polarographic analysis and its application to the determination of impurities in metals. The use of a streaming-mercury electrode and cathode-ray oscillograph in quantitative analysis by the polarographic method is outlined.—H. J. A.



**Polarographic Methods of Analysing Metals.** M. Spalenka (*Fonderie*, 1946, 1, (12), 466–467).—A review.—M. E.

**\*Spectrochemical Analysis by the Copper Spark Method.** Mark Fred, Norman H. Nachtrieb, and Frank S. Tomkins (*J. Opt. Soc. Amer.*, 1947, 37, (4), 279–288).—Spark spectra of solutions in HCl evaporated to dryness on Cu rods are recommended to combine the sensitivity usually obtained from arc spectra with the reproducibility of spark sources. Freshly turned, hard-drawn Cu rods,  $\frac{1}{4}$  in. in dia., with flat ends were treated with 0.1 ml. of acid solution and dried in coils of Nichrome resistance wire, heated so as to evaporate the liquid without visible boiling. Spectra were examined by visual comparison with test plates from solutions of known concentration; the sensitivity for 64 elements is tabulated. Quantitative results obtained with Mo, Pb, or other suitable internal-standard elements had average deviations of 3–8% and less sensitivity. The method has been applied to the purification of plutonium; the precautions needed to get optimum results are described in detail.

—E. VAN S.

**\*Flame Spectra of Copper Salts.** Nand Lal Singh (*Proc. Indian Acad. Sci.*, 1947, [A], 25, (1), 1–21).—A paper on visual lines which might be of use in the selection of internal standards for the visual estimation of impurities in Cu.

—E. VAN S.

**A Method of Semi-Quantitative Spectrographic Analysis.** C. E. Harvey (*J. Opt. Soc. Amer.*, 1946, 36, (12), 719).—A summary of a paper, read before the Optical Society of America (later published as a booklet), on a rough quantitative spectrographic method, applicable to any metallic or non-metallic material, for the estimation of about 40 metallic elements, as well as B, P, and Si. The method depends on the complete vaporization of the sample in an arc, and on measurements of selected spectrum lines, which are compared with the background for intensity and evaluated by means of factors which depend on the matrix of the mixture arced.—E. VAN S.

**\*Use of Incompletely Consumed Samples to Illustrate Effect of Fractional Distillation in Carbon-Arc Spectrochemical Analyses.** Lester W. Strock and George E. Heggen (*J. Opt. Soc. Amer.*, 1947, 37, (1), 29–36).—When mixtures of NaCl with alkali earth salts are being vaporized in the carbon arc for analysis, either BeO or Cr<sub>2</sub>O<sub>3</sub> may be added as an internal standard. If the arcing is continued till the samples are completely consumed, tolerable results are obtained with Be, but if the exposure is stopped before all the Na is burnt off, Cr comparison lines give better results. The reasons for this difference are examined in detail and explained.—E. VAN S.

**The Calculation of Line Strengths from Laboratory Data.** William Petrie (*Canad. J. Research*, 1947, [A], 25, (1), 42–48).—The line strength is one of the parameters on which the calculation of line intensities is based, and data are provided which may be of use in the selection of lines for comparison in analysis.—E. VAN S.

**\*A Slit-Less Spectrograph.** Raymond Ricard (*Compt. rend.*, 1946, 222, (19), 1091–1092).—After enumerating some of the practical difficulties of a normal spectrograph, R. describes how the slit may be replaced by the brilliant virtual focus of a polished cylinder forming a cylindrical convex mirror. In preliminary work, a steel needle, dia. 1 mm., was used as reflecting cylinder, with a sodium lamp and an iron arc as sources. Very clear spectra were obtained for all positions of the sources, without refocusing. The instrument possesses the following advantages for spectrum analysis: the spectrum of all the source is observed; small displacements of the source produce only second-order effects on the illumination of the prism; the reflecting power of the cylinder does not enter the calculations; and spectra may be obtained from moving sources, or sources whose position cannot be adjusted beforehand (e.g. lightning). By coating the cylinder with Al, spectroscopy in the ultra-violet

may be carried out. Being achromatic, the reflecting cylinder is better than the cylindrical diverging lens described by King (*J. Opt. Soc. Amer.*, 1946, **36**, 164; see *Met. Abs.*, 1946, **13**, 305).—D. P. M.

**A New Device of Quantitative Spectrography.** Aymé Cornu (*Compt. rend.*, 1946, **222**, (23), 1341–1342).—A slightly modified Feussner circuit is used to supply two sparks, one between standard alloys and the other between the samples examined, the electrode separation being equally regulated. The slit-less spectrograph of Ricard (see preceding abstract) is used, the separation of the pseudo-slits being about 1 mm. Irregularities in the emulsion are eliminated, the sparks are excited under identical conditions, and the times of exposure are equal. For complicated spectra, the dispersion may be increased without sacrificing the simplicity of the system.—D. P. M.

**General Review of the Present Position of Quantitative Spectrographic Analysis.** J. Gillis (*Anal. Chim. Acta*, 1947, **1**, (1), 38–49).—[In English]. A review of recent published information.—R. W. R.

## XII.—LABORATORY APPARATUS, INSTRUMENTS, &c.

(See also "Testing" and "Temperature Measurement and Control".)

**\*A Simple Method for Producing Good Front-Surface Mirrors [Aluminium Films on Glass].** Noel W. Scott (*J. Opt. Soc. Amer.*, 1946, **36**, (12), 711).—A summary of a paper on the deposition of aluminium films on glass, for use as front-surface mirrors, and on the protection of these films by a transparent surface film of SiO. After the aluminium has been deposited, a mixture of Si and SiO<sub>2</sub> is evaporated from a boat of tungsten or tantalum in the vacuum apparatus, and the mixture gives vapour of SiO, which forms a more adhesive film on aluminium than SiO<sub>2</sub> can form. The abrasion resistance is tested with a steel ball wrapped in linen and loaded with a 500-g. weight, which would mark an aluminium surface in a single movement. Similar mirrors can be formed on metal bases, with an intermediate film of SiO between the base metal and the aluminium, or even on some plastics.—E. VAN S.

**Instrument Threads [Standardization].** G. A. Whipple, W. O. Davis, and A. D. Snutch (*Proc. Inst. Mech. Eng.*, 1946, **155**, (18), 152–154).—The authors present the results of discussions at an Anglo-Canadian-American series of conferences on the standardization of instrument threads. The conference considered instrument threads as: (1) fastening screws, (2) fine-motion screws, (3) bearing-adjustment screws, (4) optical mounting threads, and (5) attachment threads. The results of the discussions are tabulated, and, in general, with the possible exception of some micrometer screws, it is suggested that a Whitworth-form thread be adopted.—H. J. A.

**Optical Methods for Evaluation of Metal Surfaces.** Arthur A. Vernon and John Broomfield (*Metal Finishing*, 1947, **45**, (4), 70–71).—A brief review of the optical instruments available for the evaluation of the degree of smoothness of metal surfaces. Reference is made to instruments which show a picture of the surface and to one which measures accurately the light reflected from the surface.—G. T. C.

**Optical and Allied Instruments Shown at the Physical Society's Exhibition [1947].** J. L. Haughton (*J. Sci. Instruments*, 1947, **24**, (6), 142–143).—Reference is made to an optical surface finish comparator made by Machine Shop Equipment, Ltd. This consists of a double microscope which enables the surface under examination to be seen beside a standard surface. A set of 8 standard surfaces are provided. Numerous travelling microscopes were shown.—W. H. R.

**Thermal and Mechanical Instruments Shown at the Physical Society's Exhibition [1947].** C. R. Barber (*J. Sci. Instruments*, 1947, 24, (6), 144–145).—No outstanding achievements are noted, but references to pyrometers, furnaces, and dilatometers are of direct metallurgical interest. These include a colour-temp. pyrometer developed by H. Tinsley and Co. in conjunction with the B.I.S.R.A., and pyrometers based on lead sulphide photo-conductive cells. A "photo-electric brightness telepyrometer" made by the General Electric Co., Ltd., indicates differences of temp. as small as 2° C. at 1050°–1000° C., using about 1 in.<sup>2</sup> of source at 15 ft.; in its present form it is unsuited to absolute temp. measurements. A modified mercury-in-steel thermometer made by Negretti and Zambra, Ltd., can be used up to 650° C. A molybdenum furnace by the Wild-Barfield Electric Furnaces, Ltd., can be used up to 1700° C., and a new type of gas muffle using a recuperative system, exhibited by the Gas Light and Coke Co., enables a temp. of 1450° C. to be reached without forced draught.—W. H.-R.

**Recorders and Controllers Shown at the Physical Society's Exhibition [1947].** K. M. Greenland (*J. Sci. Instruments*, 1947, 24, (6), 146–148).—A new thermocouple recorder by Sunvic Controls, Ltd., has a full-scale deflection of  $\pm 10^\circ$  C., and registers up to 1500° C. with a probable accuracy of  $\pm \frac{1}{4}^\circ$  C. It is provided with automatic standardization of the potentiometer voltage, and automatic protection in the event of failures. Courtauld's, Ltd., show a method of recording which is photographic but is visible without development. A high-intensity spot of light from a galvanometer mirror is focused on a moving sheet of printing-out paper. Several recorders using electronic amplification are mentioned. New cine-camera instruments are described, including applications for visible, infra-red, and X-ray radiation.—W. H.-R.

**Electrical and Acoustical Instruments Shown at the Physical Society's Exhibition [1947].** T. B. Rymer (*J. Sci. Instruments*, 1947, 24, (6), 148–151).—Much of general but little of direct metallurgical interest is included, although the following may be noted: (1) Cobalt-steel magnets formerly used in moving-coil meters have been completely replaced by materials such as Alcomax and Ticonal. (2) A few commercial applications of supersonics were shown. In one of these, the thickness of a metal plate is gauged by placing a piezo-electric supersonic generator in contact with one face and adjusting the frequency until resonance is obtained; the thickness of the plate can then be determined from the known velocity of sound in the metal. Another application is the detection of flaws in a metal. A supersonic generator is placed in contact with one face of the specimen under test. Flaws cause scattering of the resulting sound waves in the metal, and the scattered sound waves are picked up on a detector, and shown on a cathode-ray tube.—W. H.-R.

**Summarized Proceedings of Conference on the Electron Microscope, Oxford, 1946.** V. E. Cosslett (*J. Sci. Instruments*, 1947, 24, (5), 113–119).—The first session dealt with the construction of electron microscopes of different types. G. D. Preston contributed a paper on the principles of the imaging process. M. E. Haine dealt with the limiting factors of a practical nature encountered in designing electron microscopes, and described the instrument being produced by the Metropolitan-Vickers Electrical Co. In the projection-lens system, the limiting factor is distortion of the field, and this is eliminated by employing a double projection lens. The main magnification is provided by the first lens, while the second lens runs at const. power, using a small area only of the field between the poles. J. B. Le Poole described the electron microscope developed during the war at Delft. This was a 3-stage instrument employing 5 water-cooled magnetic lenses. A. C. Van Dorsten described the microscope now being developed by Philips of Eindhoven on the basis of Le Poole's work. W. J. Oosterkamp gave an account of an experimental electron microscope for 400 kV., in order to obtain greater penetrating power of the beam. G. Dupouy



(University of Toulouse) has developed a 2-stage magnetic microscope which exhibits many ingenious mechanical ideas. *H. Bruck* (Paris) spoke of work on electrostatic lenses, and of an electrostatic microscope in which they were used. The best resolution obtained was about 80 Å. *D. Gabor* (Rugby) discussed phase-contrast phenomena in electron microscopy, and the possibility of revealing the structure of objects too thin to be revealed by differential scattering. It might be possible to correct electron optical systems by drying a thin film over a fine aperture; if the form of the film were a fourth-order parabola, the aberrations of the electron microscope could be reduced to about half the limit calculated for existing systems. He summarized a long communication from *O. Scherzer*. The session closed with a lively discussion of the points raised. The next session was concerned mainly with problems of specimen preparation. *D. G. Drummond* (Manchester) gave a general review, including surface-replica methods. *W. M. Jones* (Harwell) described a method of taking surface replicas, using methyl methacrylate instead of polystyrene; this enables the impression to be taken at room temp. and pressure. A silica film is then prepared, and good results have been obtained with pearlite. *R. Garrod* read a paper on the measurement of surface topography, and *R. V. Scott* made critical remarks on the technique of shadow casting. The final session was concerned with papers on applications of electron microscopy. *W. H. Walton* and *W. J. Harris* dealt with applications to particle-size measurements, and *G. Dupouy* described investigations including anodic replicas of aluminium surfaces after different treatments. The degree of etching of aluminium specimens of different purity was strikingly demonstrated.—*W. H.-R.*

**A Magnetic Electron Microscope with High Resolving Power.** *Gaston Dupouy* (*J. Phys. Radium*, 1946, [viii], 7, (11), 320-329).—An illustrated account of the first electron microscope entirely built in France. Its characteristics are: 70-250 kV.; vacuum,  $10^{-5}$  mm. of mercury; and resolving power, 20-30 Å.—*M. E.*

**New Developments in Electron Microscopy.** *J. L. Farrant* (*Elect. Eng. Merchandiser*, 1947, 23, (10), 315-319).—A review.—*H. J. A.*

**\*Apparatus for Measuring Power Loss in Small Ferromagnetic Samples Subject to an Alternating Magnetic Field.** *K. H. Stewart* (*J. Sci. Instruments*, 1947, 24, (6), 159-162).—An apparatus is described which enables loss measurements to be made at known flux densities on flat strip specimens, about  $15 \times 1 \times 0.03$  cm., of soft magnetic materials. Frequencies of between 10 and 150 c./s. can be used.—*W. H.-R.*

**\*Photo-Electric Spectrometer to Record Faint Illumination.** *Alexandre Dauvillier* (*Compt. rend.*, 1946, 222, (18), 1042-1043).—*D.* describes the use of a photo-electric cell in the spectrometry of faint illuminations, thus eliminating the sensitometric and photometric operations necessary with a photographic plate. Electronic amplification enables spectra which require prolonged photographic exposures to be obtained in a few minutes, and the method is thus very useful for studying comets and auroræ. The application to a normal spectrograph with glass prisms and a dispersion of 109 mm. between 3800 and 8300 Å is described. The photo-electric cell uses a rubidium or silver cathode, giving good sensitivity between 2500 and 11,000 Å. The large dispersion enabled wide slits to be used, and the spectrum of neon, with a minimum illumination of 0.08 lumens, was obtained in a few minutes. A photographic plate is more sensitive below 5000 Å, but the photo-electric cell is better in the red and infra-red. With illumination by cathodic luminescence of neon, argon, and mercury, the spectra do not pass beyond 5900 Å with panchromatic plates (Agfa Isopan) or 7800 Å with special infra-red plates (Kodak). The photo-cell method picks up rapidly the fluorescence curves of organic substances or of crystals illuminated with ultra-violet light.—*D. P. M.*



### XIII.—PHYSICAL AND MECHANICAL TESTING, INSPECTION, AND RADIOLOGY

**\*A Rapid Method for Accurate Yield-Strength Determination Without Stress-Strain Curves.** L. J. Ebert, M. L. Freid, and A. R. Toole (*Bull. Amer. Soc. Test. Mat.*, 1947, (145), 50-52; discussion, 52-53).—The authors describe a method by which the yield strength of non-ferrous materials may be determined rapidly by a single operator and without an automatic load-elongation recorder. Charts have been constructed relating yield loads and total elongations to yield strengths, for test specimens of various cross-sectional areas. From these charts, it is possible to estimate the correct extensometer reading (total elongation) at which to read the actual yield load from the tension machine dial. The errors involved in the rapid method are of the same order as those involved in making the measurements. In discussion, it is stressed that the method is suitable only for use with metals of known elastic moduli.—H. J. A.

**Proof Loading—An Essential Non-Destructive Test.** John C. New (*Indust. Radiography Non-Destructive Test.*, 1947, 5, (4), 37-40).—A review of the scope of proof testing, with special reference to the simultaneous use of strain-sensitive brittle lacquers or wire resistance strain-gauges on the surface of the specimen.—L. M.

**Observations on Conducting and Evaluating Creep Tests.** W. Siegfried (*J. Iron Steel Inst.*, 1947, 156, (2), 189-207).—S. stresses the importance of a knowledge of the creep properties, especially under multi-axial stressing, of the heat-resisting materials used for gas-turbine blades, and discusses testing procedure. The dangers of extrapolating to long periods of time, tests which only occupy a few hundred hours are emphasized, and it is pointed out that the stress/time-to-rupture curve frequently shows a discontinuity at long times. It is suggested that the presence of such discontinuities may sometimes be deduced from observations of changes taking place in other physical properties (e.g. magnetic saturation) during short-time tests. It is important that creep tests be made on both smooth and notched specimens, so that the separate influences of notching and multi-axial stressing may be assessed. S. reviews the work of Kuntze, McAdam, and others, presents the results of sustained-load tests at elevated temp. on a number of steels, and makes some deductions with regard to the notch sensitivity and the behaviour of these materials under multi-axial stressing. S-shaped tenacity curves (true stress/reduction of area) are given which show that time of testing exerts a profound influence on the deformation characteristics of the materials. Some practical recommendations are made for testing materials at elevated temp.

—R. W. R.

**Some Aspects of Hot-Hardness Testing.** K. G. Robinson (*Metallurgia*, 1947, 36, (211), 45-46).—Impact and indentation methods of testing hot hardness are discussed critically. Static testing is recommended, using steel balls up to 200° C., and sintered carbide balls up to 800°-900° C. At higher temp., impact testing may be preferable.—M. A. V.

**\*A New Micro-Hardness [Testing] Apparatus, the "Microscélromètre L.C."** Robert Girschig (*Rev. Mét.*, 1946, 43, (3/4), 95-112; and (summary) *Indust. Diamond Rev.*, 1947, 7, (80), 208-211).—Presented to the Société Française de Métallurgie. G. has invented a new hardness-testing machine which makes impressions 0.01 mm. in size with a load of 10 g. The load is applied by a plate spring which is loaded with weights. G. studies the possibility of measuring the hardness of single grains. Only one grain must be involved. The probability of a good hardness measurement is :

$p = \frac{1 - d/r_0 \sqrt{1 - (a/r_0)^2}}{2}$ , where  $2d$  is the dia. of the affected zone, and  $2r_0$  the grain dia. If  $P$  is the load,  $P/d^2$  is not a constant.—M. E.

†**Magnetic Testing.** Raymond L. Sanford (*U.S. Nat. Bur. Stand. Circ.*, 1946, (C456), 40 pp.).—The subject matter is dealt with under these headings: (1) introduction, (2) magnetic quantities and units, (3) magnetic characteristics of materials, (4) tests with D.C., (5) tests with A.C., (6) magnetic properties of typical materials, (7) magnetic analysis, and (8) tables of data and references to the literature.—H. J. A.

**Testing Materials for Internal Discontinuities with Supersonic Echoes [Reflectoscope].** J. W. Dice (*Indust. Radiography Non-Destructive Test.*, 1947, 5, (4), 29–33).—A detailed account of the (Sperry) supersonic Reflectoscope, which utilizes supersonic waves (from 0.5 to 12 Mc./s.) to detect discontinuities in metals. The apparatus permits a penetration in aluminium of 28 ft. (24 ft. in steel).—L. M.

**Use of "Magnaflex" and "Zygo" for Non-Destructive Testing.** K. E. Glover (*Indust. Radiography Non-Destructive Test.*, 1947, 5, (4), 41–44).—A review of the scope and technique of (1) magnetic methods for detecting surface cracks and sub-surface discontinuities in magnetic materials, and (2) the use of fluorescent salt suspensions, in conjunction with viewing by near-ultra-violet light, for the detection of surface discontinuities, including cracks, in both magnetic and non-magnetic materials.—L. M.

#### RADIOLOGY

**Gun-Metal Castings. Radiographic Tests.** William H. Baer (*Amer. Foundryman*, 1947, 11, (4), 111–116).—A general illustrated review, in which is described the appearance of radiographs of defects due to gas porosity, shrinkage, and blow-holes.—J. E. G.

**Some Photographic Aspects of Industrial Radiography.** Herman E. Seemann (*Indust. Radiography Non-Destructive Test.*, 1947, 5, (4), 9–15).—A detailed description of image formation and film processing, with special reference to X-ray films. In discussing lead and salt intensifying screens, reference is made to the use of a lead screen in making electron micrographs. The thin specimen is placed between a front lead screen and the film. X-ray radiation incident upon the lead generates electrons which, passing through the specimen, produce the image.—L. M.

#### XIV.—TEMPERATURE MEASUREMENT AND CONTROL

**A Symposium on the Contamination of Platinum Thermocouples.** — (*J. Iron Steel Inst.*, 1947, 155, (2), 213–234; and (summary) *Iron and Steel*, 1947, 20, (6), 232–241).—An introduction to a symposium consisting of six papers (abstracted below) covering work done during the last few years to elucidate the causes of the contamination of platinum thermocouples at steel-making temp. The conclusions reached are briefly reviewed in this introduction.—R. W. R.

\***An Investigation of the Embrittlement of Platinum-Rhodium Wire in the Heads of Liquid-Steel Pyrometers.** T. Land (*J. Iron Steel Inst.*, 1947, 155, (2), 214–215).—Lengths of platinum-rhodium wire were heated at 1000° C. for 1 hr. in thermocouple sheath assemblies, the different parts of which were made from the following materials: steel, graphite, silica, Sairset cement, and asbestos. The experiment was repeated with different combinations of materials omitted so as to isolate the contaminant. The couple wires were subsequently tested for embrittlement by a tension test, the load being

measured on a spring balance. Brittle fractures were obtained with all combinations of materials involving new steel tubes, but, when well burnt-out steel tubes were used, ductile fractures were generally obtained. The embrittlement is ascribed to the presence of oil and other carbonaceous materials in the steel tubes.—R. W. R.

**\*Fracture of Platinum and Platinum-13% Rhodium Wires Used in the Immersion Thermocouple.** L. Reeve and A. Howard (*J. Iron Steel Inst.*, 1947, 155, (2), 216-220).—The intercrystalline attack commonly experienced by wires in the Schofield-Grace immersion thermocouple was investigated by heating specimens of platinum-rhodium wire in the presence of oil, in a graphite block, the wires being placed in a silica insulator. Attack was found to be rapid above 1200° C., and a standard test was evolved in which specimens  $\frac{3}{8}$  in. long were heated at 1400° C. for 15 min. in the presence of 0.05 c.c. oil, of sulphur content 0.2%. Specimens of wire in the fully cold-worked, partially annealed, and fully annealed states were examined, and it was found, by subsequent micro-examination, that the partially annealed wires were most susceptible to attack, and also showed more marked grain coarsening. A further observation was that, in the presence of the silica insulator, swelling frequently occurred at the portion of the wire just leaving the insulator: max. attack was found to have occurred at this point. Spectrographic analysis showed the presence of the following elements in the contaminated wires: copper > silicon > manganese > nickel > iron. The conclusions of Chaston *et al.*, that contamination is caused by the co-presence of sulphur and silica (see abstract below), was confirmed by substituting an alumina insulator for the silica one, and by replacing the oil by flowers of sulphur or by liquid paraffin. Attack was only observed with those combinations of materials containing both sulphur and silica. Oil tests carried out on pure platinum revealed a similar kind of intercrystalline attack. It was found that, in practice, attack could be prevented by baking the iron protection tubes at a dull-red heat in the presence of a current of air.—R. W. R.

**\*An X-Ray Investigation of the Embrittlement of Platinum and Platinum-Rhodium Wires.** H. J. Goldschmidt and T. Land (*J. Iron Steel Inst.*, 1947, 155, (2), 221-226).—The authors describe the results of X-ray diffraction studies of the following thermocouple materials: platinum, platinum-13% rhodium, and pure rhodium wires artificially contaminated by a method similar to that used by Reeves and Howard (see preceding abstract), a platinum-13% rhodium wire accidentally contaminated with quenching oil, and a thermocouple which had failed in service. A 9-in.-dia. Debye-Scherrer camera was used, employing cobalt radiation for the platinum-rich materials and copper radiation for the rhodium-rich materials, and a preliminary study of the system platinum-rhodium was made, using prepared standards. Observed lattice parameters for the pure materials were 3.9161 ( $\pm 0.0001$ ) $a_0$  kX. (platinum) and 3.7968 ( $\pm 0.0001$ ) $a_0$  kX. (rhodium). Measurement of the parameters of a series of standard alloys revealed a small divergence from Vegard's Law in the direction of larger values of the parameter. Within the range 0-30% rhodium it is possible to determine the rhodium content to within  $\pm 0.1\%$  from lattice-parameter measurements, using the following formula:  $C = 903.1(3.9161 - a_0) - 1296(3.9161 - a_0)^2 + 6300(3.9161 - a_0)^3$ , where  $C$  is the atomic percentage of rhodium. A brief investigation of the system platinum-silicon was made. Silicon was found to have a small solid solubility in platinum, and two silicides were detected, these being formed by the addition of 1% silicon to pure platinum (silicide "P"), and to platinum-13% rhodium (silicide "R"). Phases detected in the artificially contaminated wires were: "P" (in pure platinum) and "R" (in the 13% alloy). The platinum leg of the couple which failed in service showed an additional, unidentified phase, "Q", and the alloy leg of this couple contained "Q" only.



The matrix of the accidentally contaminated wire was found to have split into two phases, one rhodium-free (platinum black), the other containing 19.6% of rhodium; phases "P" and "Q" were also present, these being confined to the platinum- and rhodium-rich parts respectively. The phase splitting in this wire is clear evidence of reaction with another element which enters into solid solution in pure platinum. Embrittlement is thought to be caused by the presence of phases "R" and "Q", while phase "P" is considered relatively ductile.—R. W. R.

**\*Contamination and Failure of Rare-Metal Thermocouples.** D. Manterfield (*J. Iron Steel Inst.*, 1947, 155, (2), 227-229).—M. describes the results of visual and X-ray examinations of six thermocouples which failed in service. The couples had been used to measure temp. in solidifying steel ingots, and had been protected from the molten metal with silicon insulators and sheaths. The X-ray examination showed that the following impurities were present near the points of failure in the platinum wires: (1) a platinum-silicon compound, (2) magnesium silicate, (3) magnesium hydroxide, (4) a silicon-rich phase, and (5)  $\gamma$ -iron; contaminants found in the platinum-rhodium wires were (4) and (5) above, and a platinum-rhodium-silicon compound. Some suggestions as to the origins of the contaminants are made.

—R. W. R.

**\*Embrittlement of Platinum/Platinum-Rhodium Thermocouples.** J. C. Chaston, R. A. Edwards, and F. M. Lever (*J. Iron Steel Inst.*, 1947, 155, (2), 229-231).—The authors describe tests in which pure platinum and platinum-13% rhodium wires were heated in contact with carbon, sulphurous vapours, and hydrocarbon vapours (*(a)* xylol and *(b)* white spirit). None of these substances gave rise to embrittlement when employed alone, but severe attack occurred when sulphur and carbon were present simultaneously at 1200° C. and above. No attack occurred at 1100° C. or below. Chemical analysis of the attacked wires revealed the presence of silicon, but no carbon or sulphur. The explanation advanced is that the sulphur combines with the silicon in the refractory of the furnace lining, forming volatile  $\text{SiS}_2$  which attacks the platinum; the presence of carbon is necessary to catalyse this reaction. This explanation was confirmed by substituting fused alumina for the siliceous furnace lining, when no attack occurred.—R. W. R.

**\*Examination of the Microstructure of Contaminated and Embrittled Platinum and Platinum-Rhodium Wires.** R. C. Jewell (*J. Iron Steel Inst.*, 1947, 155, (2), 231-234).—J. reports the results of micrographic and spectrographic examination of *(a)* the wires examined by Manterfield, *(b)* the wire accidentally contaminated by quenching oil and examined by Goldschmidt and Land, and *(c)* the service failure referred to by Goldschmidt and Land (see preceding abstracts). The failure of wire *(a)* was due to severe attack by silicon (greatest for the platinum-rhodium wire), a brittle eutectic being formed. The silicide phase in the platinum-rhodium wire was microscopically distinguishable from that formed in the pure platinum wire, showing more relief and a bluish tinge, and exhibiting considerable segregation. These two constituents are respectively the "R" and "P" phases of Goldschmidt and Land. Contamination of wires *(b)* and *(c)* was similar, taking the form of intercrystalline attack; similar unidentifiable constituents were present in both instances (probably the constituent "Q" referred to by Goldschmidt and Land).—R. W. R.

**\*A Photo-Electric Pyrometer for a Small High-Frequency Induction Furnace.** T. Land and H. Lund (*J. Iron Steel Inst.*, 1947, 156, (1), 75-77).—The authors describe a photo-electric pyrometer designed primarily for the measurement of temp. of molten permanent-magnet alloys in a high-frequency induction furnace of 100-lb. capacity. Radiation from the surface of the metal is focused by a simple optical system on to a barrier-layer-type photo-cell. The output from the photo-cell is measured by a micro-ammeter calibrated in



° C., and a variable shunt resistance enables the sensitivity of the instrument to be varied so as to compensate for variations in emissivity of between 0.33 and 1.0. The dial of this resistance is graduated in terms of emissivity. The estimated accuracy of the instrument is  $\pm 10^{\circ}$  C.—R. W. R.

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\***Tomashov, N. D.** *The Corrosion of Metals with Oxygen Depolarization.* [In Russian.] 29 × 19 cm. Pp. 258, illustrated. 1947. Moscow and Leningrad: Academy of Sciences of the U.S.S.R. (17 roubles.)

[The book summarizes the results of a number of years' work by the author on the corrosion of metals. The main divisions of the book are: (1) Theory of the Electrochemical Corrosion of Metals, (2) Theory of Cathodic Processes in the Corrosion of Metals with Oxygen Depolarization, (3) Experimental Study of Various Cathodic Materials, (4) The Effect of the Size, Shape, and Position of the Cathodic Surface on the Effectiveness of the Work of the Cathode, and (5) The Application of Polarization Curves to Problems of the Corrosion of Metals. The principal conclusions are given in a seven-page English summary.]

\***Wickman, A. C., Ltd.** *The Wimet Tool Manual.* Demy 8vo. Pp. 92, illustrated. [1947.] Coventry: A. C. Wickman, Ltd. (10s. 6d.)

\***Zinc Development Association.** *A Report for 1945-46.* Cr. 8vo. Pp. 11. [1947.] Oxford: The Association, Lincoln House, Turl Street.



## XXV.—BOOK REVIEW

**Aluminium Alloy Castings : Their Founding and Finishing.** By E. Carrington. Med. 8vo. Pp. viii + 326, with 121 illustrations and 12 tables. 1946. London : Charles Griffin and Co., Ltd. (25s.)

The past decade has been one of steady development in the aluminium foundry industry and the author has attempted in this book to give a full and up-to-date account of the processes involved in the production and after-treatment of aluminium sand castings, gravity die-castings, and pressure die-castings. The chapter headings may be quoted to show the range of the book : (1) Quotations; (2) Pattern and Die Making; (3) Melting; (4) Sand Control; (5) Moulding and Sand Casting; (6) Die Preparation, Gravity and Pressure Die-Casting; (7) Fettling and Repairing; (8) Heat-Treatment; (9) Inspection and Laboratory Assistance; (10) After-Treatment; (11) Machining; (12) Corrosion; (13) General Organization; and (14) Future Outlook.

It will be appreciated at once that the author has set himself a very formidable task, and the treatment of some of the sections must, in a book of ordinary dimensions, necessarily be superficial. As stated in the preface, the author's aim, however, is to give a broad picture of aluminium foundry work for the benefit of the *foundryman*, and to this end he explains heat-treatment, degassing, and other metallurgical processes in simple and homely terms. The description and illustrations of foundry machinery, moulding, and casting methods are appropriate to a book of this type. The inclusion of a long and detailed statement of the duties of the metallurgist is less in keeping with the author's declared intentions, as also is the chapter devoted to corrosion. There is quite a lot of information in the book relating to foundry plant, equipment, and methods, and in the hands of, say, a foundry apprentice it may serve a useful purpose.

Still bearing in mind that the author did not set out to write a text-book but a book for foundrymen, the reviewer is left with a feeling that the foundrymen have not been particularly well served in the present volume. The selection, arrangement, and presentation of the subject matter certainly leave much ground for criticism. There are instances of undue emphasis on features which, it may be inferred, are of particular interest to the author, and the reader may form an unbalanced view of things as a result; the section dealing with the alloys used in the foundry may be used to illustrate this point. The 4.5% copper alloy (D.T.D. 298, 304, and 361) is introduced first of all, and described as one of the "most important of all the alloys". The aluminium-magnesium alloys to specification D.T.D. 300A and 165 are dealt with next, these being followed by a miscellany of alloys, including 3L5 (scarcely used at the present time, but referred to "as probably the best all-round sand-casting alloy"). Not until the end of the section do we find mention of the materials D.T.D. 424, L.33, and L.A.C. 112A, on which the greater part of our present-day casting production is based, and only the first two of these alloys are dealt with adequately.

The section on anodic treatment and dyeing which is based, presumably, on the results obtainable with wrought materials, is little less misleading and may give rise to some embarrassment to casting producers and metal finishers. The various anodic treatments are described and a long list of dye-stuffs quoted, and one would gather that it is the simplest matter to secure uniform coatings of any required colour on castings. No mention is made of the difficulties which arise from those small traces of porosity in castings, which at the present state of the art appear to be generally unavoidable; nor is it clearly stated that the alloys most readily cast and most generally used are suitable only for dark colours.

There are a host of other features in the book which will distract, puzzle, or irritate the reader. He will be told that melts of D.T.D. 300A which have been allowed to exceed a temperature of 720° C. in the melting process should not be used for casting production but should be cast into ingots (no reason for this recommendation is given); and that L.33 which becomes contaminated with copper-bearing alloys should be discarded because it falls "outside specification" (the consequences of contamination with copper are not stated). Macrographs and micrographs are given and labelled "metal" (type of alloy and composition unstated); aluminium bronze is dealt with as though it were an aluminium-base alloy; the terms contraction and shrinkage are confused in a reference to billet casting; and pin-holing is said to be peculiar to aluminium alloys. Supervising inspectors will raise their eyebrows on reading that "the production of test bars (covering melts made in the production foundry) is a very important part of the work of the laboratory". Even the English could be improved: segregation is defined as "a collecting together locally of some of the metals used to make up the alloy".

Metallurgists and metallurgical students will find little useful to them in this book.—  
R. J. M. PAYNE.





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