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Symposium on Micro-Metallurgy

MICRO-METALLURGY relating to the role of minute additions to metals and alloys presents a fascinating metallurgical spectrum ranging from basic and fundamental research studies to applied research themes covering the development of alloys and super alloys dependant chiefly for their metallurgical characteristics and physical properties on minute additions of certain elements. The subject of micro-metallurgy, however, is not new. Illustrations of minute additions exercising major damaging effects whilst others conferring equally beneficial properties in the vast and almost unlimited series of ferrous and non-ferrous alloys are abundant—yet the mechanism of how so much is conferred by the so small and the recurring explanations, hypotheses, theories, etc. thereof present to the theoretical scientists and practical metallurgists scope for ceaseless research activity and development applied work. The vast technical literature on the subject, though rich in its technical wealth, presents a wide array of loosely dispersed and scattered technical references in manuals, text books and scientific and technical journals. At the National Metallurgical Laboratory research and development themes to study the role of minute additions to ferrous and non-ferrous metals for the production of diverse ranges of alloys to exacting specifications and needs represent a major field of the laboratory's research activities. The technical and research contributions to this Symposium received from many countries will it is hoped, prove of significant value to the participating Delegates.

It is our privilege and pleasure to welcome the distinguished Delegates from home and overseas to the National Metallurgical Laboratory and a happy landing in our midst. To the warmth of the warming Indian weather, we wish to add the warmth of our welcome.

SYMPOSIUM ON MICRO-METALLURGY—THE ROLE OF MINUTE ADDITIONS TO FERROUS AND NON-FERROUS METALS AND ALLOYS

PROGRAMME

INAUGURAL SESSION

Monday, March 29, 1965

9.00 a.m. to 10.30 a.m.	Registration of Delegates at the NML Main Building
10.30 a.m. to 1.15 p.m.	Visit round the National Metallurgical Laboratory and the Pilot Plants

Break for Lunch

3.30 p.m.	GREETINGS	Dr B. R. NIJHAWAN, *Director,* *National Metallurgical Laboratory,* *Jamshedpur.*
3.35 p.m.	OPENING ADDRESS	Sir JEHANGIR GHANDY, Kt. C.I.E., *Chairman,* *Executive Council,* *National Metallurgical Laboratory,* *Jamshedpur.*
3.45 p.m.	INAUGURAL ADDRESS	Shri ASOKA MEHTA, *Deputy Chairman,* *Planning Commission,* *Government of India, New Delhi.*
3.55 p.m.	ADDRESS	Dr S. HUSAIN ZAHEER, *Director-General,* *Scientific and Industrial Research, New Delhi.*
4.05 p.m.	ADDRESS	Shri M. S. RAO, I.C.S., *Chairman,* *Hindustan Steel Ltd.,* *Ranchi.*
4.15 p.m.	GROUP PHOTOGRAPH	In front of the Main Building of the National Metallurgical Laboratory.
4.25 p.m.	Opening of the New Foundry Block of the National Metallurgical Laboratory	Shri ASOKA MEHTA, *Deputy Chairman,* *Planning Commission, Government of India,* *New Delhi.*

4.45 p.m. Tea at the Lawn of the National Metallurgical Laboratory

SPECIAL LECTURES

Monday, March 29, 1965

5.00 p.m.—5.45 p.m.

Chairman	... ...	Shri ASOKA MEHTA, *Deputy Chairman,* *Planning Commission,* *Government of India, New Delhi.*
Recorder	... ...	Dr T. BANERJEE, *National Metallurgical Laboratory,* *Jamshedpur.*

1. Micro-Production in the Steel Industry	Mr J. R. MILLER, *Special Technical Adviser,* *Centre for Industrial Development,* *United Nations* *New York, U.S.A.*
2. Bismuth in Malleable Cast Iron	Dr ROBERT H. ABORN, *Consultant to American Smelting and Refining Co., U.S.A.* (To be presented by Mr C. Sharma, National Metallurgical Laboratory)

6.45 p.m. Dances of India—at the NML Auditorium, organised by the NML Club (by invitation)

TECHNICAL SESSIONS

Tuesday, March 30, 1965

Session I

9.30 a.m. to 1.00 p.m.

Chairman	... ...	Mr J. R. MILLER, *Senior Technical Adviser,* *Centre for Industrial Development, United Nations.* *New York, U.S.A.*
Recorder	... ...	Dr A. B. CHATTERJEA, *National Metallurgical Laboratory,* *Jamshedpur.*

PAPERS

Titles	*Authors*
1. **Pearlite free structural steels**	J. A. CHAPMAN, R. PHILLIPS, Colvilles Ltd., and Dr R. L. CRAIK, *Head of Laboratories, BISRA, England.*
2. **Rare earth metals in stainless steel making.**	L. NEMETHY, *Manager,* *Atlas India Project, Atlas Steels Company,* *Ontario, Canada.*
3. **Techniques for the study of micro-phases and their influence on material properties.**	D. SCOTT and A. I. SMITH, DSIR, *National Engineering Laboratory, Glasgow, England.*
4. **The purification of iron by selective prepurification and oxidation zone melting; property measurements and Minor impurity contents.**	B. F. OLIVER, F. GAROFALO, S. ARAJS and R. PRIESTNER, *United States Steel Corpn., Monroeville, U.S.A.*
5. **The Role of Residual trace elements in alloy steels.**	Dr J. M. CAPUS, *Research Laboratories, Gillette Industries Ltd., Reading, Berkshire, England.*

Break for lunch.

Tuesday, March 30, 1965

Session II

2.30 p.m. to 5.30 p.m.

Chairman	... ...	Mr J. L. HARRISON, *Manager,* *Iron and Steel Processes* *British Oxygen Co. Ltd.,* *London.*
Recorder	... ...	Dr S. S. BHATNAGAR, *National Metallurgical Laboratory,* *Jamshedpur.*

PAPERS

	Titles	*Authors*
6.	**On the transformation of austenite to martensite and the tempering process of martensite in iron-nitrogen system.**	Prof. Y. IMAI, M. IZUMIYAMA and M. TSUCHIYA, *Metallurgical Research Institute,* *Tohoku University,* *Japan.*
7.	**Effect of Molybdenum on the grain boundary relaxation of iron.**	Dr T. R. S. GOEL, *Research and Advanced Development,* *Delco Radio Division, Kokomo and* Dr F. H. VITOVEC, *Departments of Minerals and Metals and Nuclear Engineering,* *University of Wisconsin, U.S.A.*
8.	**The effect of a small amount of vanadium and niobium on the micro-structures and the strength of pro-eutectoid ferrite of iron-carbon alloys.**	M. TANINO, T. NISHIDA, T. OOKA and K. YOSHIKAWA, *Tokyo Research Institute,* *Yawata Iron and Steel Co. Ltd.,* *Japan.*
9.	**Effect of minor elements on normal grain growth in singly oriented silicon iron.**	M. IMAI, T. SAITO, K. TSURUOKA and M. NISHIDA, *Technical Research Laboratory,* *Kawasaki Steel Corporation,* *Kobe, Japan.*

5.40 p.m. TEA

Wednesday, 31 March, 1965

Session III

9.30 a.m. to 1.00 p.m.

Chairman	... ...	Dr R. L. CRAIK, *Head of Laboratories, Metallurgy Division, BISRA, Sheffield.*
Recorder	... ...	Dr RAJENDRA KUMAR, *National Metallurgical Laboratory, Jamshedpur.*

PAPERS

Titles	*Authors*
10. **Scope of Research in Micro-Metallurgy at the National Metallurgical Laboratory.**	Dr B. R. NIJHAWAN, *Director, National Metallurgical Laboratory, Jamshedpur.*
11. **Micro-Metallurgy of abnormality in steels**	Dr B. R. NIJHAWAN, Dr A. B. CHATTERJEA and Dr S. S. BHATNAGAR *National Metallurgical Laboratory, Jamshedpur.*
12. **Micro-Metallurgy of austenitic grain size control of steels.**	Dr A. B. CHATTERJEA and Dr B. R. NIJHAWAN, *National Metallurgical Laboratory, Jamshedpur.*
13. **Effect of aluminium additions on the primary grain size of steel castings.**	Dr K. B. MEHTA, *Dy. General Manager (Tech.), Heavy Engineering Corpn. Ltd., Ranchi.*
14. **Effect of Micro Impurities and Alloying Elements on Zirconium and Zircaloy-2.**	H. C. KATIYAR, P. PANDE, K. B. MOORTHY and N. K. RAO, *Atomic Energy Establishment, Trombay.*
15. **Distribution and function of micro-phases and micro-additions in metals and alloys.**	Mr L. J. BALASUNDARAM and Dr RAJENDRA KUMAR, *National Metallurgical Laboratory, Jamshedpur.*

Break for lunch

Wednesday March, 31 1965

Session IV

2.30 p.m. to 5.00 p.m.

Chairman	... ...	Mr D. SCOTT, *Head of Metallurgy Section,* *Department of Scientific and* *Industrial Research,* *National Engineering Laboratory,* *Glasgow, England.*
Recorder	... ...	Mr B. N. DAS, *National Metallurgical Laboratory,* *Jamshedpur.*

PAPERS

	Titles	*Authors*
16.	**Morphology and Polygonisation of copper whiskers in the form of ribbons.**	Mrs B. MINARI, Dr H. J. LATIERE and Mr R. MICHAUD, *Centre de Recherches Physiques CNRS,* *France.*
17.	**Importance of Micro-Metallurgy in Non-ferrous metals and alloys used in Navy.**	G. VENKATESWARLU, *Naval Chemical and Metallurgical Laboratory,* *Bombay.*
18.	**Influence of small amounts of Boron and Zirconium on Hot working characteristics and creep rupture properties of some vacuum melted nickel base alloys.**	Lt. Cdr. V. N. MADHAV RAO and Dr R. V. TAMHANKAR, *Defence Metallurgical Research Laboratory,* *Hyderabad.*
19.	**Effect of minor additions of alloying elements on irradiation behaviour and mechanical properties of natural metallic uranium.**	K. BALARAMAMOORTHY, H. C. KATIYAR and N. K. RAO, *Atomic Energy Establishment,* *Trombay.*

5.40 p.m. TEA

Thursday, April 1, 1965

Session V

9.30 a.m. to 1.00 p.m.

Chairman	...	...	Mr J. L. YOUNG, *Management Consultant,* *The Ford Foundation* *New Delhi.*
Recorder	...	...	Mr P. K. GUPTE, *National Metallurgical Laboratory,* *Jamshedpur.*

PAPERS

Titles	*Authors*
20. Effect of Bismuth Additions on the Graphitization of spheroidal graphite cast iron.	Dr CARL R. LOPER, *University of Wisconsin,* *Department of Minerals and Metallurgical Engineering, U.S.A.*
21. Surface tension of cast iron with additions of different elements.	JATINDER MOHAN, *National Metallurgical Laboratory,* FRANCIS MOSTOR and Dr Ing. C. PELHAN, *Metalurski Institute, Ljubljana, Yugoslavia.*
22. Determination of the distribution of impurities in commercial beryllium by electron microscopy.	M. CROUTZEILLES and J. MORICEAU, *Metallurgical Research Centre,* *Pechiney, France.*
23. Studies on micro constituents in metals and alloys by the electron microprobe and by some specialised chemical technique aided with x-ray diffraction.	Dr G. P. CHATTERJEE, Dr B. P. SEN and Dr J. BHATTACHARJEE *Hindustan Steel Ltd., Durgapur.*
24. Effect of Trace Elements on Ageing of some aluminium-base alloys.	Dr VED PRAKASH, *National Metallurgical Laboratory,* *Jamshedpur.*
25. Study of micro-additions to Al-Mg wrought alloys.	Dr Y. N. TREHAN, S. P. BHADRA, P. K. GUPTE and Dr B. R. NIJHAWAN, *National Metallurgical Laboratory.* *Jamshedpur.*

Break for lunch.

Thursday, April 1, 1965

Session VI

2.30 p.m. to 5.00 p.m.

Chairman	... ...	Dr B. R. NIJHAWAN, *Director,* *National Metallurgical Laboratory,* *Jamshedpnr.*
Recorder	... ...	Mr R. M. KRISHNAN, *National Metallurgical Laboratory,* *Jamshedpur.*

PAPERS

Titles	*Authors*
26. Some observations on recent methods of analysis of gases in metals and alloys.	N. G. BANERJEE, *National Metallurgical Laboratory,* *Jamshedpur.*
27. A spectroscopic method of determination of micro-amounts of elements in steels.	R. D. NAIDU and Miss M. NIRMALA, *Defence Metallurgical Research Laboratory,* *Hyderabad.*
28. Spectrographic analysis of micro-elements in Metals aud Alloys.	M. K. GHOSH, P. C. DEBNATH, Miss SUMITRA DASGUPTA, and Dr B. C. KAR, *National Metallurgical Laboratory,* *Jamshedpur.*
29. Analytical methods in micro-metallurgy.	P. KOTESWARA RAO, S. L. N. ACHARYULU and M. K. JOSHI, *Defence Metallurgical Research Laboratory,* *Hyderabad.*
CLOSING REMARKS	Dr B. R. NIJHAWAN, *Director,* *National Metallurgical Laboratory,* *Jamshedpur.*

5.10 p.m. TEA

Abstracts of papers—Symposium on Micro-Metallurgy—The Role of Minute Additions to Ferrous and Non-ferrous Metals and Alloys

1

Pearlite free structural steels

J. A. CHAPMAN, *BISRA*, R. PHILLIPS, Colvilles Ltd., and Dr R. L. CRAIK,
Head of Laboratories, BISRA, England.

THE effects of grain refinement and precipitation hardening conferred by minor additions of Nb and V have been studied in both as rolled and normalised low carbon experimental steels. The mechanical properties of such steels are outlined and related to their structures.

2

Rare earth metals in stainless steel making

L. NEMETHY,
Manager, Atlas India Project, Atlas Steels Company, Ontario, Canada.

A considerable amount of research has been done between 1950 and 1960 in the US and Canada using various types of Rare Earths to improve certain properties of Cr-Ni austenitic stainless steels. After encouraging research work, almost all Stainless S teel Producers started field application of Rare Earths primarily to improve yield by eliminating poor 'Hot Workability' of certain chemically 'unbalanced' alloys. Only a very few papers were published in those years basically because of contradicting results. Several patents have been granted in the U.S. and the Special Alloy Manufacturers anticipated a very bright future for the future use of these elements. Rare Earths are plentiful and chemists were working with them during the past 150 years. Their separation was expensive and scarce. With the wide use of Thorium in nuclear technology, large quantities of Rare Earths became available as a by-product after World War II.

Despite the relative abundancy, the use of Rare Earths is more and more limited in the Steel Industry and was practically withdrawn by 1960 from the manufacture of stainless steels in North America. Suprisingly, authors from other countries claimed recently that the addition of Rare Earths to stainless steels can improve 'Hot Workability' and permit the reduction of Ni content from 10·6% to 9·6% resulting in significant cost saving.

It appears proper to review the history of making stainless steels with the addition of Rare Earths, particularly in light of recent work on properties of Rare Earths.

It is unfortunate that most of the scientific work on phase diagrams, thermodynamic properties of Rare Earth alloys were not known at the time the Stainless Steel Industry experimented with their use, as most of the work of this nature was published after 1960. The information avilable to-day is still far from being complete but it is certainly confirmative for the discontinued use of Rare Earths for stainless steels.

The present paper will discuss: (i) The Chemistry of Rare Earths (ii) Historical Review of Experimental work in Connection with Stainless Steels (iii) Physical Properties of Rare Earth metals (iv) Alloying Behaviour of Rare Earth metals (v) Rare Earths—Stainless Steel experiments done by ATLAS (vi) Interpretation of Results in light of thermodynamical data available.

It can be concluded that further specific work is needed to investigate some properties, the improvement of which could justify the expensive use of Rare Earths additions.

3

Techniques for the study of micro-phases and their influence on material properties

D. SCOTT and A. I. SMITH, DSIR,
National Engineering Laboratory, Glasgow, England.

IN modern materials research the necessity of studying processes in great detail requires the application of special techniques. Several advanced techniques are available for the study of micro-phases and their influence on material properties. Specific information from each technique enables comprehensive data to be obtained. Various techniques and equipment are briefly described.

Electron microscopy is used for the study of fine-scale structural details and electron microfractography for the investigation of failure initiation, mode of failure and the influence of minor constituents. Elegant extraction replica techniques are available for the study of precipitates and electron and X-ray diffraction is used for their identification. Thin foil techniques provide a quantitative approach and a three dimension structural picture. The use of heating, straining and cooling stages in the electron microscope allows events to be followed as they occur and to be recorded by sequential and cine-photography.

Electron probe X-ray microanalysis is used for the study of solution segregation problems, diffusion couples, composition of oxide films, non-metallic inclusions, precipitates, precipitate and nucleation processes, grain boundary and denudation effects, etc.

Potentially powerful new techniques such as the application of probe analysis to the light elements and ion-beam micro-analysis for the identification of isotopes are being developed. New developments such as emission electron microscopy for observing solid surfaces and extending the range of hot-stage microscopy to the electron optical range and the study of temperature-dependent changes as they occur are being assessed.

The potential advantage of the development of new equipment such as the combination of scanning, transmission electron microscopy and probe analysis in one instrument is discussed.

The paper reviews, with a comprehensive list of references to published work, the techniques for study of fine-scale physical processes in metals. The advantages of several techniques are discussed and illustrated by relevant work NEL. This includes studies of the effect of N in Al and Si killed steels, negative creep, structural changes in creep, vacuum melting reduction of impurities in ball bearing materials, tin oxide problem in bearings and H embrittlement, etc.

4

The purification of iron by selective prepurification and oxidation zone melting; property measurements and Minor impurity contents

B. F. OLIVER, F. GAROFALO, S. ARAJS and R. PRIESTNER,
United States Steel Corpn., Monroeville, U.S.A.

GAS-metal oxidation reactions and zone-melting (OZM) were simultaneously employed to produce several high-purity irons in a levitating zone melter. Vacuum melted commercial electrolytic iron and iron produced from hydrogen reduction of distilled ferric chloride were given the OZM treatments. Initial and final analyses for substitutional elements were obtained by mass spectrometric methods.

Irons ranging in purity from 99·89 to approximately 99·998+ with low interstitial contents have been prepared. The major uncertainty in the purity is related to the inability to reliably analyze for oxygen below 20 ppm.

No measureable gradient in the residual carbon content was observed along the entire zone-melted length (6-10 inches) in one-inch diameter bars. The absence of a carbon concentration gradient in the solid indicates an apparent distribution coefficient of 1, an unusual condition.

Internal friction, hot hardness data, and solid state extraction analyses indicate that the level of mobile carbon in α-iron depends on the gas-metal heterogeneous reactions employed. Purification under an oxidizing atmosphere resulted in the immobilization of nearly all of the carbon present at levels of 10 ppm or less. Continuation of purification in a reducing atmosphere returned the carbon to a state of mobility which was again reversed by further purification in an oxidizing atmosphere. The immobilization of carbon at 'residual levels' is believed to be associated with a strong interaction between carbon and oxygen atoms.

Irons with low carbon and nitrogen contents which did not exhibit a hot-hardeness peak were produced after subsequent zone-melting in vacuum.

Tensile stress strain measurements at temperatures below 300°K revealed yield point phenomena both in those irons which did and in those which did not exhibit carbon mobility in hot hardness tests. Although the nature of the upper yield differed among the two categories, in both of them the upper and lower yield points were highly temperature sensitive in the temperature range where deformation twinning was absent. Return of the yield point by low temperature ageing was, in the one case, attributed to carbon diffusion, and in the other case to the diffusion of lattice defects created during prior straining.

Thermal conductivity (λ) of a high-purity iron (99·998+%Fe) has been studied as a function of temperature (T) between 7 and 200°K. Equivalent data is presented for an iron of 99·8% purity. The λ vs T curve for the iron of higher purity reaches a maximum of about 15 W cm^{-1} $°K^{-1}$ at 21°K. This value is considerably larger than that of 4·5 W Cm^{-1} $°K^{-1}$ previously observed by Kemp et al in 1959. Our measurements are in qualitative agreement with the theoretical expectations for a high-purity iron. The problem, associated with accurate determination of the Lorenz number and the thermal resistivity due to electronphonon scattering at low temperatures in iron, is briefly discussed.

5

The Role of Residual trace elements in alloy steels

Dr J. M. CAPUS,

Research Laboratories, Gillette Industries Ltd., Reading, Berkshire, England.

RECENT work in the U.K. on the influence of residual trace elements on the mechanical properties of constructional alloy steel is reviewed. The most fruitful approach has been through the 'synthesis' of high-purity steels. This has enabled the effects of individual trace elements to be studied by making separate small additions.

Thus temper-brittleness can be caused by minute traces of phosphorus, arsenic, antimony or tin, or by much larger amounts of silicon or manganese. The embrittlement of quenched and tempered alloy steels around 350°C can be promoted also by nitrogen.

On the other hand, in high-temperature embrittlement and cast brittleness, as well as the brittleness encountered in welding, sulphur is a contributing element. These phenomena are not well understood.

The deleterious effect of minor residual elements limits the development of alloy steels, particularly for high stength applications, and efforts are now being directed towards purer as well as cleaner steels.

6

On the transformation of austenite to martensite and the tempering process of martensite in iron-nitrogen system

Prof. Y. IMAI, M. IZUMIYAMA and M. TSUCHIYA,
Metallurgical Research Institute, Tohoku University, Japan.

THE transformation of austenite to martensite and the tempering of martensite in iron-carbon system, have been investigated by several workers but very few studies have related to the problem in iron-nitrogen system, in spite of the fact that both nitrogen and carbon have similar atomic volumes and that a forced interstitial solid solution is formed in iron.

In the present work the authors have studied the transformation of austenite to martensite based on statistical thermodynamics and crystallography using Fe-N alloys, and have compared the results to those obtained with Fe-C alloys. Studies on tempering of martensite and austenite in Fe-N alloys were carried out by X-ray diffraction analysis.

7

Effect of Molybdenum on the grain boundary relaxation of iron

Dr T. R. S. GOEL,
Research and Advanced Development, Delco Radio Division, Kokomo and
Dr F. H. VITOVEC,
Departments of Minerals and Metals and Nuclear Engineering, University of Wisconsin, U.S.A.

GRAIN boundary segregation of molybdenum in iron at a range of composition from 0·0006 a/o (0·001 w/o) molybdenum (zone refined iron) to 0·24 a/o (0·41 w/o) molybdenum was studied by internal friction techniques and X-ray microradiography. The activation energy for grain boundary segregation of molybdenum in iron was calculated to be 5 300 cal/mole. This information was used to select the appropriate heat-treating conditions which would promote grain boundary segregation. The segregation of molybdenum in the test samples was verified by X-ray microscopy using Co-Kα radiation.

The internal friction spectrum was determined from flexural resonant vibrations of cylindrical specimens during cooling in a protective atmosphere. Magnetic damping effects were suppressed during the internal friction measurements.

The internal friction peaks observed fall in the same general temperature range as the grain boundary relaxation peaks of iron which are reported in the literature. The activation energy for grain boundary relaxation increases from 54 400 cal/mole for zone refined iron rapidly at first with increasing molybdenum addition and levels off at higher concentrations. This variation indicates that grain boundary saturation by molybdenum is reached at approximately 0·065 a/o (0·11 w/o) Mo.

The temperature of the internal friction peaks increases somewhat whereas the magnitude of the internal friction peak values decreases rapidly with increasing molybdenum concentrations. A new peak could be attributed to molybdenum was not observed within the range of compositions investigated.

The data agree with the concepts of Mott's island model of the grain-boundary structure in general. The solute atoms when adsorbed at the grain bounaries improve the fit at the boundaries and reduce the lattice and grain boundary strain energies. The velocity of sliding decreases, while the grain boundary viscosity, the number of atoms surrounding an 'area of good fit' and the rate constant increase with increasing bulk, concentration of molybdenum in iron.

8

The effect of a small amount of vanadium and niobium on the micro-structures and the strength of pro-eutectoid ferrite of iron-carbon alloys

M. TANINO, T. NISHIDA, T. OOKA and K. YOSHIKAWA,
Tokyo Research Institute, Yawata Iron and Steel Co. Ltd., Japan.

AN investigation was carried out to elucidate the strengthening effect of vanadium and niobium added to the 0·2% carbon steels by means of the transmission electron microscope.

It was found that the strength of proeutectoid ferrite in steels containing vanadium and niobium was generally higher than that in plain carbon steels, and that the maximum strength could be obtained when cooled approximately at 60–80°C/min.

It was concluded that the strengthening of proeutectoid ferrite by addition of vanadium and niobium was attributable to the formation of substructures containing various types of lattice defects and extremely fine particles of vanadium and niobium carbide. In the substructures we have (1) dislocation loops and carbide particles uniformly distributed in ferrite matrix, (2) dislocation dipoles, (3) rows of carbide particles and (4) micro-twins which seem to be a kind of transformotion twin.

The mechanism of formation of such substructures is discussed. The fact that a considerable number of micro-twins were found suggests that the stacking fault energy of alpha iron is considerably lowered by addition of vanadium.

The crystallographic orientation relationship between vanadium carbide and ferrite matrix has been determined. The result is as follows ;

$$(100)V_4C_3//(100)\alpha-Fe,\ (010)V_4C_3//(011)\alpha-Fe,$$
$$(001)V_4C_3//(01\bar{1})\alpha-Fe$$

9

Effect of minor elements on normal grain growth in singly oriented silicon iron

M. IMAI, T. SAITO, K. TSURUOKA and M. NISHIDA,
Technical Research Laboratory, Kawasaki Steel Corporation, Kobe, Japan.

THE normal grain growth behaviour of cold rolled 3% Si–Fe sheet was investigated. The rate of primary grain boundary migration was strongly retarded by the existence of minute quantities of impurity elements such as Ag, Au, Cu, Ni, Ti, Nb, Sb, S, Se, and Te. The free energy of activation for primary grain boundary migration ($\triangle F_A$) was calculated. In specimens containing sufficient addition amount of these elements, $\triangle F_A$ takes a value in close agreement with the free energy of activation for lattice self-diffusion of alpha iron. Impurity atoms with a low intragranular solubility will segregate preferentially at grain boundary because they can there reduce the misfit energy, and then the free energy of activation for boundary migration will tend towards a value approaching that characteristic of lattice self-diffusion.

10

Scope of Research in Micro-Metallurgy at the National Metallurgical Laboratory

Dr B. R. NIJHAWAN,
Director, National Metallurgical Laboratory, Jamshedpur.

THE scope of research in 'micro-metallurgy' bridges across the work of fundamental research studies and that of applied development themes thereby providing the link between theory and practice. The subject of 'micro-metallurgy', however, is not new-almost a decade back, at one of the N.M.L. Symposia, the role of 'micro-metallurgy' in relation to alloy steels was discussed with particular reference to some pioneering work undertaken on the subject at the National Metallurgical Laboratory. Basic and applied researches on diverse facets of 'micro-metallurgy' have since been pursued at the National Metallurgical Laboratory; some of the themes have related to effects of micro-additions on grain-size control of steel and on abnormality, strain-ageing effects in steel in relation to residual micro-constituents, micro-additions of rare earths to stainless steels and aluminium-magnesium alloys, micro-metallurgy of liquid metals, ageing of aluminium alloys in relation to trace elements, studies on phase changes in relation to micro-constituents, micro-metallurgy of aluminium-silicon alloys and heat resistant alloys and analytical techniques for measuring micro-additions to alloys. In this paper, a review has been made of some of the highlights of research results obtained in the foregoing broad themes whilst indicating current thinking on the subject and outlining fresh fields for future work the perplexing problem of melt to melt variations of some of these alloys and the conclusions drawn thereof with respect to influence of Boron and Zirconium have

along the line. The subject of 'micro-metallurgy' is of-course, vast indeed and as such, requires priorities to be assigned, cataloguing of observed hypotheses and defining those needing focussed attention, particularly in relation to research facilites and equipment avilable for introducing specialized research techniques. Research studies undertaken so far have been fully rewarding whilst the search and research on, how so much is conferred by the so minute, present a fascinating metallurgical spectrum for unending research programming, in the planing and execution of which, the National Metallurgical Laboratory currently is actively engaged upon.

11

Micro-Metallurgy of abnormality in steels

Dr B. R. NIJHAWAN, Dr A. B. CHATTERJEA and Dr S. S. BHATNAGAR

National Metallurgical Laboratory, Jamshedpur.

THE subject of 'abnormality' in steels has of late, somewhat receded in the background, even though its practical importance has not diminished. Factors influencing abnormality are related to the micro-presence of aluminium, oxygen, nitrogen, etc. in hyper-eutectic zones of carburized steels; each of these factors has been well investigated and reported upon this paper. The degree of abnormality in different depths of the case-carburized zone of the steel was observed to be affected by the formation of Al_2O_3 as also possibly of AlN in the relevant zones. Carburization treatments with and without oxygen and nitrogen were carried out to substantiate some of the observations made. Steels containing micro-contents of aluminium optimum for austenitic grain growth inhibition, were likewise investigated. The subject of 'abnormality' in steel which perhaps owed its origin to case-carburization treatments to promote high surface hardness and came to limelight following the presence of 'abnormal soft spots', is none-the-less important and interesting from both theoretical and practical stand points, despite the development of alternative modes of surface-hardening other than case-carburization.

12

Micro-Metallurgy of austenitic grain size control of steels.

Dr A. B. CHATTERJEA and Dr B. R. NIJHAWAN,

National Metallurgical Laboratory, Jamshedpur.

MICRO-ADDITIONS of aluminium and nitrogen to steels exercise significant effects on their austenitic grain growth characteristics and physical properties. Effects of such micro-additions judiciously made, in relation to the properties, isothermal transformation characteristics, MS points and grain-growth characteristics of steels have been outlined in this paper. Grain refinement enhanced the impact toughness without affecting the tensile strength of some plain carbon steels. In nickel-chrome steels, grain refinement reduced the susceptibility to temperbrittleness. The phenomenon of abnormal grain growth was comprehensively investigated and earlier hypotheses based on the role therein of sub-microscopic Al_2O_3 particles, residual aluminium and aluminium sulphide were found untenable. Intergranular precipitation was observed on soaking the steels in nitrogen and the precipitated phase was identified as aluminium nitride. The aluminium nitride contents of the steels decreased with rise in temperature. The solution temperature of aluminium nitride in austenite fairly agreed with the grain-coarsening temperature of the steels. In steels containing optimum micro-addition of aluminium, isothermal grain growth below the grain coarsening temperature was not conspicuous. The size, distribution and agglomeration of AlN at higher temperatures were significantly affected by the residual aluminium contents of the steel. It was established that in

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source of data for evaluating certain important properties of the transition region between the liquid and solid phase, surface energy and interfacial energies.

In the present study, surface tension of the Swedish iron with and without 0·05 and 0·5 percentage additions of some elements was studied by capillary-rise method. It was observed that 0·05 percentage addition of Si, Mo, P, Ca, Va, Ni and Cr caused rise in surface tension than that of base Swedish iron; whereas with the same percentge addition of Al, Ti, Pb, Mn, S and Zn to the

microscopic precipitated phase to be AlN-an observation which finds scanty reference in the technical literature on the subject.

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Effect of aluminium additions on the primary grain size of steel castings

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THEORIES of grain-refinement of castings have been put forward on the basis of liquid-solid nucleation by the process of 'Kindred' and 'Foreign' nuclei and crystal growth in the melt. A theory has been developed on the basis of foreign nuclei due to small additions of titanium and other metals in the case of aluminium alloy sand castings. This accounts satisfactorily for the way in which grain refinement takes place and appears to be of more general application. The present investigation is initiated to find out the extent to which this theory can be applied to the refinement of the macrostructure and inherent as - solidified grain size of steel during casting operations by the addition of aluminium and, in particular, whether there is a close relationship between the lattice of nucleating particles and that of the solidifying metal. Attempts have also been made to study the casting conditions snch as the state of deoxidation of the melt, casting temperature, pouring rates and turbulence of the melt during solidification and to relate these to the as-cast primary structure in steel castings.

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Effect of Micro Impurities and Alloying Elements on Zirconium and Zircaloy-2

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RESISTANCE to corrosion in high temperature water and to radiation damage and retention of strength to moderately high temperatures are some of the important factors that have promoted zirconium and its alloys as canning and structural materials for atomic reactor cores.

These properties are very much affected by impurities like oxygen, hydrogen and nitrogen etc. An attempt has been made to review the effect of these impurity levels in zirconium and zircaloy-2. The effects of other alloying elements are also described.

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Distribution and function of micro-phases and micro-additions in metals and alloys

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THE paper begins with an introduction to the fundamental thermodynamic factors responsible for the hunger of metals for solutes. In addition to the discussion of micro-additions, the influence of micro-phases and imperfections is also discussed.

The distribution of micro-constituents is discussed in the liquid and solid states. It is shown that in liquids, solute atoms are usually present in the form of clusters. The influence of the clusters and other heterogenities on solidification and subsequent mechanical properties is then brought out.

In the solid state, micro-constituents in metals and alloys exert their influence either by themselves when present in solution in the matrix, or as micro-phases. After discussing the distribution of micro-constituents on imperfections and grain boundaries, their influence on crystal structure, plasticity, electrical and mechanical properties, phase transformations (including pre-precipitation phenomena, internal oxidation and internal

Swedish iron, the surface tension was lowered; Sb, Sn and Cu however, did not show any important effect thereon. On increasing the percentage from 0·05 to 0·5 of Ti, Mn, Zn and Cu to the base iron, the latter showed higher surface tension. Vanadium which caused somewhat higher surface tension upto 0.05% addition, did not affect the surface addition when added singly upto 0·5%. With the increase from 0·05% to 0·5% of Sb, Sn, S, Al to the base iron, the surface tension was further lowered.

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Determination of the distribution of impurities in commercial beryllium by electron microscopy

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ELECTRON microscopy and microdiffraction techniques were applied to the study of the distribution of impurities in the alloys Be-Fe, Be-Al and Be-Si, and in several varieties of commercial beryllium.

Heat-treatments have no influence on some of the impurities which always remain out of solution either as aluminium or silicon, or as the intermetallic compounds $Be_{13}M$ or $Be_{12}X$.

Iron plays an importont part. At 800°C (1472°F), beryllium can dissolve 0·25% iron. Ageing treatments between 500°C (932°F) and 700°C (1292°F) precipitate the oriented phase $Be_{11}Fe$, mainly in the grain boundaries.

In presence of aluminium, the solubility of iron in beryllium is greatly decreased. The natures of the precipitate appearing during ageing treatments at 500°C and 700°C depend upon the temperature of the solution heat-treatment: when the metal is quenched from 950°C (1742°F), only Be_5 (Fe, Al) precipitates, but a solution treatment at 1100°C (2012°F) induces the precipitation of a mixture of Be_5 (Fe, Al) and Be_{11}-Fe. This phenomenon occurs even when the iron and aluminium contents are very small (35 ppm).

The precipitates appear in the boundaries and decorate the dislocations within the grains.

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Studies on micro constituents in metals and alloys by the electron microprobe and by some specialised chemical technique aided with x-ray diffraction

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DIFFERENT micro-constituents in a metallic specimen can be studied with optical micrcscope and by X-ray or electron diffraction and microscopy. All these techniques provide specific informations with regard to the different phases and micro-constituents present in metals and alloys. In recent years another powerful technique namely the technique ef Electron Microprobe analysis has been successfully utilised in getting informations about the micro-constituents in metals and alloys as well as non-metallic materials.

An attempt has been made in this paper to study the nature of different micro-constituents in metals and alloys with particular reference to steel. The so-called ferrite banding in steel is often attributed to micro segregation of phosphorus. With the help of the Electron Microprobe it was proved that banding may occur for reasons other than phosphorus segregation. The informations obtained from Electron Microprobe studies have been of considerable help to devise ways and means to eliminate the so-called banding in the micro-structructure of steel. By specialised chemical technique it has been possible to study the nature of some of the inclusions in steel.

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Effect of Trace Elements on Ageing of some aluminium-base alloys

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AGEING of an alloy is affected by minute additions of an element, which may result in retardation of natural ageing effects or acceleration of induced ageing. Lowering of ageing capacity in an alloy is purely related to phase changes that the minute additions of an element can bring about. Such changes may result in marked depletion of the alloying element mainly responsible for ageing. Other two effects may be purely physical in nature and are discussed in this paper in some details. Hardy and others had observed that when elements, such as tin, bismuth, lead, magnesium, cadmium or silver are present in quantites$\sim$0·1% in Al-4% copper alloys, the former can almost wholly suppress the natural ageing of the latter and accelerate the induced ageing effects by a factor of 3 to 8 ; such effects have been discussed in this paper in terms of (a) interaction of these elements with vacancies, and (b) creation of additional nucleation sites for pre-cipitation.

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Study of Micro-additions to Al-Mg wrought alloys

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THE solubility of magnesium in aluminium decreases from 15·3 per cent at 450°C to 3 per cent at 200°C and further to about 1 per cent at room temperature, which should render the aluminium-magnesium alloys susceptible to precipitation-age-hardening particularly in the range of 7–10% magnesium ; in practice, however, the latter alloys reveal abnormally low response to precipitation-age-hardening. Increased hardening response may, however, be induced by small additions of zinc, silver and copper. This paper outlines some of the research results obtained at the National Metallurgical Laboratory on the effects of additions of misch-metal upto 3%, silver upto 0·1% and copper upto 0·1% to Al-7% Mg alloys in relation to their response to precipitation-age-hardening. It has been observed that the additions of misch-metal do not cause any significant change in the hardness of the alloys after ageing. Additions of silver and copper improve the response to age-hardening at 250°C of Al-7% Mg alloys ; Such improvement is perceptible on ageing for 24-100 hours after quenching from the solution-treatment temperature. After a peak hardness is reached, the latter drops ultimately attaining the hardness value almost equal to that of the solution-treated alloy. Changes in the tensile strength and 0·1% proof stress have also been studied in the alloys treated under similar conditions, or kept for long periods at room temperature. An increase in the 0·1% proof stress has been observed on ageing. These results have been discussed in relation to some of the theoritical implications of micro-additions to Al-Mg wrought alloys.

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Some observations on recent methods of analysis of gases in metals and alloys

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VACUUM fusion gas analysis has long been used as a standard method for the determination of oxygen, nitrogen and hydrogen in metals and alloys. Conventional methods in use for determination of nitrogen and hydrogen, their merits and demerits are discussed in this paper. Some modifications in vacuum fusion analytical techniques recently introduced have also been outlined. One modification involves the use of emission spectrography to estimate oxygen, nitrogen and hydrogen in metals by melting the sample and extracting the gaseous elements with a d.c. carbon arc discharge in pure argon. Attempts have been made recently to use radio-activation, isotopic dilution and isotopic balancing methods for estimation purposes. Determination of oxygen in beryllium has successfully been achieved by activation with a wide variety of bombarding particles. The method of isotopic dilution has been developed for the determination of oxygen and nitrogen in copper, titanium, zirconium, steel etc. The method of isotopic balancing has been developed for the determination of hydrogen only. The paper also gives the description of the vacuum fusion gas analysis apparatus and hot extraction hydrogen determination apparatus fabricated at the National Metallurgical Laboratory. Some information on the new method for the determination of nitrogen in solid solution by electro-chemical method has also been furnished.

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A spectroscopic method of determination of micro-amounts of elements in steels

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THE paper describes a method of spectrographic determination of small amounts of elements of importance in metals and alloys which sometimes exert a great influence on the metallurgical properties. Such elements in metals and alloys may either be residues with deleterious or beneficial effects or additions made to obtain special properties in the alloys.

A brief survey of the influence of small amounts of elements in certain alloys of importance to defence has been made and a method of Spectrographic determination of these trace amounts of some elements in steel has been described. The co-efficient of variation to assess the precision of the method is given.

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Spectrographic analysis of micro-elements in Metals and Alloys

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Miss SUMITRA DASGUPTA
nad Dr B. C. KAR,
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MINUTE additions of certain elements to both ferrous and non-ferrous metals and alloys exercise pronounced effects on their metallurgical characteristics and physical properties. To study such effects, accurate analyses of the minute additions are required. Methods for the spectrographic analysis of micro-constituents in ferrous and non-ferrous metals and alloys based on some new techniques have been developed at the National Metallurgical Laboratory. In this paper, methods of spectrographic determination of boron in steel, aluminium in steel, magnesium in nodular cast iron, copper and magnesium in zinc base die-casting alloys and antimony in tin-lead non-antimonial solders in use at the N.M.L. have been outlined. Accuracy of such methods, type of spectrograph, source of excitation used and other conditions maintained to obtain reproducible results have been outlined in this paper.

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Analytical methods in micro-metallurgy

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IN this review salient features of the various procedures which have recently appeared in literature during 1960–63 have been discussed at some length also indicating in brief the effects of the elements, which when introduced in very small quantities into metals and alloys produce beneficial properties or modify them.

The occurence of the elements in the metallic materials being of much importance, the method of attack in opening the sample and the subsequent techniques for separating the elements from interfering substances are also considered before the actual determinations are discussed.

The discussions of the various procedures appear in alphabetical sequence of elements in (1) Iron and steel (2) Aluminium and its alloys, (3) Copper and its alloys, etc.

Special methods such as micro and ultramicro-techniques have not come into general use. Even the

instrumental methods are used in their simpler forms and in a somewhat conventional manner. The discussions covered here are as much based on analytical methods currently in vogue.

Although some of the procedures were originally devised for analysis of elements occurring as impurities in metallic materials their utility in the present context is realised and cited where appropriate.

The references cited in the case of some inaccessible literature were read only in the abstract form and given for completeness as well as for ready informatoin of analysis engaged in application of analytical methods in micrometallurgy projects and industries producing or using such materials.

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