The Journal of the INSTITUTE OF METALS

and

METALLURGICAL ABSTRACTS

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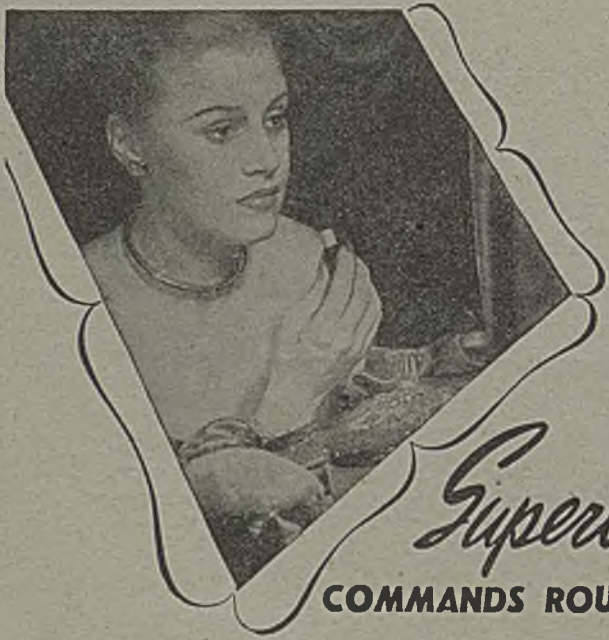

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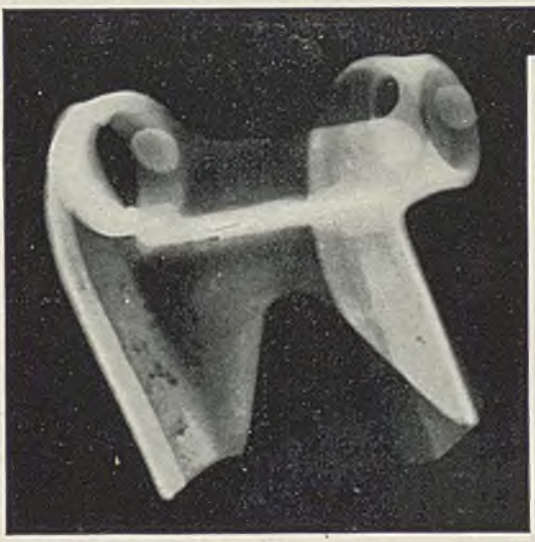

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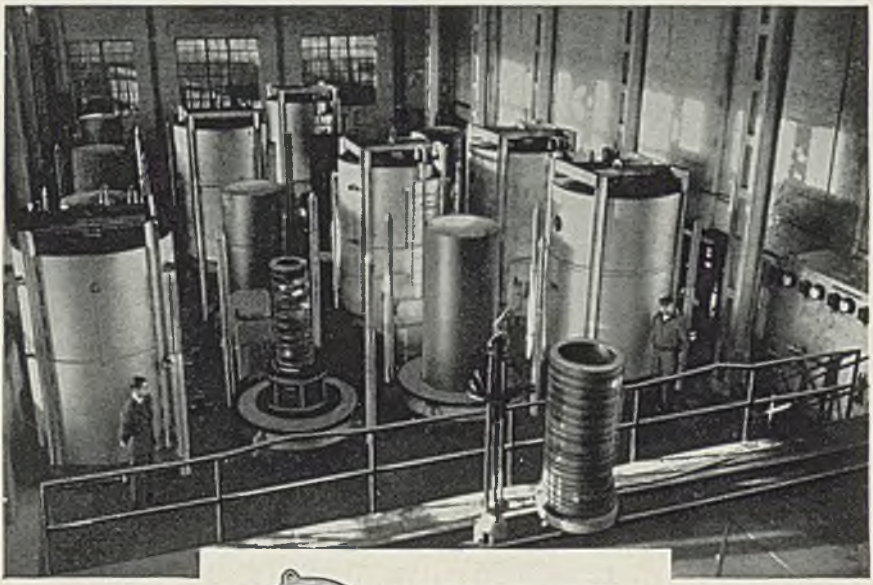

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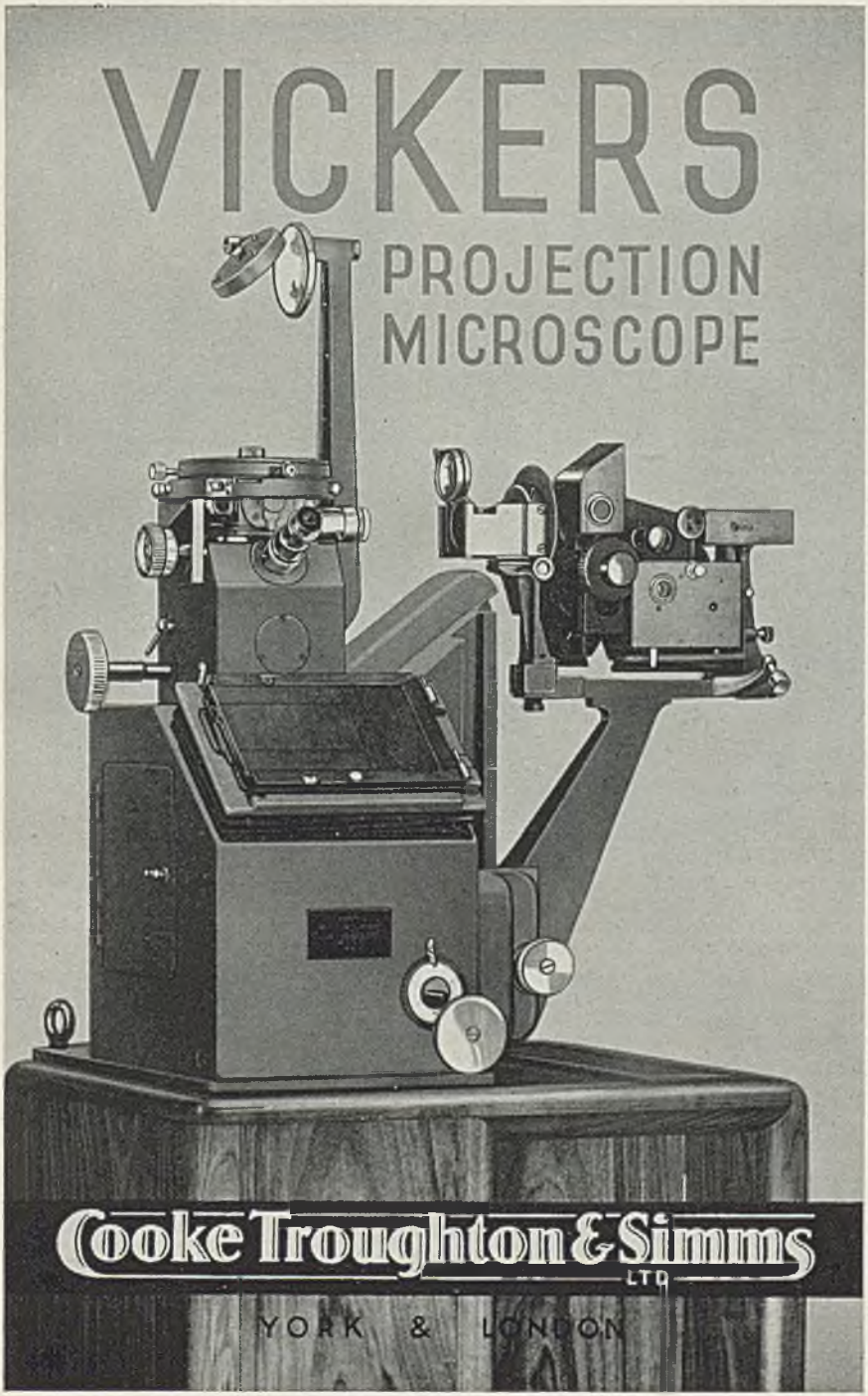
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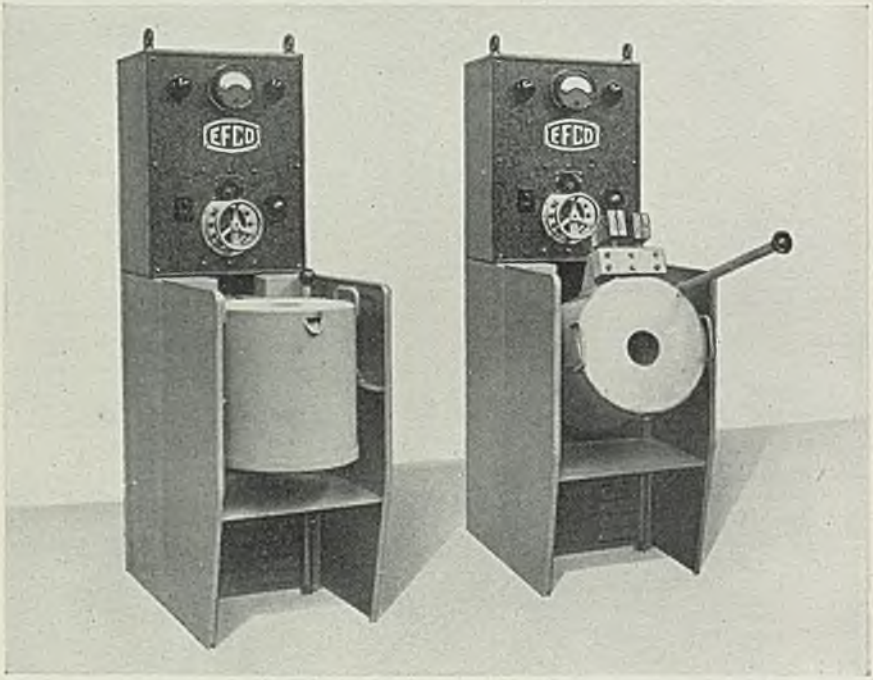

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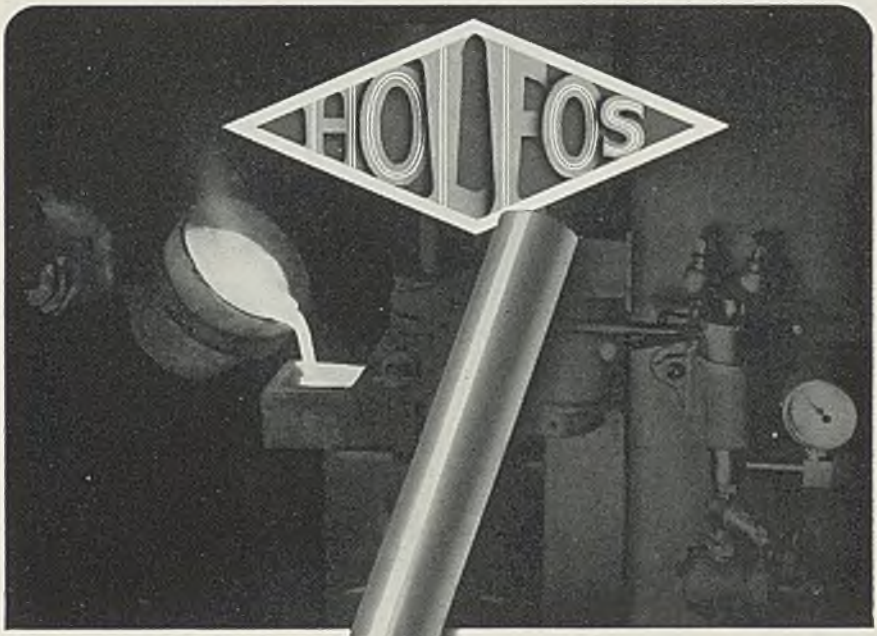

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THE INSTITUTE OF METALS

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Autumn Meeting, London.

September 10 *and* 11, 1946.

A detailed programme of the Autumn Meeting of the Institute has been despatched by post to all members.

Council.

The undermentioned officers will retire from the Council in March, 1947, and, with the exception of the President, are not at that time eligible for re-election in their present capacities:

President.

Colonel P. G. J. GUETERBOCK, C.B., D.S.O., M.C., T.D., J.P., M.A.

Vice-President.

G. L. BAILEY, M.Sc.

Members of Council.

Sir CLIVE BAILLIEU, K.B.E., C.M.G., O.B.E., M.A.
J. CARTLAND, M.C., M.Sc.
A. G. C. GWYER, B.Sc., Ph.D.
C. SYKES, D.Sc., Ph.D., F.R.S.

To fill the vacancies caused by these retirements, the Council makes the following nominations; previous service on the Council is indicated against each name (P. = President, V.P. = Vice-President, M.C. = Member of Council).

As President:

Colonel P. G. J. GUETERBOCK, C.B., D.S.O., M.C., T.D., J.P., M.A. (M.C., 1940–43; V.P., 1943–46; P., 1946–47).

As Vice-President:

Mr. JOHN CARTLAND, M.C., M.Sc., Director, Fry's Metal Foundries, Ltd., and Eyre Smelting Co., Ltd., London (M.C., 1943–47).

As Members of Council:

Professor LESLIE AITCHISON, D.Met., B.Sc., Department of Industrial Metallurgy, University of Birmingham.

Mr. JOHN ARNOTT, Chief Metallurgist, G. & J. Weir, Ltd., Glasgow.

Dr. MAURICE COOK, Delegate Director and Research Manager, Metals Division, Imperial Chemical Industries, Ltd., Birmingham (M.C., 1939–43; V.P., 1943–46).

Mr. A. J. MURPHY, M.Sc., Chief Metallurgist, J. Stone & Co., Ltd., London (M.C., 1939–43; V.P., 1943–46).

In accordance with Article of Association No. 22, any ten members of the Institute are entitled to nominate a candidate for election to the Council, such nominations to be made in writing, to be received by the Secretary of the Institute at or before the Autumn Meeting, which is to be held in London on September 10 and 11, 1946. In the event of such nominations being received, a ballot paper will be circulated to members in due course.

Local Sections.

The undermentioned Chairmen of Local Sections of the Institute have been elected for the session 1946–7. The Chairmen of Local Sections are *ex officio* Members of Council of the Institute.

Birmingham.
Mr. E. A. BOLTON, M.Sc.

London.
Mr. S. V. WILLIAMS, B.Sc.

Scottish.
Mr. A. B. GRAHAM.

Sheffield.
Mr. FRANK MASON.

Swansea.
Mr. HARRY DAVIES.

Staff Changes.

Lieut.-Colonel S. C. GUILLAN, who has been on service with H.M. Forces since August 24, 1939, returned to duty on the Institute's staff on July 24, 1946, and re-assumed his duties as Editor of the Institute's publications. He also took over from Mr. A. E. Chattin the functions of Assistant Secretary of the Institute of Metals.

Mr. A. E. CHATTIN is now Assistant Secretary of the Iron and Steel Institute and Technical Editor of its publications.

Mr. N. B. VAUGHAN, M.Sc., has resigned his post of Assistant Editor of the Institute's publications to take up the position of Information Officer and Librarian, Aluminium Development Association.

Major W. G. ASKEW, M.C., was appointed Assistant Editor with effect from June 12, 1946.

The Council has recorded its warm appreciation of the services which Mr. Chattin has rendered to the Institute as Joint Assistant Secretary and Mr. Vaughan has rendered to the Institute as Acting Editor of its publications during the war years.

Atomic Theory for Students of Metallurgy.

By Dr. W. Hume-Rothery, F.R.S.

The Institute will shortly have ready for issue the third of the Monograph and Report Series: "Atomic Theory for Students of Metallurgy," by Dr. W. Hume-Rothery, M.A., D.Sc., F.R.S. This is a book of 286 pages, bound in stiff boards. The price is 7*s*. 6*d*., post free, but any member or student member may receive one copy free on application to the Secretary.

Object and Scope of the Monograph.

In recent years the influence of physics and chemistry of a fundamental type on the development of the science of metals has been pronounced. As more data have accumulated on the characteristics of intermetallic systems, it has become increasingly plain that these characteristics may be traced back to the atomic structures of the metals concerned, and to the peculiar nature of the metals themselves. While it is possible for any metallurgist who seeks to understand the physics of metals to refer to original papers and physical text-books, it is not always possible to read such works with profit unless an able interpreter is available to point out the meaning of conclusions which are probably expressed mathematically, and to indicate the principles upon which the conclusions are based. In the present monograph, Dr. Hume-Rothery has combined the rôles of teacher and interpreter. The monograph is not a popular exposition, but is written primarily for serious study by students of metallurgy who are growing to maturity in the universities and colleges of to-day, and who have the necessary background in physics and mathematics and wish to become thoroughly acquainted with this aspect of the fundamental background of their science. The book, though so far as possible non-mathematical, involves material and conceptions with which the student may not be comfortable until he has reached an advanced stage. Much of the book should be of value to students of physics, chemistry and engineering, and there is, of course, much to repay the older metallurgist who is sufficiently interested to wrestle honestly with a subject which had developed since his own student days.

Parts I, II, and III of the monograph are of general interest. The success of the modern theory of metals has been largely due to the application of wave-mechanics to the problem of the metallic state, and the student will find the difficulties squarely met, and puzzling points fully discussed. With this material as a basis, the author deals with the structures of free atoms and describes the "probability patterns" or "electron clouds" of the electrons. Attention is then given to the problem of assemblies of similar and dissimilar atoms, and the wave-mechanical interpretation of cohesive forces in solids attacked. The normal metallic link, ionic and covalent linkages, van der Waals forces and exchange forces all form part of the picture, and the general relationships between these are brought out, and

illustrated by a review of metal and alloy structures.

Parts IV and V are more specialized and are devoted to the electron theory of metals and alloys. First the "free electron" model is discussed from the point of view of the Fermi–Dirac statistics, and it is shown how several of the typical metallic properties (*e.g.*, electronic conduction and emission; paramagnetism) are interpreted in terms of this theory. It is a good approximation only for the alkali metals, but serves to illustrate the wave-mechanical principles involved, and the manner in which these principles are made to "give results." The influence of the periodic nature of the fields of force in which electrons move in real crystals is then fully developed, and the so-called "Brillouin zone" theory and its implications are dealt with in detail. The more recent developments of the theory, and the assumptions involved in the mathematical procedures adopted in the attempt to calculate exact wave-functions, are fully described.

Part VI describes some applications of the theories to selected metals and alloys. The examples illustrate the theoretical work and give practical point to much which at first sight might seem to be purely academic.

This work may be regarded as complementary to Dr. Hume-Rothery's previous monograph, "The Structure of Metals and Alloys," and provides in admirable detail almost all that is necessary to allow the student to appreciate the fundamental basis of metal theory, and to acquire a sufficient knowledge of the modern approach to follow subsequent developments in the subject.

Membership Additions.

There were elected on July 25:

As Members.

Aal, Willem, Amsterdam, Holland.
Ball, Capt. George Raymond, Barrackpore, India.
Boag, David James Wilson, B.Sc., Chaldon, Surrey.
Brandt, David Julius Oscar, B.Sc., A.R.S.M., London.
Cann, Harold, Solihull, Warwickshire.
Dunn, Francis, Liverpool.
Foster, (Miss) Sybil Pritchard, Stoke Poges, Bucks.
Glinkov, Professor Mark, D.Sc., Moscow, U.S.S.R.
Greaves, Frank W., Birmingham.
Grönwall, Olof Rickard Alexis, Stockholm, Sweden.
Hart, Edmund S., B.Sc., A.R.S.M., Newcastle-under-Lyme, Staffs.
Haughton, Malcolm A., M.A.Sc., A.R.S.M., Bristol.
Jeníček, Professor Ladislav, S.D., Prague, Czechoslovakia.
Misra, H. N., B.Sc.(Eng.), Bombay, India.
Parks, Philip Barry, Kowloon, Hong Kong.
Payne, Charles Arnold, B.Sc., Derby.
Pouvreau, Jean Marie, Paris, France.
Race, Russell Edgar, Stonehouse, Glos.
Ramanujam, S., M.A., Khargpur, India.
Saksena, Jwala Prasad, B.Sc., Khargpur, India.
Salah-ud-Din, B.A., M.Sc., Lahore, India.
Sastri, Hebbalam Srinivasa, B.A., B.E., Sheffield.
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Waters, Alexander James Garland, B.Sc., Wraysbury, Bucks.
White, Gerald Langdale, B.A.Sc., Toronto, Canada.
Whitfield, Thomas A., Gateshead, Co. Durham.
Wright, Edwin Williams, Cape Town, South Africa.
Wright, Norman Percy, B.Sc., Bristol.

As Student Members.

Ashton, Stanley James, Bebington, Wirral, Cheshire.
Davies, John Kenneth, B.Met., Swinton, Manchester.
Day, (Miss) Margaret Katharine Bricknell, M.A., Gerrards Cross, Bucks.
Harding, Alan R., Birmingham.
Hedger, Harry John, Liverpool.
Helliwell, Ralph, Liverpool.
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SUBRAMANIAM, V., B.Sc., B.Met., Jamshedpur, India.
SUTCLIFFE, David Alan, B.Sc., Farnborough, Hants.
TOTTLE, Charles Ronald, B.Met., Newcastle-upon-Tyne.
WHYTE, Matthew, B.Sc., Gerrards Cross, Bucks.

PERSONAL NOTES

DR. EDWIN GREGORY, Chief Metallurgist of Edgar Allen and Co., Ltd., has been elected President of the Institution of Engineering Inspection.

SIR FREDERICK REBBECK, D.L., J.P., Chairman and Managing Director of Harland and Wolff, Ltd., Belfast, received the Honorary Degree of Doctor of Science of Queen's University, Belfast, on 10 July, 1946.

MR. FRANK TWYMAN, F.R.S., has resigned his position as Managing Director of Adam Hilger, Ltd., to become Technical Advisor to the firm and to its associates E. R. Watts and Son, Ltd. Mr. Twyman remains Chairman of Adam Hilger, Ltd.

DR. S. WERNICK has been elected President of the Electrodepositors Technical Society for the year 1946–47.

DEATH.

The Editor regrets to announce the death, suddenly, on 7 August, of Mr. S. V. WILLIAMS, B.Sc., Chairman, and formerly Honorary Local Secretary, of the London Local Section. Mr. Williams was Chief Metallurgist of the General Electric Co., Ltd., Wembley.

APPOINTMENTS VACANT AND REQUIRED.

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EXPERIMENTAL PHYSICIST required, preferably with some chemical or metallurgical knowledge, for research and development of new equipment with London metal spraying company. Salary £500–£700. Box No. 176, Institute of Metals, 4 Grosvenor Gardens, London, S.W. 1.

THE COLD WORKING OF A HIGH-PURITY ALUMINIUM ALLOY CONTAINING 4% OF COPPER AND ITS RELATION TO AGE-HARDENING.* 1031

By MARIE L. V. GAYLER,† D.Sc., MEMBER.

SYNOPSIS.

By means of microscopical analysis and hardness measurements, the effect of cold work on a high-purity aluminium alloy containing 4% of copper has been studied in relation to its age-hardening properties.

The data obtained allow the following conclusions to be drawn:

(1) Cold work accelerates the rate of ageing by an amount determined by the degree of cold work given.

(2) Microstructural changes have shown that the effect of cold work on a quenched specimen is to bring it into the fully aged state normally produced by ageing at room temperature; the effect of further ageing at room or higher temperatures has been related to conditions determined by the normal time–hardness curves.

(3) Lattice strain caused by cold work has been shown to be relieved by the formation of sub-microscopic (or microscopic, depending on the previous condition of the alloy and its ageing temperature) "crystallites" and, simultaneously, sub-microscopic precipitates of copper-rich particles.

(4) The course of ageing curves of cold-worked and aged material has been considered in relation to the amount of lattice strain the material is capable of withstanding. The inference has been drawn that measurement of lattice strain, resulting from the cold rolling of a soft-annealed specimen, may be associated with Brinell hardness.

(5) The mechanism of the relief of a strain caused by cold work is similar to that which occurs in relief of strains set up during age-hardening, *i.e.*, by the formation of "crystallites."

INTRODUCTION.

IN a recent paper[1] on the age-hardening of high-purity aluminium–4% copper alloy, the present author published new data regarding the changes in microstructure occurring during the process of age-hardening at room and higher temperatures.

Briefly, the age-hardening of the alloy is considered to be due to the precipitation of copper-rich particles from solid solution, and to the simultaneous formation of "crystallites" of the solid solution of aluminium, stable at the temperature of ageing, and *not* to the precipitation of either α- or β-$CuAl_2$.[2]

Furthermore, it has been shown that:

(*a*) The first increase in hardness on time–hardness curves is due to the segregation of copper atoms to form plates on (100) planes.

* This paper was written in the first years of the war; manuscript received February 18, 1946.

† Late Metallurgy Division, National Physical Laboratory, Teddington.

(*b*) The "flat" on time–hardness curves is associated with the formation of copper-rich aggregates of a critical size depending on the temperature of ageing, which, by their precipitation, relieve the strains set up in localized areas in the surrounding matrix. The precipitate is sub-microscopic; simultaneously, sub-microscopic "crystallites" of the new solid solution are formed.

(*c*) The second increase in hardness is due to two processes occurring simultaneously:

(i) the growth of sub-microscopic, copper-rich particles to visible size;

(ii) the formation of fresh aggregates of copper-rich particles in strain-free areas by further diffusion of copper atoms.

(*d*) After the maximum of the second increase in hardness has been reached, and when softening has set in, particles of α-$CuAl_2$ are observed in strain-free areas.

(*e*) The polymorphic transformation of α-$CuAl_2$ to β-$CuAl_2$ [2] takes place in the softened alloy with no increase in hardness.

The above facts help to throw light on the effect of cold work on the age-hardening of high-purity aluminium–4% copper alloy and on the relationship between the two processes.

Experimental.

It has already been shown that the use of high-purity aluminium could introduce anomalous results during the ageing of an aluminium–4% copper alloy, which could be associated with the presence of rounded particles of $CuAl_2$ in the forged 1-in. chill castings, and it has been found that the best method of ensuring complete solid solution of this phase in the aluminium is to cold forge using a light blow on alternate faces.[22] This "standard" method of forging has been used throughout for the breaking-down of the 1-in. chill castings, before the required solution heat-treatments. In Table I are given data regarding the material used.

Cold working is well known to accelerate the age-hardening of alloys capable of being age-hardened and to be accompanied by visible changes in microstructure, such as change in shape of individual crystals and in the appearance of slip-bands, and also by changes in the X-ray spectra.[3–11] So far as the author is aware, no record has been published which gives information regarding any microstructural changes which can be specifically related to the age-hardening of the alloy in its relation to cold working.

The data described in this paper are based on the cold working of a high-purity aluminium–4% copper alloy previously subjected to specific

TABLE I.—*Material Used in Experiments.*

Experimental Series (see below).	Fig. No.	Material.
I	1	Bars cold forged to a thickness of 0·19 in.; solution heat-treated for 4 days at 540° C., quenched as indicated in I (p. 546), and cold rolled, 50% reduction, before ageing at room temperature.
II	2	Bars cold forged to a thickness of 0·3 in., solution heat-treated for 2 hr. at 530° C., quenched in water at room temperature, and immediately cold rolled and aged as indicated in II (p. 547).
III	32, 33	Bars cold forged to a thickness of 0·19 in., solution heat-treated for 4 days at 540° C., quenched in water at room temperature, aged various amounts, and then cold rolled and aged as indicated in III (p. 552).
IV	35	Bars heat-treated for 4 hr. at 450° C., cold forged to a thickness of 0·3 in., solution heat-treated for 2 hr. at 530° C., quenched in water at room temperature, and immediately cold rolled to give the required reduction as indicated in IV (p. 557). The final thicknesses were as follows: 50% reduction, 0·15 in.; 75%, 0·074 in.; 87%, 0·038 in.; 96%, 0·012 in. After initial reduction to a thickness of 0·176 in. and heat-treatment for 1 hr. at 540° C., the specimen reduced 10% was cold rolled to a final thickness of 0·159 in.

ageing heat-treatments. Correlation between the observed changes in hardness and the microstructure has led to further information regarding the effect of cold work on the age-hardening process.

In order to obtain as complete a picture as possible experiments were carried out in five series, in each of which all factors were constant save one, which was varied. The five series are:

I. The effect of rate of cooling from the solution-heat-treatment temperature before cold working, when the degree of cold working and temperature of ageing are fixed.

II. The effect of the temperature of ageing when both the condition of the alloy before cold working and the degree of cold working are fixed.

III. The effect of a definite amount of cold work applied to an alloy previously aged to a fixed degree (as determined by time–hardness curves), on subsequent ageing at two specified temperatures.

IV. The effect of varying the degree of cold work, given immediately after quenching from the solution-heat-treatment temperature, on the subsequent ageing of the alloy at a specified temperature.

V. The effect of the degree of cold work before, and of the time exposed to, the solution-heat-treatment temperature, on subsequent ageing at a fixed temperature.

The properties of the alloy after each of these specific treatments, respectively, have been determined by microscopical analysis and Brinell hardness measurements.

I.—*Alloy Cooled at Different Rates from the Solution-Heat-Treatment Temperature; Cold Rolled,* 50% *Reduction; and Aged at Room Temperature* (15° C.).

The alloy was cooled at one of the following rates from the solution-heat-treatment temperature:

(1) Quenched in water at room temperature.
(2) Quenched in water at 30° C.
(3) Quenched in water at 50° C.
(4) Air-cooled.
(5) Air-cooled in a 2-in. dia. aluminium block, thickness of specimen 0·19 in.
(6) Furnace-cooled.

The Brinell hardness numbers obtained on ageing at room temperature, after cooling the alloy at each of the above rates, are plotted in Fig. 1. In each case a considerable increase in hardness occurs

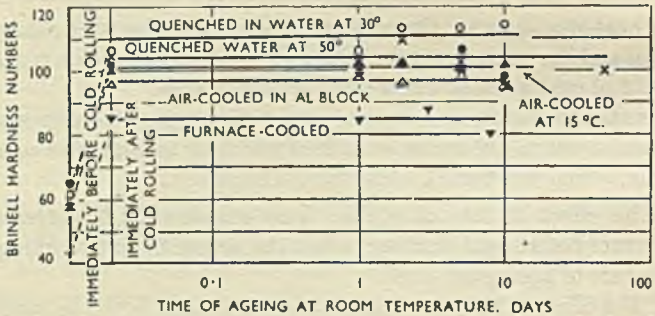


FIG. 1.—Effect of Cold Rolling, 50% Reduction, on the Ageing at Room Temperature (15° C.) of Aluminium–4% Copper Alloy, Cooled at Different Rates from the Solution-Heat-Treatment Temperature.

KEY.

●	Quenched in water at room temperature (15° C.)	Cold rolled, 50% reduction. Aged at room temperature.
○	" " " 30° C.	
×	" " " 50° C.	
▲	Air-cooled	
△	Air-cooled in aluminium block	
▼	Furnace-cooled	

immediately on cold rolling, but no further change in hardness takes place on ageing at room temperature up to a period of at least 10 days.

Furthermore, those specimens cooled at slower rates from the solution-heat-treatment temperature show the same *increase* in hardness, but a lower *final* hardness. The latter is probably due to the fact that sub-microscopic and/or microscopic precipitation of copper-rich aggregates has already taken place during the slow rates of cooling before cold working.[1] In all cases, the hardness is higher than that observed in specimens not cold worked before ageing.[12] The microstructure of the alloy cold worked immediately after quenching in water at 15° C. and aged over 2 months at room temperature is shown in Figs. 4–5 (Plate XLIX) under magnifications of 1000 and 2750 dia., respectively. The specimen has been etched with 25% nitric acid in water at 70° C., which has developed minute "pits" within the grains and in the grain boundaries. This phenomenon indicates that the alloy is in an advanced state of ageing,[12] *i.e.*, that corresponding with a stage approaching the end of the "flat" on time–hardness curves. These "pits" have been shown to be indications of the presence of sub-microscopic, copper-rich aggregates and of "crystallites" of the new solid solution, stable at the temperature of ageing.[1]

II.—*Alloy Quenched in Water at Room Temperature, Immediately Cold Rolled, 40% Reduction, and Subsequently Aged at Different Temperatures.*

The alloy was aged at each of the following temperatures:

(1) Room temperature (15° C.).
(2) 130° C.
(3) 149° C.
(4) 187° C.

The results of the Brinell hardness measurements are plotted in Figs. 2 and 3, and typical microstructures are given in Figs. 6–31 (Plates XLIX–LV). In comparison with material aged normally,[12] it is apparent that cold working has accelerated the rate of ageing.

(a) *Room Temperature Ageing.*

The acceleration of ageing at room temperature (15° C.) is indicated by the presence of etching "pits" in the planes of slip and in the grain boundaries, as seen in the microstructure of the cold-rolled and aged specimens shown in Figs. 6–8, under magnifications of 150 and 1000 and 2500 dia., respectively. Deformation bands crossed with slip-bands, characteristic of cold working, are seen in Fig. 6, while the presence of "pits," developed by the etching reagent in planes of slip

and in the boundaries, is shown in Fig. 7. In Fig. 8, "pits" in a grain boundary are seen under a still higher magnification.

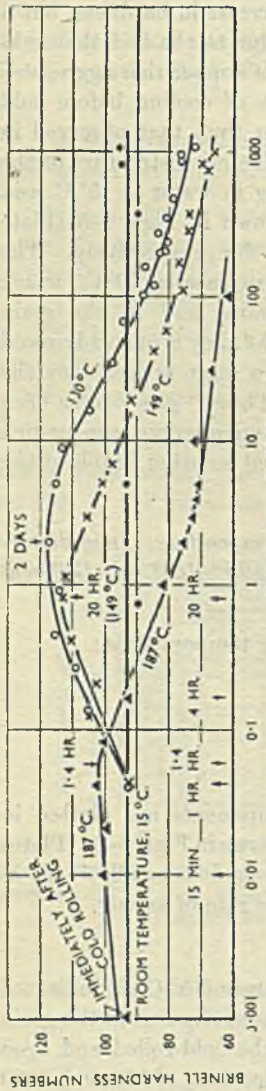


FIG. 2.—Effect of Cold Rolling, 40% Reduction, before Ageing Heat-Treatment.

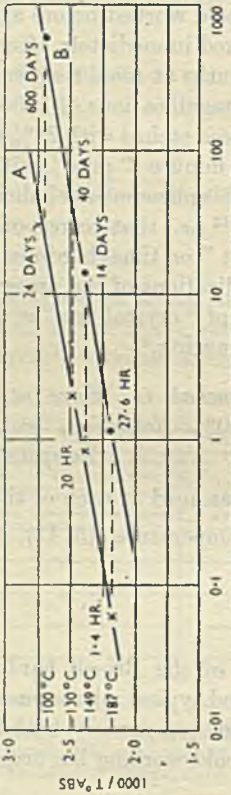


FIG. 3.—Effect of Cold Work on Time taken to Attain Second Maximum Hardness.
A. Cold rolled, 40% reduction, immediately after quenching and before ageing.
B. Normal ageing after quenching at the temperature indicated.

Although no increase in hardness is observed on ageing cold-worked material at room temperature, the development of etching "pits,"

which increase in number with time of ageing, indicates that the first stage of the ageing process is proceeding at room temperature and is initially in a comparatively advanced state.[12]

The presence of these "pits," indicating precipitation of sub-microscopic, copper-rich particles, and of the formation of "crystallites" of a new solid solution, is associated with the relief of strain [1] at localized areas in the lattice, hence, their occurrence in cold-worked material indicates a similar relief of strain by a sub-microscopic precipitate and "crystallites," in regions where maximum stress has been present, *i.e.*, in planes of slip and in the boundaries of the grains.

(b) *The Effect of Higher Ageing Temperatures.*

The age-hardening curves of cold-rolled specimens, aged at higher temperatures, show only one increase to a maximum value on time–hardness curves, followed by a decrease (Fig. 2). This is contrary to what occurs during normal ageing at high temperatures, when two increases are observed up to a certain temperature of ageing.[12] The increases in hardness shown on the curves in Fig. 2 correspond with the second increase on normal time–hardness curves. Cold working alone, as already mentioned, brings the alloy into the first stage of ageing.

Examination of the microstructures of the cold-rolled and aged specimens supplies the evidence on which the above deduction has been made.

Specimens have, therefore, been examined after the following ageing times, *i.e.*, before and after the maxima on the curves in Fig. 2 have been reached:

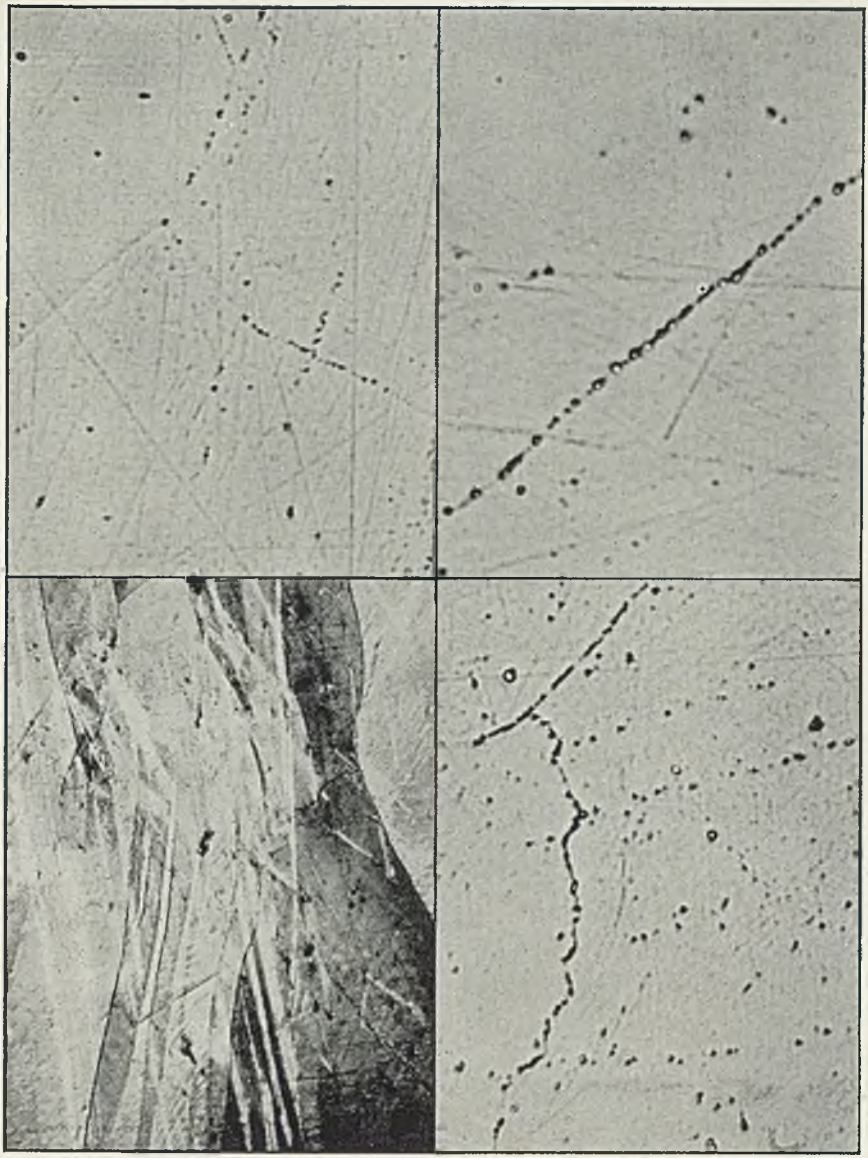
(i) Ageing temperature, 130° C.: 1, 10, 121 days.
(ii) Ageing temperature, 187° C.: ¼, 1, 4 hr., 1, 10, 30 days.

Figs. 9, 12, and 13 give the microstructures of the specimens aged at 130° C. for 1, 10, and 121 days, respectively. These have been etched with 25% nitric acid at 70° C. In the case of the specimen aged 1 day at 130° C. (Fig. 9), "pits" appear in the grain boundaries and along the planes of slip, as shown under a magnification of 2750 dia. in Figs. 10 and 11; "pits" also appear similarly in the alloy aged for 10 days (Fig. 12). A longer period of ageing, however, *i.e.*, 121 days, causes a further change in structure to take place and be developed by the hot nitric acid etch, as seen in Fig. 13, *viz.*, the appearance of a white material associated with a precipitate in the grain boundaries and also within the grains, particularly along the planes of slip, as shown under a magnification of 2750 dia. in Figs. 14–15. This light phase must be

the new solid solution, stable at the temperature of ageing, which only appears when the second stage of ageing has begun.[1] The presence of this phase is more easily detected in its early stages by etching with ½% hydrofluoric acid only, when minute "crystallites" appear; their presence is, however, masked when the alloy has previously been etched with hot nitric acid. The series heat-treated at 187° C. has been more completely examined, since the changes taking place on ageing occur more rapidly. Figs. 16, 19, 21, 22, 23, and 25 show the changes in microstructure, under a magnification of 300 dia., of the alloy aged ¼, 1, and 4 hr., and 1, 10, and 30 days at 187° C., etched with 25% nitric acid at 70° C. Comparing these structures with reference to the ageing curve (Fig. 2), it can be seen that the presence of "pits" alone is observed (with the specific reagent used) in the specimens aged ¼ and 1 hr. (Figs. 16, 19), *viz.*, before the maximum on the time–hardness curve is reached. On ageing 4 hr. or more (Figs. 21, 22, 23, and 25), *viz.*, after the maximum increase has been passed, the character of the structure changes, however, and, instead of a predominance of "pits" in the boundaries and slip planes, the white material appears in increasing amount as ageing proceeds.

After 10 days' ageing, a visible precipitate can be detected in the specimen under a higher magnification of 2750 dia. (Fig. 24) and this precipitate grows in amount as time of ageing increases. Figs. 25–26 show the precipitate in the white substance in the alloy aged 30 days at 187° C., under magnifications of 300 and 2500 dia., respectively.

Further evidence that the maxima on the curves shown in Fig. 2 may be attributed to the second stage of ageing is obtained by etching specimens for 2–3 sec. in ½% hydrofluoric acid. The alloy aged ¼ hr. at 187° C., etched with 25% nitric acid at 70° C., is seen under a magnification of 2000 dia. in Fig. 17; in Fig. 18 the same specimen is shown etched with ½% hydrofluoric acid. In the former case, "pits" have been developed by the reagent on the slip planes and on the boundaries, while in the latter, minute "crystallites" of the new solid solution have appeared in the grain boundaries and in the slip planes. These two facts together prove that microscopic copper-rich particles and microscopic "crystallites" of the new solid solution, stable at the temperature of ageing, have been formed, and since these can only be associated with the second stage of ageing,[1] the first stage must have been accelerated. Similarly, the alloy aged for 1 hr. at 187° C., when etched with ½% hydrofluoric acid, shows, under a magnification of 2000 dia., the presence of "crystallites" in the slip planes (Fig. 20). These "crystallites" are the precursors of the white substance developed to a greater extent by prolonged ageing (Figs. 21–26).



High-Purity Aluminium Alloy Containing 4% of Copper.

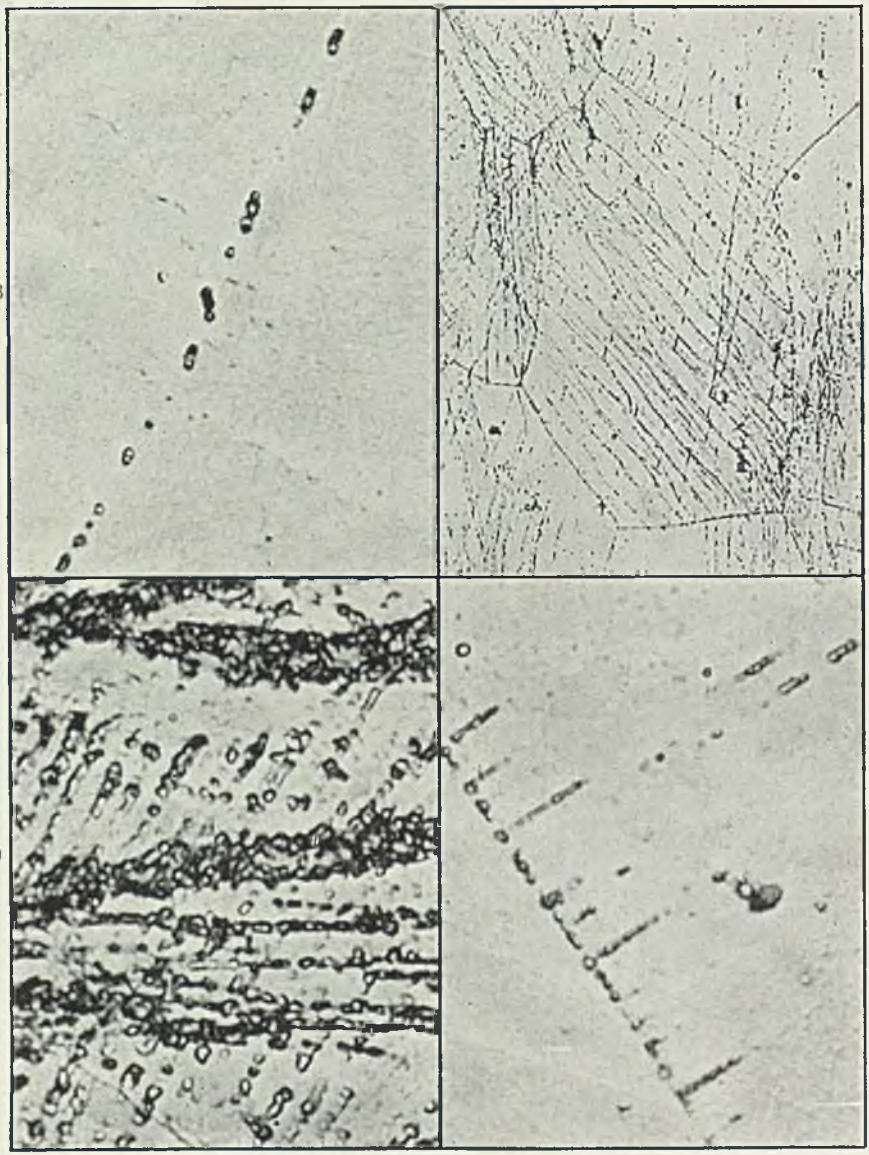
FIG. 4.—Cold rolled, 50% reduction, immediately after quenching in water at 15° C. Aged 2 months at 15° C. × 1000. Etch : 1½ min. 25% HNO_3 at 70° C.

FIG. 5.—As Fig. 4. × 2750.

FIG. 6.—Cold rolled, 40% reduction, immediately after quenching in water at 15° C. Aged 4 months at 15° C. × 150. Etch : 1 min. 25% HNO_3 at 70° C. and then 3 min. Keller's reagent.

FIG. 7.—Cold rolled and quenched as Fig. 6. Aged 2 years, 2 months at 15° C. × 1000. Etch : 1½ min. 25% HNO_3 at 70° C.

[*To face p.* 550.

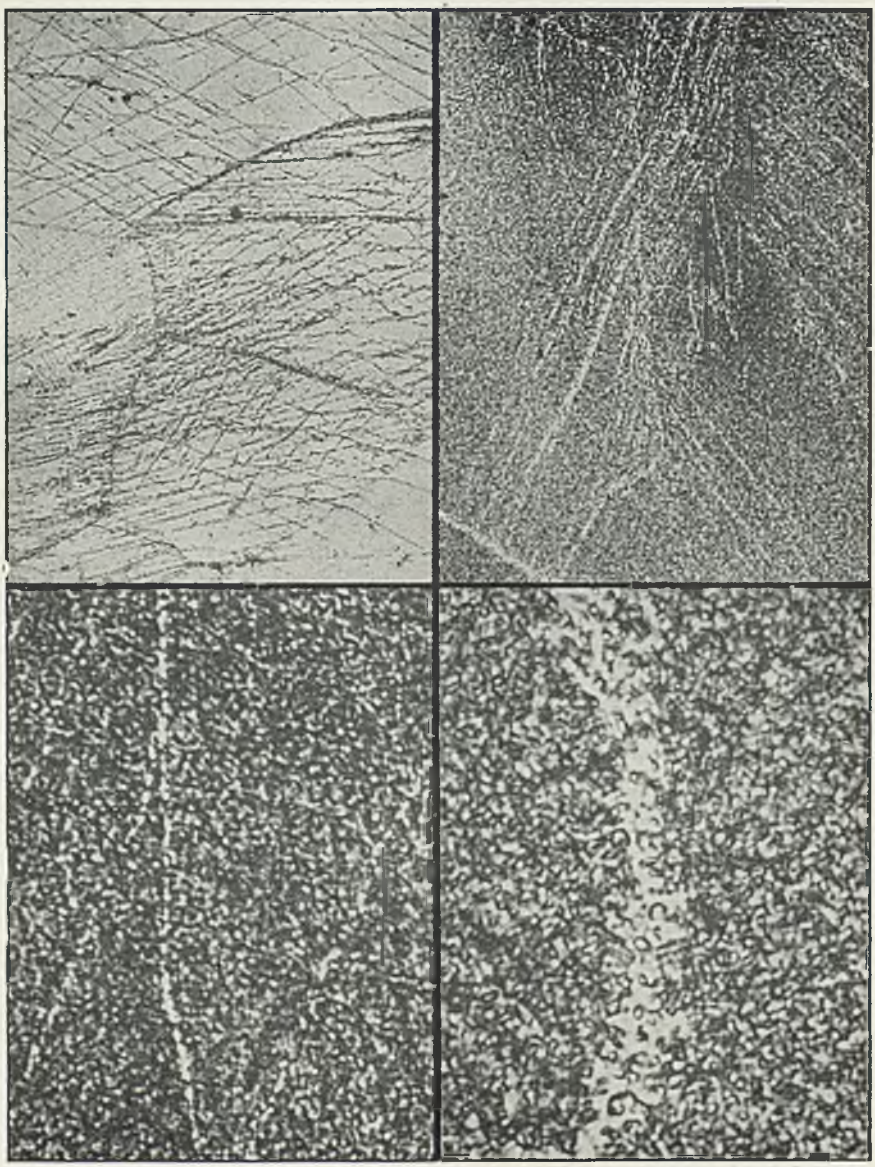


High-Purity Aluminium Alloy Containing 4% of Copper.

FIG. 8.—Cold rolled, 40% reduction, immediately after quenching in water at 15° C. Aged 2 years, 2 months at 15° C. × 2500. Etch: 1½ min. 25% HNO_3 at 70° C.

FIG. 9.—Cold rolled, 40% reduction. Aged 1 day at 130° C. × 300. Etch: 1 min. 25% HNO_3 at 70° C.

FIGS. 10 and 11.—As Fig. 9. × 2750.



High-Purity Aluminium Alloy Containing 4% of Copper.

FIG. 12.—Cold rolled, 40% reduction. Aged 10 days at 130° C. × 300. Etch: 45 sec. 25% HNO_3 at 70° C.

FIG. 13.—Cold rolled, 40% reduction. Aged 121 days at 130° C. × 300. Etch: 45 sec. 25% HNO_3 at 70° C.

FIGS. 14 and 15.—As Fig. 13. × 2750.

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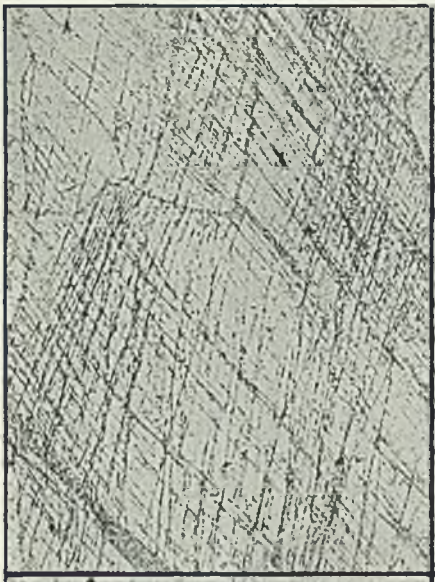

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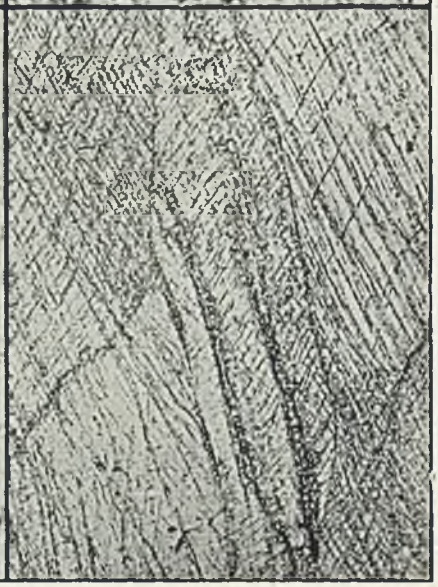


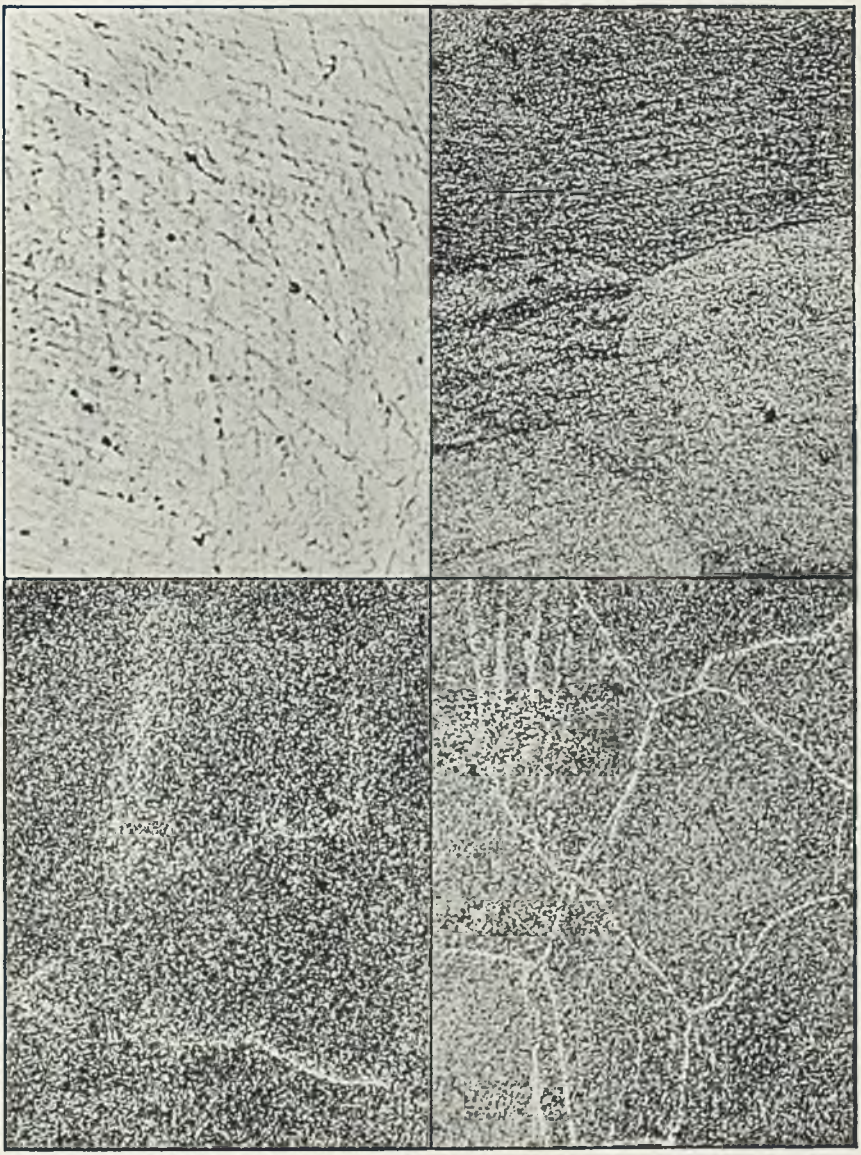
High-Purity Aluminium Alloy Containing 4% of Copper.

FIG. 16.—Cold rolled, 40% reduction. Aged ¼ hr. at 187° C. × 300. Etch: 1 min. 25% HNO_3 at 70° C.

FIG. 17.—As Fig. 16. × 2000.

FIG. 18.—Same specimen as Fig. 16. × 2000. Etch: 4 sec. ½% HF.

FIG. 19.—Cold rolled, 40% reduction. Aged 1 hr. at 187° C. × 300. Etch: 1 min. 25% HNO_3 at 70° C.



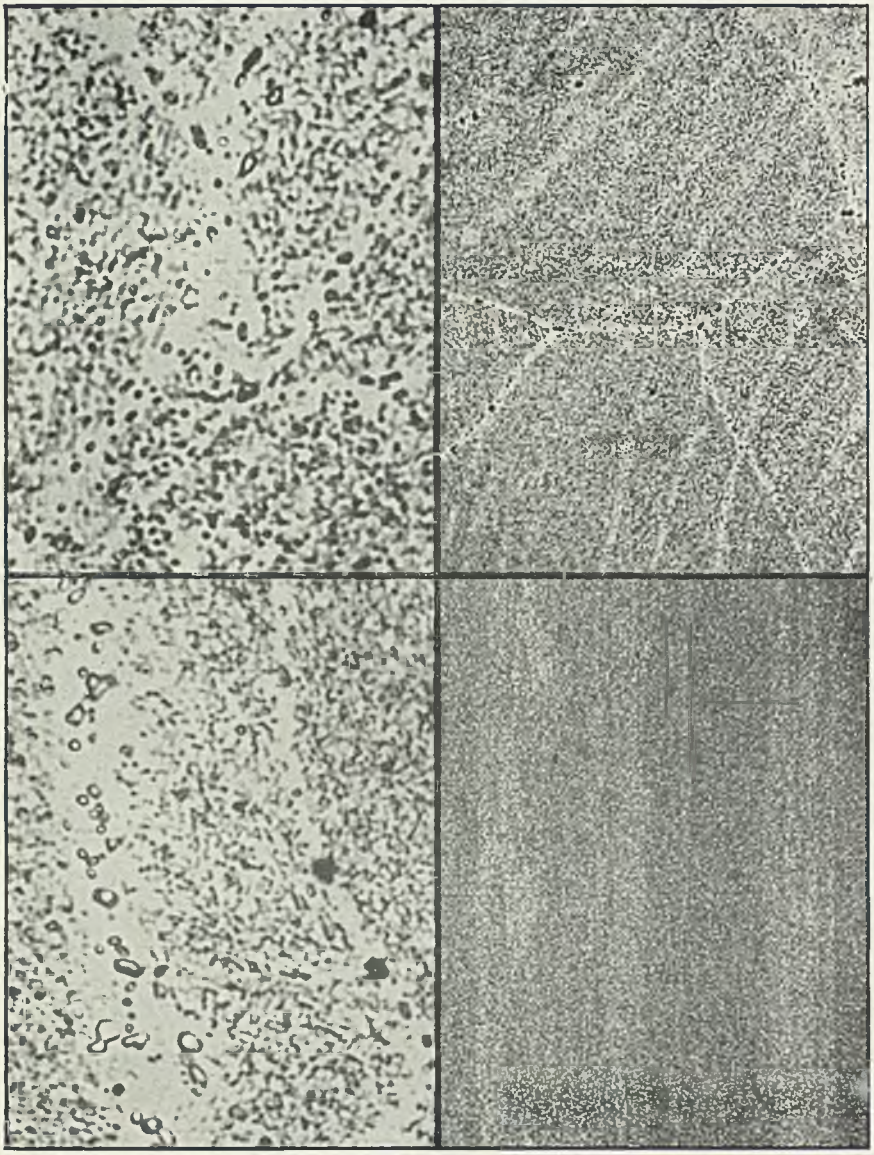
High-Purity Aluminium Alloy Containing 4% of Copper.

FIG. 20.—Cold rolled, 40% reduction. Aged 1 hr. at 187° C. × 2000. Etch: Swabbed ½% HF.

FIG. 21.—Cold rolled, 40% reduction. Aged 4 hr. at 187° C. × 300. Etch: 1 min. 25% HNO_3 at 70° C.

FIG. 22.—Cold rolled, 40% reduction. Aged 1 day at 187° C. × 300. Etch: ½ min. 25% HNO_3 at 70° C.

FIG. 23.—Cold rolled, 40% reduction. Aged 10 days at 187° C. × 300. Etch: ½ min. 25% HNO_3 at 70° C.



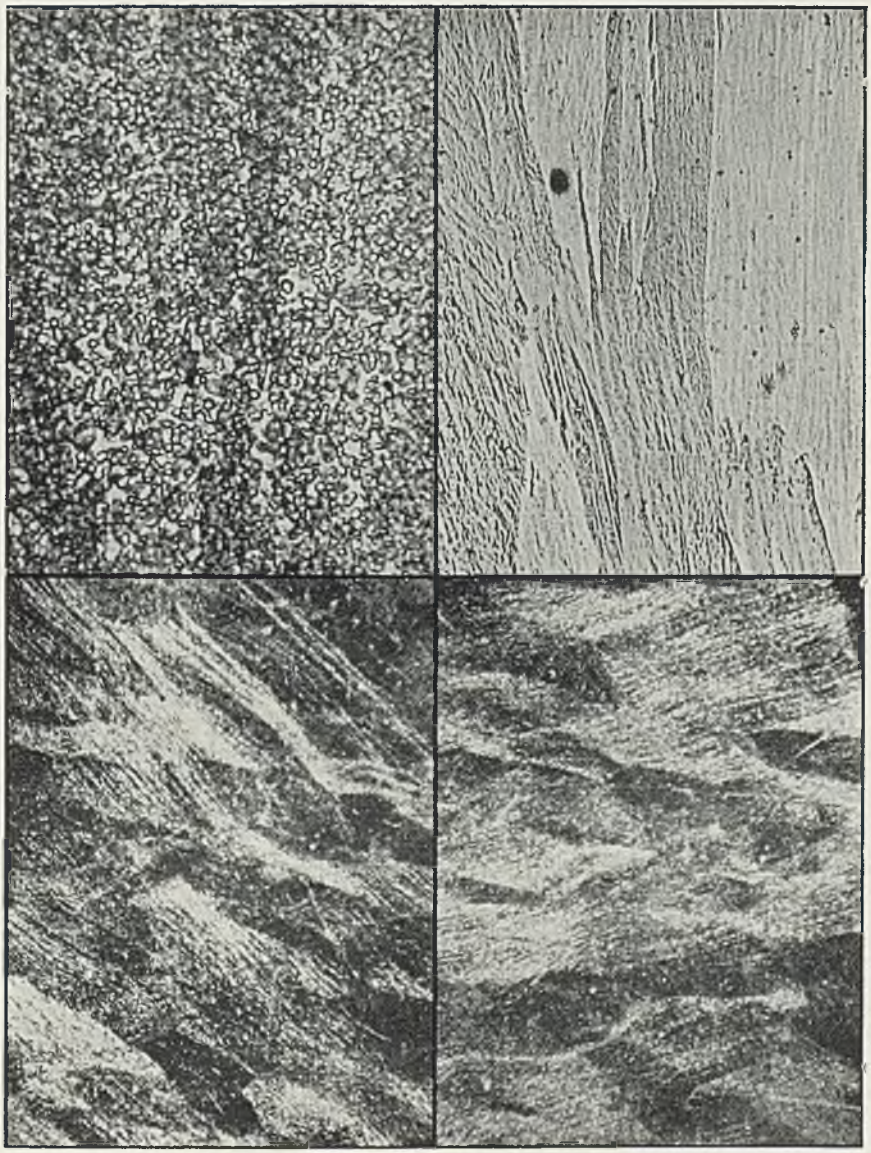
High-Purity Aluminium Alloy Containing 4% of Copper.

FIG. 24.—Cold rolled, 40% reduction. Aged 10 days at 187° C. × 2750. Etch: ½ min. 25% HNO_3 at 70° C.

FIG. 25.—Cold rolled, 40% reduction. Aged 30 days at 187° C. × 300. Etch: ½ min. 25% HNO_3 at 70° C.

FIG. 26.—As Fig. 25. × 2500.

FIG. 27.—Cold rolled, 75% reduction. Aged ¼ hr. at 290° C. × 200. Etch: 1 min. 25% HNO_3 at 70° C.



High-Purity Aluminium Alloy Containing 4% of Copper.

FIG. 28.—Cold rolled, 75% reduction. Aged ¼ hr. at 290° C. × 2000. Etch: 1 min. 25% HNO_3 at 70° C.

FIG. 29.—Cold rolled, 75% reduction. Aged ¼ hr. at 290° C. × 150. Etch: ½% HF.

FIG. 30.—Cold rolled, 40% reduction. Aged 1 hr. at 187° C. (Same specimen as Fig. 20.) Oblique lighting. × 60. Etch: 2–3 sec. ½% HF.

FIG. 31.—Same place as Fig. 30, but angle of light altered. × 60.

(c) *Ageing after 75% Reduction.*

An alloy more heavily cold rolled, *i.e.*, reduced 75%, was aged for 15 min. at 290° C. and etched in 25% nitric acid at 70° C.; the microstructure is given in Fig. 27 under a magnification of 200 dia. Here the presence of the light phase is clearly to be seen in bands associated with the direction of rolling. Under a magnification of 2000 dia., particles of precipitate appear which are, most probably, β-$CuAl_2$ [13] (Fig. 28). If, however, the specimen is etched with ½% hydrofluoric acid, "crystallites" are seen (Fig. 29) in deformed grains along the planes of slip.

That the structures shown in Figs. 18, 20, and 29 are genuinely due to the formation of "crystallites" of the new solid solution, stable at the temperature of ageing, and not to a "flowed" surface, is shown on examining a specimen under oblique lighting. The structure of the specimen in Fig. 20 was viewed under dark-background, oblique illumination; Figs. 30–31 show the same place in the specimen, under a magnification of 60 dia., under two angles of lighting. Two important facts stand out: first, the "crystallites" observed on slip planes (Fig. 20) reflect light while the matrix remains dark and, second, the reflection of light by the "crystallites" depends on the orientation of the slip planes, cf. Fig. 30 with Fig. 31. It may be concluded, therefore, that, (i) the small "crystallites" seen in Figs. 18, 20, and 29 are real, (ii) they are similarly oriented (or very nearly) with respect to each other along slip planes, (iii) their orientation depends upon the orientation of the slip plane, and (iv) their orientation differs from that of the matrix.

This microscopical examination demonstrates the reason why no grain boundaries can be etched up in aged Duralumin: the formation of "crystallites" replaces the original grains.

The above data show that the maxima on the ageing curves seen in Fig. 2 are associated with the second stage of ageing, the first stage having been brought about by the cold work immediately after quenching from the solution-heat-treatment temperature.

In Fig. 3 is plotted the log of the time taken to attain the maximum of the second increase in hardness accompanying ageing against the reciprocal of the absolute temperature of ageing of alloys cold rolled, or not cold rolled, immediately after quenching and before ageing. It may be seen that the points obtained lie respectively on two lines which are parallel, showing that the acceleration of the second stage of ageing is related to the degree of cold work and not to the temperature of ageing.

III.—*Alloys Aged Specified Amounts Before Cold Rolling*, 50% *Reduction, and Subsequently Aged at* (a) *Room Temperature* (15° C.), (b) 130° C.

The question arises, what is the effect of cold work on the subsequent ageing of a specimen which has previously been aged to various stages, as shown by time–hardness curves? Since cold work accelerates ageing, it would be expected that any increase in hardness caused by cold rolling a specimen previously in a state corresponding with a completely softened alloy would be due to the imposed strains on the lattice and not to any ageing process. On the other hand, specimens aged to stages intermediate between the as-quenched and the fully softened conditions would probably show increases in hardness, due to accelerated ageing of those parts of the alloy in which the ageing process had not been completed. Fig. 32 shows the results obtained when

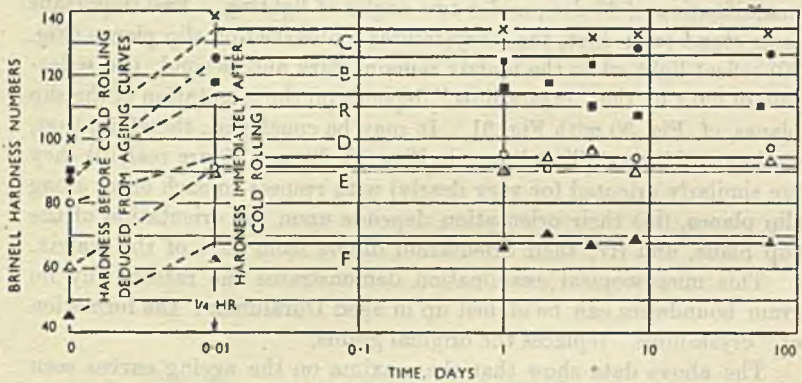


Fig. 32.—Effect of Ageing before Cold Working, 50% Reduction, on the Subsequent Ageing at Room Temperature (15° C.).

previously heat-treated alloys are cold rolled, 50% reduction, and aged at room temperature; while those from specimens similarly treated, but aged at 130° C., are plotted in Fig. 33. The conditions of the alloys before cold rolling were as follows:

B.—Quenched in water at 15° C., aged 6 hr., 150° C.
C. ,, ,, ,, 3 days, 150° C.
D. ,, ,, ,, ½ hr., 250° C.
E. ,, ,, ,, ½ hr., 350° C.
F. ,, ,, ,, 4 hr., 350° C.
R. ,, ,, ,, 8 days, 15° C.

From time–hardness curves,[12] the degree to which these alloys should have been age-hardened is deduced as follows :

B.—Aged just to the maximum of the first increase in hardness.
C.—Aged to rise to the second increase in hardness.
D.—Aged to the maximum of the second increase in hardness.
E.—Aged well past the maximum of the second increase in hardness.
F.—Fully softened.
R.—Aged just to the maximum of the first increase in hardness.

Cold rolling causes, in all cases, an immediate increase in hardness, which does not change during prolonged ageing at room temperature.

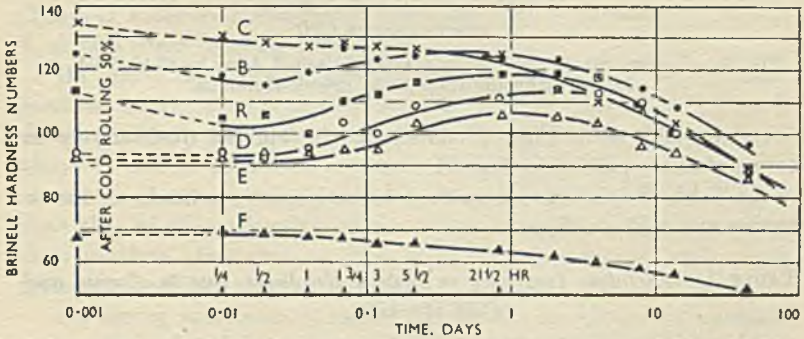


Fig. 33.—Effect of Ageing Before Cold Working, 50% Reduction, on the Subsequent Ageing at 130° C.

The effect of cold work on the fully softened specimen F is important, since any change in hardness can be attributed to strain-hardening of the lattice alone, and not to any ageing process. In order to determine the maximum increase in hardness which could be imposed on such a fully softened alloy by cold work, a specimen was soft-annealed for 1 hr. at 370° C., air-cooled, and then cold rolled to give thicknesses corresponding with 10, 40, 60, and 80% reduction, respectively. The increase in Brinell hardness obtained is plotted in Fig. 34 against the percentage reduction. It was not possible, using the same specimen, to obtain by further rolling material of suitable thickness for accurate Brinell measurements with the same $\frac{P}{D^2}$ ratio; the higher portion of the curve in Fig. 34 has not, therefore, been determined experimentally. By extrapolation, it appears that the maximum lattice strain caused by cold work is of the order 40 Brinell; after this, further cold working will

result either in a breakdown (locally) of the lattice, or else in relief of strain by some "recrystallization" process.

Since cold working and age-hardening both produce lattice strain,

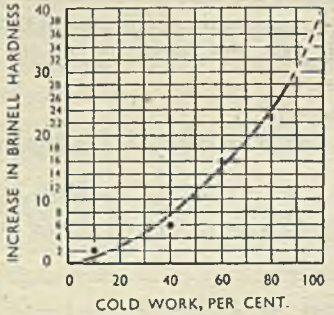


FIG. 34.—Lattice Strain Induced in a Fully Softened Alloy by Cold Work, as Measured by the Increase in Brinell Hardness.

the data plotted in Figs. 32–33 can be interpreted qualitatively in terms of those plotted in Fig. 34.

In Table II are summed up the relative increases in hardness due to ageing and cold working.

TABLE II.—*Relative Increases in Brinell Hardness, due to Ageing and Cold Working.*

1	2	3	4	5	6	7	8	9	10
Specimen.	As Quenched, B.H.	Ageing Heat-treatment.	B.H. after Ageing.	Increase in B.H.	Cold Rolled, 50% Reduction, B.H.	Total Increase in B.H.	Cold Rolled, 90% Reduction, B.H.	Total Increase in B.H.	Difference in B.H. between 50 and 90% Reduction by Cold Work.
C	60	3 days, 150° C.	100	40	134	74	103	43	−31
B	,,	6 hr., 150° C.	87	27	126	66	92	32	−34
R	,,	8 days, 15° C.	90	30	114	54	108	48	− 6
D	,,	½ hr., 250° C.	80	20	94	34	102	42	+ 8
E	,,	½ hr., 350° C.	60	0	92	32	107	47	+15
F	,,	¼ hr., 350° C.	45	−15	68	23	78	33	+10

Examination of Table II and Fig. 32 shows that the greatest increase in hardness caused by cold rolling, 50% reduction, occurs in the alloy C,

which had been aged previously to a stage about half-way between the "flat" on the time-hardness curve and the maximum hardness value associated with the second ageing process.[12] Alloys B and R, both aged previously to the stage corresponding with the beginning of the "flat" on time–hardness curves, show the next greatest increase due to 50% reduction by cold work, while alloys D, E, and F, whose conditions before cold working corresponded with further progressive stages of ageing, are increasingly less affected by the same amount of cold work.

If the amount of cold work given is increased to 90% reduction, two most interesting facts appear, *viz.*, that (*a*) in the case of the first three alloys mentioned above, C, B, and R, no further hardening occurs at room temperature, indeed, a definite reduction of hardness takes place (Table II, columns 6, 8, and 10); and (*b*) the last three alloys, D, E, and F, show a slight increase in hardness.

On ageing the alloys cold rolled, 50% reduction, at 130° C., time–hardness curves are obtained (Fig. 33). Briefly, alloy C and alloy F show a gradually increasing softening and no increase in hardness, while alloys B and R both show a small initial softening followed by an increase in hardness to a maximum value which is only slightly higher than that of the cold-rolled alloy. These increases in hardness reach their maxima after about 1–2 days' ageing, respectively, after which softening gradually sets in.*

On ageing specimens D and E at 130° C., no change in hardness occurs until after ½ hr., when an increase takes place. The hardness rises to a maximum value which is greater than the hardness of the material in its original, cold-rolled state.

The following explanations of the changes in hardness, given in Table II and seen in Figs. 32–33, are put forward:

(1) *Specimen C.*—Ageing this specimen for 3 days at 150° C. produces an increase in hardness of 40 Brinell (measured at room temperature). In terms of lattice strain (Fig. 34), this increase corresponds with that percentage caused by the maximum reduction by cold work; hence, an additional strain imposed on the lattice by still further cold work must cause either localized "cracking" or a localized "recrystallization" in relief of this strain.

It has been shown in Part II (p. 547) that relief of strain set up by cold working is brought about by the formation of sub-microscopic,

* There is evidence in the literature that, after a definite amount of cold work has been applied to certain materials, further cold work causes a decrease, rather than an increase, in hardness. It is suggested that this may be due to the fact that the maximum lattice strain has been exceeded and relief of strain is brought about by a localized "recrystallization" process in the matrix.

copper-rich particles and "crystallites" of the solid solution, stable at the temperature of ageing; hence, if an *aged* alloy be cold worked, it is likely that the stresses set up will again be relieved by a similar process. The condition of the present alloy before cold rolling corresponds with a state intermediate between that on the "flat" on time–hardness curves and the maximum of the second increase in hardness. It is most probable, therefore, that the ageing process will be so greatly accelerated that the second stage of ageing will at least be attained, together with the accompanying increase in hardness to a maximum value. If this be so, the formation of a visible precipitate of copper-rich particles, together with visible "crystallites" of the new, stable aluminium solid solution, will take place during cold rolling, thereby relieving stresses in those areas where the strain is greatest.

The Brinell hardness will also be affected by the number, size, and growth of precipitated, copper-rich particles which have been formed.

That this deduction is correct is shown by the facts that (*a*) further cold working (90% reduction) produces no increase, but a decrease, in hardness; (*b*) ageing at 130° C. the alloy previously cold worked, 50% reduction, causes no increase in hardness but a gradual softening (Fig. 33).

(2) *Specimens B and R.*—These have both been aged to the stage when the "flat" on the time–hardness curve is reached, though possibly to slightly different degrees, the former by ageing for 6 hr. at 150° C., and the latter by ageing for 8 days at room temperature. From Table II, column 5, it is seen, by comparison with specimen C, that the increase in hardness due to ageing is not so great, and that 50% reduction by cold work also increases the hardness of both to a lesser extent. The total growth in hardness of both specimens B and R (Table II, column 7) corresponds with about the increase to maximum lattice strain; hence, any additional cold work would be expected to produce no further increase in hardness, but to promote still further the ageing process which relieves strain, *i.e.*, increase the rate of precipitation of the copper-rich particles and the formation of "crystallites" of the new solid solution. That this probably occurs is seen from Table II, columns 8–10, and from the results of ageing the 50%-reduction cold-worked specimens at 130° C. (Fig. 33).

In contrast with specimen C, specimens B and R show a small decrease in hardness as the first effect of ageing at 130° C.; this decrease must be associated with the relief of strain by the formation of visible copper-rich particles and visible "crystallites" of the new solid solution. This is followed, in both cases, by an increase in hardness to maximum values in about ½ day and 1½ days, respectively, after which softening sets in

progressively. This increase in hardness can only be attributed to the second stage of ageing.

(3) *Specimens D and E.*—These specimens have been aged to a stage in which the second maximum increase in hardness should have been first attained, or passed, respectively. Consequently, the effect of cold working would be expected to be considerably less than that observed in specimens C, B, and R, since the amount of hardening attributable to ageing has been considerably reduced by the ageing treatments. From Table II, column 7, it is seen that the increase in hardness due to 50% reduction by cold work is about half that attained in the specimens B and R. Hence, if the specimens D and E be cold worked, 90% reduction, the lattice could be further strained to the maximum value, giving a total increase in Brinell hardness of 50 or more. From Table II, column 9, it is seen that the hardnesses of both specimens D and E have been raised to approximately the maximum value and from column 10 it is seen that this increase is greater in the case of the alloy which was originally in the less hard condition.

It is difficult to offer any explanation for the increase in hardness which occurs on heat-treating the cold-worked specimens D and E at 130° C. (Fig. 33). It is possible that re-solution and re-precipitation of copper-rich particles is responsible for the change.

(4) *Specimen F.*—From Table II, column 7, it is seen that the total increase in hardness is 23 Brinell, which is due entirely to lattice strain, cf. Fig. 34. Accordingly, by further cold working, it should be possible to increase the hardness to the total maximum increase of about 40–50 Brinell. The effect of 90% reduction by cold rolling is seen in columns 9 and 10; the total increase in hardness shows that the lattice has not yet been strained to the uttermost, cf. Fig. 34, and that, unlike the previous specimens, there are no "residual" ageing effects to cause any additional increase or decrease in hardness. The softening on heat-treating at 130° C. specimen F when cold rolled, 50% reduction, is probably due solely to relief of lattice strain caused by the cold rolling, brought about by a crystallization process.

IV.—*The Effect of Varying the Degree of Cold Work Applied Immediately after Quenching on the subsequent Ageing at* 100° C.

The alloy was given 10, 50, 75, 87, and 96% reduction by cold rolling (Table I) and specimens were cut from the bar after each of the respective stages of cold work and aged at 100° C. This ageing temperature was chosen so that the initial effects could be studied. The changes in hardness observed are plotted in Fig. 35 and summarized in Table III.

From previous experiments (Part I, p. 546), it is known that 50% reduction by cold work on a quenched alloy accelerates the rate of ageing to such an extent that the formation of sub-microscopic, copper-

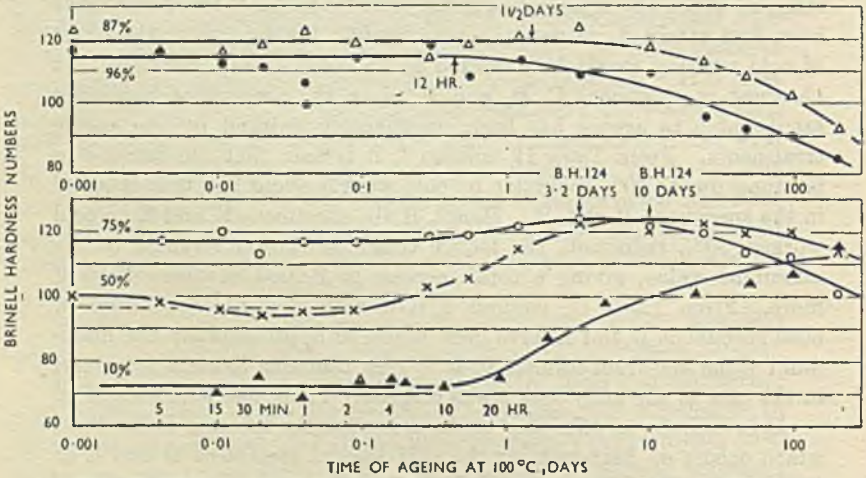


FIG. 35.—Effect of Cold Working, Immediately after Quenching in Water at Room Temperature, and then Ageing at 100° C.

(Brinell hardness numbers plotted are the means of 3 impressions across the specimens.)

TABLE III.—*Changes in Brinell Hardness with Varying Degrees of Cold Work Given Immediately after Quenching.*

1	2	3	4	5	6
B.H., Before Cold Work.	Reduction, %.	B.H., After Cold Work.	Increase in B.H.	Maximum B.H. on Ageing at 100° C.	Maximum Increase due to Ageing.
(*a*) 60	10	Not measured. (Probably about 70.)	*Ca.* 10	*Ca.* 115	*Ca.* 45.
(*b*) 60	50	100	40	124	24
(*c*) 60	75	120	60	124	4
(*d*) 60	87	122	62	Softening set in.	Nil.
(*e*) 60	96	114	54	Softening set in.	Nil.

rich particles and sub-microscopic "crystallites" of the new solid solution takes place, which indicates that the stage corresponding with the "flat" on ageing curves has been reached. Hence, the increases in hardness caused by ageing at 100° C. specimens reduced by 50%

and over (Fig. 35 and Table III) must be attributed to the second ageing process.

The effect of the first 10 hr. of short times of ageing is of great interest. The specimen reduced 10% by cold work shows no change in hardness, within experimental error, over a period of 10–20 hr., after which an increase in hardness sets in which appears to be reaching a maximum value of 115 Brinell after 203 days. The alloy cold worked, 50% reduction, possibly decreases in hardness very slightly, but this is not certain since the change is within experimental error, as shown by the dotted line in Fig. 35. A maximum value of 124 Brinell is, however, attained after about 10 days at 100° C., this being replaced by a gradual softening on prolonged ageing. No change in hardness, followed by a very small increase to approximately 124 Brinell in about 3 days, accompanies the ageing at 100° C. of the alloy cold worked, 75% reduction.

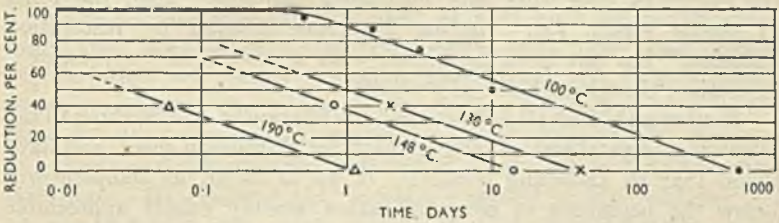


Fig. 36.—Relationship between the Percentage Reduction by Cold Work Immediately after Quenching, and the Time Taken to Attain Second Maximum Hardness (or Beginning of Softening), on Ageing at Various Temperatures.

As in the previous case, this increase is followed later by a decrease in hardness.

The specimens cold rolled, 87 and 96% reduction, show no change in hardness over a period of ageing of 1½ days and ½ day, respectively, after which progressive softening sets in.

The explanation of these ageing curves is similar to that already set out in Part III (p. 552), the degree of cold work given causing the lattice of the matrix to be strained either up to, or beyond, its fullest extent (Table III and Fig. 34) and relief of strain setting in on heat-treatment.

The relation between the log-time to attain the second hardness on ageing at 100° C. and the degree of cold work is shown to be a straight line (Fig. 36) up to between 75 and 87% reduction by cold work, after which the curve probably approaches the time axis asymptotically. From Fig. 36 it can be seen that the rate of ageing at any temperature is determined by the amount of cold work given.

V.—*The Effect of the Degree of Cold Work before the Solution Heat-Treatment and the Time Exposed at the Heat-Treatment Temperature on Subsequent Ageing at Room and Higher Temperatures.*

Preliminary experiments indicated that, in the case of the high-purity alloy, the degree of cold work before the solution heat-treatment and the time of exposure at the heat-treatment temperature had little or no effect on the subsequent ageing at room or higher temperatures. Further experiments to check this were not carried out owing to the intervention of the war.

After this paper was written, Harrington and Jester [14] published data on aged, cold-rolled brasses, the general conclusions of which support the results of the present research. While these investigators associate the observed changes in physical properties with a precipitation process, they have not correlated ageing temperature with, for example, the degree of cold work and maximum hardness. Also, the relief of elastic strain, *i.e.*, "lattice distortion relieved by restoration diffusion," has not been associated with a process of "crystallite" formation, though with one of precipitation.

Hansen and Moritz[15] have studied the relation of cold working to the recovery, recrystallization, and softening of Duralumin sheet, and have found that an annealing treatment of up to 25 hr. at temperatures below the beginning of recrystallization already effects appreciable softening, but that complete softening occurs only at temperatures above the beginning of recrystallization. The softening is said to be due partly to recovery and partly to recrystallization. The results of the present investigation show, however, that softening occurs progressively at temperatures of heat-treatment below that of recrystallization.

To sum up:

(1) Cold work accelerates the rate of ageing at room and at higher temperatures by an amount determined by the degree of cold work given.

(2) Microstructural changes have shown that the effect of cold work on a quenched specimen is to bring it into the condition of the fully hardened alloy normally aged at room temperature. Subsequent ageing at room temperature discloses that the condition of the alloy must correspond with that some way along the "flat" of the room-temperature time–hardness curve of the alloy; ageing at higher temperatures shows that the second stage of the ageing process sets in immediately.

(3) The lattice strain caused by cold work has been shown to be relieved by the formation (depending on the temperature of ageing

and condition of the alloy before cold working) of a sub-microscopic, or microscopic, precipitate of copper-rich particles and of "crystallites" of the aluminium solid solution, stable at the temperature of ageing. Both of these are formed simultaneously during the ageing process. This relief of strain occurs, first, in those regions which have been most heavily stressed by the cold work, *i.e.*, in planes of slip and in the grain boundaries, and is dependent on the degree of cold work and on the temperature of heat-treatment.

(4) The course of the ageing curves of cold-worked and aged material has been considered in relation to the amount of lattice strain the material is capable of withstanding. The inference has been drawn that measurement of lattice strain produced by cold rolling a soft-annealed specimen may be associated with Brinell hardness.

Discussion.

Adcock,[16] investigating the effect of cold work on cupro-nickel, found that, after annealing, recrystallization took place along lines of slip and that new grains were always associated with (i) etch bands, which represented the traces on the surface of the specimen of the sloping "slip" planes; (ii) striations running parallel to the direction of rolling; or (iii) boundaries of deformed crystal grains and mechanical inclusions. He observed that samples annealed at temperatures just too low to bring about visible recrystallization showed both the usual etch bands and horizontal striations, the latter becoming very pronounced and, after deep etching, resembling ordinary crystal grain boundaries. Adcock also made the following important observation: "On the other hand, the deep etching of areas of the partially annealed specimen where the 'slip' planes came to the surface merely resulted in the formation of chains of rather irregular etch-pits." Unfortunately, he did not study the changes at constant temperatures for various time intervals.

Crampton, Burghoff, and Stacy [17] found, during the cold drawing of copper alloys, "... in the age-hardened state, the decrease of conductivity was roughly proportional to the degree of draw. ... The magnitude of the effect was also dependent upon the degree of precipitation, for the change became less as ageing time increased and, also, as ageing temperature increased."

Andrade,[18] examining plastic deformation of single crystals of sodium by means of X-ray analysis, found that a severely strained crystal is no longer a single crystal, and that, in the case of sodium, recrystallization has occurred. Bragg [19] has suggested that if a metal is thrown into an amorphous state by distortion, it will rapidly self-anneal up to a certain coarseness of crystallite size, but beyond that

point the process will become so slow that the metal remains in an approximately constant condition, though not in true equilibrium. Wood [20] has shown by X-ray analysis that the breakdown of the lattice of certain metals on cold working increases with the amount of cold work and leads to the formation of "crystallites" whose size is characteristic of each specific metal, and whose first appearance depends upon the degree of cold work given.

The present author has previously suggested that, during the age-hardening of a high-purity aluminium–4% copper alloy [1] at room temperature, the lattice becomes locally strained by the segregation of copper-rich particles and that, ultimately, when the lattice has been strained a critical amount, relief of this strain takes place by the precipitation of sub-microscopic, copper-rich particles and by the simultaneous formation of sub-microscopic "crystallites" of a new aluminium solid solution.

The present data have shown that when such an alloy is cold worked, either as-quenched or as previously age-hardened, and then aged at room or higher temperatures relief of strains set up by the cold work occurs in the most highly stressed regions, *i.e.*, in the planes of slip and in the boundaries.

These facts, together with the observations of other investigators mentioned above, can be explained on the assumption, which is now put forward, that *the mechanism of the relief of strain caused by cold working is similar to that which occurs in relief of strain set up during age-hardening*, i.e., *by the formation of "crystallites."* If the alloy age-hardens, this formation of "crystallites" takes place more rapidly when the alloy is cold worked since the already strained matrix can only withstand a limited amount of further strain. Hence, the imposition of cold work on such localized, highly strained areas increases the degree of strain, relief of which then takes place in a shorter time than would have occurred in the absence of added cold work.

In an alloy undergoing age-hardening, the localized, highly strained areas (due * to copper-rich aggregates) exist within the matrix of the grains and in the grain boundaries; in a cold-worked material the localized, highly strained regions are in planes of slip as well as in the boundaries. Hence, "crystallite" formation, in an alloy which age-hardens, will start within the grains and on the boundaries, while in cold-worked material it will start in the planes of slip and in the boundaries. In both cases, their initial appearance is indicated by the presence of "pits" developed on etching. This relief of strain occurs

* During ageing, the strain is caused by copper-rich aggregates and during cold working the strain is caused by an *applied* pressure.

at temperatures below the recrystallization temperature of the material, as the present data show. Smith and Wood,[21] in their examination of the atomic lattice of iron under stress, remark that "recovery of the lattice can be produced by mild heat-treatment at a temperature much lower than that required to renew the properties of the metal by recrystallization." This recovery is probably due to the formation of the "crystallites" which Wood [20] observed appearing on the X-ray spectra of heavily cold-worked material and which the present author now shows to be formed during the cold working of an age-hardenable alloy.

ACKNOWLEDGEMENTS.

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INVERSE SEGREGATION IN CAST MAGNESIUM ALLOYS.* 1032

By R. J. M. PAYNE,† B.Sc., Member.

Synopsis.

Inverse-segregation effects have been observed in magnesium alloy castings (ingots, sand- and die-castings) of the compositions ordinarily used in the foundry (essentially magnesium–aluminium alloys containing 8–10% of aluminium).

The resulting enrichment of the surface layer of cast parts with alloying elements and impurities raises difficulties of a practical nature by interfering with the development of chromate films of normal colour. Using the R.A.E. ½-hr. hot chromate bath the effects are particularly marked, segregated castings acquiring an unattractive, piebald appearance.

It is shown that the grain-size of the material, the rate of cooling in the mould, and the presence of thermal gradients are the main factors governing the appearance of these segregation effects. The use of coarse-grained metal, low casting temperatures, and severe chilling are all conducive to the occurrence of inverse segregation. With sand castings, and using the very fine-grained metal such as is obtained with the normal melting, superheating, and casting practice, no significant inverse segregation is encountered. With increasing grain-size the tendency to segregate becomes more marked, and with very coarse-grained material, such as may be obtained by the addition to the alloy of small proportions of beryllium, exudations of eutectic occur at the surface of unchilled sand castings. With gravity die-castings the effects are similar, but the speedier cooling brought about by the use of the metal mould makes slight segregation difficult to avoid, even with fine-grained material.

It is shown that, in segregating, the aluminium is generally accompanied by other elements present as minor alloying additions or as impurities; silicon in particular, which is present in the alloy as magnesium silicide, may be strongly segregated at the surface of the casting and make its presence manifest by its bright blue colour.

The fact that a magnesium alloy cast under controlled conditions can behave in two ways, exhibiting or failing to exhibit inverse-segregation effects according to its grain-size, is of some theoretical interest; the significance of the observation is briefly discussed.

Introduction.

The alloys most widely used in magnesium foundries in this country and elsewhere are essentially magnesium–aluminium alloys containing 8–10% of aluminium; small proportions of zinc and manganese appear as the only other intentional additions. In the course of the production of sand- and die-castings in these materials over a period of years it had been observed, from time to time, that certain castings exhibited a patchy appearance after chromating in the R.A.E. 30-min. hot chromate

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bath.* This non-uniformity of colour was seen on castings in the as-cast and solution-treated conditions; the colour contrast was, however, more marked with the parts which had been solution heat-treated (Specifications D.T.D. 281 and 289). Instead of acquiring the uniform, black colour which is normal for materials to these specifications, the castings referred to took on a piebald appearance, due to the apparent inability of certain areas to accept a chromate film. Improper preparation or inadequate cleaning of the castings had played no part in the occurrence of these features, which were reproduced exactly when the parts were rechromated. The effects were seen most often in gravity die-castings and photographs of two types of casting illustrating the feature mentioned are given in Figs. 2 and 3 (Plate LVI). With components of a given design, the light patches appeared with great regularity on particular parts of the casting. With sand castings the effects were small and much less frequently observed and occurred only on, or close to, those parts of the casting which had been chilled.

Examination under the microscope of sections taken from areas yielding light and dark chromate films had shown that this inability of certain parts of the casting to develop a chromate film of normal colour was to be attributed to a localized enrichment of the surface with aluminium. The segregated layer was in some cases as much as $\frac{1}{32}$ in. in depth. As a remedial measure, some attempts had been made to disperse the excess aluminium by repeated solution treatment: these had been rejected as impracticable in view of the long time required to effect solution of the magnesium–aluminium compound.

While inverse-segregation effects had thus been recognized as operative in magnesium alloy castings, early attempts to reproduce the phenomena in the laboratory had repeatedly failed. Beyond the realization that chilling played some part therein, no clear understanding of the conditions causing inverse segregation was gained, and for some time no effective control over its appearance was possible. At this period, however, castings showing these segregation effects appeared comparatively infrequently and as their only apparent shortcoming was their somewhat unattractive appearance no really serious attention was directed towards finding a cure.

The re-appearance of the phenomena at a later date in an acute form in sand castings led to a further investigation of the problem being made with the results described below. For clarity the subject matter is divided into two parts: Part I records how difficulties encountered in the production foundry were studied and overcome;

* Bath No. iii, Process Specification D.T.D. 911.

FIG. 2.—Small Tail Landing Wheel. Weight approximately 3 lb. Approximately × ½.

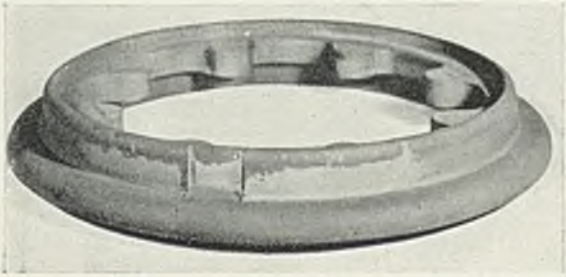

FIG. 3.—Flange for Larger Aircraft Landing Wheel. Weight approximately 4 lb. Approximately × ½.

Figs. 2 and 3 are magnesium alloy gravity die-castings in Elektron alloy AZ91, having a nominal composition of: aluminium 9·5, zinc 0·5, manganese 0·3%, magnesium remainder, solution heat-treated to conform to specification D.T.D. 281. The photographs represent castings after chromate treatment in the R.A.E. alkaline bath. The normal colour of the film resulting from such treatment is black; the light areas are caused by inverse segregation.

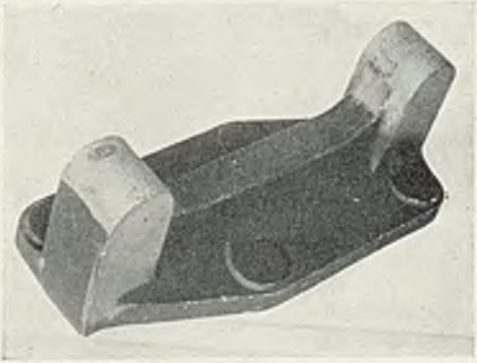

FIG. 4.—Bracket Casting. Weight 3 lb. 9 oz. Approximately × ⅕.

Magnesium alloy sand casting in Elektron alloy A8, solution heat-treated to comply with specification D.T.D. 289. Nominal composition: aluminium 8, zinc 0·5, manganese 0·3%, magnesium remainder. Chromate treated and showing marked inverse-segregation effects.

[*To face p.* 566.

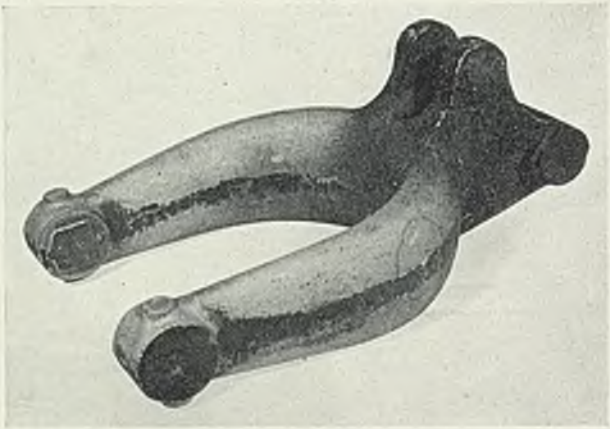

FIG. 5.—Tail Wheel Fork Casting. Weight 7½ lb. Approximately × $\frac{1}{10}$.

FIG. 6.—Tail Wheel Leg Casing Casting. Weight 12 lb. 8 oz. Approximately × ¼.

Figs. 5 and 6 are magnesium alloy sand castings in Elektron alloy A8, solution heat-treated to comply with specification D.T.D. 289. Nominal composition, aluminium 8, zinc 0·5, manganese 0·3%, magnesium remainder. Chromate treated and showing marked inverse-segregation effects.

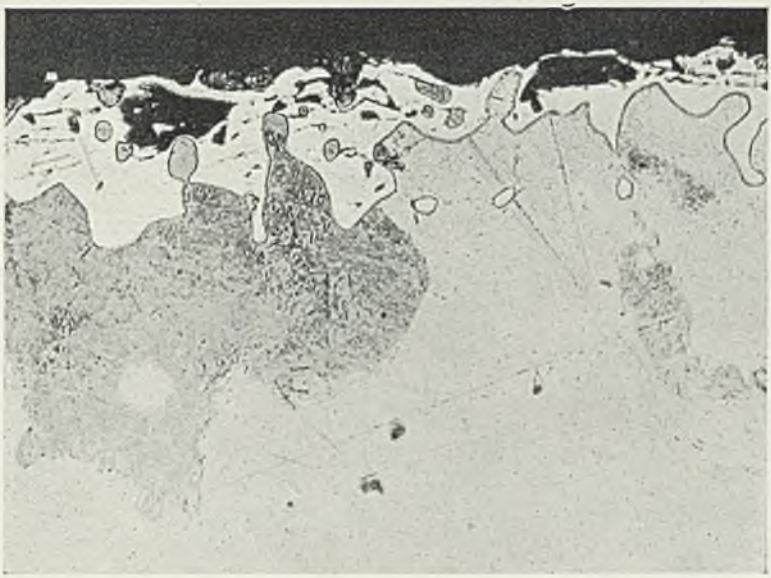

FIG. 7.—Micrograph Through a Segregated Area in a Magnesium Alloy Sand Casting. × 150.

Material.—Solution heat-treated Elektron A8 alloy (D.T.D. 289). Showing marked concentration of Mg_4Al_3 in the surface of the casting. Some of the free compound originally present has been dissolved by heat-treatment, with consequent rounding of the masses; precipitation has occurred on cooling in some supersaturated areas.

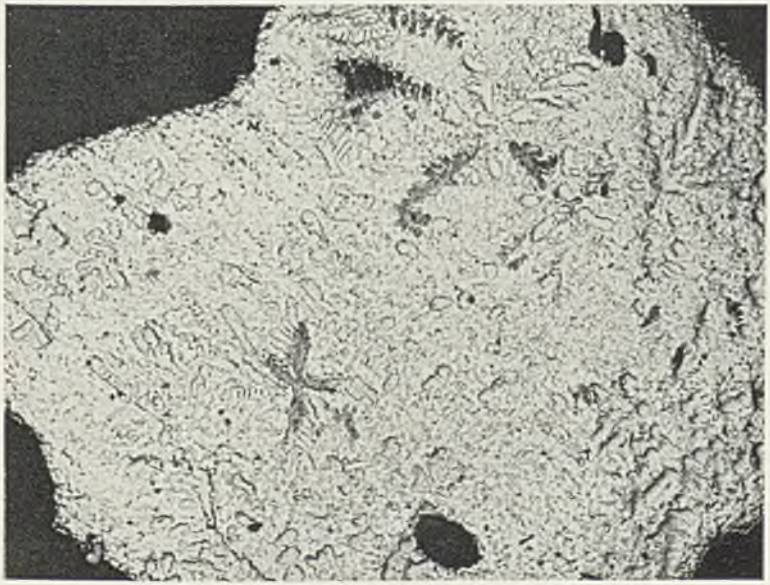

FIG. 8.—Micrograph of Section through Exudation from Castings shown in Fig. 13. × 100.

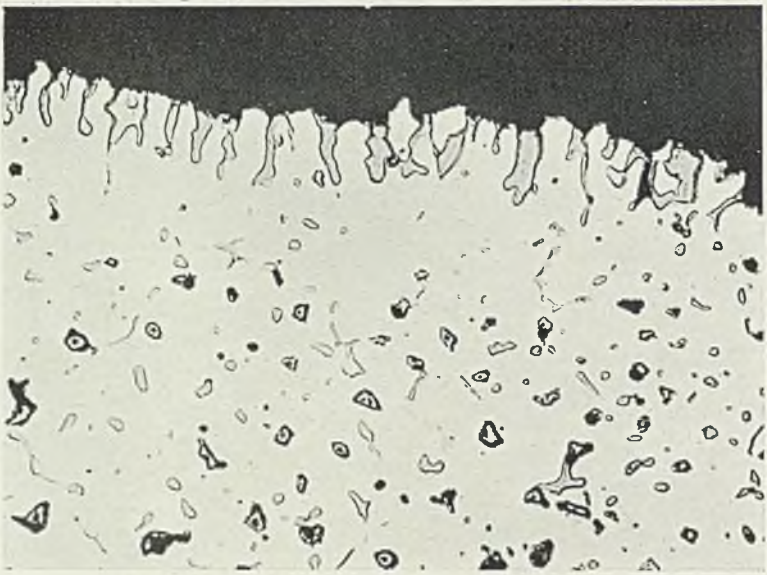

FIG. 9.—Micrograph of Magnesium Alloy Bracket Casting Showing Inverse Segregation Produced Deliberately. × 100.

Material.—Elektron A8 alloy as cast. The configuration of the Mg_4Al_3 suggests that solidification started with the growth of columnar α crystals. The part of the surface of the casting depicted above was not itself chilled, but was in the neighbourhood of one of the chills used.

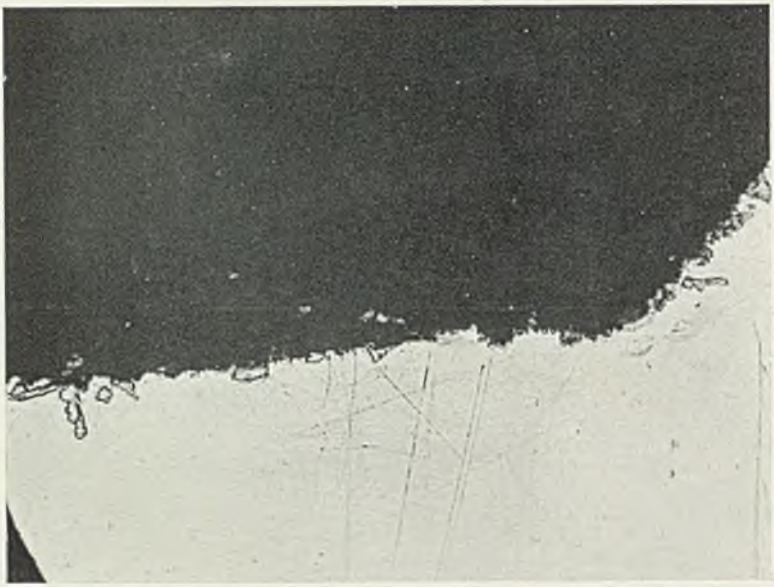

FIG. 10.—Same Area as in Fig. 9, but with Higher Magnification. × 400. Showing presence of magnesium silicide at the cast surface. Unetched.

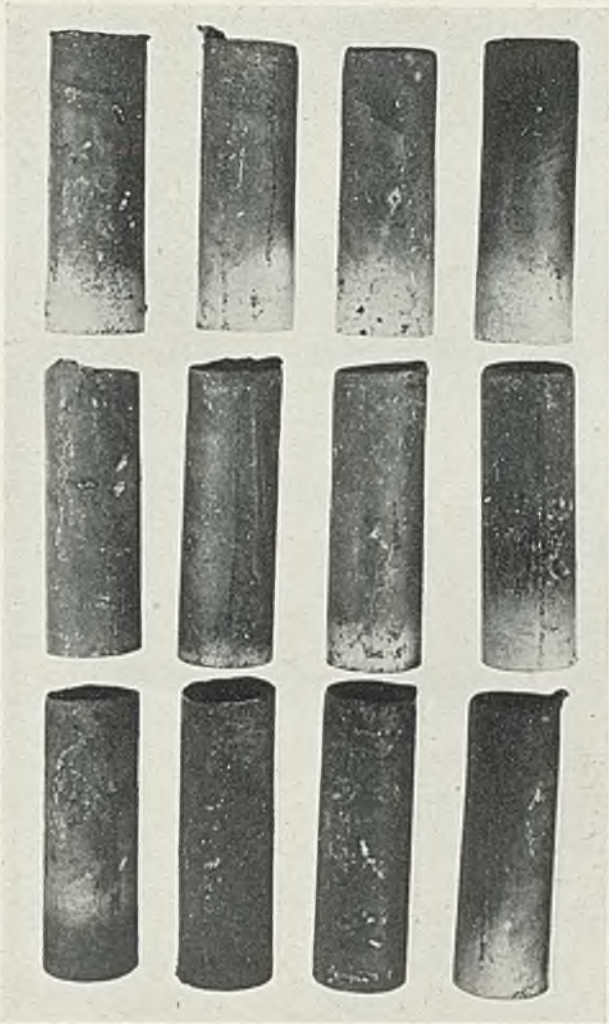

FIG. 11.—6 in. × 2 in. dia. Blocks Cast in Sand Moulds with Chills 1 in. Thick on Bottom. Photograph shows Castings after Heat-Treatment and Chromating.

Casting temperatures, left to right: 800°, 750°, 700°, 650° C.

Top row.—Melt 77F—Coarse-grained Elektron A8 alloy, unsuperheated.

Middle row.—Melt 78F—Coarse-grained Elektron A8 alloy from the same original melt as above, but superheated.

Bottom row.—Melt 79F—Fine-grained foundry scrap, superheated.

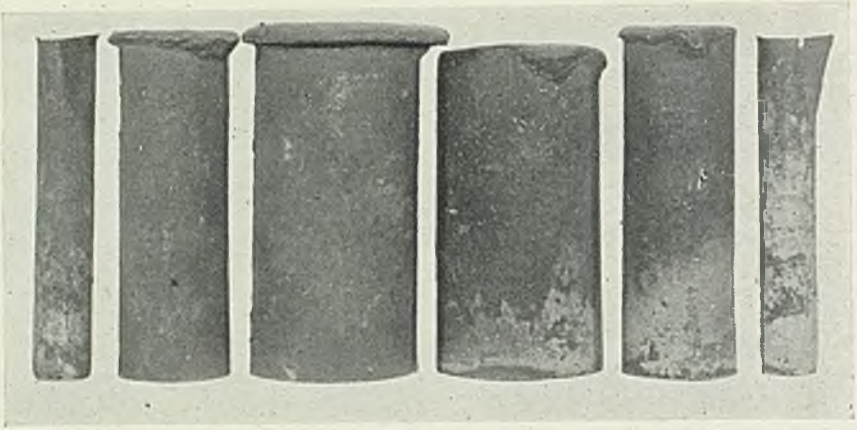

FIG. 12.—Showing Influence of Cast Section upon Occurrence of Inverse Segregation. Approximately × ½.

Material.—Elektron A8 alloy, unsuperheated. Left to right: 1, 2, and 3 in. unchilled; 3, 2, and 1 in. chilled, blocks.

FIG. 13.—Chilled and Unchilled Cylindrical Sand-Cast Blocks, Showing Intense Inverse-Segregation Effects. Approximately × ½.

Material.—Elektron A8 alloy + 0·1% of beryllium. Left to right: 1, 2, and 3 in. unchilled; 3, 2, and 1 in. chilled, blocks.

FIG. 14.—Same Test Castings as in Fig. 13 above, Showing Chill Face.

FIG. 15.—Same Test Castings as Shown in Fig. 13. Approximately × ¼.
Condition.—As cast, showing marked silicon segregation in chilled samples on left.

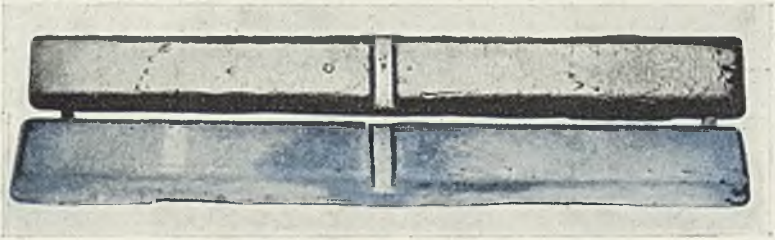

FIG. 16.—Elektron A8 Alloy Ingots D.T.D. 59A. Approximately × ⅛.
Top.—Fine-grained ingot.
Bottom.—Coarse-grained ingot, showing silicon segregation.

FIG. 17.—Showing Fractures through Fine- and Coarse-grained Elektron A8 Alloy Ingots. Approximately × ½.
The fine-grained ingots (left) were white in colour, those of coarse structure (right) were very blue (see Fig. 16 above).

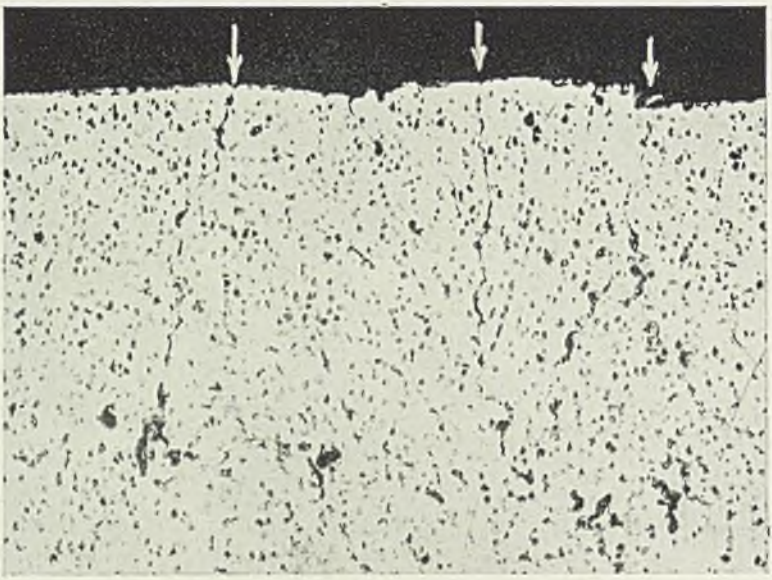

FIG. 18.—Micrograph Showing Structure of 2-in.-dia. Block Casting. × 50.
Material.—Magnesium–8% aluminium alloy, containing a nominal 0·1% of beryllium. Section taken through the chilled face, showing strong columnar growth. Grain boundaries indicated by arrows.

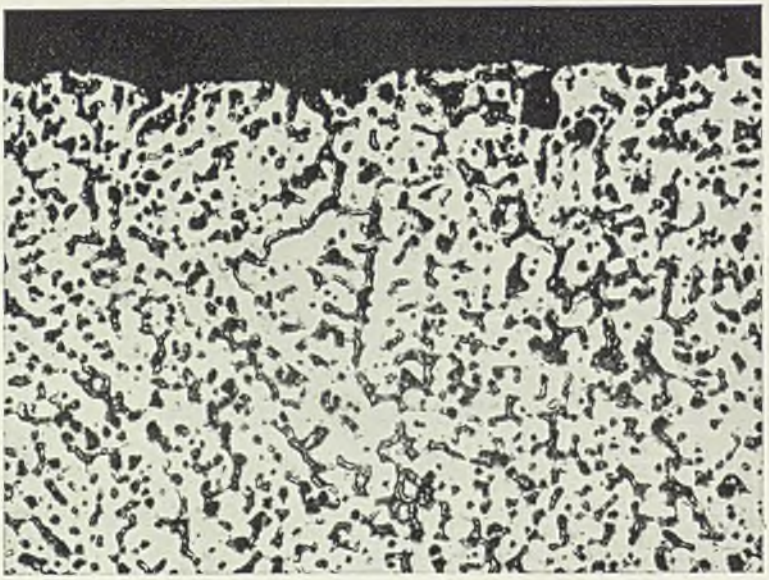

FIG. 19.—Micrograph Showing Structure of 2-in.-dia. Sand-Cast Block. × 50.
Material.—Magnesium–8% aluminium alloy, containing a nominal 0·1% of beryllium. Section through the (unchilled) bottom face.

Part II contains a more detailed description of the observed phenomena and a record of further work of an experimental character.

The effects with which the present paper is concerned appear to have attracted very little attention and only two references to inverse segregation in magnesium alloys have been found in the literature. In 1931 Bauer and Vogel [1] reported that Elektron alloys showed very little tendency to segregate. The other reference occurs in "Metallography of Magnesium and its Alloys," by Bulian and Fahrenhorst.[2] A macrograph of a magnesium alloy slab is given therein, together with the bald statement that it represents an example of inverse segregation due to "unfavourable solidification conditions." No further information is given.

Part I.—Examination of Sand Castings.

As has been stated, it was the occurrence of inverse segregation in sand castings (previously considered relatively immune from such troubles) which concentrated attention on the segregation phenomena. It is, therefore, appropriate to begin by describing and illustrating some typical examples of segregation in such castings.

Three photographs of sand castings showing inverse-segregation effects, all in Elektron A8 alloy, heat-treated to comply with specification D.T.D. 289, are given in Figs. 4, 5, and 6 (Plates LVI–LVII). The photographs show the castings after subjection to the normal processing schedule involving solution heat-treatment, shot-blasting, and chromate treatment in the R.A.E. ½-hr. alkaline chromate bath. In examining these parts it was observed that, with the bracket casting (Fig. 4), the segregation occurred only around those parts (the lugs) which had been chilled; the base of the casting, the cooling of which had been retarded by the heavy feeding head attached, showed no signs of segregation. The method of production used in the foundry for this casting is given in Fig. 1. Evidence that the rate of cooling of the metal had a bearing upon the occurrence and distribution of segregation was similarly seen in the tail wheel fork casting (Fig. 5): here, however, the segregation was more general in character and appeared upon the limbs of the fork which had not been chilled; the effects were seen to become less intense as the heavy, central mass was approached. With the tail wheel leg casing casting, shown in Fig. 6, the segregation was again general in character and was seen on all parts of the cast surface (chilled or unchilled) where this had not been disturbed in fettling. It should be mentioned at this stage that the castings concerned had shown, in routine fracture tests (carried out for purposes

of production control), normal soundness, strength, and ductility. Four miniature tensile test-pieces, cut from the side members of a tail wheel fork casting (Fig. 5), had, for example, shown values for ultimate stress and elongation exceeding those specified for a separately cast test bar.

To confirm that the effects observed were similar in character to those seen earlier in die-castings, sections through affected areas were prepared for microscopic examination. It was at once seen that a high concentration of the magnesium–aluminium compound Mg_4Al_3 existed at the surface of the castings. A typical microstructure is

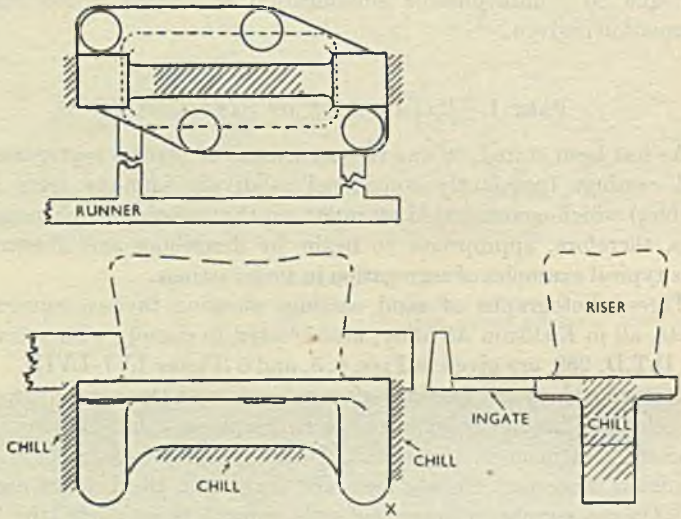

Fig. 1.—Method Used in Foundry for Production of Bracket Casting.

× Specimen for micrographic examination taken here (see Figs. 9 and 10).

shown in Fig. 7 (Plate LVIII). Millings for analysis were taken from the surface of the casting to which Fig. 7 relates and their aluminium content compared with that of a sample taken from the body of the casting. Values for aluminium content of 10·28% at the surface and 7·51% in the body of the casting were obtained. (A more detailed description of the segregation is given in a later section of the present paper. In so far as interference with the formation of chromate films is concerned, it is not proposed, for the moment, to take account of the segregation of elements other than aluminium.) No abnormalities were observed when complete analyses of the material were made on a number of castings, *e.g.*, the sample from the body of the casting of Fig. 7 gave the following values: aluminium 7·51, zinc 0·45,

manganese 0·19, copper 0·07, silicon 0·04, iron 0·02%, and magnesium remainder.

A careful search for unusual impurities (*e.g.*, alkali metals), using the spectrograph, yielded no clue to the reason for the segregation. When studying certain castings which were in quantity production at the time, however, it was observed that all the castings originating from a single crucible of metal behaved similarly in their response to chromate treatment: one melt gave castings showing no segregation whatever, while another yielded castings which all showed the effect. It was thus established that, although the average alloy composition was in all cases apparently normal, the occurrence of the segregation effects was connected in some way with the metal used. Further attempts were carried out at this stage to reproduce the effects in the laboratory; these are described below.

Attempts to Produce Inverse Segregation Deliberately.

(*a*) With other materials, the presence of gas has been recognized as an important, if not decisive, factor in the production of inverse segregation. A test was, therefore, made to discover whether inverse segregation could be induced in magnesium alloy castings (Elektron A8) by gassing the metal.

A 15-lb. charge of the alloy was melted and coal gas bubbled through it for 10 min. at 800° C. The metal was then superheated to 900° C. and cast into sand moulds after cooling to 760° C. No evidence of gas evolution was observed on solidification. As a test for segregation, the test castings (chilled and unchilled pieces of various forms) were subjected to the same finishing scheme as is applied to production castings, *viz.*, solution heat-treatment (16 hr. at 415° C., air-cooled), shot-blasting, and R.A.E. chromate treatment. No evidence of segregation was obtained, the test castings being of a uniform, black colour.

The test reported above was considered unsatisfactory, in that no indications were obtained that the metal used actually held any hydrogen in solution. The experiment was therefore repeated, a sample of the metal being tested for gas by solidification under reduced pressure, using an apparatus of the type described by Baker.[3] The test castings (chilled and unchilled 2-in. dia. sand-cast blocks) were this time cast at 800° C. The gas test showed that very large quantities of gas had been introduced into the metal, but, as in the experiment described above, no evidence of inverse segregation was seen in the final castings.

(*b*) Test (*a*) above was made using ingot metal of standard composition drawn from laboratory stocks and not, therefore, of precisely

the same composition as the alloy in current use in the foundries. For the next test it was decided to utilize castings which themselves exhibited marked segregation effects. These were melted, fluxed, and superheated to 900° C. in the ordinary way under close control and made into bracket castings of the type shown in Fig. 4. The method of manufacture was identical with that used in the production foundry. The casting temperature was 760° C. No evidence of segregation was detected in the castings produced.

(*c*) At this stage it was observed that, with all the castings exhibiting segregation effects thus far examined, the grain-size of the material, while seldom really coarse, was, nevertheless, less fine than is obtained with the best melting, superheating, and casting practice. It was, therefore, decided to make a further set of test castings under conditions conducive to the production of coarse-grained material. A charge of segregated castings was melted and cast into bracket moulds as in test (*b*) above, but this time without raising the temperature of the metal any higher than was required for making the castings; in other words, the usual superheating was omitted. The casting temperature (and, therefore, the maximum temperature) was, as in test (*b*) above, 760° C. The test castings were heat-treated, shot-blasted, and chromated as before and, this time, were seen to produce exactly the segregation phenomena illustrated in Fig. 4 (Plate LVI).

Experiments (*b*) and (*c*) above (afterwards confirmed by many repeat experiments) showed that grain-size could be a controlling factor in the occurrence of inverse segregation in magnesium alloy castings and pointed the way in which the chromating difficulties could be overcome. Finally, it may be mentioned that an examination of ingot stocks, made immediately following the discovery that the chromating troubles were related to the grain-size of the castings, showed that certain batches of ingot possessed an extremely coarse structure (a photograph of a fractured ingot is given in Fig. 17, Plate LXII). The use of this very coarse-grained ingot, together with inadequate superheating in the foundry, had yielded castings showing the severe segregation effects. In justice to the foundrymen, it must be recorded that the chromating difficulties occurred at a particular stage in the war when conditions caused some disorganization in the foundry. With the return of more ordered working, and with a stricter observance of superheating and pouring temperatures, the chromating difficulties disappeared.

This completed the investigation so far as it was concerned with overcoming production troubles. The opportunity was, however, taken to continue the study of inverse-segregation phenomena in

magnesium alloy castings with the object of gaining a better understanding of the controlling factors; the results of this further work are recorded below.

Part II.—Further Observations and Tests.

A.—*Fuller description of Laboratory Test (c) Above.*

It has been briefly recorded in the report on test (*c*) in Part I how inverse-segregation effects were deliberately produced in the laboratory. A fuller description of the observations made in connection with this experiment is, however, merited, and is given below.

The bracket casting upon which the test was made was carefully examined by eye after stripping from the mould. Two features were at once noted: (1) the texture of the cast skin of the lugs was different from that of the base, being rough in the former and smooth in the latter case; and (2) the lugs of the casting were of a clear blue colour while the remainder of the casting was white. After this examination, the test casting was sawn longitudinally into two equal pieces, one of which was subjected to the heat-treatment/shot-blasting/chromating process, the other being retained for reference purposes in the as-cast condition. A comparison made after the processing had been completed showed that the rough surface and the blue colour had been accurate indicators of the extent and degree of segregation, this being confirmed by microscopic examination of sections taken from the casting in the as-cast condition. The micrographs given in Figs. 9 and 10 (Plate LIX) were obtained. Fig. 9, which represents a section through the casting at a point which was close to, but not in actual contact with, a chill (point $\times$, Fig. 1) suggests that freezing started with the growth of columnar α crystals away from the mould face, the Mg_4Al_3 visible in the micrograph having at some later stage in the freezing process taken up its position in the spaces between the crystals. A zone relatively deficient in Mg_4Al_3 is also to be observed between the enriched surface layer and the more normal structure of the interior of the casting. No evidence of the segregation of elements other than aluminium is to be seen in Fig. 9. The micrograph given in Figure 10, however, representing the same area under a higher magnification, shows quite clearly the presence of an abnormal amount of Mg_2Si in the surface layer. It is to the concentration of this constituent in the surface that the blue colour of the casting is to be attributed. It can be deduced from the micrograph that the enrichment of the surface with Mg_2Si extends to a depth of approximately $\frac{1}{4000}$ in. As has been

stated, Fig. 9 represents the structure of the material at a point where the metal had been cast against sand. At the chill face no evidence of enrichment with aluminium was observed; on the contrary, there seemed, in this particular case, to be indications of a localized impoverishment of Mg_4Al_3. This observation is in keeping with others made subsequently: the segregating element does not invariably appear in excess, or even in its normal proportions, at the chilled face.

B.—*Factors Influencing the Occurrence of Inverse Segregation.*

In the investigations already described, it was seen that the grain-size of the metal and the rate of cooling of the castings were factors influencing the occurrence of inverse segregation. With the object of gaining a better insight into the influence of these and other variables a number of further experiments was made; these are described in the following paragraphs.

Test castings of simple form were used in order that the casting conditions might be strictly controlled and the segregation phenomena studied to the best advantage. Simple cylindrical bars 1, 2, and 3 in. in dia. and 6 in. long were employed, these being cast vertically in sand moulds, with and without chills on the bottom face. Where used, the chills were 1 in. thick and of the same diameter as the bar to which they were applied: they were made of mild steel.

The test cylinders were made by pouring the metal directly into the tops of the open moulds, the latter being tilted at the beginning of the operation to ensure quiet filling. For convenience in pouring, the 1-in. dia. bars were made in the form of D.T.D.-pattern test bars; the 2- and 3-in. bars were cast without feeder heads. Once again, the appearance of the heat-treated and chromated castings was taken as indicative of the degree and extent of segregation. The material used for the tests was basically the same throughout—a magnesium–8% aluminium alloy—but, as will be seen, certain modifications of alloy composition were made for some of the experiments. In general, the standard alloy Elektron A8 was employed.

The significant tests are described below.

(1) *Influence of Casting Temperature and Grain-Size of Metal.*

A 20-lb. melt of Elektron A8 was taken and its temperature adjusted to 800° C. A pair of 2-in. dia. × 6 in. test blocks was cast and the temperature allowed to fall to 750° C. Another pair of test blocks was cast at that temperature and, after further cooling, other test blocks were poured at 700° and 650° C.

Sets of blocks were obtained in this way from metal whose origin and treatment is given below :

Melt 77F. Coarse-grained A8 ingot—melted, fluxed, and heated to 810° C. (only) before casting.

Melt 78F. Coarse-grained A8 ingot from the same original melt as in Melt 77F—melted, fluxed, superheated to 900° C., and cooled to 800° C. for casting.

Melt 79F. Fine-grained A8 foundry scrap—melted and superheated as in Melt 78F.

The object in using the above treatments was to obtain castings with coarse, fine, and very fine structures, respectively. That this was achieved was confirmed by preparing sections of the test castings for microscopic examination. Mean grain-size values, as measured on the chilled blocks at a point ½ in. above the chill face and ½ in. from the cylindrical surface, are given in Table I.

TABLE I.—*Mean Grain-Size Values of Test Castings.*

Melt No.	History of Melt.	Mean Grain Dia., mm.	
		Measured on Block Cast at 800° C.	Measured on Block Cast at 650° C.
77F.	Coarse-grained ingot, not superheated.	0·218	0·224
78F.	Coarse-grained ingot, superheated.	0·087	0·135
79F.	Fine-grained scrap, melted and superheated.	0·076	0·129

The test castings were given the homogenizing and chromating treatments and then examined by eye. None of the unchilled blocks showed evidence of segregation; the chilled blocks are depicted in Fig. 11 (Plate LX).

It will be seen that with the coarse-grained castings Melt 77F (Fig. 11, top row) the samples poured at all temperatures on to chills showed evidence of marked segregation. The effect of superheating this material (Fig. 11, middle row) is to prevent any segregation in the casting poured at 800° C. and to permit only a trace with the sample cast at 750° C.; with the lower temperatures the effects are more severe and at 650° C. they are little less intense than those obtained with the coarse-grained metal. The castings prepared from foundry scrap (which, with repeated melting of the metal, had become very fine-grained) show noteworthy segregation only in the block cast at 650° C. (Fig. 11, bottom row).

Reference to Table I will show that with the superheated melts (Nos. 78F and 79F) the grain-size of the blocks cast at 650° C. was distinctly less fine than that of those cast at 800° C. It is plain, therefore, that on cooling to the lower casting temperature a partial reversion to the coarse-grained condition resulted and it is to this that the re-appearance of segregation with falling temperature is due.

It was thus demonstrated that coarse-grained metal is liable to yield segregated castings, whatever the temperature used (within the range 800°–650° C.). The refinement of grain that results from superheating and repeated melting is effective in preventing segregation in test-pieces made under the same conditions but fails, through the reversion of the melts to the coarse-grained condition, with very low pouring temperatures. It may be remarked here that the casting temperatures ordinarily used in the production foundry lie between 700° and 800° C.; segregation effects are, therefore, for practical purposes, avoided by superheating.

(2) *Influence of the Cast Section.*

It will have been observed that, with the tests described above, the rate of cooling and presence of thermal gradients had a marked influence upon the occurrence of inverse segregation. The effects were not noticed in any of the unchilled castings and in the chilled castings were concentrated around those parts in the immediate neighbourhood of the chill.

The effects of varying the cast section are apparent in Fig. 12 (Plate LXI), which represents chilled and unchilled test castings, 1, 2, and 3 in. in dia., cast from unsuperheated and consequently coarse-grained A8 alloy; the casting temperature was 680° C. It will be observed that the effects are no longer confined to the chilled samples, but appear to some slight degree upon the 1-in. dia. unchilled bar as well. With the chilled bars the effects are more pronounced, particularly in the 1-in. dia. bar, where the rate of cooling is greatest.

(3) *Tests with Very Coarse-Grained Material.*

All the tests so far described have been made using the standard A8 alloy, the most marked segregation effects being seen with unsuperheated melts made from coarse-grained ingot. While castings produced in this way may be considered far from fine grained when judged by the standards ordinarily applied to production castings, they still do not approach the extremely coarse structures which may be obtained by the use of certain alloying additions. Zirconium and beryllium are known as elements which possess grain-coarsening powers in magnesium

alloys containing aluminium and small additions of each of these metals were made (separately) to the basic alloy for experimental purposes. Chilled and unchilled blocks 1–3 in. in dia. were made, the casting temperature being 680° C. The addition of a nominal 0·05% of zirconium to the A8 alloy yielded castings showing only slightly more intense segregation than is depicted in Fig. 12. The addition of 0·1% of beryllium, however, produced some very striking results. As will be seen from the photographs (Figs. 13 and 14, Plate LXI), representing the test castings in the chromated condition, marked segregation has occurred in all the samples, chilled and unchilled. In this instance, the effects have progressed far beyond the slight enrichment of the surface layer previously seen and liquid constituents have been forced out of the casting and into the interstices in the moulding sand. These exudations projected $\frac{1}{16}$–$\frac{1}{8}$ in. from the surface of the sample and were found on all parts of the test casting, not excluding the top free surface of the as-cast metal. While the intensity of the effects is again somewhat more marked in the chilled samples, the difference in behaviour between chilled and unchilled pieces is, with this very coarse material, less sharp than before. The intensity of segregation cannot always be satisfactorily expressed by quoting the results of analyses or in any other quantitative manner, by reason of the non-uniform way in which the effects appear; comparison must, in such cases, be based mainly upon the visual appearance of the samples. In this particular case the effects, as judged by the amount of exudations, appear to be most marked in the 3-in. dia. bars, a result in conflict with the effects previously encountered and illustrated in Fig. 12. The effects are still concentrated in the neighbourhood of the chills (where used) but, as will be seen from Fig. 14 showing the undersides of the bars, segregation at the chilled face itself is by no means uniform.

Fig. 8 (Plate LVIII) shows a micrograph of a section through a piece of exuded material detached from the main casting. The structure of the sample is obviously not very different from that of the Mg–Mg_4Al_3 eutectic which contains 32·2% of aluminium; analysis indicated an aluminium content of 28·6% in the exuded sample. A spectrographic examination of the limited quantity of material available showed that the elements zinc, manganese, silicon, and copper were present in roughly the same proportions, relative to the aluminium, as in material of normal composition; the beryllium content appeared to be very low.

The test blocks were examined microscopically and the grain-size estimated. These points are referred to later in the discussion of results.

It must be recorded that some of the test castings showed evidence

of silicon segregation to a very marked degree in the as-cast condition, the surface of the lower halves of the chilled castings being of an intense blue colour. The effects are reproduced in Fig. 15 (Plate LXII).

(4) *The Effects of Gas.*

The experiments with gassed melts described in Part I had shown that the presence of gas could not of itself lead to inverse segregation. The metal used for these tests had been superheated and cast at relatively high temperatures (760°–800° C.) and for this reason yielded fine-grained castings. In these circumstances, the presence of large quantities of gas did not bring about any inverse segregation.

It remained to be shown whether gas played any part in the segregation effects observed with coarse-grained material and a number of tests were carried out with the object of settling this point. The evidence obtained did not lead to a firm conclusion, mainly owing to the difficulty of ascertaining whether the last traces of gas had been removed by a given treatment. It may, however, be recorded that coarse-grained metal which had been twice "pre-solidified" with the object of expelling dissolved gases gave castings showing marked inverse-segregation effects. In the same way, melts treated with volatile chlorides for the purpose of gas removal yielded segregated castings. On the other hand, the deliberate introduction of large amounts of gas into metal which was subsequently held for a prolonged period at a low temperature (650° C.) so as to yield coarse-grained castings did not lead to any particularly severe segregation; the effects were far less intense than those observed with the alloy containing beryllium.

C.—*Silicon Segregation in Ingots.*

Reference has been made to the segregation of silicon in sand castings, such segregation usually accompanying the segregation of aluminium. A micrograph showing a section through a sand-cast surface which had been enriched in this way with Mg_2Si has been given in Fig. 10.

While examining ingot stocks of Elektron A8 alloy in the course of the investigation, an interesting correlation between the appearance of the ingots and their internal structure was observed. It was seen that the ingots derived from certain melts were quite white, while those from other melts were blue. The effect seen, which was recognized as evidence of silicon (and aluminium) segregation, was not usually of a general character and occurred in patches on the centre portion of the ingots and on the two ends. Photographs of a "white" and a "blue" ingot are given in Fig. 16 (Plate LXII).

On fracturing the ingots showing these differences in colour, it was found that all the "white" ingots were fine-grained and all the "blue" ingots coarse-grained. A photograph of fractures of the two ingots shown in Fig. 16 is given in Fig. 17 (Plate LXII). The relatively smooth, fine fracture of the "white" ingot may be compared with the rough, broken surface of the "blue" ingot.

A number of analytical tests was made on sample ingots taken from the two batches, with the following objects:

(1) To confirm that the blue patches seen on the ingots were due to silicon segregation and to establish, if possible, the concentrations of silicon and possibly other elements at the surface.

(2) To establish that the mean compositions of the "white" and the "blue" ingots were sufficiently similar to justify the conclusion that the differences in colour were solely attributable to differences in structure.

The surface tests were made mainly by means of the spectroscope, as that instrument was thought to be particularly well suited to the determination of the composition of very thin surface layers. To determine quantitatively the concentrations of all the elements present would, however, have necessitated establishing several new spectrographic standards and the development of new or modified techniques. In view of the large amount of work involved this was not attempted, save in the case of silicon. The results of the tests may be summarized as follows:

(i) With both "blue" and "white" ingots the chill-cast surface was found to contain a greater proportion of the elements aluminium, zinc, copper, and silicon than the interior; the degree of segregation was, however, much higher in the "blue" than in the "white" ingot. An enrichment of the chill-cast surface with manganese was reported only in the case of the "white" ingot, the surface value for the "blue" ingot being found to be normal. This observation is contrary to earlier experience with the sand-cast blocks where exuded material had been shown by analysis to contain a high proportion of manganese.

(ii) The concentration of silicon in the surface layer appeared to be of the order of 4·0 and 2·4% in the "blue" and "white" ingots, respectively.

(iii) A complete bulk-analysis of the materials comprising the two ingots was made, with these results:

	"Blue" Ingot, Melt No. 7461.	"White" Ingot, Melt No. 7465.
Aluminium, %	7·87	7·48
Zinc, %	0·51	0·51
Manganese, %	0·20	0·26
Iron, %	0·03	0·03
Silicon, %	0·11	0·09
Copper, %	0·11	0·05

The compositions of the two batches of ingots will be seen to be so nearly identical as to justify the statement that the only significant difference between the two ingot samples lies in their internal structure.

It will be seen from the foregoing that the colour of an ingot can act as a guide to its internal structure, a marked blue colour being indicative of silicon (and aluminium) segregation and thus of a coarse grain. While it cannot be claimed that the point has been tested in an exhaustive manner, it nevertheless appears to hold good so far as material of normal silicon content is concerned (0·1–0·2%). It can be seen that with alloys containing considerably higher proportions of silicon a very slight amount of inverse segregation might yield castings blue in colour, in spite of a reasonably fine grain-size.

As a further point of interest, it may be recorded that abnormal amounts of Mg_2Si in the surface of a casting (resulting from inverse segregation) make their presence known in a very clear-cut manner should the casting be pickled in acid—a widely used method of preparing the surface for chromate treatment. The magnesium silicide reacts with the dilute acids employed for this purpose, with the generation of spontaneously inflammable silicon hydride. By contrast with unsegregated castings, which dissolve quietly in the acid used (generally 10% nitric acid in water), a sequence of mild explosions results from the immersion of a segregated casting in the same reagent.

Summary and Discussion.

It has been shown that marked inverse segregation may be encountered in the magnesium alloys ordinarily used in the foundry. Pronounced effects may be met in castings made in sand, as well as in chill, moulds. The conditions which favour the development of the effects appear to be:

(1) Coarse-grained metal.
(2) Low casting temperatures.
(3) Rapid cooling and steep temperature gradients.

The effects may be produced in an exaggerated form by the addition of grain-coarsening agents, such as zirconium and beryllium.

In production castings, inverse segregation raises difficulties of a practical nature by affecting the response of cast parts to chromate

treatment. By avoiding the conditions set out above, the tendency to segregate is greatly reduced, if not entirely eliminated; in these circumstances, castings are obtained in which the segregated skin is so thin as to be removed in the cleaning processes ordinarily applied to production castings.

The constituents which may concentrate at the surface of the casting are aluminium, zinc, manganese, copper, and silicon. The first-named constituent (and probably the second also) is responsible for the chromating difficulties; the last-named (when present) gives the parts a marked blue colour and makes them particularly reactive in a pickling bath.

The earliest attempts to reproduce segregation effects in the laboratory failed for the reason that the test castings were, in all cases, prepared from superheated (and consequently fine-grained) melts.

Apart from the foregoing statements, all of which have a practical significance, it is of interest to examine whether the work now reported presents any new features for consideration so far as the theoretical aspect of inverse segregation is concerned.

The influence of the rate of cooling and of strong thermal gradients (resulting from heavy chilling) in aggravating a tendency to segregate is common experience with other materials and a sensitivity to casting temperatures (interlinked, of course, with the rate of cooling and with thermal gradients) is not unknown.[4] With the magnesium-base alloys, the influence of casting temperature upon the degree of segregation in the casting is not, however, a simple one. Cooling to low casting temperatures necessarily takes place slowly with large melts and in the process the beneficial effects of superheating are largely or wholly lost. It is probable that the changes in the grain-size of the castings which result are of more account in promoting inverse segregation than is the direct influence of low casting temperatures upon the rate of cooling and magnitude of temperature gradients during solidification. The simultaneous segregation of a number of elements (particularly those forming low melting-point constituents) in a given alloy is common [4] and would be expected on theoretical grounds. The only new feature which emerges from the present work is the demonstration that a given material can behave in two distinct ways under certain casting conditions, its response being governed by its grain-size. If fine-grained, the material will furnish castings in which inverse segregation, if not entirely absent, will be of negligible proportions; if coarse-grained, the castings may be segregated severely.

An explanation of this behaviour is thought to lie in the essential difference between the structures of castings made from unsuperheated

and superheated melts of magnesium–aluminium alloy. The characteristics of the two types of structure have been well brought out and illustrated by Fox and Lardner,[5] who showed that unsuperheated melts yield large grains with a marked dendritic growth of the α-phase, the β-constituent (Mg_4Al_3) being partly held within the branches of the dendrites and partly at the grain boundaries. By contrast, melts refined by superheating yield very small crystals with little evidence of dendritic growth, the β-constituent being largely confined to the grain boundaries. Under the influence of unidirectional chilling, unsuperheated melts crystallize with a columnar structure at the chill face; this tendency is almost completely suppressed with grain-refined material. Magnesium–aluminium alloys containing small proportions of beryllium or zirconium possess microstructures which are essentially the same as those of unsuperheated melts containing no such additions; the grain-size is, however, even coarser and the tendency to a strong columnar growth even more pronounced. These features are illustrated in the micrographs given in Figs. 18 and 19 (Plate LXIII), which represent sections taken through the undersurface of chilled and unchilled 2-in. dia. blocks in the beryllium-containing alloy; the long, columnar crystals growing from the chill face (Fig. 18) are particularly to be noted. The grain-size of these castings was measured at the same point in the casting as were the values given in Table I. A mean grain diameter of about 1·5 mm. was obtained. The grain-size at points well removed from the chill face was about 3 mm.

It seems, therefore, that the difference in the casting behaviour of coarse- and fine-grained material lies simply in the fact that, in the one case, comparatively large dendrites are formed on cooling, with interspaces providing a relatively free passage for the counter-flow of low melting-point constituents; in the other case, the opportunities for such movements are considerably restricted, due to the small size of the primary crystals. It will, of course, be appreciated that the magnesium alloys used in industry for castings are, by reason of their long freezing range (600°–435° C.), inherently liable to the exhibition of inverse-segregation effects.

The part that dissolved gases play in the segregation phenomena has not been firmly established. It has, however, been clearly shown in Part I that gas cannot cause segregation in melts which have been handled in such a way as to yield fine-grained castings. Whether the presence of gas is essential to the exhibition of inverse-segregation effects with coarse-grained castings, or whether the movement of low melting-point constituents takes place for entirely different reasons remains to be determined. If (as is believed to be the case with tin-

bronzes) gas is the governing factor with magnesium alloys, then the critical amount required to cause inverse segregation is extremely small.

It is not proposed to examine here the many theories which have been advanced to account for inverse-segregation phenomena; to be generally acceptable such theories will, however, need to explain satisfactorily the effects observed with magnesium alloy castings, as reported in the present paper.

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ANTIMONIAL 70 : 30 BRASS.*

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Synopsis.

A study has been made of the effect on 70 : 30 brass of antimony, lead, and sulphur. The results reported in the literature were found to be inconsistent, suggesting that the effect of antimony depends on the operative conditions.

Part I of the present work deals with the hot- and cold-rolling behaviour and some mechanical properties of three series of 70 : 30 brass containing antimony, antimony plus lead, and antimony plus sulphur, respectively. Antimony severely impairs cold-rolling behaviour and impact strength in the cast condition but an annealing treatment at the ingot stage, by promoting homogenization, reduces these deleterious effects of antimony considerably. Hot-rolling behaviour is affected to a lesser extent. The simultaneous presence, with antimony, of lead or sulphur, within reasonable limits, did not influence greatly the effect of antimony. Conclusions are also reached about the individual effects of lead and sulphur.

In Part II the microstructure of the three series is studied and the influence of heat-treatment described. Slowly cooling antimonial brass or maintaining it within a certain temperature range induces intergranular brittleness; this is associated with reduced solid solubility of antimony at low temperatures but may not be due to precipitation. Comparison with previous work shows that the effect of antimony on brass is intensified by increase in zinc content. A portion of the copper–zinc–antimony equilibrium diagram has been determined.

Part III deals with the removal of antimonial embrittlement by treatment of the melt. Phosphorus and lithium additions produce the desired result, probably by forming innocuous compounds with antimony; lithium, however, embrittles on its own account. Certain other elements may also be effective.

The results are discussed in Part IV. The inconsistency of previous work is ascribed to the influence of three factors: heat-treatment, zinc content, and casting segregation. Attention is directed to the similarity between antimonial embrittlement of brass and temper-brittleness of steels. The technique of electrolytic polishing of samples for micro-examination is described in the Appendix.

Introduction.

The availability of brass scrap in the form of fired cartridge cases had raised the questions of (*a*) possible contamination of the melt when such scrap was used as part of the charge and (*b*) the effect of any impurities so introduced upon the properties and working behaviour of the brass. The principal impurity is known to be antimony, but small quantities

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of lead and sulphur may also be present. The literature on the subject shows considerable diversity of opinion as to the permissible contents of these impurities and a study has therefore been made of the properties of three series of 70 : 30 brass alloys containing, respectively, antimony, antimony plus lead, and antimony plus sulphur. Following this work, the possibility was examined of counteracting the effect of antimony by treatment of the melt.

Previous Work.

Effect of Antimony.

Many references to the effect of antimony on brass are to be found in the literature, covering the effect on working, mechanical properties, and structure. Widely varying limits of antimony content are suggested, the lowest being derived from a study of the working behaviour of the brass. Brannt [1] early specified a limit of 0·001 to 0·006% of antimony and is supported by some later writers. Thus, Severgin [2] found, in an industrial investigation, that in lead-free brass of 62% copper content 0·009% of antimony caused cracking on cold rolling. StJohn [3] infers that 0·009% of antimony makes brass practically unworkable. According to Jevons,[4] experience during and after the 1914–18 war with brass made from charges incorporating ammunition fuses proved that 0·004% of antimony was sufficient to cause splitting during rolling or subsequent deep-drawing or pressing; he considers, however, that this low limit may be due to the simultaneous presence of sulphur. These low limits are qualitatively supported by Gibbs,[5] who states that antimony has always been of ill repute in brass mills.

Two groups of workers reached less-restrictive conclusions. Amsterdamskiy, Gagen-Torn, and Grashtschenko,[6] in an investigation carried out under industrial conditions, found that 70 : 30 brass behaved satisfactorily on hot or cold rolling with up to 0·055% of antimony; and Hull, Silliman, and Palmer,[7] in laboratory tests on 70 : 30 brass, also found that 0·05% of antimony did not interfere much with cold rolling although the limit was being reached, but concluded that only 0·01–0·02% could be tolerated for hot rolling. In works tests, apparently employing cold rolling, 0·02% of antimony caused slight splitting, but the results are rather erratic. Both these groups of workers give clues to the divergent opinions existing by showing the influence of other factors. Amsterdamskiy, Gagen-Torn, and Grashtschenko [6] found that the antimony limit for cold rolling was decreased to 0·01% when the zinc content was increased to 40%. Hull, Silliman, and Palmer [7] found the effect of antimony on cold rolling to be reduced by phosphorus,

although silicon, magnesium, and manganese were without effect; in their works tests, they record, splitting occurred with only 0·007% of antimony when 0·07% of lead was also present. In connection with the influence of zinc content on the effect of antimony it is interesting to note that Archbutt and Prytherch [8] found that copper containing as much as 0·47% of antimony could be hot rolled and that for cold rolling even 0·85% caused no difficulty.

The effect of antimony on the mechanical properties of 70 : 30 brass is subject to the same uncertainty as that on working behaviour. On the one hand, Keese [9] reported that the tensile properties of 70 : 30 brass were very seriously affected by antimony; for example, 0·11% of antimony reduced the ultimate tensile strength to 12·8 tons/sq. in., the percentage reduction of area to zero, and elongation to 8%. Hull, Silliman, and Palmer [7] similarly found that the impact-resistance of cast material was seriously impaired by antimony and that other factors were also involved, since the impact strength was increased by annealing almost to the pure brass value unless lead were present, in which case annealing had no effect. On the other hand, Lynes [10] showed that 0·1% of antimony reduced the elongation of 70 : 30 brass only from 80 to 77% for the same ultimate tensile strength figure as pure brass. The effect on resistance to impact was, however, more serious: specimens of 70 : 30 brass with no antimony and with 0·03% of antimony were both unbroken in the impact test, whereas with 0·1% of antimony fracture occurred. Lynes is supported by Amsterdamskiy, Gagen-Torn, and Grashtschenko,[6] who found the ultimate tensile strength to be slightly decreased and the elongation slightly increased to a maximum at 0·03–0·05% antimony content.

There is evidence that the harmful effect of antimony on the mechanical properties of brass is greater, the higher the zinc content. Thus, the last-named workers found that 0·01% of antimony had a noticeable adverse influence on 60 : 40 brass and are supported by the work of Rae [11] and Robertson.[12] Rae found that the detrimental effect of the small quantities of antimony he tried (up to about 0·02%) was practically confined to the impact values and to the bending and cold-riveting properties. Robertson concluded that 0·01% of antimony reduced the properties of manganese bronze below the specification limit. Conversely, with low zinc contents more antimony seems permissible. Gardner and Saeger [13] found that additions of 0·10–0·25% of antimony to red brass (nominal composition: copper 85, zinc 5, tin 5, and lead 5%) had little effect on tensile properties and hardness and Archbutt and Prytherch [8] decided that the tensile and impact properties of copper were slightly improved with up to 0·22% of antimony. Work

on the effect of antimony on bronzes may be mentioned here [14–18] and leads to the conclusion that 0·5–1·0% of antimony is without harmful effect.

As to constitution, Keese [9] reported that the solubility of antimony in brass varies from zero at 37% zinc to 8% in pure copper. This is supported by the fact that Amsterdamskiy, Gagen-Torn, and Grashtschenko [6] placed the solubility of antimony in 70 : 30 brass at about 0·5%, while other workers report much higher values for the solubility in copper.[8, 19, 20] The results of Mertz and Mathewson [20] are probably the most reliable. They report that the solid solubility of antimony in copper diminishes gradually from 11·3% at 630° C. to 10% at 450° C., at which latter temperature a phase change occurs in the separating constituent,[21–25] the solubility then decreasing more rapidly to 2·1% at 200° C.

There is thus a wide diversity of results on the effect of antimony on brass. In general, industrial experience sets a limit of not more than 0·01% antimony whereas, in controlled experiments, 0·05% has been found to have little effect. This discrepancy is doubtless partly to be explained by (i) an increasing tolerance for antimony as the copper content increases, (ii) the influence of other impurities, and (iii) differences in processing technique, but the degree of responsibility of these factors is unknown.

Effect of Lead.

General experience is that lead in brass is considerably more harmful during hot rolling than during cold rolling and three workers [2, 26, 27] indicate a limit of about 0·01% of lead in 70 : 30 brass intended for hot rolling. Two investigators [2, 26] report that the permissible amount of lead increases with zinc content.

Ductility of 70 : 30 brass was found by Croft [28] to be decreased by lead rather more than tensile strength was increased and he states that deep-drawing properties are somewhat reduced. Jevons [29] introduces a qualification with the statement that the effect of lead on the deep-drawing properties of brass depends, within limits, more on particle size and distribution than on actual content. Jevons also states that good-quality brass sheet usually contains from 0·01 to 0·05% of lead.

Effect of Sulphur.

No quantitative results on the effect of sulphur in 70 : 30 brass have been found in the literature. However, Gardner and Saeger [30] observed a 10% reduction in the tensile strength of cast red brass

(copper 85, zinc 5, tin 5, and lead 5%) after adding 0·1% of sulphur; while Smith and Bolton [17] found no deterioration in the properties of leaded bronze with 0·3% of sulphur alone, or with both sulphur and antimony together, if the contents were kept below 0·15 and 0·3%, respectively.

Present Work.

The main interest of the present work has been the harmful influence of antimony on 70 : 30 cartridge brass and the removal of this influence. The work on lead and sulphur has been chiefly concerned with the manner in which these elements modify the effect of antimony.

The paper is divided into four parts. Part I covers the examination of rolling behaviour and mechanical properties of 70 : 30 brass containing 0–0·22% antimony and 0–0·12% each of lead and sulphur. Part II deals with physical tests performed on the plain antimonial series and with the constitution of the alloys with up to 2·3% of antimony. Part III is concerned with the elimination of the harmful effects of antimony by means of additions to the melt. Finally, the experimental results are discussed in Part IV.

Part I.—Antimonial Brass Containing Lead or Sulphur.

The alloys comprised three series *viz.*, those containing (*a*) antimony only, (*b*) antimony with lead, and (*c*) antimony with sulphur.

Preparation of Ingots.

The raw materials were: electrolytic copper; 99·99 + % zinc; antimony containing lead 0·095, iron 0·14, and sulphur 0·19%; lead containing antimony 0·12, tin 0·37, and copper 0·05%; and rock sulphur. Copper–antimony and copper–lead alloys, containing 1% of antimony or lead respectively, were prepared for the additions to series (*a*) and (*b*).

The charges were melted under a glass cover in a gas furnace, cast into notched-bar moulds under burning coal gas, remelted, and cast at 1090°–1110° C., again under coal gas, into soot-coated, cast-iron moulds, 12 × 7 × 1 in., fitted with hot tops. The ingots were sectioned transversely into two or three slabs, depending on rolling requirements, and a transverse slice for macro- and micro-examination. Antimony and sulphur were added as the elements to series (*c*) which was not remelted but cast direct into 12 × 5 × ¾-in. moulds.

Chemical Composition.

Samples for analysis were obtained by machining the full transverse section of the ingot or rolled sheet. The compositions are given in Table I. In general, the copper–zinc ratio was within the limits 69 : 31 to 71 : 29. A few castings were completely analysed for impurities: in series (*a*) and (*b*) the only impurities present in more than traces were iron, not more than 0·004%, and sulphur in the range 0·001–0·006%, but in series (*c*) one sulphur-free ingot was contaminated with sulphur.

Rolling of Antimony [*Series* (*a*)] *and Antimony plus Lead* [*Series* (*b*)] *Alloys.*

Cold rolling was carried out in two stages, with an intermediate anneal of 1 hr. at 650° C. followed by air-cooling. The first stage consisted of a 57% reduction and the second of a 54% reduction, both in 7–11 passes. The total reduction was, therefore, 80% and the final thickness 0·20 in. Although small reductions per pass were necessitated by the equipment available, they probably have the advantage of proving more sensitive in revealing rolling defects than would the normal commercial rolling schedule. Heavier reductions were, in fact, tried on one ingot as described later. A cross-flattening pass was given at the end of each stage.

In Table II, the (*a*) results show the surface cracking revealed by visual examination; longitudinal cracks at the ends also appeared occasionally. In view of antimony micro-segregation in the as-cast material, portions of several ingots were pre-annealed for 1 hr. at 750° C. followed by air-cooling and then processed in the usual way. In Table II, the (ii) results refer to this material. Edge and surface cracking were much reduced by pre-annealing.

The as-cast ingots containing 0·11 or 0·12% of antimony cracked badly, and the pre-annealed ingots slightly. The as-cast ingots containing 0·15–0·22% of antimony cracked so badly after 10–15% reduction as to be useless; the 0·20% of antimony pre-annealed ingot was comparable with the 0·10% of antimony as-cast ingot.

It is clear that antimony alone has a more serious effect than lead on cold-rolling behaviour, but small amounts of lead considerably reduce the permissible antimony content *e.g.*, in the absence of lead, 0·05% of antimony is on the border-line for satisfactory cold rolling, but if 0·01% of lead is present, antimony must be restricted to 0·03%.

The bottom half of the ingot containing 0·05% each of antimony and lead was reduced cold by 43% in two passes. No visible surface

TABLE I.—*Chemical Compositions of Alloys.*

Series (*a*)—Antimony.

Calculated Composition. Sb, %.	Composition by Analysis on Full Section.	
	Cu, %.	Sb, %.
0	70·09	Nil
0·01	69·57	0·009
0·015	70·15	0·015
0·020	71·14	0·021
0·025	70·27	0·024
0·05	69·38	0·05
0·075	69·67	0·075
0·10	69·25	0·10
0·10	70·19	0·11
0·10	70·08	0·12
0·15	70·27	0·15
0·20	68·47	0·20
0·25	69·83	0·22
0·40	69·59	0·35
0·80	69·01	0·75
2·5	68·39	2·31

Series (*b*)—Antimony plus Lead.

Calculated Composition.		Composition by Analysis on Full Section.		
Sb, %.	Pb, %.	Cu, %.	Sb, %.	Pb, %.
0·035	0·005	69·45	0·035	0·005
0·035	0·005	69·65	0·06	0·005
0·01	0·01	69·78	0·012	0·009
0·015	0·01	69·88	0·012	0·009
0·02	0·01	69·46	0·021	0·009
0·025	0·01	69·80	0·031	0·010
0·035	0·01	69·30	0·03	0·010
0·05	0·01	71·49	0·06	0·010
0·035	0·015	69·22	0·03	0·015
0·075	0·015	69·41	0·075	0·015
0	0·02	69·42	...	0·02
0·01	0·02	69·59	0·009	0·019
0·015	0·02	69·42	0·015	0·019
0·02	0·02	70·36	0·02	0·02
0·025	0·02	69·35	0·027	0·019
0·05	0·02	70·60	0·06	0·02
0·075	0·02	70·63	0·075	0·02
0	0·05	70·81	...	0·05
0·01	0·05	69·41	0·01	0·05
0·02	0·05	71·08	0·02	0·05
0·035	0·05	69·63	0·03	0·05
0·05	0·05	70·50	0·05	0·05
0·075	0·05	70·51	0·08	0·05
0	0·10	69·87	...	0·09
0·02	0·10	70·55	0·03	0·12
0·035	0·10	69·23	0·03	0·10
0·05	0·10	71·89	0·06	0·12

Series (*c*)—Antimony plus Sulphur.

Calculated Composition.		Composition by Analysis on Full Section.		
Sb, %.	S, %.	Cu, %.	Sb, %.	S, %.
0	0	69·84	0·004	0·004
0·05	0	69·44	0·06	0·002
0·10	0	70·05	0·10	0·002
0·25	0	70·82	0·23	0·014
0	0·01	70·19	0·005	0·006
0	0·02	70·48	0·004	0·015
0	0·03	70·67	0·004	0·026
0	0·05	70·50	0·005	0·032
0·02	0·02	69·43	0·02	0·011
0·05	0·05	68·64	0·04	0·024
0·10	0·10	71·04	0·06	0·054
0·25	0·25	71·32	0·18	0·12

cracks developed and edge cracks were $\frac{1}{16}$ in. deep. The duplicate ingot after being cold reduced 57% in seven passes exhibited general surface cracking and had edge cracks $\frac{1}{8}$–$\frac{1}{4}$ in. deep. This supports the view that heavier passes than those normally given in this work reduce cracking and consequently a commercial rolling schedule should tolerate a higher antimony content than the limit indicated above.

After annealing, the pure brass slabs and those containing up to 0·05% of lead, antimony nil, or 0·01% of antimony, lead nil, were free from cracks. The other slabs showed fine, longitudinal cracks which were ascribed to fire-cracking and occasioned some concern. The

TABLE II.—*Surface Cracking During Cold Rolling* [*Series* (*a*) *and* (*b*)].

(i) As-cast; (ii) Pre-annealed 1 hr. at 750° C.

Pb, %.	Sb, %.								
	0	0·01	0·015	0·02	0·025	0·035	0·05	0·075	0·10
0	(i) None. (ii) ...	None. None.	None. None.	None. None.	None. None.	... ...	None. ...	Bad. Slight.	Bad. Slight.
0·005	(i) ... (ii) ...	... ...	... ...	... ...	... ...	Slight. ...	... ...	Bad. ...	... ...
0·01	(i) ... (ii) ...	None. None.	None. None.	None. None.	None. None.	Slight. ...	Slight. ...	... ...	... ...
0·015	(i) ... (ii) ...	... ...	... ...	... ...	... ...	Slight. ...	... ...	Bad. ...	... ...
0·02	(i) None. (ii) ...	None. None.	None. None.	None. None.	None. None.	... ...	Slight. ...	Bad. ...	... ...
0·05	(i) None. (ii) ...	None. ...	... ...	Slight. ...	... ...	Bad. Slight.	Bad. Slight.	Bad. ...	... ...
0·1	(i) None. (ii) None.	... ...	... ...	Slight. ...	... ...	Bad. Slight.	Bad. ...	... ...	... ...

incidence of these cracks was not reduced by decreasing the rate of heating to the annealing temperature but, if anything, was increased. These cracks were not taken into account in the assessment given in Table II.

Hot rolling was also carried out in two stages, both beginning with the metal at 750° C. The first stage consisted of a 57% reduction in three passes and the second of a 54% reduction in four passes. The final thickness was again 0·20 in.

The extent of visible surface cracking is given in Table III, together with the results of bend tests in which $\frac{1}{2}$-in. strips of the annealed material, cut transversely, were bent to and fro through 180° over rollers of radius three times the thickness of the strip in the machine shown

in Fig. 1. The bend test is evidently more sensitive than visual examination. Subsequent tests showed that, except for the alloys containing 0·15% of antimony or more, the bend value was a measure of surface-rolling cracks.

It is clear that in hot rolling, lead is the more detrimental element

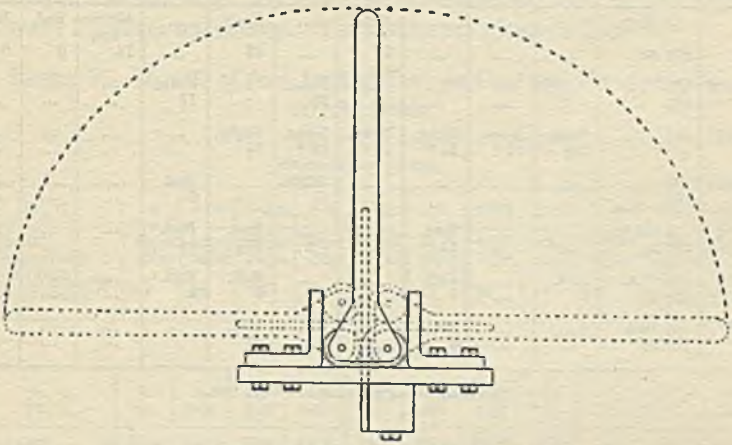


FIG. 1.—Bend-Test Machine.

and its influence is increased by the presence of antimony. In the absence of antimony the maximum permissible lead content for hot rolling was shown by interpolation to be 0·011%; this limit is halved by the addition of 0·04% of antimony. In the absence of lead the maximum permissible antimony content is about 0·075%.

Rolling of Antimony plus Sulphur [*Series* (*c*)] *Alloys.*

One of the duplicate ingots of each composition was cold rolled to 0·14 in. in three stages, with two intermediate anneals of ½ hr. at 650° C. The reduction per stage ranged from 35 to 47%. The other ingots were heated to 700° C. and hot rolled to about 0·15 in., some in two stages with an intermediate reheat to 700° C. As the bend-test machine was not available while the work on series (*c*) was in progress, transverse tensile tests were performed; in Table IV elongation figures are given as a measure of surface cracking during rolling.

Antimony is more injurious on cold rolling than on hot rolling. Sulphur is harmful on hot and cold rolling and in both cases the limit is 0·01–0·02%. Antimony up to 0·04% does not intensify the effect of sulphur on hot rolling but does so on cold rolling; sulphur increases

TABLE III.—*Surface Cracking During Hot Rolling of Series (a) and (b).*

Gauged (i) visually and (ii) by number of bend reversals of specimens annealed 1 hr. at 650° C.

Pb, %.	Sb, %.										
	0	0·01	0·015	0·02	0·025	0·035	0·05	0·075	0·11	0·15	0·22
0	(i) None.	...	...	...	None.	...	None.	...	Moderate.	Bad.	Bad.
	(ii) 25	...	...	...	29	...	23	...	14	0	0
0·005	(i) ...	...	...	...	...	None.	...	None.	...	...	...
	(ii) ...	...	...	...	...	26	...	17	...	...	...
0·01	(i) ...	None.	None.	None.	None.	None.	Slight.	...	...	...	...
	(ii) ...	23½	23 *	21 *	23 *	15 *	9½	...	...	...	...
0·015	(i) ...	...	...	...	...	Slight.	...	Bad.	...	...	...
	(ii) ...	...	...	...	...	15	...	3	...	...	...
0·02	(i) Slight.	...	...	Bad.	...	...	Bad.	Bad.	...	...	...
	(ii) 9½	...	...	11	...	...	10½	5½	...	...	...
0·05	(i) Bad.	Bad.	...	Bad.	...	...	Bad.	Bad.	...	...	...
	(ii) 8½	9	...	7	...	...	7	4	...	...	...
0·1	(i) Bad.	...	...	Bad.	...	...	Bad.	...	...	...	...
	(ii) 5½	...	...	8	...	...	6½	...	...	...	...

* Cracks revealed during the bend test.

TABLE IV.—*Surface Cracking During Rolling of Series (c).*

Gauged visually and by transverse elongations of tensile specimens annealed ½ hr. at 650° C.

Composition.		Cold Rolled.		Hot Rolled.	
Sb, %.	S, %.	Surface Cracking.	Elongation, %.	Surface Cracking.	Elongation, %.
0·004	0·004	None.	74	None.	59
0·06	0·002	Slight.	59	None.	74
0·10	0·002	Bad.	49	Slight.	65
0·23	0·014	Bad.	16	Bad.	44
0·005	0·006	None.	67	None.	66
0·004	0·015	None.	74	Slight.	45
0·004	0·026	Slight.	50	Slight.	58
0·005	0·032	Slight.	44	Bad.	32
0·02	0·011	None.	61	Slight.	56
0·04	0·024	Slight.	62	Slight.	48
0·06	0·054	Slight.	29	Slight.	27
0·18	0·12	Bad.	6	Bad.	25

the effect of antimony. The sulphur contents of commercial alloys are, however, insufficient to account for the divergent results reported in the literature on the effect of antimony.

Mechanical Properties.

Izod impact tests were performed on some of the alloys of series (*a*) and (*b*) in the cast state and also after pre-annealing for 1 hr. at 750° C. followed by water-quenching. The results are given in Table V.

TABLE V.—*Results of Izod Impact Tests on Cast and Pre-Annealed 70 : 30 Brass.*

Antimony Series (*a*).											
Sb, %.	0	0·01	0·015	0·02	0·025	0·05	0·075	0·10	0·12	0·15	0·20
As-cast	36(*u*)	34(*u*)	32(*u*)	35(*u*)	34(*u*)	15(*f*)	10(*f*)	6(*f*)	11(*f*)	3(*f*)	5(*f*)
Pre-annealed	35(*u*)	36(*u*)	34(*u*)	36(*u*)	36(*u*)	32(*u*)	32(*u*)	31(*u*)	33(*u*)	14(*f*)	16(*f*)
As-cast / Pre-annealed, %	102	96	94	98	96	47	31	19	33	23	32
Antimony plus lead series (*b*).											
Sb, %.	0	0	0	0	0·025	0·03	0·03	0·03	0·075	0·075	0·06
Pb, %.	0	0·02	0·05	0·10	0	0·02	0·05	0·12	0	0·02	0·12
As-cast	36(*u*)	34(*u*)	35(*u*)	32(*u*)	34(*u*)	23(*p*)	19(*f*)	21(*p*)	10(*f*)	9(*f*)	16(*f*)
Pre-annealed	35(*u*)	31(*u*)	34(*u*)	34(*u*)	36(*u*)	30(*u*)	34(*u*)	33(*u*)	32(*u*)	34(*u*)	30(*u*)
As-cast / Pre-annealed, %	103	110	103	94	94	77	56	64	31	27	53

(*u*) Unbroken.
(*p*) Partial fracture.
(*f*) Complete fracture.

Many of the specimens, including pure 70 : 30 brass, absorbed 32–34 ft. lb. of energy without fracturing. Presumably the test does not discriminate until embrittlement reaches a certain level. It is clear, however, that antimony much in excess of 0·025% embrittles cast brass very markedly, that this embrittlement is reduced by pre-annealing, and that lead has a much smaller effect than antimony. These results accord with the cold-rolling behaviour.

In order to determine the effect of antimony and lead on the tensile and bend properties of rolled but crack-free brass, tests were carried out on samples from which the surface layers containing cracks had been removed by machining. Duplicate transverse specimens were prepared from annealed sheet; 0·06 in. was removed by machining from each side, leaving a specimen 0·08 in. thick originally the middle zone of the ingot. Analyses made on the parallel portion of some of these specimens showed the antimony content to be 20–30% higher than the corresponding values for the full thickness in Tables I and VI but the lead content

was unaffected. The gauge length of the tensile specimens was 2 in. and the width ½ in. Since the tensile tests showed the surface machining to have decreased the ductility slightly, the bend specimens were machined before annealing. The results are given in Table VI.

TABLE VI.—*Transverse Tensile and Bend Properties.*

Annealed 1 hr. at 650° C. and air-cooled.

Composition.		Cold Rolled.				Hot Rolled.			
Sb, %.	Pb, %.	U.T.S., tons/sq. in.	Elong., % on 2 in.	Redn. in Area, %.	Bend.*	U.T.S., tons/sq. in.	Elong., % on 2 in.	Redn. in Area, %.	Bend.*
nil.	nil.	20·7	61	66	24	19·1	72	68	26·3
0·05	nil.	19·6	69	67	26·7	18·3	74	67	30
0·06	0·01	19·9	67	59	22	18·4	69	68	...
nil.	0·02	20·7	61	66	23·5	20·1	68	64	27
0·02	0·02	19·8	63	63	...	19·6	48 †	60	...
0·06	0·02	19·4	70	58	22·7	19·0	69	68	25·3
0·075	0·02	19·4	67	58	16·3	19·0	70	66	14
nil.	0·05	20·4	59	64	...	20·1	66	65	...
0·01	0·05	20·0	60	66	21	19·6	65	64	...
0·03	0·05	19·9	66	65	18·6	19·4	70	68	19·3
0·05	0·05	20·0	66	66	19	...	...	...	21
0·08	0·05	...	...	...	11·5	19·5	73	68	15·7
nil.	0·09	20·7	60	64	...	...	...	...	...
0·03	0·12	20·1	61 †	60	19·3	19·7	68 †	61	20·7
0·06	0·12	20·1	46 †	61	17·3	19·4	74	62	18·7

* Number of complete reversals in bend test.
† Broke outside the gauge length.

Thus, antimony alone reduces strength and increases ductility and bend value, while lead alone has the opposite tendency, but to a smaller extent. When present together the two elements tend to nullify each other's effect on tensile properties over the range of compositions tried. The combined effect on bend value is more adverse, 0·02% of lead eliminating the improvement in bend value brought about by 0·05% of antimony.

Vickers diamond pyramid hardness tests were made on the rolled surface of sheet which had been annealed for 1 hr. at 650° C. The results for cold- and hot-rolled sheet were almost identical. Antimony slightly reduced and lead slightly increased the hardness and, together, the elements tended to cancel each other's effects.

Average grain-sizes were determined on the transverse cross-section

of the same specimens. Antimony tended to increase and lead to decrease the grain-size. Grain-sizes of hot-rolled sheet were 10–20% larger than those of cold-rolled material. The reduction in grain-size due to sulphur was noticed to be quite considerable but no quantitative measurements were made.

Part II.—Brass Containing Antimony.

Influence of Heat-Treatment on the Effect of Antimony on the Mechanical Properties of 70 : 30 *Brass.*

To determine the influence of the rate of cooling from the annealing temperature, bend, tensile (both on "thinned" specimens), and Izod impact tests were carried out on samples which had been water-quenched or furnace-cooled after heating for 1 hr. at 650° C. The furnace required 6 hr. to cool through the range 450°–200° C., the rate of cooling at 450° C. being $1\frac{1}{3}$° C./min. and at 200° C., $\frac{1}{3}$° C./min. The

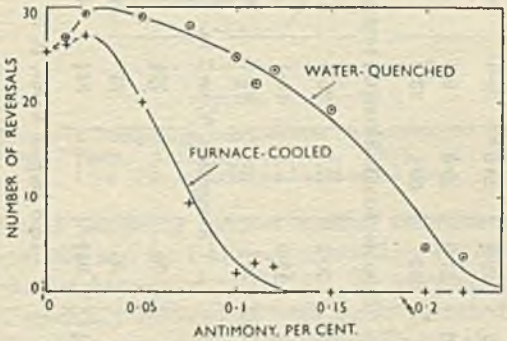


Fig. 2.—Influence of Rate of Cooling and Antimony Content on Embrittlement, as Shown by Bend Tests.

results of all tests are given in Table VII and the bend values (average of three tests) are plotted in Fig. 2.

It is clear that the rate of cooling has a pronounced influence on the effect of antimony, particularly in the bend test. Deterioration in properties of slowly cooled specimens was first noticeable with the tensile or impact tests at 0·075% of antimony and with the bend test at 0·05% of antimony. Quenching from the annealing temperature gave good results with all tests up to about 0·12% of antimony. The bend value of both water-quenched and furnace-cooled material is actually mproved by the presence of small quantities of antimony, say 0·01 to 0·02%. Micro-examination of transverse sections through the fracture

TABLE VII.—*Influence of Rate of Cooling on Embrittlement.*

Treatment.	Antimony, %.										
	Nil.	0·01	0·02	0·05	0·075	0·10	0·11	0·12	0·15	0·20	0·22
	Bend test, average number of reversals.										
(*A*) Water-quenched from 650° C. . . .	25·3	26·7	29·3	29·0 *	28·0	24·7	22·0	23·3	19·3	4·7	3·7
(*B*) Furnace-cooled from 650° C. . . .	25·3	26·0	27·0	20·0	9·3	2·0	3·0	2·7	0	0	0
Ratio $\frac{B}{A}$, % . .	100	97½	92	69	33	8	13½	11½	0	0	0
	Ultimate Tensile Strength, tons/sq. in.										
(*A*) Water-quenched from 650° C. . . .	20·1	20·0	20·2	...	20·0	19·7	...	...	...	14·5	...
(*B*) Furnace-cooled from 650° C. . . .	19·7	19·9	19·9	...	19·4	18·3	...	...	...	8·4	...
Ratio $\frac{B}{A}$, % . .	98	99½	98½	...	97	93	...	...	...	58	...
	Elongation, % on 2 in.										
(*A*) Water-quenched from 650° C. . . .	70	71	70	...	72½	73	...	...	...	28	...
(*B*) Furnace-cooled from 650° C. . . .	70	70	66	...	61	50	...	...	...	11	...
Ratio $\frac{B}{A}$, % . .	100	98½	94½	...	84½	68½	...	...	...	39½	...
	Izod Impact Value, ft. lb.										
(*A*) Water-quenched from 650° C. . . .	41·5(*b*)	41·1(*b*)	43·0(*b*)	41·6(*b*)	41·9(*b*)	...	44·0(*b*)	43·1(*b*)	...	35·2(*p*)	...
(*B*) Furnace-cooled from 650° C. . . .	41·9(*b*)	42·0(*b*)	42·2(*b*)	42·1(*b*)	40·9(*b*)	...	27·0(*f*)	22·5(*f*)	...	3·9(*f*)	...
Ratio $\frac{B}{A}$, % . .	101	102	98	101	98	...	61	52	...	11	...

(*b*) Bent. (*p*) Partial fracture. (*f*) Complete fracture. * Air-cooled: 26·7 = 92% of (*A*).

showed that grain-boundary cracking and fracture had occurred in the embrittled specimens. Figs. 7, 8, and 9 (Plate LXIV) illustrate the transcrystalline fracture in the water-quenched, and the intercrystalline fracture and cracking in the furnace-cooled, specimens, both containing 0·11% of antimony.

One series of " thinned " specimens, containing 0·11% of antimony, was annealed at 650° C. for 1 hr., water-quenched, and tempered for 1 hr. at various temperatures, while another series was furnace-cooled from 650° C. and tempered. The bend values are plotted in Fig. 3.

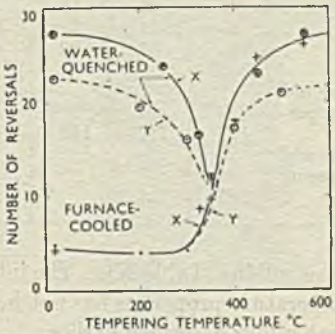


Fig. 3.—Influence of Tempering Temperature on Antimonial Embrittlement, as Shown by Bend Tests. Antimony content, 0·11%. *X*: Hot-rolled sheet. *Y*: Pre-annealed and cold-rolled sheet.

It is clear that tempering induces marked embrittlement in water-quenched material and for 1-hr. treatments the most harmful temperature is in the neighbourhood of 350° C. At temperatures above this, ductility is gradually restored in both water-quenched and furnace-cooled specimens, demonstrating the reversibility of the embrittlement process.

It was expected that embrittlement at low tempering temperatures would be accentuated by prior straining, so cold-worked samples in the form of Izod notched bars were tested, with the results given in Table VIII.

Antimony induces marked embrittlement, which is manifest at lower tempering temperatures than for the unstrained bend specimens of Fig. 2. The specimens tempered at 350° C. recrystallized to a fine grain-size and consequently the resistance to impact of those of lower antimony content was increased by this treatment.

Information relating to the onset of brittleness as a function of temperature and antimony content was provided by the various results of mechanical tests performed at room temperature on heat-treated

samples. A curve showing the onset of brittleness is drawn in Fig. 4. At high temperatures it was fixed approximately by the results of bend

TABLE VIII.—*Resistance to Impact (ft. lb.) of Quenched and Cold-Worked Antimonial Brass after Tempering.*

Tempering Treatment.	Antimony, %.					
	Nil	0·025	0·05	0·075	0·11	0·20
None	47	45	46	43	30	6
1 hr. at 150° C. . . .	47	42	44	41	26	5
1 hr. at 250° C. . . .	47	45	38	35	21	5
1 hr. at 350° C. . . .	65	63	60	46	23	3
Vickers diamond pyramid hardness before tempering	178	182	180	179	182	186
Vickers diamond pyramid hardness after tempering at 350° C. . . .	85	...	...	...	89	87

tests made to locate the solidus (Table X). The influence of antimony on the actual high-temperature properties has not been examined, apart from the hot-rolling behaviour described earlier.

Stress–Strain Behaviour.

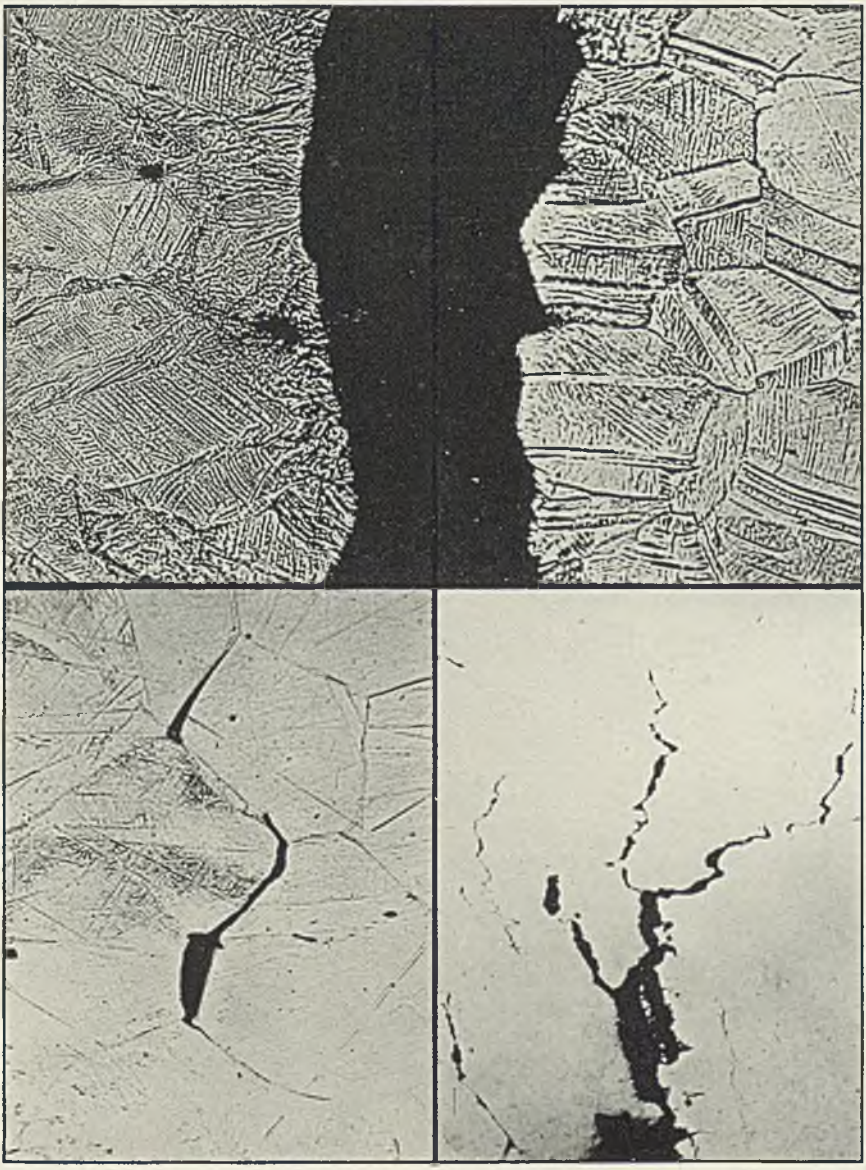
The stress–strain relationships of four alloys were examined to obtain an indication of their deep-drawing behaviour. The alloys were pure 70 : 30 brass and brass containing, respectively, 0·04% of antimony, 0·04% of lead, and 0·04% each of antimony and lead. The test specimens were annealed to the same grain-size and tested in the water-quenched condition.

When plotted on the same graph, the stress–strain curves overlapped almost up to the maximum-load stages: beyond this point the lead-bearing brass showed slightly lower elongation. Judging from these tests, therefore, 0·05% of antimony in solid solution should have a negligible effect on deep-drawing behaviour.

Structure.

Lead decreased the crystal size of as-cast brass, but antimony and sulphur had little effect.

Under the microscope, a pale-blue constituent shown in Fig. 11 (Plate LXV) was found in the antimonial brasses; it first appeared at an antimony content of 0·02–0·05% in the cast ingots, the limiting content



Fracture in Bend-Test Specimens Containing 0·11% of Antimony.

FIG. 7.—Water-quenched from 650° C. Transcrystalline (bend value 22). × 250.
FIG. 8.—Furnace-cooled from 650° C. Intercrystalline (bend value 3). × 250.

Intercrystalline Cracking.

FIG. 9.—Intercrystalline cracking near fracture of Fig. 8 specimen. × 500.
FIG. 10.—Intercrystalline cracking in cold-rolled ingot containing 0·22% of antimony. × 20.

[*To face p.* 598.

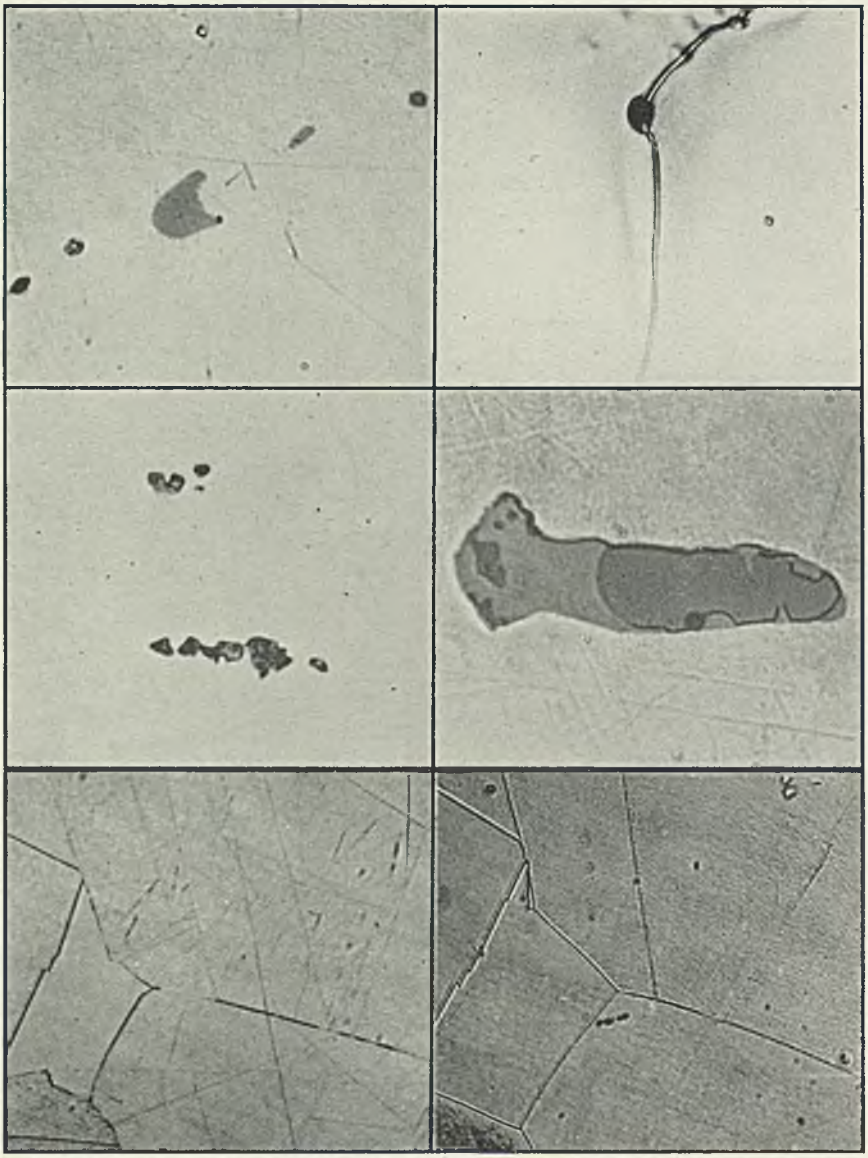


FIG. 11.—Pale-Blue Copper–Antimony Compound in Cast Brass Containing 0·10% of Antimony. × 2000.

FIG. 12.—Grain-Boundary-Film Appearance in Same Specimen as Fig. 11. × 500.

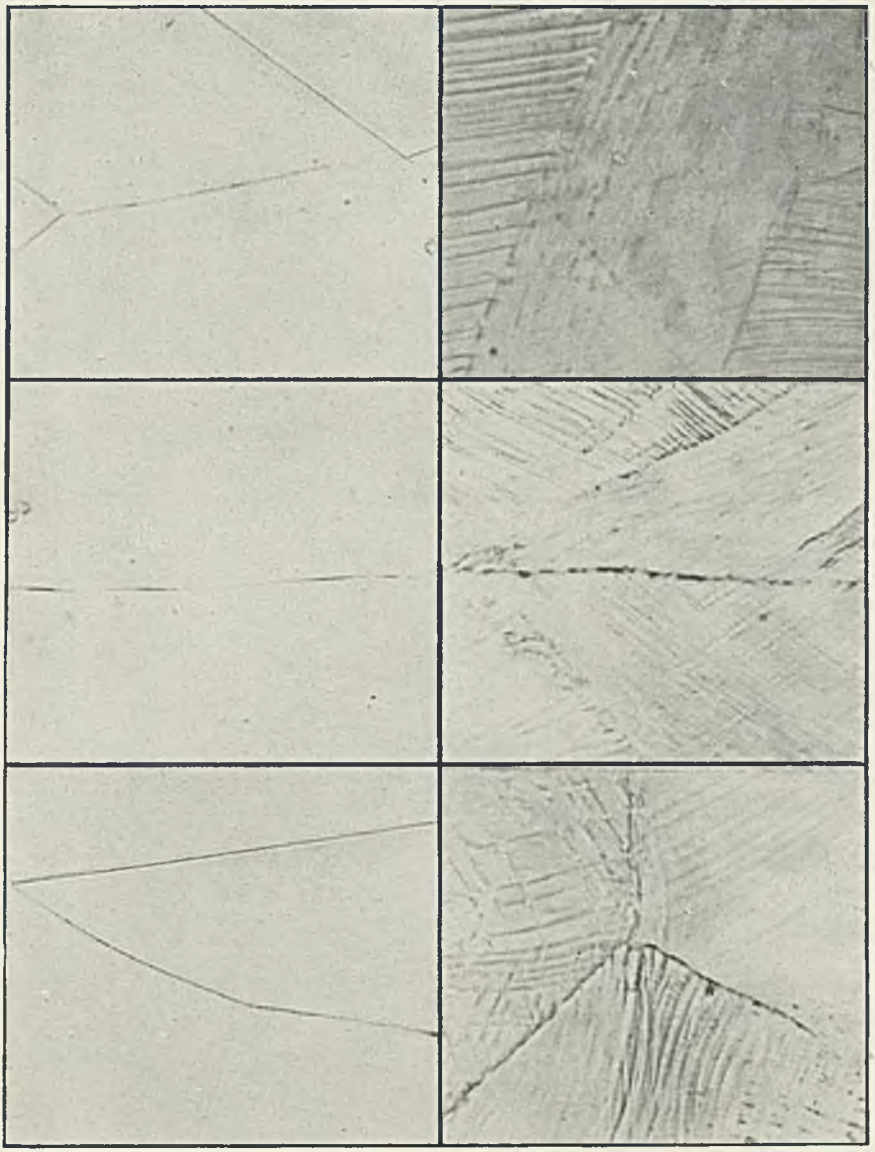
FIG. 13.—Sulphide Particles in Rolled Brass Containing 0·12% of Sulphur. × 2000.

FIG. 14.—Phosphorus–Antimony Compound (dark) in Copper–Antimony Compound. Cast brass containing 0·11% of antimony and 0·041% of phosphorus. × 2000.

Brass Containing 0·15% of Antimony, Water-Quenched from 650° C., and Heated 1 week at 200° C. Both etched in ammonium persulphate.

FIG. 15.—Hand-polished. × 500.

FIG. 16.—Electrolytically polished. × 500.



Discontinuous Precipitate in Antimonial Brass Free from Lead and Sulphur.
Electrolytically polished and etched. All × 2000.

Rolled and Annealed.

FIG. 17.—0·10% of antimony, furnace-cooled from 650° C.

FIG. 18.—0·10% of antimony, water-quenched from 650° C., heated 1 hr. at 250° C.

FIG. 19.—0·024% of antimony, furnace-cooled from 650° C.

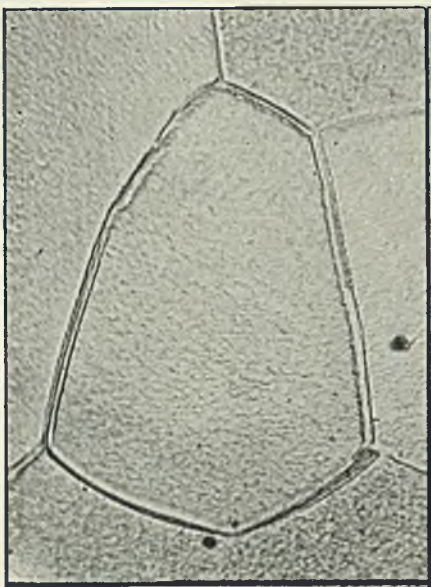
Water-Quenched from 650° *C.*; *Cold Rolled*, 40% *Reduction*; *Heated* 1 *hr. at* 250° *C.*

FIG. 20.—Antimony nil. No precipitate.

FIG. 21.—0·024% of antimony.

FIG. 22.—0·11% of antimony.

23



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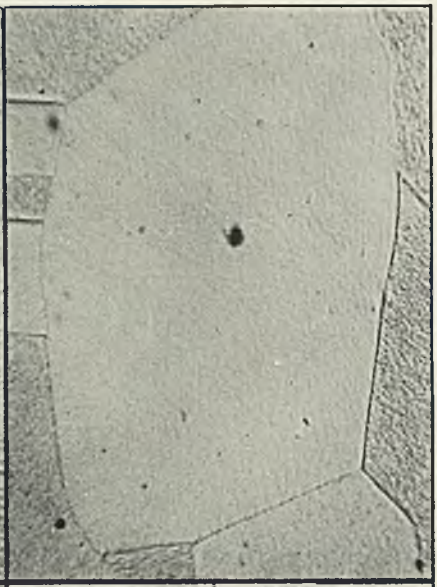

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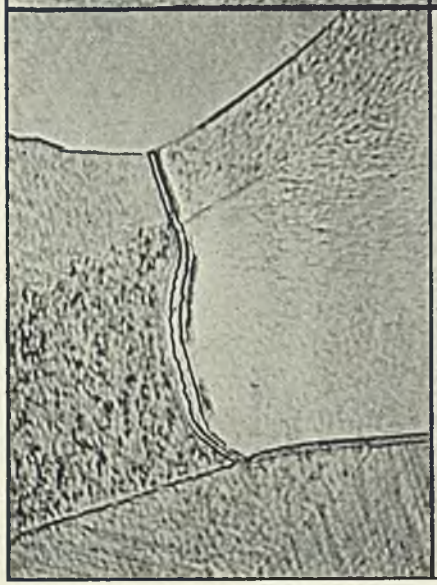

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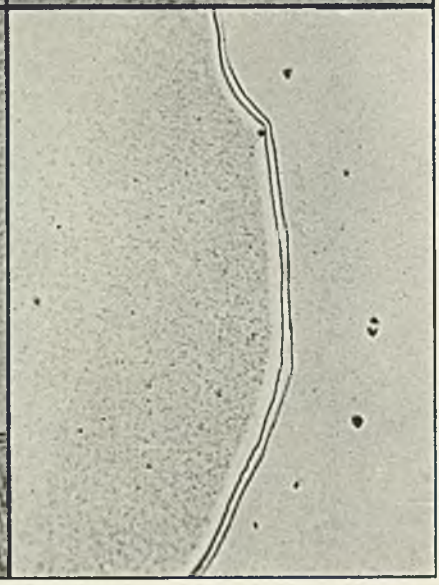

Effect of Rate of Cooling on Grain-Boundary Etch; Brass containing 0·11% of Antimony (Lead and sulphur not present).

FIG. 23.—Furnace-cooled from 650° C. (Izod impact, 27 ft. lb., complete fracture.) × 1000.
FIG. 24.—Water-quenched from 650° C. Same etch as Fig. 23. (Izod impact, 44 ft. lb., no fracture.) × 1000.

Continuous Grain-Boundary-Film Appearance in Furnace-Cooled Brass Containing Antimony (Lead and Sulphur not present).

FIG. 25.—0·10% of antimony. × 1000. FIG. 26.—0·22% of antimony. × 1250.

not being regularly affected by lead or sulphur. It was not, however, present in the rolled or annealed sheet having the maximum content of 0·22% of antimony. The pale-blue constituent was similar in appearance to the β- and ε-phases of the copper–antimony system.[21–25] Thermal analysis confirmed that it is based on these phases, since cast 70 : 30

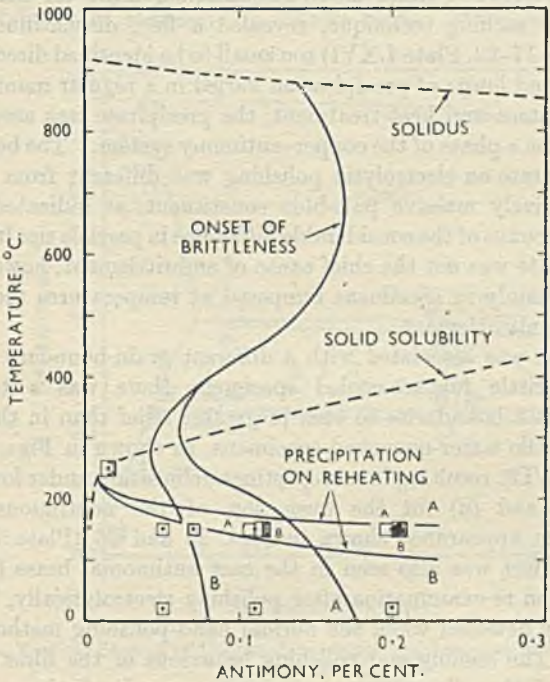

FIG. 4.—Precipitation on Reheating for 1 hr. after Quenching from 650° C. and Onset of Brittleness. (Key as for Fig. 5.) *A*: Annealed. *B*: Cold rolled, 40% reduction.

brass containing 2·3% of antimony showed arrests on heating at 463° and 597° C., corresponding to the β ⇌ ε transformation and melting of the α–β-eutectic, respectively, found to occur in a copper–18% antimony alloy at 466° and 635° C. The eutectic temperature in the copper–antimony system at about 32% of antimony is thus lowered by addition of zinc, but the transformation temperature of the compound is hardly affected.

Lead occurred as globules and could be detected when 0·005% was present. Sulphur occurred as a dark-grey, angular constituent (Fig. 13, Plate LXV) in all samples containing not less than 0·004% of sulphur.

Compared to lead, a small amount of sulphur produced a large number of particles, the quantity and appearance being unaffected by rolling and annealing.

The evidence so far presented suggests that the solubility of antimony diminishes with temperature, causing grain-boundary precipitation of a brittle constituent. Careful micro-examination, using an electrolytic polishing and etching technique, revealed a fine, discontinuous precipitate (Figs. 17–22, Plate LXVI) too small to be identified directly. As the amount and limits of precipitation varied in a regular manner with antimony content and heat-treatment, the precipitate was assumed to be based on the ε-phase of the copper–antimony system. The behaviour of the precipitate on electrolytic polishing was different from that of the comparatively massive pale-blue constituent, as indicated below, perhaps on account of the considerable difference in particle size involved. This precipitate was not the chief cause of embrittlement, however, for it occurred mainly in specimens tempered at temperatures too low to cause much embrittlement.

Brittleness was associated with a different grain-boundary appearance. In brittle furnace-cooled specimens there was a tendency (i) for the grain boundaries to etch in greater relief than in the corresponding ductile water-quenched specimens, as shown in Figs. 23 and 24 (Plate LXVII), resulting in more distinct delineation under low-power examination and (ii) for the formation of the continuous grain-boundary-film appearance shown in Figs. 25 and 26 (Plate LXVII). This latter effect was also seen in the cast antimonial brass (Fig. 12, Plate LXV) on re-examination after polishing electrolytically, but was not originally detected when the normal hand-polishing methods were employed. The etching and polishing behaviour of the films differed from that of the discontinuous precipitate and of the pale-blue constituent. The differences are summarized as follows:

Treatment.	*Nodular Pale-Blue.*	*Discontinuous.*	*Film.*
(i) Hand-polish; no etch.	Pale-blue.	Not detectable.	Not detectable.
(ii) Electrolytic polish.	Dissolved out.	Not detectable.	Not detectable.
(iii) Electrolytic etch.	Dissolved out.	Blackened (Figs. 17–22).	Not detectable with certainty.
(iv) Ammonium persulphate etch.	Darkened.	Obscured by the general etching which occurs.	Detectable with more certainty (Figs. 25 and 26).

Further, the discontinuous precipitate could be consistently detected in an appropriately treated specimen containing as little as 0·02% of antimony, whereas films were not consistently revealed, even with

0·2% antimony content. These observations raise doubts about the nature of the films.

Copper–Zinc–Antimony Constitution.

The solid solubility of the discontinuous precipitate has been determined up to 350° C. and the results are given in Figs. 5 and 6 as the solid solubility of antimony in 70 : 30 brass. Three series of heat-

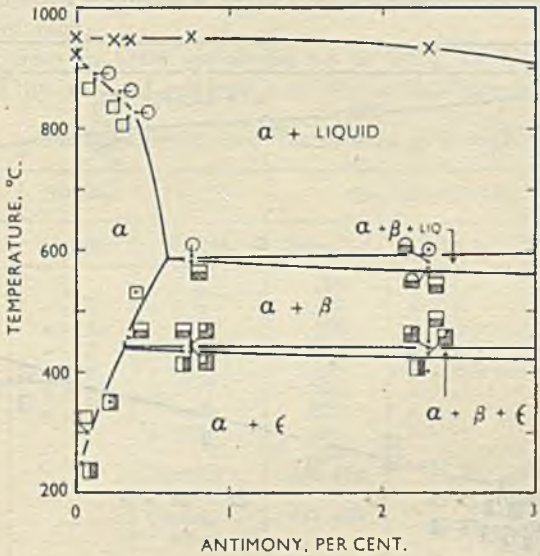

FIG. 5.—Vertical Section of Copper–Zinc–Antimony Diagram at Copper : Zinc Ratio of 70 : 30.

KEY.

× THERMAL ARREST
α SOLID SOLUTION
α + LIQUID
α + ε
α + β + LIQUID
α + β
α + β + ε

treatments were performed: (1) specimens were heated for 1 hr. at different temperatures after a preliminary homogenization of 1 hr. at 650° C. and then water-quenched, (2) cold-worked specimens were heated for 1 hr. at different temperatures (comparison with series (1) shows the effect of prior straining—see lines *A* and *B* in Fig. 4), (3) specimens were homogenized at 600°–700° C. for 1–2 hr., furnace-cooled to temperature, and held for one week.

The solubility at 200° C. does not exceed 0·015% of antimony. Below 250° C. precipitation is accelerated by prior cold work. The discontinuous precipitate occurred only at grain boundaries except possibly in the strained specimens, where a suggestion of it was present along strain lines. The specimens treated for one week at the lower temperatures contained approximately the same amount of precipitate as the corresponding specimens treated for 1 hr., but one week at the higher temperatures resulted in less discontinuous precipitate. Where a

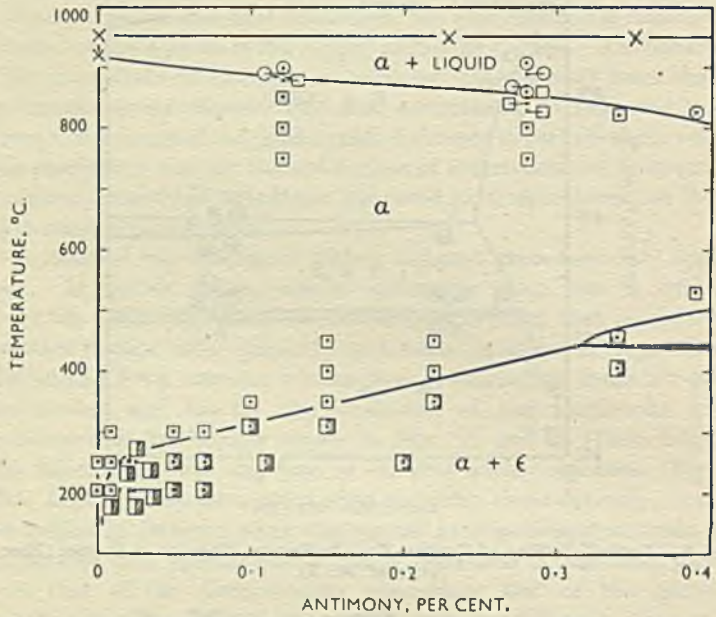

FIG. 6.—Enlarged Version of the Low-Antimony Portion of Fig. 5. (Key as for Fig. 5.)

specimen treated for 1 hr. contained a precipitate and the corresponding specimen treated for one week did not, the point concerned on the equilibrium diagram is considered to be in the two-phase region, in accordance with the hypothesis advanced in the discussion.

To locate the solid-solubility curve above 350° C., heat-treatments were carried out on the alloy containing 0·35% of antimony. Specimens were cold reduced approximately 20% and heated for 14 or 28 days at various temperatures. A layer $\frac{1}{32}$–$\frac{1}{16}$ in. in thickness was removed from the surface to be micro-examined. Precipitate, when present, was in the form of fine dots fairly uniformly distributed. The specimens

were then analysed. The results of the examination are plotted in Fig. 6.

The temperature ranges of the β–ε transformation * and the eutectic were determined by thermal curves and micro-examination of samples of the alloys containing 0·7 and 2·3% of antimony, quenched after a 1-hr. anneal. The results are given in Table IX. The small amount of the blue compound in the lower antimony alloy when quenched from near the eutectic temperature located the solid-solubility limit at about 0·6% of antimony.

The liquidus in brass containing up to 2·3% of antimony was determined by thermal analysis and the solidus mainly by micro-

TABLE IX.—*Thermal Arrests and Constitution of Quenched Specimens.*

Eutectic.			Transformation.		
Temp., ° C.	0·7% Sb.	2·3% Sb.	Temp., ° C.	0·7% Sb.	2·3% Sb.
620	...	α + L	480	...	α + β
600	α + L	α + L	470	α + β	...
590	α + L	...	450	α + β	α + β
580	α + β	α + β + L	445	α + β + ε	...
570	α + β	α + β + L	440	α + β + ε	α + β + ε
560	α + β	α + β	435	α + ε	...
550	α + β	...	430	α + ε	α + β + ε
540	...	α + β	410	α + ε	...
			400	...	α + ε
576–602	...	Heating arrest.	446–474	...	Heating arrest.
556–592	...	Cooling arrest.	447–400	...	Cooling arrest.

examination, giving the curves shown in Figs. 5 and 6. Corrections for deviations from the 70 : 30 copper–zinc ratio were applied from Hansen's [21] diagram for the copper–zinc system; for 1% excess zinc 1° C. was added to the liquidus and 6° C. to the solidus. A few bend tests were performed on quenched specimens, giving the results shown in Table X. The results are in agreement with the microscopic work.

Comparison of the present work on 70 : 30 brass with that reported in the literature on pure copper shows that 30% of zinc reduces both the antimony solubility and the antimony content required to cause a given deterioration in properties of the α-phase, by a factor of 10–100. As mentioned in the review of literature, there is evidence that higher zinc contents operate still further in the same direction. Comparison of previous work [6, 11, 12] with the present investigation indicates that the

* This refers to the phase based on the β-constituent in the copper–antimony system and not to β-brass.

amount of antimony required to cause a given effect in 60 : 40 brass is of the order of $\frac{1}{5}$th of that which would be required in 70 : 30 brass; the solubility of antimony in 60 : 40 brass is, therefore, considered to be about $\frac{1}{5}$th of that in 70 : 30 brass.

Electrical-conductivity measurements were made on annealed samples, water-quenched or furnace-cooled from the annealing tempera-

TABLE X.—*Results of Bend Tests on Quenched Specimens to Determine the Solidus.*

Average number of reversals.

Sb, %.	Quenching Temperature, ° C.					
	500	650	750	800	850	900
0·025	...	29·3	...	25·3	...	25·7
0·12	...	23·3	22·7	21·7	18·0	17·8
0·20	6·2	8·7	11·7	11·7	8·2	0

ture. The electrical conductivity was reduced progressively by increases in antimony content (6% by an addition of 0·2% of antimony) and was slightly higher in the furnace-cooled samples, but the difference did not increase with antimony content between the two treatments. This indicates either a high resistance of the continuous intercrystalline film, or that the latter is not a true precipitate.

PART III.—ADDITIONS TO OVERCOME THE HARMFUL EFFECT OF ANTIMONY.

Influence of Phosphorus, Lithium, and Mould Dressing.

It was thought that the harmful effects of antimony could be avoided by addition elements which formed with it innocuous stable compounds. Elements forming antimonial compounds of higher melting point than Cu_2Sb (681° C.) seemed promising ones to consider. Reference to Hansen [21] showed the following to be in this category: Li, Na, K, Mg, Al, Ni, Co, Fe, Mn, and Pt. Since antimony is weakly electronegative, the strongly electropositive members of this list seemed particularly promising. A rough test confirmed this for lithium but not for sodium as none could be introduced into a melt, probably owing to the low boiling point of sodium (880° C.). There are disadvantages in the use of some of the other elements, none of which was tried.* It

* After this work had been completed a patent specification [31] was received, claiming that nickel or cobalt added to β or $\alpha + \beta$ copper–zinc-base alloys offsets the adverse effects of antimony and arsenic.

has been reported that phosphorus is beneficial in this and similar applications [7, 32, 33] and its beneficial effect on antimonial 70 : 30 brass was confirmed by a rough test. Lithium and phosphorus were, therefore, selected for more extensive trials.

It had also been suggested * that a finer grain-size and greater dispersal of any second phase might result from casting into a mould coated with a flaming dressing. Two series of castings were, therefore,

TABLE XI.—*Chemical Composition of Ingots in Phosphorus and Lithium Series.*

Made up :			P or Li : Sb Ratio (atomic).	Analysed :			
Sb, %.	P, %.	Li ,%.		Cu, %.	Sb, %.	P, %.	Li.†
0·025	nil.	nil.	Sb only.	69·93	0·025	nil.	...
			P : Sb.				
0·025	0·0031	nil.	1 : 2	69·6	0·03	0·0024	...
0·025	0·0062	nil.	1 : 1	70·2	0·02	0·006	...
0·025	0·0125	nil.	2 : 1	70·0	0·02	0·011	...
			Li : Sb.				
0·025	nil.	0·0021	3 : 2	69·5	0·025	...	...
0·025	nil.	0·0043	3 : 1	70·4	0·02	...	...
0·025	nil.	0·0086	6 : 1	70·02	0·03	...	... §
0·10	nil.	nil.	Sb only.	70·19	0·11	nil.	...
			P : Sb.				
0·10	0·0125	nil.	1 : 2	69·4	0·11	0·010	...
0·10	0·025	nil.	1 : 1	69·3	0·12	0·025	...
0·10	0·05	nil.	2 : 1	68·9	0·11	0·041	...
nil.	0·05	nil.	P only.	70·9	nil.	0·040	...
			Li : Sb.				
0·10	nil.	0·0086	3 : 2	69·5	0·12	...	... §
0·10	nil.	0·017	3 : 1	70·08	0·10	...	... §
0·10	Flaming dressing used in mould.		Sb only.	70·2	0·11	nil.	...

† It was not found possible to determine the lithium contents accurately. Spectrographic analysis gave possible limits of 0·005 and 0·01% lithium for those marked §, although the order was uncertain. This suggests considerably greater loss on melting than the 30% anticipated, but for simplicity the intended Li : Sb atomic ratios are used.

prepared, of constant antimony content of 0·1%, but of varying phosphorus and lithium contents; and a further single casting of 0·1% antimony content was made in a mould coated with a flaming dressing. The compositions by weight and the approximate atomic compositions are given in Table XI, which includes phosphorus and lithium series, based on 0·025% of antimony, for Izod tests described later.

* Thanks are due to Mr. G. L. Bailey, British Non-Ferrous Metals Research Association, for this suggestion.

The grain-sizes of the antimony-free phosphorus composition and the ingot cast with a flaming dressing were about one-half and one-third, respectively, that of the other alloys.

Rolling Behaviour.

The rolling schedules were similar to those employed for series (*a*) and (*b*) (see Part I, p. 588), except that the cross-flattening pass was omitted. Probably for this reason no fire-cracks occurred.

Phosphorus considerably reduced surface- and edge-cracking during cold rolling of as-cast ingots, but increased it during hot rolling and also slightly increased it during cold rolling of pre-annealed ingots. The improvement on cold rolling was most marked up to the phosphorus–antimony ratio of 1 : 1. Lithium considerably reduced surface- and edge-cracking during both hot and cold rolling, in the latter case whether

TABLE XII.—*Influence of Phosphorus, Lithium, and Mould Dressing on Cracking during Rolling, as Indicated by Bend Tests on Unmachined Annealed Specimens.*

Antimony content 0·1%.

Treatment.	Number of Reversals in Bend Test.								
	P and Li nil.	Phosphorus Series. P : Sb Ratio (atomic).				Lithium Series. Li : Sb Ratio (atomic).			Flaming Dressing.
		1 : 2.	1 : 1.	2 : 1.	Sb nil.	3 : 2.	3 : 1.	3 : 1, annealed at 800° C.	
Cold rolled .	4½	7½	13	11½	14	6	4½	7½	6
Pre-annealed and cold rolled .	13	13	13	10½	16½	9	6	6	14
Hot rolled .	14	11½	15	13	18	11	5	11	14½

pre-annealed or not. The improvement on cold rolling with the lithium–antimony ratio of 3 : 1 was similar to that with the phosphorus–antimony ratio of 1 : 1. The casting prepared with a flaming dressing contained more, but finer, cracks after all three treatments than did its soot-dressing counterpart.

To obtain a quantitative measure of surface cracking, transverse bend-test specimens were tested after annealing followed by rapid cooling. The results are given in Table XII.

The results for the phosphorus series and for the alloy cast with a

flaming dressing agree with the observed rolling behaviour. The results for the lithium series, however, do not. The worse results in each pair are given by the higher lithium-content material, which rolled with least apparent cracking. Only part of this discrepancy may be accounted for by the small grain-size of this material, since tests on further bend specimens of normal grain-size produced by annealing at 800° C. still gave poor results as shown in Table XII.

Influence of Heat-Treatment.

Two further sets of bend-test specimens were prepared, with surfaces machined free from cracks as described earlier: one set was water-quenched from the annealing temperature of 650° C. and the other

TABLE XIII.—*Influence of Phosphorus, Lithium, and Mould Dressing on Embrittlement During Slow Cooling.*

Antimony content 0·1%.

Treatment.	Number of Reversals in Bend Test.								
	P and Li nil.	Phosphorus Series. P : Sb Ratio (atomic).				Lithium Series. Li : Sb Ratio (atomic).			Flaming Dressing.
		1 : 2.	1 : 1.	2 : 1.	Sb nil.	3 : 2.	3 : 1.	3 : 1, annealed at 800° C.	
Water-quenched.	24·7	21·8	23·3	21·6	22·6	25·0	11·9	13·1	22·3
Furnace-cooled .	3·4	12·4	16·5	18·6	22·2	2·5	10·6	9·2	5

furnace-cooled. As the results and particularly their trend were nearly independent of the type of rolling, mean values are given in Table XIII for each composition and heat-treatment.

Increasing phosphorus content progressively eliminates the embrittlement occurring on furnace-cooling at the expense of only a slight reduction in the bend value of the water-quenched material. Lithium above 0·01% also eliminates this embrittlement, but apparently only at the expense of a big reduction in the bend values of the water-quenched alloy. The figures in the penultimate column show that small grain-size is not responsible for the poor value of the water-quenched material.

Quenched, strained, and tempered Izod specimens were prepared in the same way as those already described in order to determine the

influence of the addition elements on embrittlement occurring on tempering strained material. A phosphorus and lithium series, based on 0·025% of antimony, was included. The results are given in Table XIV.

In the 0·025%-antimony series, the only low results are those for the maximum lithium ratio. With 0·11% of antimony, the results are complicated by the strain-embrittlement introduced by phosphorus, but the antimonial embrittlement is progressively reduced with increase in phosphorus content in specimens tempered at 350° C. In the annealed and furnace-cooled condition, the lowest phosphorus ratio of 1 : 2 has a considerably beneficial effect. Lithium introduces serious embrittlement of its own, although from the furnace-cooled specimens and those tempered at 350° C. it appears to reduce antimonial embrittlement. The specimens prepared with a flaming dressing are not significantly different from their soot-dressing counterparts.

Micro-Examination.

Three types of particle were found in the as-cast phosphorus brasses: (1) the pale-blue copper–antimony compound, possibly containing zinc, (2) a darker blue particle which increased in amount with increase in phosphorus and antimony and is probably a phosphorus–antimony compound, and (3) a still darker blue particle which occurs in the antimony-free alloy and in the others only at high phosphorus–antimony ratios and is, therefore, probably the copper–phosphide compound. Particles of types (1) and (2) frequently occurred together, as shown in Fig. 14 (Plate LXV). The compositions of the alloys examined and the occurrence of these three particles in them are shown in Table XV.

In the rolled and annealed condition, no particles were found other than a few inclusions of the angular dark-grey impurity observed in every alloy previously examined.

Two types of particle were observed, besides the impurity inclusions, in the as-cast lithium brasses: (1) the copper–antimony compound and (4) a rounded blue-grey type which, since it increases in quantity with increasing lithium and antimony content, is probably a lithium–antimony compound. The two types of particle frequently occurred together, much as in Fig. 14 (Plate LXV). The particles (4) readily blackened and tended to dissolve out during a polish attack with magnesium oxide and copper ammonium chloride and, in some cases, could then be confused with the impurity inclusions. Doubt was removed by examination between crossed polaroids, as the particles (4) then appeared blacker than the ground-mass. The alloys examined and

TABLE XIV.—*Influence of Phosphorus, Lithium, and Mould Dressing on Embrittlement of Samples Quenched, Cold Worked, and Tempered.*

Izod impact test results in ft. lb.

Antimony, %	0·025	0·03	0·02	0·02	0·025	0·021	0·030	0·11	0·11	0·12	0·11	nil.	0·12	0·11	0·11
Phosphorus, %	nil.	0·0024	0·006	0·11	...	...	...	nil.	0·01	0·025	0·041	0·04	...	...	nil.
Lithium, %	nil.	nil.	nil.	nil.	nil.	nil.	nil.	nil.	nil.	nil.	nil.	nil.	nil.	nil.	Flaming Dressing.
P or Li : Sb Atomic Ratio.	...	1 : 2.	1 : 1.	2 : 1.	3 : 2.	3 : 1.	6 : 1.	...	1 : 2.	1 : 1.	2 : 1.	...	3 : 2.	3 : 1.	
Tempering treatment:															
Room temperature (*a*)	45	41	37	44	43	42	24	30	33	29	22	32	29	12	34
1 hr., 150° C.	42	37	34	38	36	41	22	26	32	26	19	25	26	11	31
1 hr., 250° C.	45	37	35	33	36	39	22	21	22	23	19	25	14	9	17
1 hr., 350° C.	63	64	65	69	63	67	59	23	49	59	63	66	18	34	25
1 hr., 650° C. and furnace-cooled	41	46	47	48	44	47	46	27	46	48	52	53	19	44	26
Vickers diamond pyramid hardness numbers for (*a*)	182	180	178	179	187	192	191	182	182	188	193	200	187	190	186

the occurrence of particles in them in the as-cast state are given in Table XV.

In the rolled and annealed condition only the type (4) particles were observed. The impurity inclusions are, presumably, dissolved or decomposed by the lithium compound. The alloys examined in the rolled and annealed state and the occurrence of particles in them were

TABLE XV.—*Occurrence of Compounds in As-Cast Phosphorus and Lithium Alloys.*

Antimony Content, %.	Atomic % Ratio P : Sb and particles seen.					Atomic % Ratio Li : Sb and particles seen.			
	P nil.	1 : 2.	1 : 1.	2 : 1.	Sb nil.	Li nil.	3 : 2.	3 : 1.	6 : 1.
0·025	None.	None.	(1) or (2)	(2)	...	None.	(4)	(4)	(4)
0·11	(1)	(1) + (2)	(2)	(2) + (3)	(3)	(1)	(1) + (4)	(1) + (4)	...

KEY.

(1) = copper–antimony compound.
(2) = phosphorus–antimony compound.
(3) = copper–phosphorus compound.
(4) = lithium–antimony compound.

as for the as-cast specimens referred to in Table XV, except that none of type (1) was seen.

Grain-size and hardness measurements made on annealed specimens showed that phosphorus and lithium reduced grain-size and increased hardness.

PART IV.—DISCUSSION OF RESULTS.

The effect of a given amount of antimony on the mechanical properties of brass depends on three factors: heat-treatment, casting segregation, and zinc content. With homogeneous 70 : 30 brass, the chief precaution in practice, so far as heat-treatment is concerned, is to heat and cool rapidly through the dangerous range of temperature 200°–450° C. With 0·05% of antimony, uniformly distributed, air-cooling of single thin strips should be rapid enough, as the following results on 0·08-in.-thick bend specimens show:

	Method of Cooling.		
	Water-Quenched.	*Air-Cooled.*	*Furnace-Cooled.*
Number of reversals	29·0	26·7	20·0
Percentage of water-quenched value .	100	92	69

The second factor, namely, the segregation present in chill-cast ingots, is sufficient to render chill-cast material more brittle than

furnace-cooled material of the same antimony content, despite the more rapid cooling of the former; this can be seen from the Izod test results for cast material in Table V and for rolled, annealed, and furnace-cooled material in Table VII. It is probable that, if antimonial brass ingots can be satisfactorily broken down without the aid of pre-annealing, the antimony content is low enough to cause no trouble in subsequent operations. Turning to the third factor, the influence of zinc content is such as to suggest a reversal of previous policy on the disposal of antimonial brass scrap. This has been to use antimonial scrap in 60 : 40 melts, but with such a high zinc content antimony is extremely harmful. It seems preferable to use the scrap in high-copper melts, where the antimony contents involved would be well within the solid-solubility range and much less harmful.

The mechanism of antimonial embrittlement is not quite clear. The grain-boundary character of the cracking occurring in, and the fracture of, embrittled material show that an abnormal grain-boundary condition is responsible for brittleness. The discontinuous precipitate clearly plays only a minor rôle. Although a grain-boundary-film appearance has been detected in embrittled material, it is not considered certain that an ordinary precipitation mechanism is at work. There is some doubt as to whether the film appearance is a true precipitate or some other condition which cannot always be revealed under the microscope, such as a narrow zone of transformed lattice, or a pre-precipitation phenomenon. To summarize the evidence: on the one hand, facts which fit in with a precipitation hypothesis are the shape of the solid-solubility curve, the effect of heat-treatment on mechanical properties, and microscopical evidence. On the other hand, doubt is raised about the nature of the film by the peculiarities of etching behaviour and difficulty of detection described on p. 600, and also by the fact that the electrical-conductivity results do not show the increase in conductivity usually found when precipitation occurs. Despite this uncertainty, however, the assumption that the films are a brittle precipitate, identical in nature with the discontinuous precipitate but for some reason infrequently detected, provides a working hypothesis, namely, that in specimens slowly cooled or tempered above about 250° C. the precipitate becomes more continuous and naturally has an embrittling effect in proportion to its continuity.

There is an interesting comparison between this grain-boundary effect in brass and temper-brittleness in steels, where slow cooling through, or heating in, a dangerous range of temperature produces embrittlement of a type shown by the notched-bar impact test, the tensile properties being unaffected, whereas rapid cooling causes no

embrittlement. It is also of interest that the types of fracture in brass are similar to those in steel, namely, intercrystalline in the temper-brittle condition and transcrystalline in the non-temper-brittle condition. Further, a grain-boundary constituent has not been reliably detected in steel in the temper-brittle state. Similarity also exists with the embrittlement of copper by bismuth and possibly by selenium and tellurium.[34, 35]

The brittleness of antimonial brass in the cast state, as shown, for example, by the Izod test results in Table V, or by the intercrystalline cracking in Fig. 10 (Plate LXIV), is probably due to the same mechanism as grain-boundary embrittlement in recrystallized material. The rounded blue particles are unlikely to cause much harm, owing to their rounded shape and fairly uniform distribution. Antimony is so highly segregated in cast brass that, with an average antimony content of 0·02%, the blue constituent could often be seen and, therefore, the maximum segregated content in solution must have been, from Fig. 5, about 0·6%. In regions of such high antimony content the tendency to grain-boundary embrittlement must be very marked. Pre-annealing restores ductility by assisting homogenization and thus reducing the number and intensity of such regions.

The fire-cracking which occurred in the earlier cold-rolled ingots is assumed to be due to (i) embrittlement on heating through the dangerous temperature range, intensified by casting segregation and cold work combined with (ii) the transverse-tensile surface stress resulting from the final cross-rolling pass. Slow heating might be more harmful than rapid heating. Thus, Price and Bailey,[32] during their work on the effect of bismuth in brass, found fire-cracking to occur after a light break-down and not to be entirely avoided by slow heating. In the discussion on their paper Smith[36] stated that "Castings (of 70 : 30 brass) rolled 30% reduction and then annealed at 600° C. always fire-cracked if they contained bismuth (0·009–1·0%), even though they were slowly heated with the furnace."

Conclusions.

(1) The divergent opinions held on the effect of antimony on brass can be largely accounted for by variations in heat-treatment, zinc content, and casting segregation.

(2) The solid solubility of antimony in 70 : 30 brass decreases from 0·6% at 550° C. to about 0·01% at 200° C. The corresponding values for antimony in pure copper are 11 and 2%; thus, the solubility of antimony at 200° C. is reduced from 2 to 0·01% by 30% of zinc and it is fairly certain that higher zinc contents cause a further reduction.

(3) Decreased solubility is accompanied by an increased tendency to grain-boundary embrittlement. The effect of antimony in unsegregated brass is, therefore, determined largely by heat-treatment and zinc content owing to the influence of these two factors on antimony solubility. Antimonial 70 : 30 brass is embrittled by furnace cooling or holding in the temperature range 200°–450° C.

(4) In cast brass the effect of antimony is intensified by the segregation to which this element is prone and a pre-annealing treatment improves ductility by assisting homogenization. Under the conditions stated in the present work, 0·05% of antimony is about the maximum for satisfactory cold rolling of 70 : 30 brass ingots, but within the limit for hot rolling.

(5) A working hypothesis that accounts for the effect of antimony on mechanical properties is based on the assumption of a brittle intercrystalline precipitate. Embrittlement increases with the continuity of this intercrystalline precipitate and the continuity, in turn, depends on heat-treatment. The continuous form has not been consistently revealed under the microscope, although the discontinuous form can be regularly detected.

(6) Lead and sulphur both impair ductility and intensify somewhat the effect of antimony and, on the whole, antimony intensifies the effects of these elements.

(7) Antimonial embrittlement can be overcome by the addition of elements which form innocuous compounds with antimony. Phosphorus forms such a compound and eliminates antimonial embrittlement but slightly impairs hot-rolling behaviour. Lithium also forms a compound with antimony, improves hot- and cold-rolling behaviour, and eliminates antimonial embrittlement, but apparently only at the expense of a big reduction in general mechanical properties.

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APPENDIX.—*Electrolytic Polishing of Brass.*

A very simple apparatus was found to give a polish considerably better for most purposes than the best obtainable by hand polishing and

in a much shorter time. The actual conditions were not found to be at all critical.

The polishing solution was a 1 : 1 mixture of orthophosphoric acid and water. The container was an etching dish, about 10 cm. in. dia. The cathode was a piece of copper strip, the portion in the solution being surrounded by a selvyt bag to prevent the bubbles formed from floating to the anode (the specimen) and interfering with the polish. The specimen was held in a pair of brass tongs and the electrical circuit was the usual potentiometric one. The anode-cathode distance was 5 cm., the time was usually 3 min., and the polishing voltage 2–2½ V. Too low a voltage results in etching and too high a voltage causes gas evolution on the specimen; bubbles of gas adhering to the specimen prevent solution, leaving "hills" underneath them. Where long polishing or etching times were required, more uniform results were obtained when the solution was stirred. The current density is the controlling factor but, in practice, with specimens of different sizes in circulation, voltage regulation was found to be reliable and more convenient.

After polishing and washing, the specimen can be etched in the usual way. It can also be electrolytically etched without removal from the polishing solution. The latter technique gives a grain-boundary etch which was found particularly suitable for revealing discontinuous grain-boundary precipitation; the former technique, using ammonium persulphate as the etching reagent, was found useful for revealing the continuous grain-boundary films. About 7 sec. at 0·8 V. sufficed for electrolytic etching.

Washing of the specimen after removal from the solution presented some difficulties. Jacquet [37] recommends immersion in a dilute solution of orthophosphoric acid. This is satisfactory for copper but results in a pitting type of etch with 70 : 30 brass, since the film of polishing solution removed with the specimen tends to etch and also to form a white, tenacious precipitate immediately it is brought into contact with water. However, it can be removed before it has time to etch, either by means of a powerful jet of water or by shaking the specimen vigorously in very hot water for not more than 5 sec. Pure brass if left in the hot water for more than 5 sec. will begin to etch, a tendency which is reduced by antimony to such an extent that brass containing 0·1% antimony may remain in the water for 20 sec. without etching. After removal from the hot water it is preferable to cool the specimen by momentarily immersing it in cold water and placing it in acetone for about ½ min., during which time residual fragments of the film of polishing solution appear to be dissolved without causing any etching, presumably owing to the low ionizing capacity of acetone.

In addition to the complete removal of the fine scratches usually observed after polishing a soft material like copper or brass, electrolytic polishing has the advantage that the structures are developed in much greater detail on etching, no doubt owing to the less distorted state of the surface. Not only do the grain boundaries and twin boundaries show more detail, such as minute serrations, but a criss-cross hatching, probably associated with the mosaic structure, has frequently been observed inside the grains after suitable etching (Figs. 15 and 16, Plate LXV). The time taken to polish brass electrolytically is less than 5 min. from the cloth polish stage and about 15 min. from the 0000 paper stage.

The method may be unsatisfactory when more than one phase is present (*e.g.*, 60 : 40 brass), although Jacquet [37] has obtained satisfactory results using pyrophosphoric acid solution. Difficulties have also been experienced in cast single-phase alloys, where coring tends to be revealed during the polishing operation. Cast 70 : 30 brass can be polished fairly satisfactorily at a sufficiently high current density, however, although cavities are enlarged. The impurity inclusions found in nearly all specimens during the present work were obscured by a small mound of reaction products; the size of the mound was diminished by adding 5% by volume of glacial acetic acid to the polishing solution.

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METALLURGICAL ABSTRACTS

(GENERAL AND NON-FERROUS)

Volume 13 AUGUST 1946 Part 8

I.—PROPERTIES OF METALS

***On the Brittleness of Aluminium After Casting.** Henri Jolivet and Marcel Armand (*Compt. rend.*, 1946, **222**, (16), 946–948).—Experiments were carried out to determine the considerable brittleness suspected to exist in aluminium immediately after solidification down to much lower temperatures. The liquid metal was poured at 800° C. into a small metal mould heated to 650° C. The brittleness of the specimen was demonstrated by its appearance after bending through 90° at different temperatures during cooling. The metal consisted of: (*a*) iron and silicon traces, aluminium 99·99%; (*b*) iron 0·17, silicon 0·31, aluminium 99·52%; (*c*) iron 0·46, silicon 0·35, aluminium 99·19%. If t_1 is the temperature above which the specimens cracked in bending and broke, and t_2 the temperature above which the brittleness progressively disappeared and the specimens bent without cracking: for metal (*a*), t_1 and t_2 are independent of the rate of cooling and lie around 630° C., for (*b*) and (*c*) they are 580° and 570° C., respectively, for rapid cooling and 630° and 620° C. for slow cooling. After rapid cooling followed by reheating, brittleness does not recur below 620° C. Micro-examination supplied the explanation of the phenomenon. The metal containing appreciable silicon shows the aluminium–iron eutectic ($Al–Al_3Fe$) after slow cooling, and the aluminium–iron–silicon eutectic ($Al–Al_4FeSi$) after rapid cooling. The melting point of the latter is 575° C. The disappearance of brittleness on reheating is bound up with the diffusion and the solution of the silicon, which is accompanied by a coalescence of the eutectic.—J. H. W.

Metals as Getters. J. D. Fast (*Philips' techn. Rundschau*, 1940, **5**, 221–226; *Chem. Zentr.*, 1941, **112**, (I), 1451; *C. Abs.*, 1943, **37**, 1659).—In order to obtain the desired high vacuum in radio tubes, X-ray tubes, &c. and to maintain it after sealing, metals which absorb and fix gases are used, such as barium and zirconium. The properties on which the action of these getters depends are discussed. Barium and other metals used are attacked with the formation of a new phase. In this case, in contrast to the process involved in corrosion-resistant metals, it is essential that the chemical process be able to proceed continuously without the formation of a closed surface layer of the reaction products. The action of zirconium depends on the remarkably high solubility in this metal of most of the gases concerned in such evacuation of tubes. Details of the use of such metals are discussed.

***On Solutions of Metals [Cadmium] in Fused Salts.** (Karpachev) See p. 301.

***The Question of Magnetic Hysteresis in Iron and Nickel.** Fritz Förster and Hans Wetzel (*Z. Metallkunde*, 1941, **33**, 115–123; *C. Abs.*, 1943, **37**, 329).—Iron and nickel wires will show some size increase owing to magnetic hysteresis. With increasing temperature, the size of cold-drawn iron wire increases more rapidly. Evidence of this phenomenon decreases if the wire has been previously annealed. Magnetization increases with stress on either iron or nickel wires. Hysteresis curves are given for the two metals.

***Disintegration and Liquation of Hard Lead [and Lead–Antimony Alloys].** Wilhelm Stockmeyer and Heinrich Hanemann (*Z. Metallkunde*, 1941, **33**, 67–68; *C. Abs.*, 1943, **37**, 329).—Melting points and relative times for melting

* Denotes a paper describing the results of original research.
† Denotes a first-class critical review.

various lead–antimony alloys, containing up to about 13% of antimony, have been determined, as well as heat capacities and pouring temperatures for the different compositions. In setting up a standard practice for disintegration and liquation of lead–antimony alloys it is important to consider the heat capacity of the mould and ladle.

The Problem of Pure Manganese. The Chemical Balance Sheet of the Electrothermal Refining of Ferro-Manganese. M. Déribéré (*Chim. et Ind.*, 1945, **54**, (3), 172–176).—Detailed charge sheets are given showing the progress of refining ferro-manganese containing 78% of manganese (with carbon 6, silicon 1·5, and phosphorus 0·5%) when treated in an electric furnace, under an oxidizing slag, to yield a product containing 86·5% of manganese, less than 0·5% of carbon, and only traces of silicon and phosphorus.—J. C. C.

***Production and Properties of Nickel [Filaments for] Bolometers.** Frank G. Brockman (*J. Opt. Soc. Amer.*, 1946, **36**, (1), 32–35).—Nickel bolometer filaments as thin as 0·1 μ have been made, and their properties investigated. The filaments, which were made by electrodeposition of nickel on to copper foil and subsequent electrochemical solution of the copper under stated conditions, appeared to possess the properties of bulk nickel.—H. J. A.

***Diffusion of Nickel in Rock Salt.** K. N. Pogodaev (*Trudy Vostoch.-Sibir. Gosudarst. Univ.*, 1940, **2**, (1), 65–86; *Khim. Referat. Zhur.*, 1941, **4**, (4), 12; *C. Abs.*, 1943, **37**, 4608).—[In Russian.] Cf. *Zhur. Eksper. Teoret. Fiziki*, 1938, **8**, 992–997. A number of problems connected with the structure of a solid body can be solved by studying diffusion in solid media. The diffusion velocity in the crystal lattice permits the determination of the possible energy levels occupied by the diffusing particles, and characterizes the behaviour of foreign ions in the lattice under the influence of thermal motion. The fundamental physical laws of diffusion can be studied under the simplest conditions when the diffusion takes place not in the polycrystalline metals, but in alkaline earth mono-crystals. The diffusion of nickel in rock salt was used for this purpose. The temperature dependence of nickel ions was studied in the interval 627°–780° C. The relation between the coeff. of diffusion and the temperature is given by $D = 0{\cdot}42e^{-13220/T}$ sq. cm./sec. There was satisfactory agreement of the experimental results with the results obtained from the Einstein equation. The diffusion of nickel and of copper ions in rock salt cannot be explained satisfactorily by the Dushman–Langmuir and Braune equations. By means of the Born cycle it was shown, on the example of nickel and copper ions in rock salt, that the possibility of the occurrence of diffusion can be determined by the energy ratio. The experimental and mathematical parts of the studies are described in detail.

***A New Method of Producing Spectral-Pure Platinum.** I. I. Černjaiev and A. M. Rubinstein (*Compt. rend.* (*Doklady*) *Acad. Sci. U.R.S.S.*, 1945, **48**, (5), 332–333).—[In English.] Spongy platinum is dissolved in *aqua regia*, and the solution evaporated twice with HCl and once with hot water. The resulting solution of H_2PtCl_6 is diluted and treated with KCl to precipitate K_2PtCl_6, which is washed and dried, and then treated with hot KNO_2 solution. Reduction and nitrification occur, giving rise to a solution of $K_2Pt(NO_2)_4$, which is treated with ammonia to precipitate $(NH_3NO_2)_2Pt$, a salt which has negligible adsorption characteristics. Further purification is effected by oxidizing with gaseous chlorine to give $(NH_3NO_2)_2Cl_2Pt$, which is recrystallized several times and heated to produce pure metallic platinum, free from iridium, palladium, and iron.—G. V. R.

***Heat-Treatment of Semi-Conductors and Contact Rectification.** B. Serin (*Phys. Rev.*, 1946, [ii], **69**, (7/8), 357–362).—In making silicon–tungsten point contact rectifiers it is usual to heat the silicon at approx. 1000° C. for 1 hr. S. suggests that this treatment causes evaporation of impurities (*e.g.*, aluminium and boron) from the surface of the silicon more rapidly than they

are supplied by diffusion from the interior. This produces a surface layer which changes the electrical characteristics of the metal–semi-conductor contact. The conditions for the formation of this surface layer are discussed and a theoretical expression is developed. The surface layer increases the back-resistance and decreases the capacity of the contact, with improvement in the rectification efficiency.—W. H.-R.

***On the Growth of a Grain of Zinc.** Léon Guillet, Jr., and (Mme.) R. Laurent (*Compt. rend.*, 1946, **222**, (13), 735–736).—It has been shown (*ibid.*, 1943, **216**, (19), 642; *Met. Abs.*, 1944, **11**, 323) that a grain of pure zinc grows considerably at elevated temperatures, without previous cold work. As the rate of growth diminishes rapidly isothermally, it is not possible to obtain a single crystal by this means. This, however, can be easily effected if the metal is subjected to a small permanent deformation before annealing. Small bars of zinc, 99·99% pure, hot rolled and cold drawn, containing 320 grains/sq. mm., were subjected to 1% plastic elongation, brought to 300° C. at a rate of 25° C. per day in an electric furnace, and held there for 96 hr. In general, the result was that the bars contained 4–6 crystals several cm. long. In a large number of tests the max. length was 86 cm. and the mean 30 cm. The crystallographic orientation was generally random and the bars were often covered with a surface layer of five crystals 0·1 mm. thick, which disappeared on annealing for 2 hr. at 400° C. This method of making single crystals is analogous to those described by other workers for aluminium, magnesium, iron, and tungsten.—J. H. W.

†Survey of Literature on the Metallurgy of Zirconium. W. J. Kroll and A. W. Schlechten (*U.S. Bur. Mines, Inform. Circ.* No. 7341, 1946, 50 pp.).—K. and S. critically review the literature dealing with the preparation of zirconium-bearing ores, the production of zirconium metal, and the production, properties, and uses of zirconium alloys, halides, sulphides, and phosphides. A *bibliography* of 218 references is given.—H. J. A.

***The Fusing Time of Fuses.—III.** J. A. M. van Liempt and J. A. de Vriend (*Z. Physik*, 1940, **117**, 18–19; *Chem. Zentr.*, 1941, **112**, (I), 1713; *C. Abs.*, 1943, **37**, 2315).—Cf. *Met. Abs.*, 1935, **2**, 644; 1936, **3**, 37. The fusing time constant C was determined by the method of G. J. Meyer for various metals and alloys. The following values are reported for the metals (as $C \times 10^{-6}$): gold 575±17, aluminium 360±10, palladium 236±2, cobalt 210±10, zinc 200±17, magnesium 199±9, tantalum 167±6, iron 147±2, steel 105±2, cadmium 98±4, zirconium 63±2, and lead 32±1. The following values are reported for the alloys indicated: Konel (nickel 73, cobalt 17, iron 7, titanium 2·5, manganese 0·5%) 145±6, 80 : 20 copper–nickel alloy 262±1, 70 : 30 copper–nickel alloy 221±4, 55 : 45 copper–nickel alloy 135±3, 90 : 10 copper–nickel alloy 294±12, 70 : 30 nickel–copper alloy 150±4, 75 : 25 tin–lead alloy 32±1, 63 : 37 tin–lead alloy 31±1, 78 : 20 : 2 nickel–chromium–iron alloy 109±2, 58 : 42 iron–nickel alloy 79±3. Comparative measurements made on round wires and wires with edges showed that the form of the cross-section has no effect on the value found.

The Importance of Ultimate Extension as an Engineering Property of Materials. A. Fisher (*Metallurgia*, 1946, **34**, (200), 77–84).—One of the properties in the ductility group, percentage ultimate extension, is discussed. It is considered that a condition of specific strain is far more common in engineering structures than is generally supposed. It is stated that, under a condition of specific strain, it is quite useless to use a material with a higher modulus of elasticity, a higher proof strength, or any higher property which moves the stress–strain curve to the left in the hope that higher strength will result, because such a move merely results in higher stress being generated. Under conditions of specific strain, a component will fracture if its available percentage extension is below that required under the imposed strain, irrespective of

its strength. Hence, percentage ultimate extension indicates a valuable property in any material to be used where stress concentrations exist.—J. W. D.

Comparisons Between Values of Hardness and Mechanical Strength. W. Späth (*Metallwirtschaft*, 1941, **20**, (49/50), 1185–1187; *Chem. Zentr.*, 1942, **113**, (1), 2322; *C. Abs.*, 1943, **37**, 3379).—A discussion of the relation between stress and deformation and of the present state of the development of these two research fields leads to the conclusion that, in future, the recently gained knowledge of the mechanical properties will be applied also to the theory of hardness. This will influence the measurement of hardness, which will be made under consideration of the effect of deformation, hardening, and time of loading.

On the Hardening of Metals. G. V. Akimov (*Compt. rend.* (*Doklady*) *Acad. Sci. U.R.S.S.*, 1945, **47**, (8), 546–549).—[In English.] The distribution of electron density in crystals with different types of bond (ionic, atomic, molecular, and metallic) is briefly considered and correlations between the metallic bond and metallic properties are discussed. It is suggested that the deformation-hardening of metals is due to localized transition from the metallic type of bond to the atomic type, so that rigid "electron bridges" are formed between atoms. The atomic bond corresponds to a higher energy level than the metallic bond and this is responsible for the considerable absorption of energy during deformation. The regions in which the transformation has occurred are regarded as having a continuous, rigid skeleton of atoms, with high strength and hardness, and low plasticity. When these regions are sufficiently continuous throughout the metal to isolate unchanged ductile regions, the plasticity is exhausted and the structure breaks on further deformation. The implications of this theory are discussed.—G. V. R.

***Wear in Non-Ferrous Metals by Dry Friction.** W. Rädeker (*Metallwirtschaft*, 1944, **23**, (22/26), 202–215).—An apparatus is described in which a revolving disc of metal wears an impression in the flat surface of a plate of the same or dissimilar metal under controlled conditions of load, time, &c. The load was kept constant at 7·9 kg./sq. mm.; the lubricants consisted of liquid air, air, water, and oil; the temperature varied from −190° to 700° C.; two disc speeds were used, 1·8 and 9·5 m./sec., and the duration of the test was the time during which a distance of 10,000 m. was traversed peripherally by the disc. The materials tested were: lead, zinc, Duralumin, Elektron, aluminium bronze, copper, nickel, and a few steels. The specimens were weighed before and after test and the resulting surfaces were examined by macro and micro methods for the effects of flow, grinding, &c. It is shown that: (1) wear was high at low temperatures, decreasing to a min. between 0° and 200° C. and then increasing again with increase of temperature; (2) lubrication gave results which varied, depending on the extent of chemical reaction between the lubricant and the metal surfaces, but, in general, wear was greater in water than in air; (3) variation of peripheral speed gave results depending on the nature of the surfaces in contact. It is concluded that welding processes play an important part in wear; such factors as the use of similar metals, especially when they have low melting points, direct contact of surfaces, and high temperatures, all of which favour welding, tend to increase the rate of wear; factors which prevent welding, such as the use of non-alloying metals, the formation of oxide films, and polishing due to flow, tend to decrease the effect of wear.—E. N.

Friction of Pure Metals and the Influence of Adsorbed Gases. The Temperature Coefficient of Friction. M. L. Goldovsky (*Uspekhi Fiz. Nauk*, 1941, **25**, 373–376).—[In Russian.] A review.

***Researches on the Impurities in Volatile Metals, by Distillation in vacuo.** Marcel Villat (*Thesis: École Polytechnique Fédérale, Zürich*, 1942, 57 pp.).—The general principles of the vacuum distillation of volatile metals are dis-

cussed, and the application of the method to the analysis of such metals is described in detail. The apparatus required, the experimental technique, and the course of typical experiments are fully described. The metal is volatilized from an externally-heated crucible in a high vacuum; the vapour, after passing through a steel-wool filter plug, is condensed on a water-cooled tube. The non-volatile residue remaining in the crucible may be analysed; in certain cases, analysis of the gas liberated before distillation begins is possible. The analysis of calcium, magnesium, zinc, cadmium, and the alkali metals is described and typical figures for the composition of the non-volatile residues are given. The separation of zinc from brass by distillation is examined; for this to give satisfactory quantitative results the sample must be very finely divided. An analogous analysis of aluminium, in which the metal is volatilized as $AlCl_3$ in a hydrochloric acid atmosphere, is described and discussed and typical results are given. The method is convenient for the determination and identification of small quantities of non-volatile impurities.—G. V. R.

Mechanics of Plastic Working. Erich Siebel (*Arch. Eisenhüttenwesen*, 1944, **18**, (1/2), 13–22).—The basic equations of the plastic flow of polycrystalline materials are outlined and the application of these equations in the problems dealing with the strength of the material and with plastic working of metals are discussed.—V. K.

***Graphical Methods of Representing Some Conditions of Plasticity.** William Marsh Baldwin, Jr. (*Metals Technol.*, 1946, **13**, (3); and *A.I.M.M.E. Tech. Publ.* No. **1980**, 11 pp.).—The Huber–von Mises–Hencky equation defines the conditions under which plastic flow occurs in a metal subjected to multi-axial stress. The equation, when transferred to co-ordinates of a Cartesian system, describes a cylinder and the interpretation of this is discussed. The equation may be represented as a circle on an isometric plot and a rapid graphical method is given for determining relative strain rates, when the stresses are known, for a metal in the plastic state; conversely, the relative stresses may be determined if the strain rates are known.—W. H.-R.

***The Relation Between Adsorption and the Catalytic Activity of Metals.** A. van Itterbeek (*Meded. Kon. Vlaamsche Acad. Wetensch., Letteren Schoone Kunsten België, Klasse Wettensch.*, 1941, **3**, (10), 3–20; *Chem. Zentr.*, 1942, **113**, (II), 742; *C. Abs.*, 1943, **37**, 4958).—On a very pure nickel film, the adsorption of hydrogen and carbon monoxide at temperatures up to 500° C. and pressures below 1 mm. was studied with the aid of a thermal-conductivity gauge. At ordinary temperatures activated adsorption of hydrogen occurs; at higher temperatures this increases and is apparently not to be attributed to diffusion adsorption. Comparison of the hydrogen and carbon monoxide isobars shows that at 167° and 329° C. the ratio of hydrogen : carbon monoxide is 3 : 1 and at two other temperatures the ratio is 2 : 1, corresponding, respectively, to the synthesis of methane and benzene. It is concluded that the occurrence of this stoichiometric surface ratio is related to the surface reactions. On a copper film neither hydrogen nor carbon monoxide is adsorbed until a small quantity of thorium is electrodeposited. In this instance also, temperatures exist at which the ratio of hydrogen to carbon monoxide is 3 : 1 and 2 : 1.

Modern Developments of the Theory of the Liquid State. Ya. I. Frenkel (*Izvest. Akad. Nauk S.S.S.R.*, 1941, [Fiz.], **5**, (1), 10–11).—[In Russian.] A brief résumé of a review paper.—N. A.

***Surface Contacts Under High Pressure.** Dietrich Müller-Hillebrand (*Wiss. Veröff. Siemens-Werken*, 1941, **20**, 85–103; *Chem. Zentr.*, 1942, **113**, (I), 1033; *C. Abs.*, 1943, **37**, 2632).—The contact resistance of oxidized and oxide-free surfaces was determined by current and voltage measurements. Rectilinear crossed bars whose overlapping surfaces were relatively heavily loaded were used. Oxide layers of copper, aluminium, magnesium, silver, zinc, and tin were tested. After dry storage in an oven at 125° C. for a period of 10–100 hr.,

the first three metals showed a marked increase in contact resistance, but the last three only a slight increase. Contact resistance is significant only if the oxide surfaces are complete; numerous fine cracks are sufficient to furnish paths for the current. With high contact pressures these cracks become more numerous. With constant hardness of the contact material the contact resistance is inversely proportional to the contact pressure, but directly proportional to the average radius of the paths of current flow. Experiments on oxide-free surface contacts (various forms of carbon, silver, copper, and zinc) show that the contact resistance is about inversely proportional to the 0·7 power of the compressive force. The roughness of the contact surfaces has a small, but still measurable, effect, in the sense that the rougher the surface the greater is the contact resistance. With aluminium and magnesium a tough oxide skin is formed even during the cleansing, which, in spite of high pressure, reduces the conducting surface. Finely-ground copper contacts show similar behaviour. On the average, the conducting points of contact were only about $\frac{1}{2000}-\frac{1}{10}$th of the contact surface. With great pressures the contact resistance greatly decreases. From the equation $R_k = cP^{-n}$ (in which R_k is the contact resistance, P is the compressive force, $n = 0{\cdot}7$ to 2 depending on the oxidation of the surfaces) the relation between n and the number of conducting points, the ratio of bearing to conducting surfaces, and the variation in the average radius of conducting bridges can be derived.

***Theory of the Contact Potential Between Conductors in Equilibrium. II.—Contact Equilibria in Which Only the Electron Gas Enters. III.—Mathematical and Physical Laws for Equilibrium of Electrolytes. IV.—Contact Equilibrium Between a Metal or Alloy and a Solution of Its Ions.** D. Castelluccio (*Nuovo Cimento*, 1941, **18**, 209–222, 289–297, 346–357; *Chem. Zentr.*, 1942, **113**, (I), 722, 1107; *C. Abs.*, 1943, **37**, 2248).—[II.]—The differential equation for the equilibrium of an electron gas in empty space, or within a metal in isothermal equilibrium, is derived by thermodynamic means. It is applied to the calculation of the thermal electron emission from metals and to the evaluation of the p.d. between two electrolytic conductors. For thermal emission the result has the form of the Richardson relation. The electrons follow the Fermi rather than the Boltzmann statistics. The potential calculated from the Volta effect is somewhat lower than the observed value. A general theory of the Peltier effect leads to results in qualitative agreement with experiment. [III.]—The differential equation for equilibrium in an isomeric liquid electrolyte is derived and an analogy between the state of a metallic ion in a gas and in a solution of an electrolyte is pointed out. [IV.]—The contact potential between the electron "atmosphere" of a metal and the ion atmosphere of a solution of salt of the metal is calculated; this is of importance in determining the solution tension of the metal. The case of a metal that is soluble or insoluble in the solid state is considered.

The Theory of Contact Potentials Between Metals and Semi-Conductors or Insulators. J. H. Gisolf (*Ned. Tijdschr. Natuurkunde*, 1942, **9**, 49–65; *Chem. Zentr.*, 1942, **113**, (II), 865; *C. Abs.*, 1943, **37**, 5309).—A review of the work of Davydov, Mott, and Schottky on the thermodynamic equilibrium between electrons in metals and semi-conductors.

Nernst's Thermomagnetic Effect in Semi-Conductors and Metals. N. L. Pisarenko (*Izvest. Akad. Nauk S.S.S.R.*, 1941, [Fiz.], **5**, (4/5), 417–421).—[In Russian.] It is shown that the magnitude and the sign of Nernst's effect in semi-conductors and metals can be explained by the dependence of the time of free travelling of electrons on the speed and by the occurrence of two types of transfer of current in semi-conductors.—N. A.

***Contact of Semi-Conductors with Metals and Electrode Potential Differences.** S. I. Pekar (*Izvest. Akad. Nauk S.S.S.R.*, 1941, [Fiz.], **5**, (4/5), 422–433).—[In Russian.] The problem was considered theoretically in a different way

from that carried out earlier by Mott, so that the contact p.d.'s and their dependence on current density could be calculated.—N. A.

***Experimental Investigation of the Contact of Semi-Conductors With Metals.** V. I. Lyashenko, G. A. Felorus, and Z. I. Fel'vashnikova (*Izvest. Akad. Nauk S.S.S.R.*, 1941, [Fiz.], **5**, (4/5), 434–441).—[In Russian.] The theory suggested by Pekar (cf. previous abstract) was experimentally examined and confirmed.—N. A.

***Contact of Semi-Conductors With Metals.** A. V. Joffe and A. F. Joffe (*Izvest. Akad. Nauk S.S.S.R.*, 1941, [Fiz.], **5**, (4/5), 550).—[Résumé of the paper in Russian and English.] The resistance of semi-conductors was studied as a function of the contact p.d. between semi-conductors and metal electrodes. The Hole semi-conductors (Cu_2O, Cu_2S, and V_2O_5) possess an extra resistance with electrodes having a small work-function of emission (Al, Zn, Mg), while the electronic semi-conductors (WO_3, TiO_2) possess an extra resistance with electrodes having a large work-function of emission (Au, graphite).—N. A.

On the Intermediate State of Supraconductors. L. D. Landau (*Izvest. Akad. Nauk S.S.S.R.*, 1942, [Fiz.], **6**, (1/2), 79).—[In Russian.] A brief résumé of a paper including a theoretical consideration showing the possibility of an experimental confirmation of L.'s theory of an intermediate state of supraconductors.—N. A.

***An Investigation of the Intermediate State of a Supraconducting Sphere.** A. I. Shal'nikov (*Izvest. Akad. Nauk S.S.S.R.*, 1942, [Fiz.], **6**, (1/2), 80–81).—[In Russian.] A brief résumé of a paper describing experiments which show that an intermediate state exists in a supraconducting sphere of tin. This was achieved by measuring the longitudinal inhomogeneity of the magnetic field in a 12–18-μ-thick cut made in the sphere.—N. A.

Supraconductivity and Magnetic Energy Between Currents. William Band (*Phys. Rev.*, 1946, [ii], **69**, (5/6), 241).—A theoretical note.—W. H.-R.

Diamagnetism and Supraconductivity. William Band (*Phys. Rev.*, 1946, [ii], **69**, (5/6), 241).—A theoretical note.—W. H.-R.

***Electrodynamic Interaction of Two Electrons and Welker's Theory of Supraconductivity.** Vittorio Somenzi (*Nuovo Cimento*, 1941, **8**, 223–234; *Chem. Zentr.*, 1942, **113**, (I), 168; *C. Abs.*, 1943, **37**, 2259).—The exchange forces and interaction of two electrons are derived from the relativistic Hamiltonian function. The magnitude of the energy of interaction is considered for bound electrons, for quasi-free electrons, and for those bound to a single atom (alkaline earths). The results confirm the speculations of Welker (*Z. Physik*, 1939, **114**, (9/10), 525–551; *Met. Abs.*, 1940, **7**, 246), who calculated the diamagnetic moment of an electron gas on the basis of a void in the first Brillouin zone.

The Theoretical Meaning of the Justi–Zickner Investigation of Branched (Parallel) Currents in Supraconductors. M. v. Laue (*Z. Physik*, 1941, **118**, 455–460).—Cf. *Physikal. Z.*, 1941, **42**, 257–272. The experiments of J. and Z. on current distribution in coils in the supraconductive state are considered mathematically. It is concluded that the J.–Z. method offers no advantages over older methods as a means of detecting residual resistance in supraconduction. The correlation between the law of distribution and the extreme skin effect is discussed.—L. H.

Advantages of the Proposed Substitution of a Conventional ["True Elastic"] Limit. P. Rossi (*Met. Ital.*, 1941, **33**, 77–81; *Chem. Zentr.*, 1942, **113**, (I), 923; *C. Abs.*, 1943, **37**, 2316).—R. is in favour of the new elastic limit proposed by Esser and Arend (*Arch. Eisenhüttenwesen*, 1940, **13**, (10), 425–428; *Met. Abs.*, 1940, **7**, 341), which uses a logarithmic representation of the function of various stress–strain characteristics of materials. The method is said to describe the properties of metals above or below the 0·2% proof-stress limit in a relatively simple manner. The problem is discussed by means of equations and curves and correlated to Hooke's law.

II.—PROPERTIES OF ALLOYS

High-Temperature Oxidation in Aluminium Alloy Forgings. Lawrence J. Barker (*Iron Age*, 1946, **157**, (19), 60–64).—High-temperature oxidation was probably the greatest cause of rejection of aluminium alloy aircraft forgings during the war. The mechanism, causes, prevention, applications, and effect of this phenomenon are described, and a method of classifying it in terms of its effect on physical properties is discussed.—J. H. W.

***The Casting Properties of [Aluminium] Alloys.** A. A. Bochvar (*Sbornik. Nauch. Dokladov Sektsii Metallovedeniya i Termich. Obrabotki VNITO Metallurg*, **1940**, 35–59; *Khim. Referat. Zhur.*, 1941, **4**, (2), 76; *C. Abs.*, 1943, **37**, 3384).—[In Russian.] A detailed study of two casting properties (solidification and formation of cracks) of eutectic alloys (aluminium–silicon and aluminium–copper) during casting under pressure and without pressure has revealed a close relation between the casting behaviour and the equilibrium diagram. The crack-forming property in alloys of the systems investigated decreases inversely with the content of the alloying addition.

***On the Quantitative Spectrographic Analysis of Solutions, with an Appendix on the Solubility of Silicon in Aluminium.** (Walti) See p. 303.

Bronze and Phosphor-Bronze. C. Fougner (*Tekn. Ukeblad*, 1941, **88**, 213–214; *Chem. Zentr.*, 1942, **113**, (I), 102; *C. Abs.*, 1943, **37**, 2325).—F. describes the constitutional diagrams of copper–tin and copper–phosphorus alloys and discusses the ternary alloys of copper, tin, and phosphorus with reference to the effect of phosphorus on the properties of bronzes.

***Properties of Silicon Brasses and Bronzes Made by Using Ferro-Silicon.** T. K. Ryazhskaya (*Sudostroenie*, **1945**, (2), 15–19).—[In Russian.] A study was made of the effects of silicon (using ferro-silicon temper alloys) on the mechanical and casting properties of (*a*) brasses containing silicon 1·5–4·5, copper 79–81, iron 0·6, lead 1·0, tin 0·3, aluminium 0·1, manganese 0·5, antimony 0·1%, and zinc the remainder, and (*b*) bronzes containing silicon 2·75–3·5, manganese 1–1·5, iron 0·3, lead 0·03, tin 0·25, nickel 0·1%, and copper the remainder. It was found that the mechanical properties of these alloys are mainly affected by their silicon content and that iron introduced by the temper alloy slightly reduces the ductility. Linear shrinkage of the alloys is reduced by increasing their silicon content, while their fluidity is increased.
—V. K.

***Changes in the Volume and Electrical Resistance of Copper–Beryllium Alloys During Heat-Treatment.** Hans Thomas (*Z. Metallkunde*, 1944, **36**, (6), 136–140).—The volume and electrical-resistance changes occurring in a series of quenched and age-hardened alloys of copper with 0·9–3·6% beryllium and a few alloys containing up to 12% beryllium were investigated. From the data obtained it is concluded that: (1) at 170° C. there is a contraction in volume and increase in resistance due to low-temperature temper-hardening, (2) at 280° C. some reversion phenomena occur, (3) at 300°–375° C. the volume contracts and the resistance decreases quickly as a result of precipitation-hardening, (4) at 400°–600° C. reversion phenomena again occur, accompanied by a slow increase in volume and resistance. The eutectoid transformation $\alpha + \gamma = \beta$ takes place between 601° and 618° C., causing an increase in volume and resistance, and the anomalous changes which occur in alloys containing more than 3·6% of beryllium are attributed to this transformation.—E. N.

Properties of Some Cast Copper-Base Alloys at Elevated Temperatures. H. E. Montgomery (*Trans. Amer. Inst. Min. Met. Eng.*, 1945, **161**, 455–463; discussion, 463–465).—M. reviews existing literature on the high-temperature creep properties of cast copper-base alloys.—H. J. A.

***Effect of Copper and Some Other Metals on the Gold–Germanium Eutectic.** Robert I. Jaffee and Bruce W. Gonser (*Metals Technol.*, 1946, **13**, (3); and *A.I.M.M.E. Tech. Publ.* No. **1998**, 8 pp.).—In the gold–germanium system, the eutectic alloy at 12% of germanium melts at 356° C. and has an unusually fine eutectic structure. The corresponding eutectic valley in the ternary system gold–copper–germanium has been traced up to 30% of copper. Up to 10% of copper the phases present in the eutectic line are the face-centred cubic gold–copper solid solution and free germanium. At 20% of copper a new phase replaces the gold–copper solid solution. The effect of copper is to coarsen the eutectic structure. Hardness tests were made on the alloys, and also tensile tests of gold strip soldered with the ternary gold–copper–germanium alloys. Microstructures are reproduced of these alloys and also of gold–germanium alloys containing additions of silver, zinc, platinum, and nickel.
—W. H.-R.

***The Amphoteric Behaviour of PbSe as a Semi-Conductor.** F. Eckart and K. Raithel (*Naturwiss.*, 1941, **29**, 572–573; *Chem. Zentr.*, 1942, **113**, (I), 847; *C. Abs.*, 1943, **37**, 5296).—The conductivity of thin PbSe films was studied (1 mμ thickness precipitated on glass) and compared with the results of Bauer (*Ann. Physik*, 1940, [v], **38**, 84). Contrary to Bauer's observation, it was found that between 170° and 300° K. the conductivity did not decrease with increasing temperature, except at low selenium content. At low lead content the conductivity increases slightly with temperature.

***Properties of Cerium-Containing Magnesium Alloys at Room and Elevated Temperatures.** T. E. Leontis and J. P. Murphy (*Metals Technol.*, 1946, **13**, (3); and *A.I.M.M.E. Tech. Publ.* No. **1995**, 32 pp.).—The properties of magnesium–cerium alloys containing up to 10% cerium were examined at temperatures up to 700° F. (371° C.); the cerium was added in the form of "misch metal" and thus contained a large percentage of lanthanum and other rare earths which were included in the term cerium content. In general, the magnesium–cerium alloys retain much of their strength at elevated temperatures (400° F., 204° C.), and exhibit high resistance to creep over a wide range of temperatures. These properties are somewhat improved by additions of manganese and if the manganese content exceeds 1·1% the corrosion-resistance (in 3% aqueous sodium chloride) is greatly increased. Additions of aluminium tend to decrease the strength at high temperatures, but increase the ductility and electrical conductivity. Microstructures are reproduced and details given of creep tests, tensile tests, hardness tests, and measurements of the electrical and thermal conductivities.—W. H.-R.

***Water-Quenching of Some Typical Magnesium Casting Alloys.** (Busk and Anderson) See p. 310.

†Modern Hard Magnetic Materials. K. Hoselitz (*J. Sci. Instruments*, 1946, **23**, (4), 65–71).—A general review of the magnetic characteristics of modern permanent-magnet alloys including Alni, Alnico, Alcomax, Comol, Vicalloy, and other alloys including copper–nickel–iron, copper–cobalt–nickel, cobalt–platinum, and various steels. The question of magnet design and ageing stability are discussed. For purposes such as for electricity meters and electrical instruments, stability of the magnet system is of the greatest importance. With all magnet materials there is measurable change in a period of 30 min. after magnetization. In quench-hardened steels structural changes may take place slowly and affect the magnetization by 20%, but nickel–aluminium alloys show no structural changes. The most favourable stabilizing treatments and conditions of storage are discussed.—W. H.-R.

***Iron–Nickel–Cobalt Alloy for Sealing to Glass.** G. D. Redston and J. E. Stanworth (*J. Sci. Instruments*, 1946, **23**, (3), 53–57).—The differential expansion between a standard molybdenum rod and various iron–nickel–cobalt alloys was measured and the results are shown graphically; the transition

temperatures of the alloys were also determined. For sealing to borosilicate glasses, an alloy of the following composition is recommended: nickel 29 ($\pm$0·5), cobalt 17, manganese 0·3, silicon 0·15, carbon $\not>$0·05, phosphorus $\not>$0·01, and sulphur $\not>$0·01%. The total percentage of nickel + cobalt should equal 46% $\pm$0·5. Glasses can be made with coeffs. of expansion which match this alloy. Measurements were made of stresses in glass–alloy seals.
—W. H.-R.

***Creep Tests at 620° C. on a Special Material for High-Temperature Service.** Eberhard Both (*Z. Metallkunde*, 1944, **36**, (6), 149–152).—The material tested was DVL 42 (PMWC + Ti), consisting of nickel 37, cobalt 24·5, chromium 15, tungsten 5, molybdenum 5, and titanium 1·3%, with small amounts of manganese, silicon, iron, and carbon. Melting was done in a high-frequency vacuum furnace and the test specimens were cold-drawn wires 1·5 m. long and 4 mm. in dia. The specimens were loaded at 20 kg./sq. mm. for 300 hr. and thereafter given incremental loads of 5 kg./sq. mm. every 300 hr. until 35 kg./sq. mm. was reached, at which load one specimen was tested to fracture; the other specimens were tested similarly at loads of 40, 45, 50, and 55 kg./sq. mm. The results of long-time and extrapolated short-time tests are given in the form of graphs of creep strength and time–extension for 0·5 and 1% permanent extension at 620° C. It was found that the elongation at fracture, which averaged 8·3%, was independent of the final load and duration of test, but figures obtained for reduction of area were not so consistent, while no appreciable change in hardness took place. Mechanical tests on the fractured material showed that although the tensile strength and elongation were slightly less than for the original alloy, no serious embrittlement had occurred as a result of the loading at high temperature.—E. N.

***Permeability Measurements on Strip 0·1 to 0·01 mm. Thick.** Günther Rassman (*Z. Metallkunde*, 1944, **36**, (6), 131–135).—The influence of sheet thickness, cold work, and heat-treatment on the magnetic permeability of 50 : 50 nickel–iron alloy, Mumetal, and silicon–iron (3·5% silicon) was investigated. After cold reductions of 70–96% the max. permeability of nickel–iron alloys was proportional to the thickness and degree of cold work, but the influence of these factors was lessened by annealing and vanished on heating at 1100° C. for 300 min. Similar results were obtained with the other two alloys.—E. N.

***The Influence of Small Additions of Thorium on the Life of Alloys for Heating Elements.** Werner Hessenbruch and Lore Horn (*Z. Metallkunde*, 1944, **36**, (6), 145–146).—The influence of the addition of thorium, 0·015–2·1% (by analysis), on the life of 80 : 20 nickel–chromium alloy containing small amounts of manganese, silicon, and magnesium was investigated. Spiral wires 0·4 mm. in dia. were subjected to intermittent heating of 2-min. cycles at 1050° C. until final burn-through. With increase in thorium content there was a steady improvement in the oxidation properties, a corresponding decrease in the mechanical properties, and no evidence of discontinuity at 2·1% thorium. The solubility of thorium in nickel was found to be less than 0·01% at room temperature and less than 0·05% at 1000° C.; addition of chromium causes a reduction in these values.—E. N.

Symposium on Creep of Non-Ferrous Metals and Alloys. —— (*Trans. Amer. Inst. Min. Met. Eng.*, 1945, **161**, 401–477).—The individual papers are abstracted below.—H. J. A.

Application of Non-Ferrous Metals and Alloys in Stress Design. J. J. Kanter (*Trans. Amer. Inst. Min. Met. Eng.*, 1945, **161**, 402–417; discussion, 417–419).—The creep characteristics of non-ferrous metals are briefly summarized, and a *bibliography* and series of abstracts dealing with the topic are appended.—H. J. A.

***Creep Characteristics of a Phosphorized Copper.** H. L. Burghoff and A. I. Blank (*Trans. Amer. Inst. Min. Met. Eng.*, 1945, **161**, 420–438; discussion, 438–440).—The creep characteristics of copper wire (0·008% of phosphorus) as annealed to a grain-size of 0·013 mm., and as drawn to 84% reduction, are reported for temperatures of 300°, 400°, and 500° F. (150°, 205°, and 260° C.). The creep-resistance of the drawn wire decreases as recrystallization takes place.—H. J. A.

***Creep Properties of Cold-Drawn Annealed Monel and Inconel.** B. B. Betty, H. L. Eiselstein, and F. P. Huston, Jr. (*Trans. Amer. Inst. Min. Met. Eng.*, 1945, **161**, 441–452; discussion, 452–454).—Creep data have been obtained for cold-drawn annealed Monel and Inconel at temperatures between 800° and 1100° F. (430°–590° C.). Inconel has approximately twice the load-carrying capacity of Monel at any temperature, but Monel compares favourably with many low-alloy steels so far as creep performance is concerned. The tensile and Izod properties of the materials at room temperature after creep testing are recorded and a type of round Izod test-piece suitable for these alloys is described.—H. J. A.

***Creep Data on Die-Cast Zinc Alloy.** E. H. Kelton and B. D. Grissinger (*Trans. Amer. Inst. Min. Met. Eng.*, 1945, **161**, 466–471; discussion, 471).—A test method involving the bending of a die-cast zinc alloy beam is described and beam creep-test data at 25° C. and various stresses for a zinc die-cast alloy are presented.—H. J. A.

***Creep Properties of Some Rolled Lead–Antimony Alloys.** A. A. Smith, Jr., and H. E. Howe (*Trans. Amer. Inst. Min. Met. Eng.*, 1945, **161**, 472–475; discussion, 475–477).—Alloys of lead containing antimony, bismuth, and copper were cast and rolled to 0·1 in. thickness; 16-in. lengths of this material were then clamped together and creep tests conducted on the specimens at 30° and 100° C.—H. J. A.

***Flow Velocity of Zinc Alloys Under Constant Load.** G. Masing, K. Miething, and H. J. Wallbaum (*Nachr. Ges. Wiss. Göttingen, Math.-physikal. Klasse*, 1940, **2**, 1–5; *Chem. Zentr.*, 1941, **112**, (I), 1787; *C. Abs.*, 1943, **37**, 2325).—Zinc–copper alloys containing 0·75–3% of copper were studied for the purpose of explaining the surprising fact that the flow velocity of zinc under constant load is reduced with increase in the copper content so long as the copper is taken into solid solution. At higher copper contents the flow velocity increases again.

The Elastic Modulus of Metallic Alloys. Léon Guillet, Jr. (*Rev. Mét.*, 1943, **40**, (3), 91–94).—A review article in which the experimental methods for determining the elastic modulus of alloys are critically discussed. The general types of results obtained by various workers are presented and general conclusions are reached. It is difficult to obtain high moduli if the metallic properties such as malleability are to be preserved; usually the modulus of a binary alloy is intermediate between those of the components. Treatments which raise the elastic limit and the presence of impurities or additions which affect mechanical properties markedly have little effect on the elastic modulus. High elastic moduli are obtained for intermetallic compounds, but in these cases malleability is lost.—G. V. R.

***Experiments on the Proof of Damage Stress and Work-Hardening in Fatigue Tests [on Steels and Duralumin].** Erich Siebel and Gustav Stähli (*Arch. Eisenhüttenwesen*, 1942, **15**, (11), 519–527).—Wöhler curves for notched specimens were determined for two carbon and an alloy steel and a Duralumin alloy. Similar specimens were subjected to an impact test after a preliminary number of reversals at a series of bending stresses. The number of reversals at any stress at which the impact values begin to decrease were plotted to obtain "damage lines." It is shown that the more the bending stress exceeds the limiting fatigue stress values, the smaller the number of reversals at which

damage starts. The values of the damaging stress are increased by machining off 0·3 mm. from the surfaces of the notches. The same materials were then used to study "damaging" of smooth specimens by measuring the increase in the number of reversals after polishing the surfaces of the specimens which had been subjected to a preliminary number of reversals under the limiting stress. It is shown that the damaging is initiated at the surfaces and that the number of reversals can be increased several hundred per cent. by removing the damaged layer electrolytically or mechanically. It was not found possible to remove the "damaging" by intermediate annealing. Similar results were obtained in tension–compression tests.—V. K.

***Changes in Materials Produced in Fatigue Tests.** Alfred Karius, Erich Gerold, and Ernst Hermann Schulz (*Arch. Eisenhüttenwesen*, 1944, **18**, (5/6), 113–124).—Changes in the damping capacity and elastic moduli caused by reversed bend stresses were determined for a number of carbon and alloy steels and non-ferrous alloys by using the Förster instrument (*Z. Metallkunde*, 1937, **29**, 109; *Met. Abs.*, 1937, **4**, 460). The results show that all these materials behave similarly. The damping capacity of the specimens subjected to reversed bend stresses not much greater than the limiting stress increases, while the elastic modulus decreases with the increasing number of reversals, the curves being asymptotic. At still larger stresses an inflection in curves of these properties and a second rapid increase of the damping capacity or decrease of the elastic modulus are obtained. These two distinct stages in the changes of these properties are explained by the plastic deformation occurring in the first and by the formation of cracks taking place in the second stage. Additional experiments confirmed the view that the changes in the first stage depend on the stressed volume of the material, while the changes in the second stage are independent of this volume. Changes in the above-mentioned properties were not found to be suitable for the determination of the "damage lines" (see preceding abstract).—V. K.

***The Magneto-Resistance of High-Coercivity Alloys.** L. F. Bates (*Proc. Phys. Soc.*, 1946, **58**, (2), 153–164).—Using various ferromagnetic alloys of high coercivity, the change of resistance of specimens subjected to longitudinal and transverse magnetic fields was measured. The changes in the cases of the permanent-magnet materials Alni, Alnico, and Alcomax II were such as to decrease the resistance in the presence of the magnetic field and were thus in marked contrast to those for pure ferromagnetics. The results, which are fully described, are discussed from the point of view of the domain theory of ferromagnetism. It is suggested that the magnetostriction deformation of the domain boundaries or grain boundaries plays an important part.—G. V. R.

***Alloys for Electrodes of Thermoelements and Compensation Leads.** (Berkovsky and Stepanov) See p. 308.

The Representation of Quaternary Systems by Temperature–Concentration Cross-Sectional Diagrams. Günther Ritzau (*Wiss. Veröff. Siemens-Werken, Werkstoff-Sonderhefte*, **1940**, 44–49; *Chem. Zentr.*, 1942, **113**, (I), 153; *C. Abs.*, 1943, **37**, 2255).—Three ideal types of 4-component diagrams are developed geometrically. They are represented by temperature–concentration sections.

Deductions which May be Made from a Solidification Diagram. J. Luneau (*Rev. Mét.*, 1942, **39**, (7), 218–223).—The interpretation of the diagram showing liquidus and solidus curves for a continuous series of binary solid solutions is discussed. Assuming ideal diffusion conditions and the maintenance of equilibrium, equations and graphical constructions are developed for determining the mass ratios of liquid and solid present at a given temperature, the proportions of the components in the mass of solid deposited on cooling through a small temperature range, and the actual mass of solid deposited. Application of these methods to more complex forms of equilibrium diagrams is briefly considered and reference is made, without illustration, to the practi-

cal case where diffusion is limited and equilibrium is not maintained during the liquid–solid transformation.—G. V. R.

***The Zones of Immiscibility in the Liquid State in Alloys.** M. Dannenmuller (*Rev. Mét.*, 1943, **40**, (3), 85–90).—Methods for determining regions of liquid immiscibility in alloy systems are critically discussed and a method is proposed in which the homogeneous melt is allowed to cool relatively rapidly in a crucible of special shape, designed to make sure that a longitudinal section of the cold ingot, when ground and polished, will reveal small quantities of inhomogeneity. The results to be expected from the cooling of alloys of various compositions are discussed with reference to a typical equilibrium diagram and the application of the method to the ternary alloys of lead and zinc with aluminium, cadmium, copper, and tin is described. The previous results of other authors for the latter three systems are corrected by the present work.—G. V. R.

III.—STRUCTURE

(Metallography; Macrography; Crystal Structure.)

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

***Electron Metallographic Methods and Some Results for Magnesium Alloys.** R. D. Heidenreich, C. H. Gerould, and R. E. McNulty (*Metals Technol.*, 1946, **13**, (3); and *A.I.M.M.E. Tech. Publ.* No. **1979**, 22 pp.).—The general methods of electron microscopy are reviewed, with special reference to the study of magnesium and its alloys by the polystyrene–silica replica method. A special etching reagent has been developed, consisting of equal parts of methyl iodide and dry methyl alcohol, with a crystal of iodine to initiate the reaction. Photographs are reproduced showing slip-bands and twins in pure magnesium and of precipitates and other structures of magnesium alloys. In alloys containing aluminium and small amounts of iron a new "fine structure" has been discovered in which an iron–aluminium phase is present. The presence of this "fine structure" has an important effect on the stress-corrosion properties of magnesium alloys and examples are discussed.—W. H.-R.

***The Effect of Impurities on the Efficiency of $K_2Cr_2O_7$ Etching Agents.** W. Eckardt (*Oberflächentechn.*, 1942, **19**, 25–26; *Chem. Zentr.*, 1942, **113**, (II), 949; *C. Abs.*, 1943, **37**, 4999).—Magnesium alloys are not coloured in alkali bichromate solutions made up of pure salts. Addition of small amounts of CrO_3 (usually present in commercial salts) causes a dull black coloration. On further addition of traces of silver salts the coloration becomes brighter; silver salts are usually present in commercial alkali bichromate because $K_2Cr_2O_7$ is evaporated in silver-containing dishes.

***Grain-Sizes Produced by Recrystallization and Coalescence in Cold-Rolled Cartridge Brass.** Harold L. Walker (*Univ. Illinois Eng. Exper. Sta. Bull.* No. **359**, 1945, 55 pp.).—A study has been made of the grain-size and Rockwell hardness of specimens of 70 : 30 brass, cold reduced by 5–70%, after annealing in molten lead or lead–bismuth alloy at 300°–700° C. for periods up to 8 hr. Before final cold working the material was annealed at 750° C. and had a grain dia. of 0·48–0·57 mm. and a Rockwell H hardness of 74–80·5. All the hardness curves showed first a gradual loss in hardness with time (recovery), followed by a rapid decrease (due to recrystallization), and finally a slow decrease in hardness during which period the grain-size increased by coalescence, *i.e.*, by the absorption of unstrained grains by other unstrained grains. The grain-size when recrystallization was just complete (before coalescence) was dependent only on the degree of deformation and was inde-

pendent of time and temperature, being represented by: log grain-size = $-n$ (percentage deformation)$^{\frac{1}{2}} \pm b$. The constant, b, is a function of the grain-size prior to final deformation. No evidence was found of the existence of a "germinant annealing temperature range" in which large grains were produced.—J. C. C.

***Investigations of the Structure of Electrodeposited Zinc.** W. Köhler (*Korrosion u. Metallschutz*, 1943, **19**, (7), 197–199).—The crystal structures of zinc electrodeposits made from sulphate, bright sulphate, and bright cyanide zinc baths were investigated, using powder camera and Geiger counter methods. Comparison readings were made with filings of ordinary pure zinc. The rates of solution of the deposits in normal sulphuric acid were also compared. The deposits from sulphate baths showed a marked anomaly in the X-ray studies and the final rates of solution of these deposits in acid were much greater than those of the deposits from the cyanide bath.—A. B. W.

***On the Growth of a Grain of Zinc.** (Guillet and Laurent) See p. 283.

The Numerical Symbol of Close-Packing of Spheres and Its Application in the Theory of Close-Packings. G. S. Zhdanov (*Compt. rend.* (*Doklady*) *Acad. Sci. U.R.S.S.*, 1945, **48**, (1), 39–42).—In order to interpret the characteristics of structures produced by the close-packing of spheres, a system of nomenclature is developed which allows the periodicity, Bravais lattice, cell dimensions, atom co-ordinates, and space group to be described by a numerical symbol. This simplifies classification of such structures and facilitates the interpretation of complex close-packed structures.—G. V. R.

***A Theoretical Criterion for the Initiation of Slip-Bands.** Clarence Zener (*Phys. Rev.*, 1946, **69**, (3/4), 128–129).—A theoretical note. Relief of shear stress within a slip-band results in a large concentration of shear stress just ahead of its advancing edge, which may be called the spearhead. The condition for propagation of the slip-band is that this concentration of stress is sufficient to produce plastic deformation in front of the spearhead. Z. deduces the relation: yield strength $= C(d/D)^{\frac{1}{2}}G$, where d is the spacing of the atomic planes parallel to the slip-band, D is a measure of the grain diameter, G is the shear modulus, and C is a numerical coeff. whose magnitude is of the order unity. Data for copper alloys support the general views.—W. H.-R.

***X-Ray Investigation of Nitrides and Carbonitrides of Titanium.** E. P. Belyakova, A. Komar, and V. V. Mikhailov (*Metallurg*, **1940**, (4), 5–8; *Khim. Referat. Zhur.*, 1941, **4**, (1), 96; *C. Abs.*, 1943, **37**, 1358).—[In Russian.] The Debye X-ray method indicated that at 1000°–1500° C. the reduction of TiO_2 by a mixture of hydrogen and nitrogen forms Ti_2O_3 and TiO. At 1700° C. and higher, some titanium nitride is formed, as shown by chemical analysis; the lines of titanium nitride are not observed on X-ray photographs, owing to its transformation into a solid solution with TiO. Reduction by solid carbon in nitrogen at 1100°–1500° C. produces neither the free nitride nor the free carbide of titanium. At 1900° C., TiO_2 is completely reduced in 3 hr. to the nitride and carbide, which form a solid solution.

***The Accurate Determination of Cell Dimensions from Single-Crystal X-Ray Photographs.** Margaret C. M. Farquhar and H. Lipson (*Proc. Phys. Soc.*, 1946, **58**, (2), 200–206).—It is pointed out that for certain applications, such as the determination of molecular weights, it is desirable to be able to determine cell dimensions accurately from single-crystal photographs. An accuracy of the order of 0·005% may be obtained by methods of measurement and calculation analogous to those already employed in powder photography. Using the van Arkel method of film mounting, the positions of resolved α doublets with Bragg angle near 90° are measured and the value of one of the cell dimensions is deduced from each. These are plotted against $\sin^2\theta$ and extrapolated to give a final result at $\sin^2\theta = 1$. The use of the method for an orthorhombic crystal is illustrated for thallium hydrogen tartrate. The

spots used are those lying on the zero layer line of a symmetrical oscillation photograph obtained with an axis of the crystal oscillating symmetrically with respect to the X-ray beam. For the salt investigated, $a = 7{\cdot}6395$ kX., $b = 10{\cdot}9945$ kX., $c = 7{\cdot}9100$ kX., these values being correct to 0·0005 kX. The dependence of the results on the assumed camera radius and the methods of measurement are discussed.—G. V. R.

Microradiography. S. E. Maddigan (*Indust. Radiography*, 1946, **4**, (4), 22–25, 28–30).—In microradiography, enlargements are made of radiographs of thin specimens, so revealing the gross structure and supplementing photomicrography. The X-rays used may be generated by a tungsten target, operating at voltages appropriate to the specimen, or characteristic X-rays may be used. Both techniques are capable of revealing the structure, but the latter method is essential where the phases or inclusions present must be identified. The applications discussed include the examination of: Cu–Sn–Pb, Cu–Ag, Cu–Pb, and Cu–Fe alloys; Ni_3P_2 in a copper-base alloy; leaded brass; and cartridge brass. The preparation of specimens is described.—L. M.

***X-Ray Investigation of Liquids.** V. I. Danilov (*Izvest. Akad. Nauk S.S.S.R.*, 1941, [Fiz.], **5**, (1), 30–42).—[In Russian.] A number of liquids (salol, benzophenol, &c.) lose the property of crystallization when all impurities in them are completely removed. Other liquids cannot be undercooled below a temperature range characteristic of the liquid, even after a thorough purification and preliminary overheating. Metals and a number of liquids belong to the second group.—N. A.

V.—POWDER METALLURGY

General Rules for Powder Metallurgy. Henry H. Hausner (*Machinist* (*Eur. Edn.*), 1946, **90**, (8), 290–291).—The effects of processing on the physical properties of compacts of metal powders are given in 13 " trend charts " or graphs.—J. H. W.

VI.—CORROSION AND RELATED PHENOMENA

***Attack of Various Atmospheres on Copper and Some Copper Alloys at Elevated Temperatures.** A. P. C. Hallowes and E. Voce (*Metallurgia*, 1946, **34**, (200), 95–100).—Based on the first part of British Non-Ferrous Metals Research Association Report R.R.A. 527. Three high-conductivity and two arsenical coppers and six aluminium bronzes (containing from 2 to 6% aluminium), forming a group of alloys of high scaling resistance; a group of nine alloys containing small amounts of common elements added individually to copper; and three industrial copper alloys for elevated-temperature use, were tested for intermittent oxidation and scaling at 400° C. in five different atmospheres. The five atmospheres consisted of (i) dry air, free from acid impurities; and (ii) dry air as in (i) with the addition of (*a*) 10% water vapour, (*b*) 0·1% sulphur dioxide, (*c*) 5% sulphur dioxide, or (*d*) moist hydrochloric acid. The work is described and conclusions resulting from the investigation are given.—J. W. D.

Tube Corrosion Held to a Minimum by Continuous Check on [Oil Refinery] Dehydrator-Water *p*H. J. S. Pfarr and H. A. Blackstone (*Refiner*, 1942, **21**, 16–18; *C. Abs.*, 1943, **37**, 5230).—The *p*H of the waste water used for cooling condensers for crude gasoline is recorded continuously and controlled to 7·5–8 by addition of ammonia, thereby practically eliminating corrosion of iron and copper alloys.

***Causes of the Serious Corrosion of Lead in an Intensive-System Sulphuric Acid Plant.** Kurt Wickert (*Chem.-Zeit.*, 1940, **64**, 411–413; *Chem. Zentr.*, 1941, **112**, (I), 1734; *C. Abs.*, 1943, **37**, 2143).—The extensive destruction of the lead was characterized by deeply corroded drop-form cavities. The lead so destroyed was of standard quality; the bismuth content was less than the permissible limit. The corrosion was found to be due to chlorine, the action of which was further intensified by the formation of NOCl in the presence of nitric acid. Sulphur dioxide retarded the corrosion of the lead by chlorine and NOCl. The chlorine came from the tap-water in the system.

***Corrosion Studies on Lead Alloys.** K. Wickert (*Korrosion u. Metallschutz*, 1942, **18**, (11/12), 357–361).—A series of lead alloys was studied, using methods aiming at the determination of their suitability for employment in intensive-system sulphuric acid plants. A method of test with "mixed acid" (30 ml. sulphuric acid, density 1·735; 2 g. $NOHSO_4$; and 0·1 ml. conc. hydrochloric acid) at 90° C. in a sealed tube gave results in agreement with service experience. Silver was the only alloy addition that improved the behaviour of lead in this test. Silver and also nickel and copper are stated to improve alloyed leads by neutralizing the effects of deleterious constituents. The action of conc. hydrochloric acid and of sulphuric acid (density 1·735) at 90° C. on the same lead alloys is also described, as well as some electrochemical studies of protective film formation. The action of the nitrosyl compounds is discussed and their ability to increase attack attributed to the property of splitting off NO-ions, which behave in a similar manner to H-ions.—A. B. W.

***The Scaling of Nickel Containing Various Additions.** Lore Horn (*Z. Metallkunde*, 1944, **36**, (6), 142–145).—The rate of scaling of the following nickel alloys was investigated: (1) nickel with up to 1·7% cerium, (2) nickel with up to 3·4% thorium, (3) nickel with up to 2·25% beryllium, (4) nickel with up to 29·2% chromium and containing traces of manganese and silicon. Polished sheets of the alloys were exposed in air for 50–160 hr. at temperatures up to 1200° C., the increase in weight was recorded at intervals, the colours of the oxide films examined, and the composition of the films ascertained by X-ray methods. The well known parabolic law of oxidation was found to be followed by the alloys of nickel–thorium and nickel–cerium, which formed only one type of oxide film. Deviations from the law were found to occur with the alloys of nickel–chromium and nickel–beryllium, owing to the formation of more than one type of film; nickel with up to about 8% chromium formed oxide skins consisting mainly of NiO, with 8–19% chromium the skin was a mixture of NiO and Cr_2O_3, while with 29·2% chromium the skin was mainly Cr_2O_3, which was relatively thick and slowed down the rate of oxidation. Nickel–beryllium alloys with more than 1·5% beryllium had oxidation rates similarly retarded.—E. N.

***Special Phenomena in Oxidation of Heat-Resisting Steels and Alloys.** Gerhard Bandel (*Arch. Eisenhüttenwesen*, 1941, **15**, (6), 271–283; discussion, 284).—The mechanism of the formation, growth, and breaking-up of the protective oxide layer is considered in relation to the formation of the non-protective, iron-oxide-rich layer. The latter forms first and the rate of replacement by the former depends on the temperature, alloy and atmosphere composition, and the initial surface condition of the specimen. Time, temperature, atmosphere, inclusions, and alloy composition affect the breaking-up and replacement of the protective layer at higher temperatures. Experimental data on the above aspects of oxidation are given.—V. K.

Corrosion and Chemical Behaviour of Zinc and Zinc Alloys. K. Bayer (*Korrosion u. Metallschutz*, 1942, **18**, (11/12), 362–365).—The behaviour of zinc and zinc alloys when subject to atmospheric corrosion or attack by natural waters, at both normal and elevated temperatures, is surveyed and the

results of the more important German investigations in this field are briefly cited.—A. B. W.

***Contribution on the Chemical Corrosion [by Lactic Acid] of Zinc Alloys.** K. Ruttewit (*Korrosion u. Metallschutz*, 1942, **18**, (11/12), 365–368).—Tests of various zinc alloys in 1% lactic acid solutions at room temperature revealed no composition that could be recommended as a substitute for the materials hitherto used for coinage, uniform decorations, &c. Only a few additions improved the corrosion behaviour in lactic acid, *viz.*, mercury, cadmium, and magnesium and their effects were only small. Alloys containing 10% aluminium and 0·1–0·5% silicon retained a bright surface in spite of a relatively large loss in weight.—A. B. W.

***Chemistry and Morphology of Films in Corrosion Studies with Zinc.** W. Feitknecht and R. Petermann (*Korrosion u. Metallschutz*, 1943, **19**, (7), 181–188).—The conditions for formation and the more important properties of seven forms of hydroxide and oxide of zinc occurring in immersed corrosion of zinc are presented in tabular form. Diagrams of the X-ray powder lines of these forms are also given for identification purposes. In solutions more than 0·025 molar in zinc, the corrosion velocity diminishes fairly uniformly and rapidly with time. In more dilute solutions the attack is more irregular and appears only to slow down slightly with time. The corrosion velocity shows a marked min. at solutions 0·025 molar in zinc in the presence of the least carbon dioxide concentrations. Accordingly, the corrosion velocity is independent of the conductivity of the solution, but depends only on the protective action of the film. The chemical and morphological properties of the films are correlated with the corrosion velocity. The slowest attack occurs under conditions where basic chloride III is stable and is therefore related to the formation of a laminar, stable α-hydroxide. The irregularities in very dilute solutions are to be attributed to the formation of a film, composed largely of oxide in one case, and in others of ε- and β-hydroxides. In the case of oxide films the attack is very much faster than in the other cases. Although local elements play an important part, the decisive factor for the corrosion velocity is not the conductivity of the solution, but the protective action of the films, which is itself a function of the chemical nature of the corrosion product. It follows from this that, in corrosion research, greater interest should be shown in the chemistry of the oxides, hydroxides, and basic salts.—A. B. W.

Recent Work on Corrosion and Oxidation. U. R. Evans (*J. Chem. Soc.*, **1946**, 207–214).—A lecture. E. classifies corrosion and oxidation reactions as film-forming or non-film-forming, gives examples of each type, and briefly reviews the inhibition of corrosion.—H. J. A.

Corrosion Criteria—Their Visual Evaluation. Marc Darrin (*Bull. Amer. Soc. Test. Mat.*, 1946, **138**, 37–39; also (abridged) *Metal Ind.*, 1946, **68**, (18), 351–352).—When local corrosive attack takes place, loss of weight is not sufficient to specify the extent of corrosion. D. suggests standard terms for reporting various types of aqueous corrosion, especially when of a local nature, and describes the use of a standard report form to obtain "corrosion scores." The method has been used satisfactorily for six years.—H. J. A.

***Investigation of the Cavitation Phenomena.—I–II.** E. Brandenberger and P. de Haller (*Schweiz. Arch. angew. Wiss. Techn.*, 1944, **10**, (11), 331–341; (12), 379–386).—[I.]—The effects of the speed of impact, jet area and temperature, and speed of water jet on the erosion of a mild carbon steel were studied in a specially-designed apparatus (*Schweiz. Bauzeit.*, 1933, **101**, 243). It is shown that for the comparison of the erosion test results all these factors have to be taken into account. The nature of the erosion was studied in an impact-wave apparatus and was supplemented by mechanical, metallographic, and X-ray examinations. The results obtained for a number of ferrous and non-ferrous materials show that the behaviour of metals in these

tests cannot be correlated with their behaviour under any standard static mechanical tests, but that the changes in crystal structure were similar to those produced under dynamic impact loading. [II.]—A study, by back-reflection photographs, of the changes in the crystal structure at the surfaces of the specimen exposed to erosion showed that the crystals gradually break up into smaller units and finally develop small cracks. It is believed that the process of fracturing in erosion tests is different from that of the static or fatigue fracture, owing to the rôle the cleavage of crystals assumes in this case.—V. K.

***Theory of Polyelectrode Electrochemical Cells and Corrosion Problems.** G. V. Akimov (*Acta Physicochim. U.R.S.S.*, 1945, **20**, (6), 808–832).—It is pointed out that study of corrosion phenomena in alloys involves consideration of the behaviour of polyelectrode systems. The theory of such systems is considered for five electrodes connected in parallel and in series; for cases of low internal and external resistance, where polarization phenomena are important; and for high resistance, internal and external, where polarization can be neglected. It is shown how the polarity of electrodes, with initial electrode potentials intermediate between those of the extreme anode and cathode, is affected by increasing or decreasing polarization, areas and positions of electrodes, the values and ratios of the resistances in the various branches of the external circuit, and the internal resistance of the electrolyte. Experimental data illustrating the conclusions of the theory are given and discussed and the factors important for the qualitative solution of cases more complex than those rigidly treated are described. The bearing of the work on corrosion phenomena, and particularly intercrystalline corrosion, is discussed by reference to aluminium–copper alloys and certain stainless steels. Not only the initial electrode potentials, but also the relative areas and polarization characteristics of the constituent electrodes must be considered.—G. V. R.

VII.—PROTECTION

(Other than by Electrodeposition.)

***On the Formation and Estimation of Oxide Films on Aluminium.** Andreas Ulrich Obrist (*Thesis: Eidgenössische Technische Hochschule, Zürich*, **1944**, 64 pp.).—Various existing methods for the production of oxidized layers on aluminium and its alloys are discussed in detail and methods for the determination of the oxide content of aluminium are critically reviewed. Apparatus for the production of films under controlled conditions by anodic oxidation in oxalic acid is described, together with the technique of the quantitative removal of the film from the metal, which is dissolved away in ethereal hydrochloric acid. From micrometer measurements of the film produced in various conditions the rate of growth was determined. The variation with film thickness of the capacity of the film was studied and gave a dielectric constant of 7·58. An improved method for the determination of oxide in aluminium is fully described and is based on the solution of the metal in air- and moisture-free conditions in ethereal hydrochloric acid, followed by solution of the oxide residue and colorimetric estimation of the aluminium present. From results obtained, the density of the film, which is amorphous at ordinary temperatures, but crystalline after annealing at 900° C., is calculated as 3·10. The water content of the film produced in various conditions was determined and found to agree with the formula AlOOH. The effect of temperatures up to 600° C. on the rate of growth of films is also discussed.—G. V. R.

Modern Methods of Phosphatizing [of Zinc Alloys]. H. v. Rosenberg (*Oberflächentechn.*, 1941, **18**, 169–170; *Chem. Zentr.*, 1942, **113**, (I), 1806; *C. Abs.*, 1943, **37**, 3039).—The various Bonderizing and Parkerizing processes

for phosphatization produce layers of 0·003–0·015 mm. thickness. Zinc alloys containing aluminium are pickled with sodium hydroxide before phosphatizing in order to remove the aluminium from the surface. Rinsing after phosphatizing in hot, chromate-containing baths has a favourable effect. Applications of phosphatizing are discussed.

***The Tinning of Cast Iron.** R. A. Cresswell (*Iron Steel Inst. Advance Copy*, **1946**, 13 pp.; also (abridged) *Found. Trade J.*, 1946, **78**, (1549), 435–439; *Engineering*, 1946, **161**, (4194), 526–528; and *Canad. Metals*, 1946, **9**, 37–41, 44).—An investigation describing methods of hot tinning cast iron directly, without the application of an intermediate metal coating. For the dip tinning of cast iron, the nitrate process is preferred. In this process the work is mechanically cleaned by machining, shot-blasting, or sand-blasting and degreased either in trichlorethylene vapour or in a hot alkaline metal-cleaning solution. It is pickled either in 10% by weight inhibited sulphuric acid at 85° C. for 30 sec. or in cold 50% by vol. hydrochloric acid for 2–5 min., rinsed in water, and dried. The article is then immersed in a fused mixture of equal parts by weight of sodium nitrate and potassium nitrate at 350°–400° C. for 15 min. The work is removed, cooled, washed with cold water, and further pickled for 1 min. in cold 10% by vol. hydrochloric acid, fluxed in an aqueous solution containing 25% by weight of the zinc chloride–sodium chloride eutectic (82% $ZnCl_2$, 18% NaCl) and then transferred to the tin pot which is maintained at 300° C. The time of immersion should be at least 3–5 min.; longer times (up to 30 min.) give better adhesion. The work is then drained and either cooled in air or quenched in paraffin oil. The chloride process is preferred for tinning by wiping. In this process the work is mechanically prepared as in the nitrate process. The surface is then brushed with an aqueous solution containing 25% by weight of the zinc chloride–sodium chloride eutectic mixture and the article heated to 270°–300° C. Some previously melted zinc chloride–sodium chloride eutectic flux is then poured on to the surface at 300° C., washed over the surface for 1–2 min., excess of flux removed, and a quantity of molten tin poured over the surface and wiped by means of steel wire brushes or scrapers. When tinning is complete, excess metal is drained off, the article allowed to cool, and any flux residues removed by washing.—J. E. G.

***Causes and Prevention of Speck Formation on Hot-Tinned Wires.** Fritz Schellenberger (*Draht-Welt*, 1940, **33**, 611–613, 625–627; *Chem. Zentr.*, 1941, **112**, (I), 1733; *C. Abs.*, 1943, **37**, 2326).—The cause of speck formation on hot-tinned wires is usually a porous layer. The formation of an intermediate iron–tin layer does not prevent pore formation since, according to S., this intermediate layer is not formed from the iron of the part to be tinned, but rather is formed as a result of the following processes. As the part is passed through the flux, a $FeCl_2$ film is formed on the iron, which is reduced to iron upon introduction into the tin bath. It is this thin iron film which then forms the iron–tin film with the tin. The wire surface may therefore be defective, even if an intermediate film has been formed. The best protection against pore formation is a flawless wire surface. A 5% solution of H_2SO_4, to which $CuSO_4$ may be added, is recommended as a pickle. A solution of $ZnCl_2$ 45, NH_4Cl 1–2, and NaCl 1% is especially suitable as a soldering solution for a preliminary treatment. In place of the protective layer of organic material on the tin bath, a layer composed of $ZnCl_2$ 70, NH_2Cl 20, and NaCl 10% should be used. Pore formation is further reduced by the use of baths of very pure tin and by keeping clean all parts which come in contact with the wire to be tin coated.

***Influence of the Composition on the Formation of Zinc Flowers on Hot-Galvanized Steel Parts.** Werner Frölich (*Oberflächentechn.*, 1941, **18**, 142–143; *Chem. Zentr.* 1942, **113**, (I), 264; *C. Abs.*, 1943, **37**, 2701).—By adding 0·1–

0·2% of cadmium to the zinc bath, zinc coatings with large, zinc-oxide crystalline formations are obtained; antimony additions give smaller crystals. The yellow colouring caused by too high an antimony content can be eliminated by the addition of 0·001% of aluminium. Additions of tin and cadmium have the same effect, but promote at the same time the formation of zinc flowers.

Surface-Conversion Coatings. George W. Jernstedt (*Bull. Amer. Soc. Test. Mat.*, 1945, (137), 29–35; also (abridged) *Metal Ind.*, 1946, 68, (21), 407–409).—Surface-conversion coatings are formed by the chemical modification of metallic surfaces in order to secure greater resistance to corrosion. J. gives a general review of the topic, which includes the phosphate treatment of steel, oxide, sulphide, and oxalate treatments of copper alloys, anodic and chemical treatments of aluminium or magnesium, and phosphate or chromate treatment of zinc.—H. J. A.

VIII.—ELECTRODEPOSITION

Preparing Die-Castings for Plating. Herbert Chase (*Iron Age*, 1946, **157**, (21), 58–61).—The production-line polishing, buffing, and tumbling, and the use of conveyors and fixtures to expedite operations in the preparation of zinc alloy die-castings for plating in a plant with an output of 70 tons daily are described.—J. H. W.

Nickel Dipping [Before Enamelling]. G. H. McIntyre (*Found. Trade J.*, 1946, **79**, (1550), 9–11).—A paper read before the 7th Annual Forum, Porcelain Enamel Institute, Inc. (U.S.A.). It is submitted that nickel dips promote adhesion of porcelain enamels to sheet iron and steel, minimize, or, at least, decrease fish-scaling, and control or decrease copper-heading. Full working details are given of a recommended sequence of operations.—J. E. G.

Chromium-Plated Tools Have Longer Life. Andrew A. Spisak (*Machinist* (*Eur. Edn.*), 1946, **90**, (9), 343–345).—The increase in tool life produced by chromium plating is discussed and the process of slowly-controlled chromium plating required for this purpose is described.—J. H. W.

***Investigations of the Structure of Electrodeposited Zinc.** (W. Köhler) See p. 294.

Pre-Polishing Cuts Plating Time 40%. G. R. Makepeace (*Machinist* (*Eur. Edn.*), 1946, **90**, (6), 205).—The saving of time which can be achieved by smooth polishing, instead of rough grinding, before chromium plating, is described.—J. H. W.

IX.—ELECTROMETALLURGY AND ELECTROCHEMISTRY

(Other than Electrodeposition.)

***Theory of the Deposition of Metals from Aqueous Solutions.** P. Krumholz (*Natuurwetensch. Tijdschr.*, 1942, **22**, 108–111; *Chem. Zentr.*, 1941, **112**, (I), 2083; *C. Abs.*, 1943, **37**, 2980).—The effect of NaF and of $FeNH_4$ alum on the reduction of $AgNO_3$ by $FeSO_4$ in an aqueous solution was studied. In the presence of NaF the reduction proceeded practically quantitatively. In the presence of $1{\cdot}5 \times 10^{-3}$ mols. of Fe^{+++}/l. the reduction was only 45% complete. In the presence of gold the retarding effect of ferric ions could be observed, although it was less pronounced. Thus, the rate of crystallization of the silver was affected sooner than the rate of nuclei formation by the gold. It can be assumed that the reduction in the rate of nuclei produced by the ferric ions formed during the reduction is nullified by the NaF. The similar effect of the ferric ions on the rate of crystallization is likewise destroyed. By making use of the assumption of Zsigmondy, that metal in true solution is first formed

during the reduction, it is possible to derive an explanation for the effect of the ferric ions on the rates of nuclei formation and crystallization, which are determined by the supersaturation of the solution with silver. This effect is greater, the more slowly the transition from dissolved to solid silver takes place in relation to the process of reduction in the solution. In the case of gold the solubility can be assumed with certainty to be less than 10^{-12} mols./l.

***On Solutions of Metals [Cadmium] in Fused Salts.** S. V. Karpachev (*Izvest. Akad. Nauk S.S.S.R.*, 1941, [Fiz.], **5**, (1), 43–46).—[In Russian.] An investigation was carried out to determine carbon-electrode potentials as functions of the concentration of metallic cadmium dissolved in fused mixtures of potassium, sodium, and cadmium chlorides. It is shown that the relationship found obeys the thermodynamic law: $E = \text{const.} - \frac{RT}{2F} \ln C$.—N. A.

***The Motion of Solid and Liquid Metallic Bodies in Solutions of Electrolytes.—I.** A. Frumkin and B. Levich (*Acta Physicochim. U.R.S.S.*, 1945, **20**, (6), 769–808).—[In English.] In connection with electrocapillarity and electrokinetic phenomena, particularly with reference to the influence of the electrolyte on the motion of the mercury electrode in polarographic studies, the behaviour of charged particles moving in an electrolyte under the action of an external electric field is theoretically investigated. Non-conducting particles, ideally polarizable and incompletely polarizable metallic particles are separately considered. The theoretical principles involved and the results obtained are fully discussed.—G. V. R.

The Existence of Local Couples in Metal Electrodes. V. Čupr (*Chem. Listy*, 1941, **35**, 185–188, 201–205; *Chem. Zentr.*, 1942, **113**, (II), 139; *C. Abs.*, 1943, **37**, 5318).—Cf. *Korrosion u. Metallschutz*, 1942, **18**, (1), 15–20; *Met. Abs.*, 1945, **12**, 23. A theoretical study and discussion of the concept of Wagner and Traud (*Z. Elektrochem.*, 1938, **44**, 391–402; *Met. Abs.*, 1938, **5**, 491).

†The Mechanism of Electrode Measurements. H. T. S. Britton (*J. Sci. Instruments*, 1946, **23**, (5), 89–94).—A general lecture describing the measurement of electrode potential. The various standard electrodes (hydrogen electrode, glass electrode, &c.) are described and discussed.—W. H.-R.

Elementary Electrocrystallization Processes. K. M. Gorbunova and P. D. Dankov (*Compt. rend.* (*Doklady*) *Acad. Sci. U.R.S.S.*, 1945, **48**, (1), 15–17).—[In English.] The theory of the isolated growth of a face of a single crystal forming part of the electrode of an electrolytic cell is briefly discussed. The effect of overvoltage on the discharge of the ions is considered and it is postulated that, initially, a two-dimensional nucleus forms at the edge of the face, or at the respective trihedral angle. Chains of structural particles may then be deposited along the edge of the nucleus. Further accummulation of ions near the electrode causes formation of more nuclei (on the one already formed) and more chain growth. Growth therefore proceeds by multi-stage layer formation. The course of the deposition in its later stages is discussed in detail and shown to be essentially periodic. The processes are considered mathematically.—G. V. R.

X.—REFINING

The Problem of Pure Manganese. The Chemical Balance Sheet of the Electrothermal Refining of Ferro-Manganese. (M. Déribéré) See p. 282.

XI.—ANALYSIS

***On the Formation and Estimation of Oxide Films on Aluminium.** (Obrist) See p. 298.

***Spot Plate Tests for Copper, Silver, and Nickel.** E. A. Kocsis, Gy Feuer, T. Horvath, E. Kovacs, and L. Molnár (*Mikrochem. ver. Mikrochim. Acta*, 1941, **29**, 166–169; *Chem. Zentr.*, 1942, **113**, (I), 2685; *C. Abs.*, 1943, **37**, 3009).—Cf. Kocsis and R. Horvai, *Mikrochem. ver. Mikrochim. Acta*, 1941, **29**, 44–45. As reagents, use a 0·2% solution of *m*- and *p*-aminobenzoic acid and of *o*- and *p*-aminophenol. With Cu, *m*-aminobenzoic acid gives a yellowish-green spot on filter paper after drying when much Cu is present. It will detect 1 γ of Cu in 0·025 c.c. at a limiting concentration of 1 : 25,000; *p*-aminobenzoic acid behaves likewise but requires 10 γ of Cu; *p*-aminophenol gives, with more Cu, a greyish-blue spot or, with 1 γ of Cu, an olive-green spot with light brown border. The ring starts from the original colour of the reagent. With Ag, *o*-aminophenol gives a reddish-brown or dark yellow spot (2 γ), according to Ag concentration or, with 0·2 γ, a thin yellow ring; *p*-aminophenol gives a light brown spot with dark edges with 0·4 γ of Ag at a limiting concentration of 1 : 62,500. With Ni, *p*-aminophenol gives a blue-grey spot with salmon edges when as little as 0·4 γ of Ni is present at a limiting concentration of 1 : 62,500. Blank tests are made in each case.

***Internal Standard Method of Spectrographic Analysis, as Applied to the Determination of Lead in High-Purity Zinc.** Laurence Griffith and John N. Kirkbride (*J. Soc. Chem. Ind.*, 1946, **65**, (2), 39–48).—A controlled d.c. arc and solutions of Zn dried on graphite electrodes are used in the determination of Pb in Zn in routine control of electrolytic plant. The arc carries 16 amp. over a 6·1 mm. gap and the control of the ignition is by a Tesla-type transformer, which is not described in detail. The source is found sufficiently uniform for the major part of the error to arise in the photometric processes and these are examined in detail. A plate calibration pattern is made with a rotating sector, background densities are read beside each Pb line measured, and each Bi line is used as an internal standard. Each microphotometer reading is converted to an energy reading from the calibration line and line : energy ratios are plotted on a logarithmic scale against log Pb conc. The method is based on synthetic standards made from Zn containing <0·1 p.p.m. of Pb and the calibration line is straight between 2 and 125 p.p.m. The effects of varying arc length, slit width, arc current, depth of crater, exposure time, and plate development time are all carefully examined. Mechanical rocking of the developer is used instead of brush development to save labour, and this is blamed for some of the residual error, which is expressed by an average deviation of 1·6 to 2·1% of the amount present.—E. van S.

***Determination of Tin with Cupferron in the Presence of Antimony.** Élie Gray (*Compt. rend.*, 1941, **212**, 904–906; *Chem. Zentr.*, 1942, **113**, (I), 518; *C. Abs.*, 1943, **37**, 2298).—The method described is as follows: (*a*) oxidize the solution of $SnCl_2$ and $SbCl_3$ with hydrogen peroxide, boil off the excess peroxide, cool, and add a slight excess of ammonia; (*b*) add a little more (not over 5%) than twice the theoretical quantity of cupferron solution and then for every 100 c.c. of the suspension of Sn precipitate add 1 c.c. of 1% gelatin solution and a few drops of 2% tannin solution, filter, wash, ignite, and weigh as SnO_2.

The Use of β-Naphthoquinoline for the Gravimetric Determination of Tungsten. B. A. Platunov and N. M. Kirillova (*Uchen. Zapiski Leningrad. Gosudarst.-Univ., Ser. Khim. Nauk*, 1940, (5), [(54)], 269–275; *Khim. Referat. Zhur.*, 1941, **4**, (4), 73; *C. Abs.*, 1943, **37**, 4983).—In precipitating tungstic acid, β-naphthoquinoline can replace cinchonine. The determination of W is given in detail, but with no other innovation.

***Determination of Zinc in Aluminium Alloys.** A. Cohen (*Helv. Chim. Acta*, 1943, **26**, 75–88; *C. Abs.*, 1943, **37**, 5333).—Method: Dissolve 5·00 g. of alloy in 200 c.c. of 9*N*-H_2SO_4 + 0·8 c.c. conc. HCl. When, after heating, no more hydrogen is evolved, add 5–10 c.c. of 30% H_2O_2 and boil to

remove the excess. At a volume of 250 c.c., remove all Cu by electrolysis. After all Cu has been deposited, filter and wash the residue with hot, dil. H_2SO_4. Then to the filtrate add 40 c.c. of conc. H_2SO_4, cool, and, while stirring, introduce 25 c.c. of reagent (prepared by dissolving 54 g. of $HgCl_2$ and 70 g. of NH_4CNS in 1 l. of hot water). The total volume of solution from which the $Zn[Hg(SCN)_4]$ is precipitated should be about 400 c.c. If the volume is larger, add 10 c.c. of conc. H_2SO_4 for each 50 c.c. of excess volume. This concentration of acid has been found favourable for complete precipitation of the zinc compound. After an hour (or 12 hr. if only 0·01% Zn is present), filter and wash until no red $Fe(CNS)_3$ is left. Transfer the precipitate and ashless paper filter to a porcelain crucible and heat in an electric muffle furnace to light redness. Since poisonous Hg fumes are evolved during the heating, it should take place under a good hood. Cool, transfer the ash to a beaker, and dissolve the ZnO by heating with 10 c.c. of 25% NaOH solution for 10 min. Dilute to 100 c.c. and filter. To recover Zn in the residue, wash it from the filter into a beaker, digest with 5 c.c. of 9*N*-H_2SO_4, and finally add a drop of conc. H_2O_2. Cool, add NaOH in slight excess, boil a few minutes, and filter. To the combined filtrates, add 1–2 c.c. of 50% tartaric acid solution and electrolyse with stirred electrolyte, about 2 amp. of current, and with Cu-plated cathode. Rinse the deposit with water and alcohol, dry at about 90° C., and weigh. The procedure as given provides for the determination of small quantities of Zn, but, with smaller samples, it can be used with as much as 20% of Zn. When a 1-g. sample is used with 20% of Zn, the results are likely to be about 0·75% too low, although 0·2–0·3% of Pb can be precipitated with the Zn.

***Electrolytic Determination of Zinc in Magnesium Alloys.** A. Cohen (*Helv. Chim. Acta*, 1943, **26**, 89–91; *C. Abs.*, 1943, **37**, 5333).—A procedure, very similar to that given in the preceding abstract, is described, in which the Zn is first precipitated from H_2SO_4 solution as $Zn[Hg(SCN)_4]$, the precipitate is converted into ZnO, the residue is dissolved in NaOH, and from the basic solution Zn is deposited electrolytically.

Methods for the Evaluation of Metallic Zinc in Zinc Dust. D. A. Copeman (*J.S.African Chem. Inst.*, 1942, **25**, 62–69; *C. Abs.*, 1943, **37**, 1098).—The methods available for determining Zn in Zn dust are listed. The $Fe_2(SO_4)_3$ reduction method, the gas volumetric method (measurement of H_2 evolved), and the $CuSO_4$ reduction method are discussed in detail and the drawbacks noted. No method seems entirely satisfactory.

Spectrochemical Analysis with the Oscillograph. G. H. Dieke and H. M. Crosswhite (*J. Opt. Soc. Amer.*, 1946, **36**, (4), 192–195).—For spectrochemical analysis where speed is of more importance than precision, it is useful to connect several photo-electronic amplifying valves set in the focal plane of a grating spectrograph to a single oscillograph. A mechanical switch, synchronized with the time-base of the oscillograph, can then be used to show the output of each valve in turn, so that several spectrum lines appear as lines on the screen, whose relative levels indicate their relative intensities. For better quantitative work, the intensity of a line of the main constituent of an alloy is fed to the oscillograph at right-angles to the output from the lines of selected impurities, each of which gives a short, oblique line whose slope is a function of concentration of the impurity. With a transparent scale quantitative readings can be made. Examples of steel analysis are illustrated and precautions are outlined.—E. VAN S.

***On the Quantitative Spectrographic Analysis of Solutions, with an Appendix on the Solubility of Silicon in Aluminium.** Rudolf Walti (*Thesis: Eidgenössische Technische Hochschule, Zürich*, 1943, 71 pp.).—[In German.] A method of analysis for solutions of Al and its alloys is based on the use of a new type of carbon electrode for liquids, which is flooded with the solution to be

analysed at the rate of about 20 ml./min. A spark with high capacity and low self-induction, at about 12,000 V., plays between the surface of the solution and an upper electrode of pure carbon. A small quartz spectrograph is used and the method of calibration depends on the uniformity of development and of the exponent b in the equation $l = nc^b$, connecting the line intensity, l, for an impurity, with its concentration, c. Tables for the product of plate contrast-factor and b are given for Mg, Mn, Fe, Cu, and Si in Al, both alone and in the presence of various concentrations of other alloying elements. The value of n in the equation is checked by repeating the spectrum of one standard solution on each plate. Two spectrum lines are used for each element. By a modification of the method using extra NaCl in the solution Si can be estimated up to 13%, with an accuracy comparing well with that of chemical estimations. A method for distinguishing between *dissolved* and *graphitic* Si in Al alloys uses the fact that only the *dissolved* Si forms SiO_2 when dissolved in acid; the rest of the Si remains insoluble. A complete separation of the soluble and insoluble fractions is made by washing the colloidal SiO_2 off the Si with 1% HF solution. The solution of SiO_2 in acid is then dosed with $AlCl_3$ to provide an internal standard line and is tested spectrographically to determine the Si. The results obtained for the solubility of Si in Al containing about 1% of Fe are slightly higher than usual below 400° C., but lower at 550° C.—E. van S.

Spectral Intensity Measurements with Photo-Tubes and the Oscillograph. (Dicke, Loh, and Crosswhite) See p. 305.

Advances in Microchemistry. V.—Quantitative Inorganic Micro-Analysis. F. Grassner and E. Abrahamczik (*Chemie*, 1942, **55**, (39/40), 299–305; (41/42), 312–318; *C. Abs.*, 1943, **37**, 5331).—A comprehensive review and classification of specific methods for the micro-determination of the various elements. A *bibliography* is given of over 360 references.

Thermoelectric Tester for Checking the Composition of Metals. (Rogers, Wentzel, Riott, and Corbett) See p. 305.

***Researches on the Impurities in Volatile Metals, by Distillation in vacuo.** (Villat) See p. 284.

†The Mechanism of Electrode Measurements. (Britton) See p. 301.

XII.—LABORATORY APPARATUS, INSTRUMENTS, &c.

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

***Further Improvement in the Resolving Power of the Electron Microscope.** James Hillier (*J. Appl. Physics*, 1946, **17**, (4), 307–309).—It is pointed out that though theory indicates that the electron microscope should have a resolving power of 5–10 Å., only 10–15 of over 25,000 exposures made in the author's laboratory (1940–1945) definitely showed a resolving power as good as 20 Å. Owing to the recent development of a new source and telescopic viewing of the final image, magnifications of 300,000 at high intensity are obtained, with a high proportion showing resolutions of 20 Å. This work shows the limiting factor as regards resolution to be lack of axial symmetry in the magnetic fields of the lenses. Attention to this factor has produced resolutions of approximately 10·5 Å., corresponding to a useful magnification of the order of 200,000. It is concluded that the calculated limiting resolving power is obtainable in practice.—G. V. R.

***An Optical Method for Testing Surface Quality.** (Frischmuth) See p. 305.

"Micro-Profilometer." A Device for Studying the Microprofile of Surfaces by the Interferential Method. V. Linnik (*Compt. rend.* (*Doklady*) *Acad. Sci.*

U.R.S.S., 1945, **47**, (9), 630–631).—[In English.] An optical arrangement is described by means of which the topography of a surface may be studied.
—H. J. A.

A Lens to Supplant the Spectrograph Slit. Carl M. King (*J. Opt. Soc. Amer.*, 1946, **36**, (3), 164–168).—Describes the use of a concave, cylindrical lens of short focal length to replace the slit of a grating (or prism) spectrograph and the way in which a secondary aperture and lens system are combined with this lens to provide the equivalent of a continuously-variable slit width in the range 10–130 μ. The advantages claimed are ease of manufacture, durability, ease of maintenance, and high light-efficiency.—E. VAN S.

Spectral Intensity Measurements with Photo-Tubes and the Oscillograph. G. H. Dieke, H. Y. Loh, and H. M. Crosswhite (*J. Opt. Soc. Amer.*, 1946, **36**, (4), 185–191).—Describes a method of using a grating spectrograph with photo-electronic amplifying valves to register the light intensity from a single spectrum line on the oscillograph, using a time-base derived from the current used for the light source if a discharge tube is being examined. When an arc or spark is examined it may be more informative to examine light intensity as a function of the source voltage or source current, with a separate time-base for calibration. An oscillograph capable of dealing with high frequencies is necessary and the photo-electronic amplifier valves must be selected carefully. Differences between the behaviour of different lines in the same spectral source can be detected in this way, as a preliminary to the selection of lines for analysis.—E. VAN S.

Spectrochemical Analysis with the Oscillograph. (Dieke and Crosswhite) See p. 303.

The Geiger–Müller X-Ray Spectrometer. F. G. Firth (*Indust. Radiography*, 1946, **4**, (4), 17–21, 47).—Details are given of an X-ray spectrometer using an asymmetric focusing system, in which a divergent X-ray beam irradiates a flat or curved specimen oscillated at the Bragg angle. The diffracted beams are detected by a Geiger tube situated on a radial arm, with the specimen as centre, which scans an arc that is measured in terms of twice the Bragg angle by a graduated scale. The Geiger tube is linked with appropriate counter circuits and a strip-chart-pen recorder. The applications of the instrument discussed include particle-size determinations, identification of substances (*e.g.*, slags, ore minerals), and the determination of optimum sintering temperature and phase changes in powder metallurgy.—L. M.

Thermoelectric Tester for Checking the Composition of Metals. B. A. Rogers, K. Wentzel, J. P. Riott, and R. B. Corbett (*U.S. Bur. Mines, Rep. Invest.* No. **3690**, 1943, 6 pp.; *C. Abs.*, 1943, **37**, 3709).—The idea of using the thermoelectric character of metals as a means of distinguishing between them is old. An apparatus for quick and routine determinations is described and its use illustrated.

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

***An Optical Method for Testing Surface Quality.** B. Frischmuth (*Schweiz. Arch. angew. Wiss. Techn.*, 1944, **10**, (12), 386).—Discusses principles of an optical method of testing surface quality, based on simple constructional alterations on an ordinary metallurgical microscope.—V. K.

†The Supersonic Method for the Detection of Internal Flaws. E. G. Stanford and H. W. Taylor (*Metallurgia*, 1946, **34**, (200), 59–66).—The principle of the supersonic flaw detector is discussed and a description is given of the equipment used. The results of an investigation to develop a scheme whereby the equipment could be used in production on a routine basis are given, and the examination, in the laboratory, of the results obtained with

the apparatus when used to explore aluminium and aluminium alloy billets and various types of semi-finished components is reported. Special consideration is also given to the preparation of the surface of the material undergoing inspection and to the routine inspection and laboratory investigation of rectangular sections and of cylindrical bar. From the data obtained, it is concluded that the equipment achieves remarkably good results when used for the inspection of extruded samples of simple shape.—J. W. D.

***Investigation of the Heat of Friction and the Temperature Distribution in Wear Tests.** Erich Siebel and Robert Kobitzsch (*Mitt. K.-W. Inst. Eisenforsch.*, 1943, **26**, (7), 97–106).—A method of calculating temperature gradients of specimens subjected to wear test is outlined. The formulæ obtained were applied to the calculation of temperature gradients existing in steel–copper, steel–zinc, and steel–bakelite specimens, tested by dry-wear tests at a speed of 3·12 m./sec. and at surface pressures varying from 100–1000 kg./sq. cm. It is shown, in the case of steel–copper, that temperatures increase with time and surface pressures, while for steel–zinc and steel–bakelite specimens, limiting temperatures (*i.e.*, those of liquid-phase formation for zinc and the decomposition of bakelite) are reached in a thin surface layer in which wear of the specimens takes place.—V. K.

***Investigation of the Cavitation Phenomena.—I–II.** (Brandenberger and De Haller) See p. 297.

***"T.R." Machine for the Mechanical Testing of Metals at Elevated Temperatures.—I, II.** Pierre Chevenard (*Rev. Mét.*, 1942, **39**, (11), 321–330; (12), 353–359).—[I.]—The experimental aspects of the accurate measurement of the creep of metals at high temperatures are discussed in detail and a machine with several desirable features is described. The specimen is small (4 mm. dia. × 32 mm. long). Loads are transmitted to it by the lever principle and elongations are magnified 1000 times and automatically recorded as an ink tracing on a drum. During an experiment, a thermostatically-controlled furnace surrounds the specimen. The tracing is made by a pen on the end of a lever which is mechanically controlled by an electric motor. The motor is in turn controlled by a sensitive electrical contact mechanism, lying at the end of a lever which is operated by changes in the length of the specimen. The separate components of the machine, which includes apparatus for applying loads at various constant speeds, are described and specimen results for several materials are given. [II.]—The use of the machine described above for the investigation of relaxation phenomena is described. Some modifications of the arrangements are necessary, but are easily made. The specimen is subjected to a rapid extension under load and its length is then maintained constant. The relaxation of the load is recorded automatically as an ink tracing on a drum. The specimen length during the experiment is automatically controlled by an electrical device, employing the same type of contact mechanism as previously described. Any slight extension of the specimen sets in motion an electric motor, which operates in such a way as to reduce the load slightly. The consequent movement of the lever by which loads are applied is made use of for the recording device. Specimen results are given and discussed in some detail.—G. V. R.

The Importance of Ultimate Extension as an Engineering Property of Materials. (Fisher) See p. 283.

Comparisons Between Values of Hardness and Mechanical Strength. (Späth) See p. 284.

***Application and Limits of Microhardness Measurement.** Wilhelm Bischof and Berthold Wenderott (*Arch. Eisenhüttenwesen*, 1942, **15**, (11), 497–504; discussion, 505).—The effects of the surface condition and friction on hardness values obtained under loads varying from 3 g. to 100 kg. were studied on pure copper, aluminium, and iron, and martensite, using Vickers, Briviskop, and

Lipps hardness testers. It was found that with loads <100 g., the hardness value is greatly affected by the amount of surface distortion caused by polishing and that hardness values under small loads are greater than those under larger loads, owing to the surface friction of the indentor. A study of the relative magnitude of the elastic and plastic deformations which occur during an indentation was made by the method of measuring the depth or the length of the diagonals of the indentation. The results showed that these are affected by the magnitude of the load and by the structure of the material tested. The choice of loads for microhardness measurement of various phases present in the structure of an alloy depends on the grain-size of the phases present; the relation between these quantities is considered.—V. K.

The Elastic Modulus of Metallic Alloys. (Guillet) See p. 291.

***Changes in Materials Produced in Fatigue Tests.** (Karius, Gerold, and Schulz) See p. 292.

Inspection of Mass-Produced Aero-Engine Castings. P. Cook (*Machinist* (*Eur. Edn.*), 1946, **90**, (9), 325–328).—The rigid inspection system and detailed checking of proof castings required to ensure good quality and to reduce scrap components in a mechanized foundry making light alloy castings are described. —J. H. W.

Inspection in a Mechanized Foundry. T. Cook (*Found. Trade J.*, 1946, **78**, (1538), 133–145).—Read before the Scottish branch of the Institute of British Foundrymen. An illustrated account of the inspection of patterns, core-boxes, and the finished castings.—J. E. G.

RADIOLOGY

Radiographic and Fluoroscopic Interpretation of Casting Irregularities. Thomas E. Piper (*Iron Age*, 1946, **157**, (18), 46–49).—The use of X-ray and fluoroscopic methods to improve non-destructive inspection of aircraft castings is described. The relative definition produced by the fluoroscope and by radiography is discussed and the appearance of 14 types of defect is illustrated. —J. H. W.

Industrial Applications of X-Ray Stress Analysis. Don M. McCutcheon (*Indust. Radiography*, 1946, **4**, (4), 9–16).—The degree of sharpness of the diffraction spots obtained from metals provides a qualitative means of assessing surface stresses. Quantitative measurements can be made by measurement of the diffraction angles, using reference lines from a standard substance recorded on the same film. The techniques are discussed. Examples of qualitative and quantitative stress analysis are given and related to other factors, *e.g.*, applied stress and compressive stress due to surface peening by shot-blasting. The application of the Geiger-counter to this work is described.—L. M.

Microradiography. (Maddigan) See p. 295.

The Geiger–Müller X-Ray Spectrometer. (Firth) See p. 305.

XIV.—TEMPERATURE MEASUREMENT AND CONTROL

***Errors Resulting from Using Compensating Leads with Chromel–Alumel Thermocouples.** Joseph Spacek (*Instruments*, 1945, **18**, (8), 539).—Calculations are given of the errors which may arise when using nickel–copper alloy (composition not specified): copper compensating leads, for the cases when both "cold junctions" are at 300° F. (149° C.) and when they are at 290° and 300° F. (143° and 149° C.), respectively. Errors of 14°–26° F. (8°–14° C.) may be introduced. It is recommended that the lead wires should be of the same composition as the thermocouple elements.—J. C. C.

***A New Thermocouple, Unaffected by the Temperature of the Cold Junction.** Hans Thomas (*Z. Metallkunde*, 1944, **36**, (6), 140–141).—An investigation of

nickel–copper alloys suitable for use as thermocouples is reported. Particulars are given of a new couple, having limbs consisting of two nickel–copper alloys of different compositions (not exactly stated). It has a linear temperature–millivolt relationship over the range 400°–850° C. At 600° C., variation of the cold junction from: (1) −50° to 110° C. gives an error of 13° C., (2) 0° to 110° C. gives an error of 1° C. The oxidation-resistance of the couple is somewhat superior to that of Constantan.—E. N.

***Alloys for Electrodes of Thermoelements and Compensation Leads.** I. Ya. Berkovsky and F. N. Stepanov (*Tochn. Ind.*, 1940, **11**, (6), 13–17; (7), 14–18; *Chem. Zentr.*, 1941, **112**, (I), 1845; *C. Abs.*, 1943, **37**, 2322).—[In Russian.] The thermoelectric powers and their isotherms were determined for alloys of the systems copper–nickel, nickel–chromium, nickel–aluminium, nickel–manganese, and nickel–silicon. The effect of various admixtures on these properties was studied. The suitability of various alloys for the production of compensation wires and the relation between the mechanical, electrical, and physical properties of such alloys and their composition were also investigated. Fusion, casting, and rolling, and subsequent machining and working of these alloys are discussed.

XV.—FOUNDRY PRACTICE AND APPLIANCES

American Practice of Sand Casting Aircraft and Motor Components in Aluminium Alloys. Ya. E. Afanas'ev (*Aviatsion. Prom.*, 1940, (1), 12–19).—[In Russian.] Data obtained on a tour in the U.S.A. are given.—D. A.

***On the Brittleness of Aluminium After Casting.** (Jolivet and Armand) See p. 281.

The Manufacture of Plated Inlay: Steel–Lead Bronze. D. Shneider (*Vestn. Inzhen. Tekhn.*, 1941, 39; *Chem. Zentr.*, 1942, **113**, (I), 922; *C. Abs.*, 1943, **37**, 2325).—Tractor bearings in Babbitts and lead-bronze (copper 70, lead 30%) were tested for 2500 hr. The test showed the superiority of the lead-bronze. Centrifugal pouring caused lead stratification, therefore the inlays were made by hand-pouring, using a flux of borax 84, boric acid 8, and silica 8%. The output was low and up to 50% rejects resulted. As a better method, it is advisable to heat steel sheet to 1100° C., pour a 6-mm. thick lead-bronze layer, apply pressure, and cut out the inlays.

***The Casting Properties of [Aluminium] Alloys.** (Bochvar) See p. 288.

Experiments on Developing Rapid Melting of Scrap Bronze. D. P. Kucherenko (*Tsvet. Metally*, 1941, (4), 27–29).—[In Russian.] Melting was carried out by introducing the charge into a liquid melt.—N. A.

Comments and Observations on Experimental X-Ray Diffraction Stress Methods and Technique for Sand-Cast Magnesium Alloy Structures. Frank D. Klein (*Indust. Radiography*, 1946, 4, (4), 41–46, 49).—K. reaches the undermentioned conclusions, which are discussed *in extenso*: (1) the foundry equipment used for aluminium alloy castings cannot be used for magnesium castings without modification, (2) there is insufficient correlation and exchange of information between designer and foundryman, (3) sufficient study and/or knowledge of magnesium alloy castings is lacking, (4) a study of the control of grain-size is lacking, (5) refining and pouring temperatures require correlation with thickness and accessibility of section, (6) production should be controlled by physical and chemical tests, and (7) (American) designers and foundrymen should adopt alloys other than the magnesium–6% aluminium–3% zinc alloy now more or less standard.—L. M.

A New Feeding Technique for Castings. T. E. Lloyd (*Iron Age*, 1946, **157**, (17), 62–64).—The use of an exothermally-reacting compound—"Thermoto-

mic"—in feed gates for ferrous and non-ferrous castings to provide better metal feeding into the mould is described. The mixing of the compound and its use as a core and facing material and typical applications in roll and bearing castings are discussed.—J. H. W.

Duplex Pump Castings. R. H. Brown (*Found. Trade J.*, 1946, **78**, (1541), 223–233).—Read before the Bristol and West of England Branch of the Institute of British Foundrymen. A general illustrated account. —J. E. G.

The Formation of Banded Structures in Horizontal Centrifugal Castings. C. Howson (*Found. Trade J.*, 1946, **78**, (1545), 346 (letter); **79**, (1555), 135–137, 143).—Correspondence and further discussion on this paper. See *Met. Abs.*, this vol., p. 135.—J. E. G.

Use of the Cumulative Curve for Foundry Sand Control. R. E. Morey and H. F. Taylor (*Amer. Foundryman*, 1946, **9**, (1), 65–75).—See *Met. Abs.*, this vol., p. 187.—J. E. G.

Pressure Die-Casting. G. T. Vicary (*Found. Trade J.*, 1946, **78**, (1540), 201–204).—Read before the Bristol and West of England Branch of the Institute of British Foundrymen. A general account.—J. E. G.

Producing 70 Tons of Die-Castings Daily. Herbert Chase (*Iron Age*, 1946, **157**, (19), 76–80).—The equipment and procedures used in producing 70 tons of zinc die-castings daily in the world's largest single plant is described and the extensive mechanization of the system of handling castings is discussed in detail.—J. H. W.

Some Casting Troubles. J. L. Francis (*Found. Trade J.*, 1946, **79**, (1554), 103–110; discussion, 110–111).—Read before the East Anglian Section of the London Branch of the Institute of British Foundrymen. A general illustrated account in which are discussed, with the aid of typical examples, the causes of defective castings.—J. E. G.

The Technological Principles of Casting Design. Victor M. Shestopal (*Found. Trade J.*, 1946, **78**, (1539), 173–175).—Discussion on the paper. See *Met. Abs.*, this vol., p. 100.—J. E. G.

Some Pattern-Shop Methods and Their Advantages to the Foundry. H. W. Turner (*Found. Trade J.*, 1946, **78**, (1544), 315–320).—Read before the Slough Section of the Institute of British Foundrymen. A fully illustrated account.—J. E. G.

Is the Pattern-Shop an Asset or a Liability? H. Padley (*Found. Trade J.*, 1946, **79**, (1553), 83–87, 92; discussion, 92).—Read before the West Riding of Yorkshire Branch of the Institute of British Foundrymen. A general account dealing with pattern-shop equipment, the essentials of a good pattern, and the application of machinery to the construction of patterns. —J. E. G.

Inspection in a Mechanized Foundry. (Cook) See p. 307.

Foundry Education and Recruitment. Douglas H. Ingall (*Found. Trade J.*, 1946, **79**, (1551), 25–29; discussion, 29–32).—Read before the London Branch of the Institute of British Foundrymen. A general review of some typical technical courses in foundry technology in operation at the present time.—J. E. G.

Technical Records in the Foundry. R. D. Lawrie (*Found. Trade J.*, 1946, **78**, (1547), 387–390; discussion, 1946, **79**, (1553), 89–91).—Read before the Scottish and the Lancashire Branches of the Institute of British Foundrymen. A general illustrated account, in which are detailed the chief advantages to be gained from the keeping of technical records in the foundry. —J. E. G.

XVII.—FURNACES, FUELS, AND REFRACTORY MATERIALS

Development of the Industrial Vacuum Melting Furnace. Werner Hessenbruch and Karl Schichtel (*Z. Metallkunde*, 1944, **36**, (6), 127–130).—A description is given of the development of the vacuum furnace, from its inception as a laboratory appliance to the modern high-frequency furnace of 5000 kg. capacity, working at temperatures over 1800° C. with a vacuum of 0·1 mm. of mercury. Present applications of such furnaces are cited; working with controlled atmospheres and using combined reaction and degassification processes, they have great possibilities for the production of pure metals and alloys.—E. N.

XVIII.—HEAT-TREATMENT

***Water-Quenching of Some Typical Magnesium Casting Alloys.** R. S. Busk and R. E. Anderson (*Trans. Amer. Inst. Min. Met. Eng.*, 1945, **161**, 278–288; discussion, 289–290).—Time–temperature cooling curves for ¾-in. test bars of magnesium alloy indicate that there is a great difference between water-quenching and either air- or oil-quenching. Quenching magnesium-base magnesium–aluminium–zinc casting alloys from the solution-heat-treatment temperature may have a beneficial effect on the properties after subsequent ageing; the extent of this improvement in mechanical properties depends on the composition of the alloy. The possibility of the alloy cracking when being quenched is investigated. The relationship required between quenching temperature and the temperature of the water bath to avoid cracking a specially designed notched plate of magnesium alloy is indicated. The alloys considered are in the composition range: magnesium with aluminium 6–12 and zinc 0–4%, with 0·2% manganese.—H. J. A.

***Induction Heating of Hollow Metallic Cylinders.** Andrew Gemant (*J. Appl. Physics*, 1946, **17**, (3), 195–200).—It is pointed out that rigorous formulæ exist for the variation of heat input with frequency for solid metal cylinders subjected to induction heating, but that analogous equations for hollow metal cylinders are usually presented in an approximate form. A rigorous expression for the heat input in this case is derived and is valid for the whole frequency range. Examples of its use in numerical computations are given and comparisons are made between the accurate results and those obtained by the use of various approximate equations. In general, below the frequency for which the depth of penetration is equal to the wall thickness, exact and approximate equations agree accurately. For higher frequencies, the approximate formula gives a somewhat higher heat input value. For still higher frequencies (penetration markedly less than wall thickness), deviations become small again. The maximum deviation between the rigorous and approximate equations is about 10%.—G. V. R.

***Heat-Treatment of Semi-Conductors and Contact Rectification.** (Serin) See p. 282.

XIX.—WORKING

The Rolling of Aluminium Materials. P. Grüner (*Aluminium*, 1944, **26**, (4), 54–59).—An account of the factors involved in the rolling of pure aluminium, aluminium–copper–magnesium, and aluminium–zinc–magnesium alloys. Types of billets, correct rolling temperatures, permissible reductions, and edge-cracking are discussed and typical structures illustrated by micro- and X-ray photographs.—E. N.

***Grain-Sizes Produced by Recrystallization and Coalescence in Cold-Rolled Cartridge Brass.** (Walker) See p. 293.

Machining of Zinc Alloys. O. H. C. Messner (*Schweiz. Arch. angew. Wiss. Techn.*, 1944, **10**, (12), 389–390).—Machining characteristics of zinc and zinc alloys are reviewed.—V. K.

XXIV.—BIBLIOGRAPHY

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*BRITISH INTELLIGENCE OBJECTIVES SUB-COMMITTEE. **Carl Schenck Torsion Bar Testing Machine.** (FIAT Final Report No. 402.) [Mimeographed.] 9½ × 7¼ in. Pp. 5. London: H.M. Stationery Office. (6*s.*)

*BRITISH INTELLIGENCE OBJECTIVES SUB-COMMITTEE. **Ferro-Alloy Manufacture and Use.** (FIAT Final Report No. 485.) [Mimeographed.] 9½ × 7¼ in. Pp. 14. London: H.M. Stationery Office. (1*s.* 6*d.*)

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