

COUNCIL OF SCIENTIFIC & INDUSTRIAL RESEARCH

NATIONAL
METALLURGICAL LABORATORY

ANNUAL REPORT

1960-1961



JAMSHEDPUR, INDIA

CONTENTS

	PAGE
• FOREWORD	1
INTRODUCTION	7
RESEARCH PROJECTS	
Nickel-free Austenitic Stainless Steels	14
Development of Indigenous Alloy, Tool and Special Steels	20
Nodular Cast Iron	24
Study of Indigenous Foundry Moulding Materials	27
Aluminium-bronzes	29
Tinless Bearing Alloys	29
Aluminium-silicon Alloys	30
Powder Metallurgy	31
Light Metals and Their Alloys	34
Continuous Casting	36
Isothermal Transformation Characteristics of Indian Steels	37
Quench and Strain-ageing of Steel	37
Development of Nickel-free Coinage Alloy	41
Production of Magnesium by Electrolytic Method from Magnesium Chloride	43
Electrolytic Production of Chromium and Carbon-free Iron-chromium Alloy	44
Corrosion of Metals and Alloys	44
Preparation of Plating Salts	47
Liquid Gold	48
Determination of Gases in Metals	48
Non-metallic Inclusions in Steels	48
Preparation of Foundry Chemicals	50
Synthetic Cryolite	51
Spectrographic and Polarographic Studies	52
Effect of Hydrogen on the Physical Properties of Metals and Alloys and Mechanism of Phase Transformation in Alloys Deposited at Constant Cathode Potential	54
Beneficiation and Reducibility Studies of Tisco Iron Ores	55
Beneficiation Studies on a Complex Low-grade Copper-Lead-Zinc Ore from Bhotang Mines, Rangpo, Sikkim	57
Beneficiation of Low-grade Manganese Ores	58
Beneficiation of an Amorphous Gypsum Sample from Rajasthan	60
Beneficiation and Briquetting Studies of Fluorspar from Thurwali, Rajasthan	60

Reduction of Iron Oxide Content in a Bauxite Sample from Roadside Mine, Jabalpur District, Madhya Pradesh	61
Screen Analysis and Chemical Analysis of Iron Ore Sample from Kiriburu Beneficiation of Low-grade Graphite from Doomara and Lotiyana Villages, Ajmer, Rajasthan	61
Beneficiation of Low-grade Chromite	62
Beneficiation of Low-grade Garnet from Guda, Ajmer	62
Reduction of Fe_2O_3 Content in a Rutile Sample from Quilon, Travancore Beneficiation Studies on Limestone Samples from Purnapani Quarries, Sunder- garh District, Orissa	63
Flotation of Ferruginous Manganese Ores	63
Studies on the Morphology of Phosphorus in Indian Manganese Ores	64
Production of Basic Refractories from Indigenous Resources	64
Development of Super-duty Silica Bricks	65
Development of High-alumina Refractories	66
Studies on Carbon Refractories... ..	67
Development of Oxide Refractories from the Beach Sands of Kerala	67
Studies on Clays from South India	68
Base-exchange Study on Clays	68
Thermal Conductivity of Insulation Refractories	69
Suitability of Amphibole Type of Asbestos for Industrial Purposes	69
Plasticizing and Colloidizing of Non-plastic Refractory Materials	70
Correlation of Chemical and Physical Properties of Blast Furnace Refractories with Their Resistance to Carbon Monoxide Disintegration	70
Studies on Refractory Brick Forming	71
Development of Magnetic Materials	74
Transformation Studies of Austenite	76
Lattice Parameters of Iron-chromium Alloys and Study of the Sigma Phase in Alloy Steels	77
Preferred Orientation in Rolled Sheets	78
Direct Evaluation of the Effective Camera Radius in a Debye-Scherrer Photo- graph	79
Utilization of Vanadium-bearing Titaniferous Magnetite Deposits	79
Thermal Beneficiation of Low-grade Chrome Ores	80
Production of Ferro-alloys	80
Production of Magnesium from Dolomite	82
Solubility of Nitrogen in Plain Carbon and Alloy Steels	83
Studies on the Production and Recovery of Alumina from Indian Bauxite Utilization of Red Mud for the Recovery of Metallic Values	83
Catalytic Distillation of Titanium from Titanium-bearing Alloys	84
Production of Aluminium Powder	84
Measurement of Carbon Activity in Iron and Its Alloys	84
Development of Electrical Resistance Alloys for Heating Elements	86
Development of Low-alloy High-tensile Structural Steel	89
Development of Thermostatic Bi-metals	91
Development of Controlled Friction Materials	93
Internal Friction Studies	93
Development of Clad Metals	94

Photoelastic Stress Analysis	...	...	...	...	95
Wearing Characteristics of Indigenous and Foreign Grinding Media	...	...	...	...	96
Development of Photoelectric Temperature-controller	...	...	...	...	96

PILOT PLANTS

Low-shaft Furnace Pilot Plant Project	...	...	...	...	97
Pilot Plant for Production of Electrolytic Manganese Metal	...	...	...	...	104
Pilot Plant on Hot-dip Aluminizing of Steel Wire and Sheet	...	...	...	...	106
Pilot Plant for the Beneficiation of Low-grade Manganese, Chrome, Iron and Other Similar Ores	...	...	...	...	108
Pilot Plant for Refractories	...	...	...	...	112
Pilot Plant for the Recovery of Vanadium Pentoxide from Vanadiferous Magne- tite Ores	...	...	...	...	114
Pilot Plant Studies for Thermal Beneficiation of Low-grade Manganese Ores	...	...	...	...	115
Pilot Plant for Pyrometallurgical Production of Magnesium from Dolomite	...	...	...	...	116
Pilot Plant for the Production of Ferro-alloys	...	...	...	...	116

ELECTRIC FURNACE COMMITTEE	...	...	...	...	118
----------------------------	-----	-----	-----	-----	-----

INDUSTRIAL LIAISON, OPERATIONAL RESEARCH & PROJECT REPORTS

Plant Visits	...	...	...	...	120
Utilization of Low-shaft Furnace Slag	...	...	...	...	120
For Slag-cement	...	...	...	...	121
For Light Weight Aggregate	...	...	...	...	121
Investigation on Failures of Metals in Services	...	...	...	...	122
Project Reports	...	...	...	...	123

ENGINEERING SECTIONS

Design Engineering	...	...	...	...	124
Electronics Engineering	...	...	...	...	125
Electrical Engineering	...	...	...	...	125
Mechanical Engineering	...	...	...	...	126

INFORMATION SERVICE

Publications	...	...	...	...	127
Colloquia	...	...	...	...	128
Translation Service	...	...	...	...	128
Patents and Technical Information	...	...	...	...	128
Library and Documentation Service	...	...	...	...	128
Technical Aid to Industries	...	...	...	...	129
Processes Patented	...	...	...	...	131
Practical Demonstration	...	...	...	...	132
Symposium	...	...	...	...	132

AWARDS, DEPUTATIONS AND NOMINATIONS

Awards	...	...	...	...	...	...	141
Deputations	...	...	...	...	...	...	141
Nominations	...	...	...	...	...	...	141
Distinguished Visitors	...	...	...	...	...	...	143
GENERAL	...	...	...	...	...	...	144

APPENDIX I

Papers Published

Research Papers	...	...	...	...	...	146
Technical Papers	...	...	...	...	...	147
Letters to the Editor	...	...	...	...	...	149
Papers Presented at Symposia and Technical Sessions	...	...	...	...	...	149

APPENDIX II

Investigation and Research Reports	...	...	...	...	151
Survey and Special Reports	...	...	...	...	152

FOREWORD

I HAVE great pleasure in presenting the Report of the Director, National Metallurgical Laboratory for the year 1960-61.

The National Metallurgical Laboratory has now entered the Third Five Year Plan period having nearly completed the expansion envisaged during the Second Five Year Plan and has prepared itself for shouldering heavy responsibilities during the Third Five Year Plan.

The Laboratory's research, development and pilot-plant work has been suitably oriented to meet the urgent requirements of general expansion of mineral and metallurgical industries during the successive Five Year Plans. The hard core of the Second Five Year Plan has been the expansion of the iron and steel industry; in this task, as in many others, the National Metallurgical Laboratory has geared itself to render all possible assistance both to the public and private sectors and acted as a king-pin in the solution of the multitudinous short and long term problems posed before it which were all investigated with thoroughness, speed and technical efficiency, which I am glad to say is the characteristic of the National Metallurgical Laboratory. Great emphasis is being continually laid on heavy pilot plant projects and investigations and prototype trials with the objective of industrial scale implementation of important research and pilot scale processes developed at the National Metallurgical Laboratory; it is in these spheres of the Laboratory's activities that further expansion far greater and broader in scope than in the Second Five Year Plan that has been projected for the Third Plan period. This is indeed a very welcome and healthy development because pilot plant operations speed up the translation of the latest technological concepts and innovations into acceptable industrial plant operations. With the fast expanding pattern of the industrialization of our country covering today a wide spectrum, the research and development projects of the National Metallurgical Laboratory are planned and executed to provide adequate yet at times most difficult answers through research and pilot plant investigations for the ever-increasing problems of the smallest metallurgical units to the giant integrated industrial complexes now emerging. These aspects of the work of National Metallurgical Laboratory have been related to both iron and steel and non-ferrous industries where heavy expansion has been planned during the Third Five Year Plan. Significant in this context was the Annual Symposium arranged by the National Metallurgical Laboratory in February 1961, which was devoted to different aspects of 'Light Metal Industry in India'. The Symposium was a great success and went a long way in focussing attention on our deficiencies and expansion plans for the projected growth of light metal industry during the Third Five Year Plan.

In my 'Foreword' to the last Annual Report I had referred to the important new Pilot Plant Technological Block, housing several important pilot plants which was inaugurated last year. These pilot plants, it is particularly gratifying to note, are now in operation with most of the pilot plant equipment designed and fabricated in the Laboratory's workshop, effecting considerable savings in foreign exchange. Keeping up the general theme of expansion, I am glad to state that the Laboratory is now adding to its Main Building two side and one central wings at a total cost of about Rs. 1.5 million. These wings will contain the new radioactive isotope metallurgical research laboratory, new corrosion laboratories, X-ray fluorescence unit, heavy vacuum melting equipment and components for pressure leaching and hydro-metallurgical research activities of the National Metallurgical Laboratory. These extensions are expected to be completed within the next two years and will be valuable additions indeed. Concurrent with this expansion, the Governing Body of the Council of Scientific & Industrial Research has also sanctioned another heavy pilot plant to beneficiate and treat about 25 tons of low grade manganese ores a day involving a total capital and recurring expenditure of Rs. 5 million out of which Rs. 3 million will be the capital cost of heavy equipment to be imported from abroad under the U.S. Exim Bank Scheme. Land covering over 6 acres has been donated by the Tata Iron & Steel Co. Limited. This pilot plant will be located next to the Low-shaft Furnace Pilot Plant of the National Metallurgical Laboratory and will use the clean low-shaft furnace gas for reduction roast trials of the ferruginous low-grade Indian manganese ores during their beneficiation. This pilot plant is also expected to be completed during the next two years. We are sure that this pilot plant for the beneficiation of manganese ores of India will be of very considerable economic importance to this country. It is also most gratifying that the plans for the installation of a submerged electric arc ferro-alloy furnace at a total cost of Rs. 0.6 million have now been completed and the furnace is expected to go into production early next year. This pilot ferro-alloy furnace with a daily output capacity of over a ton of different ferro-alloys will be the first of its kind to be installed in India and perhaps in the whole of the East, and will be a valuable tool in research and production development work on ferro-alloys to be made from indigenous resources. This pilot plant will be inaugurated early next year and will be available to the general industry for pilot scale ferro-alloy production trials on 'Contract' basis. With these expansions to the Laboratories and different pilot plants, there is no doubt that the National Metallurgical Laboratory will be one of the largest of its kind anywhere in the world and we should feel proud of its stature and activities even as it justifiably does today, deriving its strength from the valuable research and development work completed during the Second Five Year Plan.

During the year under review, Professor Humayun Kabir, Minister for Scientific Research & Cultural Affairs, Government of India, visited the Laboratory and was greatly impressed with the equipment and facilities provided in the National Metallurgical Laboratory and spoke very highly of the excellent standard of research and development work in progress. I might say here that many of the scientists and engineers from overseas, who had visited the National Metallurgical Laboratory, had told me that there were perhaps very few places in the world, including the U.S.A., where such extensive research facilities and equipment

had been provided under one roof for metallurgical research and pilot plant trials.

To me, it appears that there is hardly any necessity to send our problems, particularly on raw materials, to outside laboratories for investigations or evaluation. It is my pleasure to state that on an assignment given to National Metallurgical Laboratory by the Tata Iron & Steel Co. Ltd. for beneficiation of iron ore, particularly with regard to the treatment of fines and a balanced alumina-silica ratio, the systematic and comprehensive investigations carried out at the National Metallurgical Laboratory has proved most valuable. Research investigations at the National Metallurgical Laboratory have shown that by washing, screening and heavy media separation, the physical characteristics of the iron ore can be greatly improved which would render the iron-making operations much more smooth and metallurgically more efficient. It is also highly gratifying that similar work has now been undertaken for the steel plants of the Hindustan Steel Limited, Government of India, as also on systematic studies of metallurgical problems, such as quality assessment of the L.D. oxygen steel including its sub-zero physical characteristics, studies on the deadburning of the dolomites in vertical shaft kilns and their stabilization for use in L.D. oxygen steel-making.

In the present stage of the Laboratory's development work considered in the context of our successive Five Year Plans, priority is naturally given to operational and developmental research projects, but researches on fundamental metallurgical problems have also made good headway and considerable fresh ground has been broken. Consolidation of the work in diverse fundamental fields already in hand has been undertaken *pari passu* with the development of different pilot plant projects.

The Laboratory has now taken in seven Fellows working on fundamental fields of research for a period of three years with the object of obtaining higher degrees from Indian universities. This is a useful innovation and should prove exceedingly useful for getting trained personnel in diverse metallurgical research fields.

During the year, the Laboratory brought out two excellent publications; one relating to the Monograph on "Foundry Moulding Sands of India" and the other relating to the Proceedings of the Symposium on "Pilot Plants in Metallurgical Research and Development" held in February 1960, which have been very well reviewed and highly spoken of in India and abroad; the former publication contains the research results on India's foundry moulding sands and bonding materials obtained during the past several years of work at the National Metallurgical Laboratory which are proving valuable to the India's foundry industry fast expanding under the tempo of the Third Five Year Plan. The Symposium publication has been classified as one of the best of its kind brought out within just five months after a Symposium and is finding a place in the technical libraries the world over. The Symposium itself was termed as a 'metallurgical summit' in having drawn scientists and technologists, specialists in their fields, from different parts of the world and providing thereby an international forum for exchange of technical 'know-how'.

During the year, the Laboratory has taken out several new patents. Practical demonstrations were arranged to acquaint interested manufacturers with the know-how of the National Metallurgical Laboratory's patented processes; some of which have been released on payment of heavy premia and production royalties — the process on the production of Electrolytic Manganese Metal and Electrolytic Manganese Dioxide will shortly go into full-scale industrial production by a leading firm in Bombay paying a lakh of rupees as the premium with $2\frac{1}{2}$ per cent production royalties. Likewise, several other processes relating to production of refractories from indigenous resources and other fields have been passed on to Indian industries through the National Research Development Corporation; these are healthy and highly encouraging developments.

Important research investigations in progress in different fields are illustrated in appropriate section of the Report of the Director, National Metallurgical Laboratory.

Highly important work on the development of substitute alloys with partial or full substitution of these metals in which India is lacking has made further significant headway. Research and development work on the nickel-free totally austenitic chromium-manganese-nitrogen-copper stainless steel has made excellent progress; the process is now covered by National Metallurgical Laboratory patents taken in India and abroad. Highly valuable results in this project have been obtained towards its production on industrial scale for multitude applications; work in fundamental fields of this project has also yielded valuable results applying X-ray diffraction techniques to 'aged' stainless steels. Studies on cold working characteristics, high temperature oxidation and creep properties in their fundamental aspects of these steels have yielded highly interesting results. Its use for pressure cooker bodies and lids, the latter requiring rigid springy characteristics, has now been established. The development work on bi-metals and other substitute alloys, such as nickel-free heating elements, is now entering its final phase prior to industrial scale implementation. Two general-purpose bi-metals have been developed: one for application up to 220°C . and the other for application up to 300°C . The nickel-free heating elements have given during industrial scale trials higher electrical resistivity than the conventional heating elements that are imported. Full use has been made in these projects of indigenous resources, such as aluminium, chromium, manganese and rare earth group of metals; it is the latter group that is now leading to the development and formulation of indigenous aluminium-based magnesium wrought light alloys containing optimum additions of rare earth metals at the National Metallurgical Laboratory.

Likewise, other interesting developments have been recorded during the year under review, such as the production of high purity iron powder from 'Blue Dust' pockets met with in Indian iron ore mines, through an ingenious process of solid state reduction developed at the National Metallurgical Laboratory. This fine iron powder has immediate market needs in India considering that the entire demands thereof are met from imports. This process is now ready for passing on to the industry towards which due steps will be taken. Work on the production of nickel plating salts from indigenous resources has yielded good results and the National Metallurgical Laboratory's plating salts have been reported to be as good as, if not better than, those imported.

The Laboratory has continued to collaborate with different Ministries of the Government of India in their research and development programmes, such as the formulation of the production technique of aluminium-bronze castings for the 25 kV. traction overhead equipment for the Ministry of Railways, production of aluminium powder of high purity and super-pure magnesium metal and ferro-alloys for the Ministry of Defence, Government of India.

Apart from the development work, the Laboratory has maintained steady progress in the operations of its various pilot plants. The pilot plant work on hot-dip aluminizing of M.S. wire has received appreciation from the British Iron & Steel Research Association, U.K., for the quality of the aluminized wire samples produced from the National Metallurgical Laboratory's pilot plant. Several miles length of aluminized steel wire have been supplied to the Post & Telegraph Department, Government of India, for field applications on their telephone lines. The National Metallurgical Laboratory's pilot plant installed for the production of electrolytic manganese and manganese dioxide has been in continuous operation meeting the requirements of manganese metal for research projects on nickel-free coinage alloys, nickel-free manganese brasses and heating elements besides nickel-free austenitic stainless steels using electrolytic manganese after nitriding it at the National Metallurgical Laboratory.

In the Low-shaft Furnace Pilot Plant, recent campaigns on the smelting of iron ores using low temperature carbonized coke from the Singareni Colliery of Andhra Pradesh have yielded very valuable results. The furnace operations have been smooth, fuel consumption has been reasonable and difficulties following hanging and high-top temperatures have also not been encountered. Successful investigations are also currently under way for the utilization directly of non-coking coals from Singareni without any prior drying or carbonization treatments. I am happy to say that the National Metallurgical Laboratory is actively collaborating with the Governments of Maharashtra, Andhra Pradesh, Rajasthan and East Punjab in conducting smelting trials with their regional raw materials in the Low-shaft Furnace Pilot Plant. Work is also being initiated to help the Government of Nepal to establish a small scale iron production plant in Nepal using their raw materials. The Low-shaft Furnace Pilot Plant has been in continuous operations round the clock practically throughout the year under review.

A demonstration of the National Metallurgical Laboratory's process on Liquid Gold was also arranged during the year to acquaint interested manufacturers with the technical 'know-how' of the process to whom this process has been leased. As metallurgical industries are becoming increasingly conscious of the importance of research in finding effective solution of the industrial problems, numerous technical operational research and investigation problems are being ever-increasingly referred to this Laboratory which are successfully handled by the operational research staff of the National Metallurgical Laboratory; particularly dominant in these fields has been the problems of Indian foundries.

The *NML Technical Journal* has stepped into the third year of its publication and has attained a stature at par with other leading Indian and overseas scientific journals. The *NML Technical Journal* is now being exchanged with the leading metallurgical journals from other countries and at home on a free basis effecting foreign exchange savings.

It is my pleasure to state that not only high research standards and tempo of work have been fully maintained at the National Metallurgical Laboratory, but also that extraordinarily valuable work is being done laboriously, systematically and comprehensively in the National Metallurgical Laboratory.

The National Metallurgical Laboratory has always enjoyed the full support and active goodwill of the Director-General, Scientific & Industrial Research, Professor M. S. Thacker, who has taken a keen personal interest in the Laboratory's activities. The Council of Scientific & Industrial Research have established their usefulness in the various fields of national development and I am indeed very happy to note that the National Metallurgical Laboratory is holding a position of pride in this organization. I would like to express on my behalf and that of other Members of the Executive Council our sincere appreciation of the extremely hard and devoted work that is being done by Dr. B. R. Nijhawan, Director, National Metallurgical Laboratory, and his able colleagues. I would like them to know that the Members of the Executive Council of the National Metallurgical Laboratory deeply appreciate their highly creditable and laudable efforts. Much of the success of the Laboratory's widening scope of work is undoubtedly due to the hard work, research and organizational ability of its Director, Dr. Nijhawan. The National Metallurgical Laboratory is today on the threshold of a great expansion in different spheres during the Third Five Year Plan, and I am sure the Members of the Executive Council and others would join me in offering Dr. Nijhawan and his colleagues our best wishes for fresh laurels and achievements.

J. J. GHANDY

Chairman

Executive Council of the

National Metallurgical Laboratory

Jamshedpur
May 1, 1961

INTRODUCTION

During the year under review, the National Metallurgical Laboratory has maintained progressively increasing and unceasing activity in many fields making notable progress over a wide spectrum ranging from fundamental, theoretical and laboratory scale research studies to round the clock pilot plant trials approaching industrial prototype production campaigns extending over several months' unbroken operations, references to which will be found in appropriate pages of this Annual Report. Under the impact of our Five Year Plans, the need for metallurgical research and development work in general in the first place and the total indispensability of associating the National Metallurgical Laboratory with national projects relating to the growth of mineral and metal industries under execution or in operation or those projected have not only been well established but today adequately appreciated in related private and public sectors of this country. A heavy pilot plant on the beneficiation of low-grade Indian manganese ores is now being established at the National Metallurgical Laboratory with a total capital and recurring expenditure of over Rs. 50 lakhs during the Third Five Year Plan. Exceptionally speedy action has been taken by the National Metallurgical Laboratory on the procurement of heavy pilot plant equipment chiefly from the U.S.A. under the U.S. Exim Bank Credit Scheme. Different items of equipment indigenous or those to be imported were more or less finalized during the year under review, including the latest analytical equipment for X-ray fluorescence spectroscopy. Acquisition of 6 acres of land from the Tata Iron & Steel Company, to whom our grateful thanks are due, has been completed. Early during the Third

Five Year Plan, this heavy and integrated pilot plant, one of the biggest of its kind anywhere in the world, would start functioning and will undoubtedly prove to be of valuable benefit to the Indian manganese industry. Action likewise to procure the pilot submerged arc electric furnace for smelting of ferro-alloys costing several lakhs of rupees has been finalized.

The Low-shaft Furnace Pilot Plant has made, during the year, valuable progress and some highly promising results have already been obtained. In this connection, it may be stated that the original Demag-Humboldt process of briquetting the raw material fines and using them as the main burden for the low-shaft furnace has proved after comprehensive trials to be of academic interest under Indian raw materials and weather condition, this original briquette smelting process is being most successfully replaced by alternative Indian processes based on the charge of sized lumpy raw materials and the use of low-temperature carbonized coke made of non-metallurgical coals, with the use of which excellent results have emerged and which should pave the way for the establishment of iron smelting plants on regional basis in different parts of the country. The Low-shaft Furnace Pilot Plant is to be further considerably expanded during the Third Five Year Plan including the use of oxygen enriched air-blast for smelting of pig iron and ferro-manganese from Indian raw materials. During the year under review, raw-materials for pilot smelting trials in the Low-shaft Furnace Pilot Plant were obtained from different parts of India.

Keeping in view the basic objectives of utilizing non-coking coals and inferior grades of raw material for iron smelting in the low-

Shaft Furnace, further continuous trials were conducted round the clock extending over several months and valuable results have been obtained. Iron ores from Orissa and Bihar have been smelted with large varieties of non-coking coals from Raniganj and Disergarh coalfields either as blended lumpy charge or by prior briquetting. The possibility of using iron ore from Chanda District with non-coking coals from Ballarpur, Kampti and Wardha (Maharashtra State) have been thoroughly investigated. Smelting trials have also been undertaken using self-fluxing briquettes made from iron-ore fines, limestone and slack coals primarily with a view to utilize run-off mine fine grained materials. Successful investigations have also been conducted with iron ores employing non-metallurgical coals from Singareni as such and low-temperature carbonized coke made from non-coking coal of Singareni Coalfields in Andhra Pradesh; these smelting trials have yielded interesting results in respect of fuel consumption, slag volume, operational characteristics and production output with increasing percentage of briquettes in the burden as well as with lumpy burden consisting of iron ore, limestone and nut-coke apart from the exclusive use of non-coking coals containing high volatile and ash contents. A complete report on smelting investigations with Andhra Pradesh raw materials is now under preparation. Smelting investigations with raw materials received from the State Governments of East Punjab and Rajasthan will also be undertaken shortly which will be followed by similar trials on raw materials for other parts of India particularly the Salem Magnetite ores and high-temperature carbonized lignite coke from Neyveli (South Arcot).

Work has also been initiated for the Government of Nepal, for establishing a small scale iron producing plant in Nepal. Smelting trials will be taken up in the Low-shaft Furnace Pilot Plant at the National Metallurgical Laboratory, utilizing regional Nepalese raw materials for iron production.

The pilot plants, relating to other subjects have also made notable progress. In this direction, efforts are constantly made to keep in touch with the latest developments abroad and also getting our results checked and confirmed from overseas research institutions. The pilot plant work on hot-dip aluminizing of M.S. wire has maintained excellent progress. Some of the initial samples of aluminized steel wire made at the National Metallurgical Laboratory in the Pilot Plant designed and fabricated at the Laboratory, were sent to the British Iron & Steel Research Association, U.K., for their detailed study of structure and properties; their detailed report on the subject has spoken highly of the National Metallurgical Laboratory's aluminized products, stating them to be "exceptional in quality and setting up a very high standard, and also that it was most gratifying that there was no evidence of failure of the alloy layer despite the fact that we had not used an active flux but a protective film on the steel wire".

Progress on pilot plant investigations has been maintained *pari passu* with other research projects under way at the National Metallurgical Laboratory both in fundamental and applied fields. Another field in which notable progress has been made is in the implementation of the processes developed at the National Metallurgical Laboratory on an industrial scale through the National Research Development Corporation. During the current year, the National Metallurgical Laboratory patented processes on the production of electrolytic manganese and manganese dioxide and Liquid Gold were released to the industry through National Research Development Corporation for commercial exploitation on payment of lump-sum premia and royalty basis. There are other processes now in the process of such release to the industry and it is hoped that the rate of such industrial scale utilization of the National Metallurgical Laboratory's patented and non-patented processes will maintain its steady progress.

The services of the Laboratory were heavily called upon by the Hindustan Steel Limited, Government of India, for systematic studies of metallurgical problems posed by their almost virgin raw materials such as iron ores and fluxes needed, including studies on the deadburning of dolomites and their stabilization for use in L.D. oxygen steel-making and study of the sub-zero and room temperature physical characteristics of the L.D. oxygen steel made. As a part of the programme for giving technical aid, detailed beneficiation investigations were conducted on complex low grade copper-lead-zinc ore from Bhotang Mines, Rangpo, Sikkim, at the request of the Government of India for Sikkim Mining Corporation, received with the comments that "the results indicated by the test in your Laboratory are certainly comparable to some of the best achieved in other parts of the world and perhaps cannot be bettered except in conditions of commercial scale processing".

Systematic work has also been undertaken on the assessment of iron ores for the National Mineral Development Corporation for Kiriburu iron ores for export to Japan and eventual use in Bokaro Steel Plant. Beneficiation and reducibility studies on iron ores which normally form the ore burden of blast furnaces of the Tata Iron & Steel Company need special mention, as in the case of Indian iron ores, the problem is twofold: one of improving the quality of the ore despatched from the mines and the other of ensuring regular supplies of properly blended and sized ores to the plant. In order to achieve a complete solution of this problem, comprehensive beneficiation studies of the iron ores were undertaken including a study of the sintering characteristics of the ore fines. A detailed report was furnished to the Tata Iron & Steel Company based on which the installation of a heavy beneficiation plant is under active consideration by them. In this connection, an extract of the speech delivered by Mr. J. R. D. Tata, Chairman, Tata Industries, in his 53rd Annual Report

— 1959-60, states "I wish to record our appreciation of the assistance we have received from the National Metallurgical Laboratory at Jamshedpur whose investigations, undertaken at our request, have formed, to some extent, the basis of our ore beneficiation programme."

Magnesium, one of our light metal assets, which is indispensable in the production of special alloys for aircraft and used in its pure form for strategic military requirements, has been successfully produced in the Laboratory for the first time in India from dolomite by silico-thermal reduction of dolomite; reserves of which are classic in our country. Magnesium so produced at the National Metallurgical Laboratory conforms to rigid specifications laid down by the Ministry of Defence.

For the establishment of alloys, tools and special steel plants proposed to be set up during the Third Plan Period, there will be a great demand for different special types of ferro-alloys, such as low-carbon ferro-chromium, ferro-vanadium, ferro-tungsten, nitrided ferro-alloys, etc. Carbon-free ferro-chrome, by aluminothermic reaction, was successfully produced with chromium ranging from 75-77 per cent, iron 22-25 per cent and carbon in traces and supplied to the Ministry of Defence conforming to rigid ordnance specifications and used successfully by them for production of special alloys.

Several important projects on the development of substitute alloys based on indigenous alloying elements to the exclusion of those whose resources are deficient or totally non-existent in India, such as nickel, cobalt, molybdenum, tin, etc., had made remarkable progress. These projects relate chiefly to the development of austenitic chromium-nitrogen-manganese-copper stainless steels, nickel-free coinage alloys, nickel-free heating elements, manganese-bearing brasses based on indigenous alloying elements such as chromium, titanium, manganese, aluminium, rare earth group of metals and the ever-abundant nitrogen. Comprehensive investigations were continued on the determination of physical,

metallurgical and corrosion resistance properties of austenitic nickel-free stainless steel in various media and their X-ray diffraction, magnetic and dilatometric studies have been completed apart from a study of their age-hardening characteristics and high temperature creep properties. The possibility of replacing nickel-bearing austenitic 18/8 stainless steels with nickel-free austenitic stainless steels for springy type pressure cooker lids which require special springy characteristics to prevent the escape of steam at medium pressure has been successfully established and it has been found that nickel-free stainless steel can successfully replace 18/8 nickel-bearing austenitic stainless steels for such applications. Several appreciative comments on these nickel-free stainless steels were received from overseas research institutions commending the use of this group of stainless steels for specific applications. This laboratory-scale research studies have now been projected on industrial-scale trials for diverse uses in industry.

Following related themes, development of bi-metals had made excellent progress; two general purpose bi-metals have been evolved: one for applications up to 220°C. and the other for applications up to about 300°C. Nickel and cobalt-free heating elements based on iron-chromium and aluminium have been successfully developed; these alloys possess much higher electrical resistivity than the conventional heating elements, such as nichrome and kanthal, which are imported at present. Nickel-free coinage alloys have been produced on large scale for detailed evaluation of their hot working and wear properties in collaboration with the Government of India Mint.

Besides, the Laboratory has undertaken several development projects for the Ministry of Railways and National Industrial Development Corporation besides others. Aluminium-bronze alloys of suitable composition have been developed including formulation of their production and casting techniques. The alloys developed by this Laboratory are

capable of taking the required load without any change in the accepted bearing designs for meeting the Railway's requirements of aluminium-bronze castings for the 25 kV traction overhead equipment in Indian Railway's electrification project.

The Laboratory has also successfully produced aluminium powder of high purity for pyro-technique applications for the Ministry of Defence, Government of India. The development of copper and dull nickel plating salts from indigenous raw materials has also likewise evoked considerable appreciation and National Metallurgical Laboratory's dull nickel plating salts were tested by the Indian plating industry to be equally good as imported plating salts under actual service conditions, if not better.

As a part of operational research for extending services to industries, laboratory-scale experiments have been successfully conducted on the preparation of iron powder which is used for scarfing, cleaning and cutting alloy steel-castings, through injection in an oxy-acetylene torch. 'Blue Dust' which is a fine iron ore powder from Indian iron ore mines was subjected to high temperature solid state reduction. The reduced powder was hand ground and sieved and had an average of 93 per cent iron content. Industrial trials using this powder have successfully established that it was equivalent in quality to imported iron powder for the purpose.

Steady and notable progress has been continually maintained on fundamental research studies such as transformation studies on austenite including its stabilization, preferred orientation in rolled sheets, isothermal transformation studies (T.T.T. curves) of Indian steels, etc. Research contributions on stabilization of austenite under cathodic hydrogen treatment appeared in renowned overseas journals during the current year.

Different industrial organizations, Government Departments and institutions have continued to call heavily upon the National Metallurgical Laboratory for technical advice,

ad hoc investigations, survey of industrial projects and initiation for undertaking long-term research projects. An active Electronic Section, set up earlier at the National Metallurgical Laboratory, is now rendering useful service not only in the design and fabrication of ancillary electronic gadgets and instruments but also in the repair and maintenance of imported equipment. The Design Section, Mechanical and Electrical Workshops and other service units have continued to perform useful service of considerable value in the research and development projects under way at the National Metallurgical Laboratory.

During the year under review, essential equipment and specialized apparatus for the pilot plant and other research projects have continued to be designed and fabricated at the National Metallurgical Laboratory, such as the side-blown basic lined pneumatic converter, brick-making attachment for Refractories Pilot Plant, agitator for extraction metallurgy, electrolytic cells for the manganese dioxide pilot plant, special alloy iron pots for aluminizing of steel wire, high temperature electric furnaces of different types. Considerable saving of foreign exchange was thereby effected by fabrication of research and pilot plant equipment at the National Metallurgical Laboratory.

With Indian metal industries becoming progressively conscious of the utility and value of research investigations, numerous technical operational research problems were referred to this Laboratory, which were successfully handled to increase productivity consistent with quality output.

Technical liaison is also actively maintained with industry through publications by use of the *NML Technical Journal*, Proceedings of Symposia, commercial exploitation of our patents, liaison with industries and scientific institutions, collection and dissemination of statistical data, through organization of International Symposia on topical metallurgical subjects, bringing popular technical brochures, translation service and last but

not the least through field investigations by operational research and other technical staff.

It is gratifying to note that the *NML Technical Journal* has attained a set-up at par with many leading technical journals in India and abroad. Several leading Indian and overseas technical journals have been acquired for the library on free exchange basis with the *NML Technical Journal*. Equally important is the fact that the Proceedings of the Symposium on "Pilot Plants in Metallurgical Research and Development" held earlier in February 1960 was brought out in a record time of a few months which was most favourably reviewed at home and abroad. The Monograph on "Foundry Sands and Bonding Clays in India" was also brought out this year embodying the results of several years of work on the indigenous moulding materials. This publication has been exceedingly well received and appreciated by the Indian foundry industry.

Early this year, the National Metallurgical Laboratory organized a Symposium on "Light Metal Industry in India" with notable success; not only delegates participated from all parts of the world, but also valuable technical and research papers from distinguished scientists and technologists were presented.

During the year under review, Technical Notes of the following processes were prepared and sent to the National Research Development Corporation:

1. An improved method for producing nitrided manganese — Indian Patent No. 62338
2. The production of dense carbon aggregate suitable for being used as base material for carbon products in general and Soderberg paste in particular (consolidated note on two processes) — Indian Patent Nos. 62938 & 65696
3. Hot-dip aluminizing of ferrous materials (consolidated note on three processes) — Indian Patent Nos. 55289, 57938 & 65230

The National Research Development Corporation of India had made proposals

for commercial exploitation of processes in the research laboratory and institutes in the public sector. The following processes have been offered for consideration of their exploitation in the public sector.

1. Indian Patent No. 49355: An improved process for the production of electrolytic manganese metal
2. Indian Patent No. 62338: An improved method for producing nitrided manganese
3. Indian Patent No. 55289: A process for the hot-dip aluminizing of ferrous materials
4. Indian Patent No. 57938: Aluminizing of iron and steel
5. Indian Patent No. 65230: Improvements in or relating to hot-dip aluminizing steel
6. Indian Patent No. 57884: Improvements in or relating to magnesium-silicate refractories and use of the same
7. Indian Patent No. 61981: A process for making completely stabilized dolomite refractories

In respect of non-patented processes, concurrent efforts are being made for their industrial scale implementation on the basis of 'Technical Sponsorship' schemes on payment of sponsorship charges through the Industrial Liaison Officer of the Council of Scientific & Industrial Research.

The following patents have been accepted and sealed during the year under review:

Patents Accepted

1. Indian Patent No. 65230: Improvements in or relating to hot-dip aluminizing steel — accepted on 4-3-1960
2. Indian Patent No. 65610: Improvements in or relating to the production of chemically bonded metal-clad or unclad basic refractories — accepted on 4-3-1960
3. Indian Patent No. 65556: A process for electrolytic production of iron-chromium alloys from chromite ore — accepted on 6-1-1960
4. Indian Patent No. 65696: Production of carbon pastes for use in the continuous Soderberg electrodes — accepted on 6-4-1960

5. Indian Patent No. 68171: Compositions and method of making welding flux — accepted on 8-2-1961
6. Indian Patent No. 68174: Refractory compositions containing non-refractory chrome ore and refractory products made therefrom — accepted on 26-12-1960
7. Indian Patent No. 68401: A pneumatic or such other forge hammer adapted for the manufacture of bricks or blocks out of ceramic mixes — accepted on 15-3-1961

Patents Sealed

1. Indian Patent No. 62352: Refractory compositions comprising graphite and aluminosilicate materials and glazes to render such compositions resistant to oxidation — sealed on 24-2-1960
2. Indian Patent No. 63904: A process for recovering zirconium dioxide from zircon — sealed on 6-4-1960
3. Indian Patent No. 65230: Improvements in or relating to hot-dip aluminizing of steel — sealed on 12-9-1960
4. Indian Patent No. 65231: An improved method for the production of chromium-manganese alloys by aluminosilicate reaction — sealed on 17-2-1960
5. Indian Patent No. 65556: A process for electrolytic production of iron-chromium alloys from chromite ore — sealed on 20-7-1960
6. Indian Patent No. 65696: Production of carbon pastes for use in the continuous Soderberg electrodes — sealed on 2-11-1960
7. U.K. Patent No. 833308: New stainless steels and methods of preparing them — sealed on 10-8-1960
8. French Patent No. 1208393: New stainless steels and methods of preparing them — sealed on 9-9-1959

The following processes have been released to the industry on lump-sum premium and royalty basis:

1. Non-patented process: Liquid Gold
2. Indian Patent No. 58869: Refractory compositions comprising graphite and silicon carbide

3. Indian Patent No. 62352: Refractory compositions comprising graphite and alumino-silicate materials and glazes to render such compositions resistant to oxidation
4. Indian Patent No. 49355: An improved process for the production of electrolytic manganese metal
5. Indian Patent No. 47982: An improved process for electrolytic production of high purity manganese dioxide

Another field of increasing activity is Operational Research undertaken at times on complex problems of metal industries, conducted with a view to improve efficiency of metallurgical operations, study of economics involved to raise productivity consistent with quality. Preparation of detailed Project Reports for different metallurgical industries both in the public and private sectors is a new theme that has been initiated during

the period under review at the National Metallurgical Laboratory. This Laboratory has now almost completed its Second Five Year Plan period and the progress it has made in different fields has been exceedingly well appreciated in Indian industry, both in the public and private sectors, at whose total disposal the National Metallurgical Laboratory is today actively functioning.

Collaborative research has been equally well maintained with other scientific organizations in the country including the Metal Research Committee of the Council of Scientific & Industrial Research, Indian Standards Institution, Ministry of Defence, Ministry of Railways, Ministry of Steel, Mines & Fuel, Government of India, Hindustan Steel Limited, Indian Bureau of Mines, etc.

A brief résumé of the highlights of important problems under progress is given in the following pages.

RESEARCH PROJECTS

1.0 Nickel-free Austenitic Chromium-Nitrogen-Manganese-Copper Stainless Steel

Following the basic themes of development of substitute alloys based on indigenous alloying elements to the exclusion of those whose resources are deficient or totally non-existent in India, such as nickel, cobalt, molybdenum, tungsten, tin, etc., research and development work conducted at the National Metallurgical Laboratory have resulted in the formulation of processes for the production of nickel-free austenitic stainless steel alloys from entirely indigenous raw materials.

Comprehensive investigations were carried out for the determination of physical, mechanical and creep properties of the new steel developed. Concurrently, comprehensive work was taken up on the industrial scale production of these stainless steels including assessment of cost of production, yield value and range of industrial products obtainable under Indian market conditions and requirements. Tests were also conducted to determine the low temperature impact property, high temperature short time tensile strength,

Physical Properties — Physical properties of nickel-free chromium-manganese-nitrogen stainless steel were determined on samples from heats having nominal composition of chromium 21 per cent, manganese 14 per cent and nitrogen 0.75 per cent. Samples on which tests were performed were either in the form of forged bar or hot rolled sheets which were solution-treated by water quenching from 1050°C.

TEST	RESULTS
Young's Modulus determined by resonance vibrating method	28.15×10^6 lb./sq. in.
Resistivity	74.69 micro ohms/cm.
Saturation induction	Less than 2500 gauss for magnetizing field of 2340 oersteds
Average permeability	1.05
Coefficient of thermal expansion	$30-100^\circ\text{C. } 15.71 \times 10^{-6}/^\circ\text{C.}$ $30-200^\circ\text{C. } 16.47 \times 10^{-6}/^\circ\text{C.}$ $30-400^\circ\text{C. } 17.57 \times 10^{-6}/^\circ\text{C.}$ $30-500^\circ\text{C. } 18.08 \times 10^{-6}/^\circ\text{C.}$ $30-1000^\circ\text{C. } 23.71 \times 10^{-6}/^\circ\text{C.}$ $100-500^\circ\text{C. } 18.50 \times 10^{-6}/^\circ\text{C.}$ $500-1000^\circ\text{C. } 29.00 \times 10^{-6}/^\circ\text{C.}$

Mechanical Properties — Mechanical properties of nickel-free chromium-manganese-nitrogen-stainless steels were determined on samples of three nominal compositions. Summary of test results is given below:

NOMINAL COMPOSITION Cr: Mn: N	HARDNESS V.P.H. No.	PROOF STRESS (0.20%) OFFSET T.S.I.	MAX. STRESS T.S.I.	ELONGATION %
B ₂ 21: 14: 0.68	270	—	58.65	45.3 (1" GL)
B ₁ 21: 18: 0.75	252	32.4	61.79	45.3 (1" GL)
B ₅ 18: 17: 0.69	239	—	58.9	46.8 (2" GL)
			57.4	54.7 (1" GL)
			56.9	51.1 (1" GL)

and ageing characteristics of the steel and the results of the investigation are given below.

Low Temperature Impact Tests — Low temperature impact test values were determined using V-notch charpy specimens made

from a forged and solution treated bar. Summary of test results is given below:

NOMINAL COMPOSITION Cr: Mn: N	TEMPERA- TURE °C.	ENERGY OF FRACTURE ft./lb.
21: 14: 0.64	25	204
	0	183
	-20	170
	-40	152
	-50	104
	-57	76
	-60	72
	-70	58, 60

Test results are represented graphically in Fig. 1 indicating a transition temperature range of the order of -47°C .

Determination of High Temperature Creep Tests — Creep test was conducted using a Denison Model T.47 creep-testing machine at 650°C . at a stress of 3 tons per sq. in. The specimen which had a test length of 1 in. and dia. of 0.1785 in., was made from a solution-treated bar having a nominal composition of chromium 21 per cent, manga-

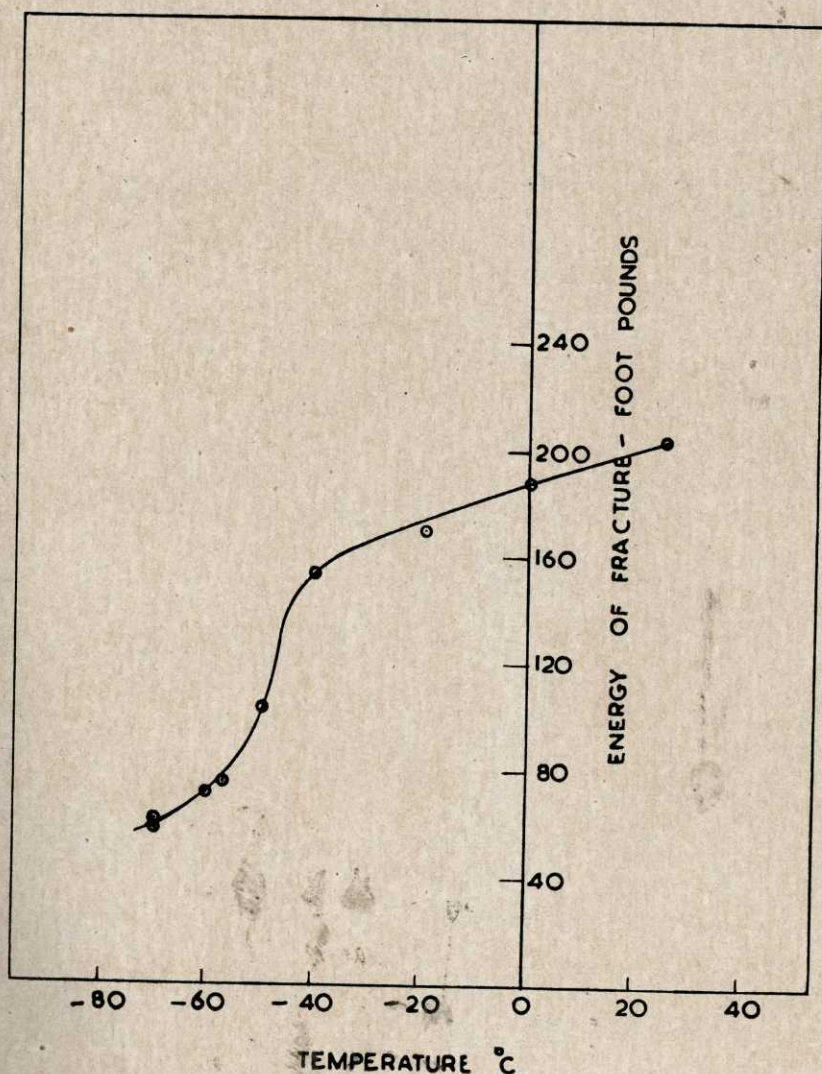


FIG. 1 — RESULTS OF LOW TEMPERATURE IMPACT TEST USING V-NOTCH CHARPY SPECIMENS MADE FROM NICKEL-FREE STAINLESS STEEL

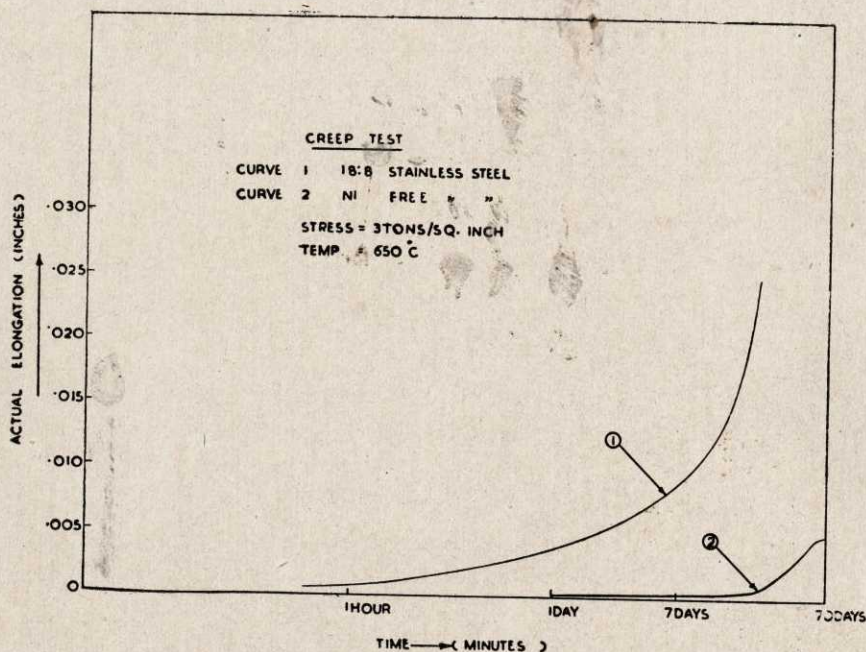


FIG. 2 — RESULTS OF HIGH-TEMPERATURE CREEP TEST

nese 14 per cent, and nitrogen 0.72 per cent. The results are plotted in Fig. 2, which also contains a creep curve for 18:8 Cr:Ni stainless steel plotted for the purpose of comparison.

High Temperature Strength — Results of high temperature tensile tests carried out on specimens prior quenched in water from 1100° and 1200°C. are shown below:

Work on 17 & 12 per cent Chromium Stainless Steels — Work was further extended to the study of the controlled transformation of stainless steels containing 17 per cent chromium; sixty-six melts were made for such detailed study. Manganese was varied in these heats from 2 to 12 per cent, nitrogen from 0.2 to 0.5 per cent and nickel from 0.5 to 4 per cent. Hardness and metallographic

NOMINAL COMPOSITION Cr: Mn: N	QUENCHING TEMPERATURE °C.	TEST TEMPERATURE °C.	TENSILE STRENGTH T.S.I.	ELONGATION %	REDUCTION OF AREA %
21: 14: 0.65	1100	600	31.30	26	30
		700	25.48	24	37
		800	17.83	31	45
21: 18: 0.70 Cu-1.19%	1200	600	31.53	26	35
		700	24.94	12	19
		800	18.03	11	17

Pressing and Deep Drawing of Vessels — Industrial trials were made for the manufacture of household utensils by deep drawing and pressing methods. Pressure cooker lids have been successfully pressed from the sheets.

study in various conditions of heat treatment including sub-zero temperature treatments were conducted on sheets of 0.05 in. thickness.

Mechanical properties and effect of cold work on the structure and properties of these

steels were also studied. Corrosion resistance tests were also carried out in boiling 65 per cent nitric acid.

Ten melts of stainless steel were made with 12 per cent chromium and varying amounts of manganese and nitrogen with the object of determining the suitability of these steels for applications where severe chemical corrosion attack is not encountered.

Several heats of age hardening stainless steels containing 17 per cent chromium, 1 per cent vanadium, 0.12-0.20 per cent nitrogen and 6-14 per cent manganese were made with carbon in the range of 0.2-0.4 per cent. Ageing characteristics of these steels are being determined.

1.1 Ageing Characteristics at Elevated Temperature of Ni-free Cr-Mn-N Austenitic Stainless Steels

With the object of determining the effect of elevated temperature ageing on tensile strength and percentage elongation of the standard composition of nickel-free Cr-Mn-N stainless steel, specimens were given ageing treatments at temperatures between 300° and 900°C. at intervals of 100°C. for extended predetermined periods. Corrosion tests in the media of boiling nitric acid, citric acid, lime juice, vinegar, and salt-spray, metallographic examination and hardness determination were carried out with the specimens

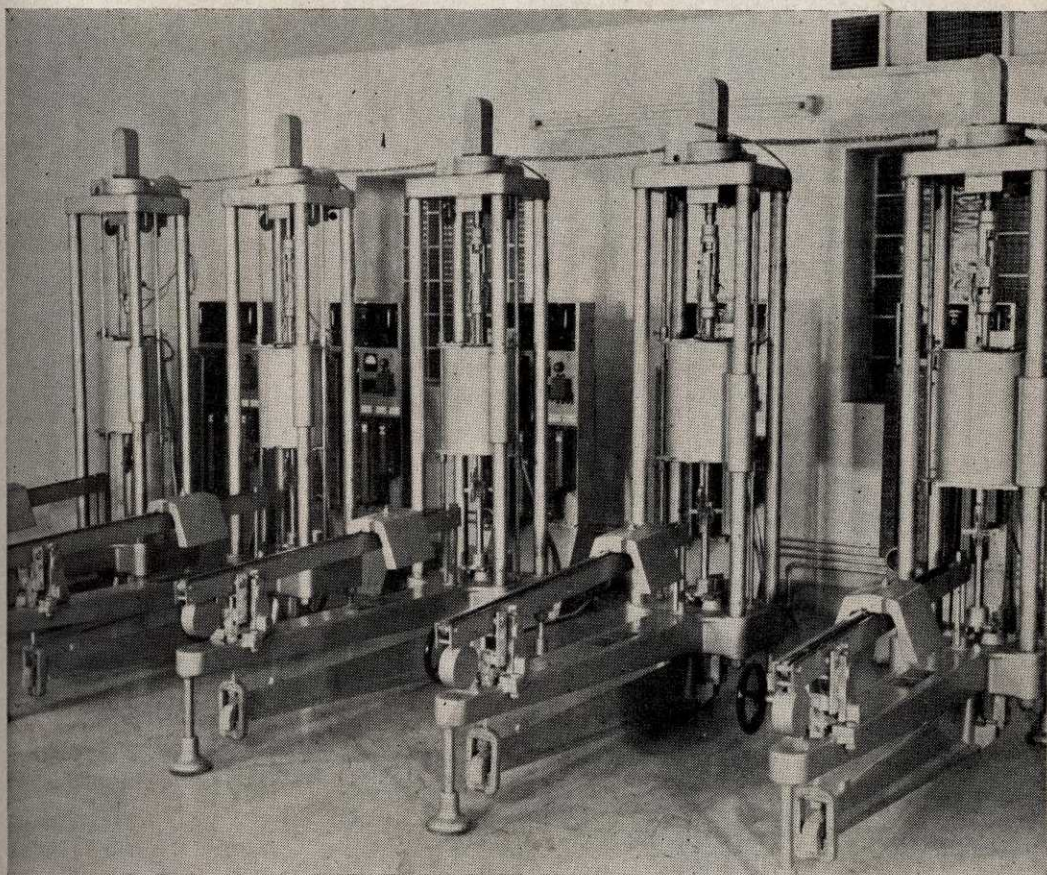


FIG. 3 — A VIEW OF THE BATTERY OF HIGH TEMPERATURE CREEP-TESTING MACHINES INSTALLED AT THE NATIONAL METALLURGICAL LABORATORY



FIG. 4 — A VIEW OF THE PHILLIPS X-RAY DIFFRACTION UNIT, DIFFRACTOMETER WITH GEIGER-MÜLLER COUNTER AND RECORDER. X-RAY SPECTROGRAPH ATTACHMENT SHOWN AT THE CENTRE TABLE

after such ageing cycles. The following ageing treatments as given below have so far been completed:

SPECIMEN NO.	AGEING TEMP. °C.	AGEING PERIOD hr.
1. B ₂ 49	300	1000
2. B ₂ 49	400	1000
3. B ₂ 49	500	1000
4. B ₂ 49	600	1000
5. B ₄	600	1000
6. B ₆	600	1000
7. B ₂ , B ₄ , B ₆	700	1000
8. B ₂ , B ₄	800	1000
9. B ₂ , B ₄ , B ₆	900	1000

Work on the identification of the phases formed in the Ni-free stainless steels during ageing at high temperatures was continued, based on the following criteria:

Studies of precipitated phases in the matrix after ageing at high temperatures

for different periods, by their electrolytic extraction and subsequent X-ray diffraction studies of the extracted residues.

Studies of the matrix (a single phase structure after solution treatment) after different periods of ageing by X-ray diffraction technique.

1.2 Studies of Precipitated Phases in the Steel Matrix after Ageing at High Temperatures

Ni-free Cr-Mn-N stainless steels designated as B₂, B₃, B₄, B₅, B₆ were selected for studies.

Heat-treatment and Metallography — The specimens taken from rolled sheets were first solution treated at 1050°C., then aged at temperatures between 300° and 700°C. for

periods up to 1000 hours; aged specimens were then metallographically examined. Ageing promoted precipitation reactions predominantly at the grain boundaries. The precipitates were in the form of lamellar nodules which formed invariably at the grain boundaries to begin with. Higher the temperature, more accelerated was the rate of precipitation. The matrix after ageing showed, in general, wholly austenitic structure. All the specimens showed increase in hardness after ageing at different temperatures with time. However, the increase in hardness value was not more than 100 V.P.H. even after 1000 hours of ageing at 600°C. and 700°C. range.

Separation of the Precipitated Phases and Their X-ray Diffraction Studies — Extraction of precipitated phases from aged specimens was done by electrolysis and chemical methods. Electrolytic separation was done using a platinum cathode and with specimen under study as the anode with 2 per cent aqueous solution of HCl as the electrolyte in most cases. However, citric acid and ammonium chloride solution were also used as electrolytes. Ultrasonic radiations were applied to the specimens for accelerating the process of electrolytic extraction of the precipitates.

Chemical separation of the precipitated phases (mostly nitrides) was undertaken by the conventional Beeghly's method. Fine cuttings of the aged specimens were digested with bromine and methyl acetate.

The precipitated residues were collected by centrifuging and were washed, dried and subjected to X-ray diffraction studies employing a 9 cm. powder camera with cobalt or chromium radiation using a suitable filter. X-ray diffraction analysis of the extracted residues showed Cr_2N type of structure. Residues from steel No. B₂ showed Cr_2N for all temperatures of ageing. The intensity and sharpness of the lines in the powder photograph, however, varied with period of ageing. The X-ray study confirmed the metallographic findings, relevant to the

increase in the amount and coarseness of the precipitated phase (i.e. Cr_2N) with increasing ageing period. The changes in the ratio of Cr:Mn:Fe concentration in this lattice structure of Cr_2N compound with period of ageing were optically ascertained from the changes in intensities. Further work on these phase studies in other nickel-free stainless steels aged for different periods is in active progress.

1.3 Studies on the Changes in the Characteristics of the Matrix after Ageing and Cold Working

Structural studies of the matrices by X-ray diffraction of solution treated specimen after ageing and cold working were carried out. Likewise, X-ray and metallographic studies of specimens from steel No. B₂ to B₆, which had been cold rolled and thus reduced by 30 per cent approximately after solution treatment, showed fully austenitic structures even after heavy cold reduction. For the purpose of X-ray diffraction analyses of the specimen as a whole (which were taken in the

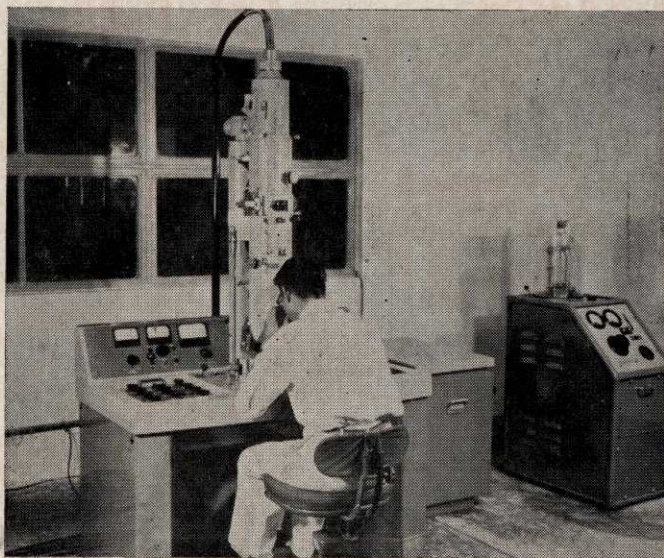


FIG. 5 — A VIEW OF THE METRO-VICK ELECTRON MICROSCOPE INSTALLED AT THE NATIONAL METALLURGICAL LABORATORY

form of a flat piece) a special device was made for scanning a large area of the specimen by chromium radiation, by rotating the specimen eccentrically to the incident beam whilst the incident beam was kept at 60° angle with respect to the specimen. The results of the X-ray diffraction and metallographic studies are shown in the Tables 1, 2 and 3.

TABLE 1—X-RAY DIFFRACTION PHOTOGRAPH DATA OF THE EXTRACTED RESIDUES FROM SPECIMEN AGED AT 600°C. FOR 1000 HR.

NO. AND NATURE OF LINES	'd'-IN A°	D-VALUES OF Cr ₂ N (A.S.T.M.)-A	
		I/I°	
1. Faint	2.397	2.38	(25)
2. Strong	2.227	2.22	(25)
3. Strong	2.113	2.10	(100)
4. Medium	1.636	1.63	(25)
5. Faint	1.587	1.38	(25)
6. Strong	1.265	1.27	(20)
7. Faint	1.178	—	—
8. Faint	1.161	—	—
9. Faint	1.117	—	—
10. Medium	0.935	—	—

TABLE 2—X-RAY DIFFRACTION PHOTOGRAPH DATA OF THE POWDERED SPECIMEN OF THE SAMPLE LEFT AFTER SEPARATION OF Cr₂N

NO. AND NATURE OF THE LINES	'd' VALUES IN A°	D-VALUES IN A°	
		α-iron (b.c.c.)	γ-iron
1. Strong	2.073	—	2.08 (100)
2. V. strong	2.034	2.0268 (100)	—
3. Medium	1.803	—	1.80 (80)
4. Med. strong	1.434	1.4332 (19)	—
5. Faint	1.275	—	1.270 (50)
6. V. strong	1.172	1.702 (30)	—
7. V. strong	1.088	—	1.083 (80)
8. strong	1.015	1.0134 (9)	—

Further work is under way on the effects of ageing on these steels after heavy cold reduction with and without prolonged ageing at high temperatures. Effects of sub-zero

treatments is also being studied after prior cold working and heat treatment cycles.

2.0 Development of Indigenous Alloy Steels

The objectives of this project are to rationalize the total number of steels to minimize the use of non-indigenous alloying elements, such as nickel and molybdenum which are not available in the country and to develop equivalent alloy steels based, as far as possible, on indigenous alloying elements in judicious combinations. Related objectives of the project are to determine the T.T.T. and C.T. curves and undertake their hardenability studies, *vis-à-vis*, ruling sections in relation to optimum heat-treatment cycles.

With these ends in view, standards in use in various countries including draft specifications prepared by the Indian Standards Institution were studied and a long-range programme of research and development work was chalked out; a broad outline thereof is given below:

- (i) *1½ per cent Manganese Steels* — These steels, though very useful, have the disadvantage that these are somewhat prone to be high in non-metallic inclusions. It is, therefore, proposed to study their melting conditions that would give a minimum of inclusions.
- (ii) *Manganese-molybdenum Steels* — It is proposed to study the following two alloy steels: C, 0.3-0.4; Si, 0.10-0.35; Mn, 1.30-1.80; Mo, 0.2-0.3; C, 0.3-0.4; Si, 0.10-0.35; Mn, 1.30-1.80 and Mo, 0.3-0.4 per cent. These steels would be identical to those proposed by I.S.I. except that these have a lower molybdenum content. If this molybdenum content proves adequate after due investigations, the existing draft specification may have to be suitably revised.
- (iii) *Molybdenum Steels* — Three alloy steel compositions have been formu-

TABLE 3 — X-RAY DIFFRACTION AND METALLOGRAPHIC DATA OF THE MATRIX AFTER COLD WORKING AND HEAT-TREATMENT

TYPE OF STEEL EXAMINED	TREATMENT	PHASES AS IDENTIFIED BY		HARDNESS (V.P.H.) AT 30 kg.
		X-ray diffraction analysis	Microscope	
B ₂	Solution treated (1050°C.-W/Q)	F.C.C. gamma phase only	Austenite and some other phase scattered on the matrix in the form of dots	260
B ₂	Solution treated and 30% cold reduced	F.C.C. gamma phase only	Austenite only broken grains visible	456
B ₂	Solution treated and aged at 500°C. for 1000 hr.	F.C.C. gamma and Cr ₂ N	Austenite and a precipitated phase starting from the grain boundaries	283
B ₃	Solution treated (1050°C.-W/Q)	F.C.C. gamma phase only	Austenite only	295
B ₃	Solution treated and 30% cold reduced	F.C.C. gamma phase only	Austenite, broken grain boundaries	495
B ₄	Solution treated (1050°C.-W/Q)	F.C.C. gamma phase only	Austenite only	302
B ₄	Solution treated and 30% cold reduced	F.C.C. gamma phase only	Austenite with broken grain boundaries	445
B ₅	Solution treated (1050°C.-W/Q)	F.C.C. gamma phase only	Austenite only	268
B ₅	Solution treated and 30% cold reduced	F.C.C. gamma phase only	Austenite only with broken grain boundaries	373
B ₆	Solution treated (1050°C.-W/Q)	F.C.C. gamma phase only	Austenite only	255
B ₆	Solution treated and 30% cold reduced	F.C.C. gamma phase only	Austenite with broken boundaries	429
18/8 stainless steel	Solution treated (1050°C.-W/Q)	F.C.C. gamma phase only	Austenite only	175
18/8 stainless steel	Solution treated 20% cold reduced	F.C.C. gamma and B.C.C. Alpha	Austenite and martensite	303

lated by I.S.I. in this group. It is proposed to limit the molybdenum content to 0.3-0.5 per cent and also to determine whether this molybdenum content is adequate for the purpose for which these steels are recommended.

(iv) *Chromium Steels* — The following seven steels have been selected for study:

C%	Si%	Mn%	Cr%
0.12-0.18	0.10-0.35	0.4-0.6	0.5-0.8
0.14-0.19	do	1.0-1.4	0.8-1.1
0.17-0.22	do	1.0-1.4	1.0-1.3
0.50-0.60	do	0.6-0.9	0.6-0.9
0.35-0.45	do	0.6-0.9	0.9-1.2
0.45-0.55	do	0.6-0.9	0.9-1.2
0.90-1.20	do	0.3-0.7	1.2-1.6

(v) *Chromium-vanadium Steels* — A study of the three compositions given below is proposed to be carried out:

(a) C, 0.45-0.55; Si, 0.10-0.35; Mn, 0.5-0.80; Cr, 0.90-1.20 and V, 0.1-0.15 per cent;

(b) C, 0.25-0.30; Si, 0.10-0.35; Mn, 1.00-1.5; Cr, 0.6-0.9 and V, 0.1-0.15 per cent;

(c) C, 0.35-0.45; Si, 0.10-0.35; Mn, 0.5-1.0; Cr, 1.5-1.8 and V, 0.1-0.15 per cent.

(vi) *Chromium-molybdenum Steels* — The following ten steels have been selected under this group:

C%	Si%	Mn%	Ni%	Cr%	Mo%
0.26 max.	0.10-0.35	0.4-0.7	—	0.90-1.20	0.20-0.30
0.35-0.45	do	do	—	do	do
0.12 max.	0.10-0.60	do	0.30 max.	do	0.40-0.60
0.10-0.20	0.10-0.35	do	do	do	do
0.35-0.45	do	do	do	do	do
0.15 max.	0.5 max.	do	do	2.0-2.5	0.8-1.0
0.10-0.20	0.10-0.35	do	do	2.90-3.40	0.40-0.60
0.20-0.30	do	do	do	do	do
0.15 max.	0.5 max.	do	do	4.0-6.0	do
0.15-0.25	do	do	do	do	do

(vii) *Nickel-chromium Steels*—The following six compositions under this group will be investigated:

- C, 0.12-0.20; Si, 0.10-0.35; Mn, 0.6-1.0; Ni, 0.7-1.0 and Cr, 0.7-1.0 per cent;
- C, 0.12-0.18; Si, 0.10-0.35; Mn, 0.4-0.8; Ni, 1.0-1.5 and Cr, 0.7-1.0 per cent;
- C, 0.3-0.4; Si, 0.10-0.35; Mn, 0.4-0.8; Ni, 1.0-1.5 and Cr, 0.7-1.0 per cent;
- C, 0.12-0.18; Si, 0.10-0.35; Mn, 0.4-0.8; Ni, 2.5-3.0 and Cr, 0.7-1.0 per cent;
- C, 0.3-0.4; Si, 0.10-0.35; Mn, 0.4-0.8; Ni, 2.5-3.0 and Cr, 0.7-1.0 per cent;
- C, 0.10-0.15; Si, 0.10-0.35; Mn, 0.4-0.8; and Ni, 3.0-3.5 per cent.

(viii) *Nickel-Chromium-Molybdenum Steels*—Study of the following five steels would be taken up under this group:

- C, 0.12-0.17; Si, 0.10-0.35; Mn, 0.6-1.0; Cr, 1.0-1.5; Ni, 1.0-1.5 and Mo, 0.10-0.20 per cent;
- C, 0.35-0.45; Si, 0.10-0.35; Mn, 0.4-0.7; Cr, 1.0-1.5; Ni, 1.25-1.75 and Mo, 0.15-0.13 per cent;
- C, 0.27-0.35; Si, 0.10-0.35; Mn, 0.4-0.7; Cr, 0.5-0.8; Ni, 2.25-2.75 and Mo, 0.4-0.6 per cent;
- C, 0.36-0.44; Si, 0.10-0.35; Mn, 0.4-0.7; Cr, 0.5-0.8; Ni, 2.25-2.75 and Mo, 0.4-0.6 per cent;

(e) C, 0.12-0.20; Si, 0.10-0.35; Mn, 0.4-0.7; Cr, 1.4-1.7; Ni, 1.8-2.20 and Mo, 0.15-0.25 per cent.

(ix) Experimental work is underway on the following alloy steels including the study of effects of boron and titanium micro additions:

(a) Si-Mn Steel: C, 0.32-0.40; Si, 1.10-1.40 and Mn, 1.10-1.40 per cent;

This steel may be suitable for heavy forging.

(b) Si-Cr-V Steels C, 0.48-0.58; Mn, 0.60-0.90; Si, 1.20-1.50; Cr, 0.70-1.00 and V, 0.10-0.20 per cent;

This steel may be used for valve springs, etc.

(c) Mn-V Steel: C, 0.38-0.45; Si, 0.15-0.35; Mn, 1.6-1.9 and V, 0.10-0.15 per cent;

(d) Silicon Steel:

(i) C, 0.40-0.5; Mn, 0.4-0.8; and Si, 1.60-2.00 per cent;

(ii) C, 0.5-0.6; Mn, 0.4-0.8; and Si, 1.60-2.00 per cent;

(e) Si-Cr-Mo Steel: C, 0.42-0.50; Mn, 0.5; Si, 1.25-1.75; Cr, 0.70-0.90 and Mn, 0.2-0.3 per cent.

2.1 Boron Steels

The objective of the investigation is to study the increase in hardenability conferred by the addition of boron to engineering steels.

Seven heats of boron-treated $1\frac{1}{2}$ per cent manganese steels were prepared with the object of making a preliminary study of the effects of boron addition on hardenability of these steels. Retention of boron in these heats was calculated from the boron actually added and comparing it with the boron content spectroscopically analysed. Hardenability characteristics of these boron-treated steels were studied by the Jominy End Quench hardenability test. It was observed that while the untreated steel showed little improvement in values of hardenability, the boron-treated steels containing 0.006-0.008 per cent boron showed a hardness value of 350-400 V.P.H. on the quenched end, which fell to about 150 V.P.H. at about 1 in. from the quenched end. Further work on these steels is actively in progress. The composition of these steels is given in the table below:

TABLE 4-COMPOSITION OF BORON STEEL

No.	C%	Mn%	Si%	B%
BS ₁	0.17	1.38	0.117	0.0010
BS ₂	0.19	1.46	0.117	0.0039
BS ₃	0.20	1.41	0.131	0.0046
BS ₄	0.14	1.46	0.135	0.0056
BS ₅	0.19	1.41	0.177	0.0057
BS ₆	0.18	1.42	0.163	0.0069
BS ₇	0.20	1.49	0.159	0.0081

2.2 Tool Steels

Work was initiated to develop tool steels based on indigenous alloying elements as

far as possible. The possibility of using nitrogen as an alloying element in low-alloy high speed steel is being investigated. The nominal composition aimed at so far was in the following range:

	%
C	0.6 0.7
W	6.0 7.0
Cr	4.0 5.0
V	1.5 2.0
N	0.25-0.3

Nitrogen was introduced in the form of nitrided ferro-chrome and in order to increase the contents of nitrogen, alloy steels containing 1 per cent aluminium were also made, but no appreciable increase in nitrogen content was noticeable therein. Additions of cobalt up to 4 per cent were also made with the same end in view. The composition of cobalt-containing steels is given in Table 5.

A few heats of 18/4/1 high speed steel were made to standardize the forging and heat-treatment cycles. The ingots were forged to a small cross-section as practicable to get the resultant bar with maximum uniformity of carbide distribution. The forging of nitrogen-bearing low-alloy high speed tool steel was carried out in a way similar to the forging of 18/4/1 high speed steel. No marked difference in the forging operation of nitrogen containing high speed steel from 18/4/1 high speed steel was noticed. Nevertheless, care was taken to maintain optimum soaking temperatures and right soaking time so that heat could soak right through the

TABLE 5 — COMPOSITION OF COBALT CONTAINING STEELS

STEEL No.	C %	W %	Cr %	V %	N %	CO %	VPH AFTER ANNEALING AT 850°C.	VPH AFTER FORGING AND ANNEALING AT 850°C.	VPH AFTER HARDENING AT 1200°C.
13	0.90	7.8	5.24	1.02	0.01	—	347	375	804
14	0.99	6.4	4.40	0.98	0.009	1.3	340	467	836
15	0.99	7.6	4.73	1.07	0.01	2.2	325	362	855
16	0.91	7.8	4.5	1.10	0.009	3.2	319	348	745
17	0.97	7.6	3.75	0.93	0.009	4.4	318	418	825

cross-section without over-heating or under-heating the ingots, so that cracking of the ingots during or after forging operation was eliminated. Investigations are under way to find out optimum conditions for obtaining maximum nitrogen contents in these steels.

2.3 Iron-Aluminium Alloys

The project on iron-aluminium alloys aims at developing heat-resistant alloys for high temperature application in the context of availability of indigenous raw materials. Work on the binary Fe-Al system was continued up to 35 per cent aluminium — the limit of solid solubility on the iron-rich end. Alloys containing more than 10 per cent aluminium were found to be mechanically unworkable. Alloys in the range of 4.5-5.5 per cent Al showed tensile strength of 42 tsi with an elongation of 4.6 per cent. With 8-10 per cent aluminium, the strength was only 30 tsi with only 2 per cent elongation (2 in. G.L., 0.1 in. sheet specimens). The low values of elongation obtained showed that the air-melted alloys lacked adequate ductility. Salt spray corrosion test undertaken, vide ASTM standard B117-54T, showed a corrosion rate of 0.09-0.12 gm./dm.²/day in the range of 4.5 per cent aluminium and 0.062-0.07 gm./dm.²/day in the range of 8-10 per cent aluminium.

Effects of Ti, Zr, V., B and Ce were comprehensively investigated as grain refining agents for iron-aluminium alloys which appeared to be inherently coarse grained. Results of 500-hour oxidation resistance test carried out in a thermal balance at 800°C. are shown in Fig. 6. A stable oxide film formed on the 12 per cent Al-alloy prevented scaling whereas the film on 5 per cent aluminium alloy permitted continuous oxidation. Results of a shorter 300-hour test at 1000°C. on the 12 per cent alloy are given in Fig. 7. A continuous steep rise in weight indicated that the 12 per cent Al-alloy behaved at 1000°C. like the 5 per cent Al-alloy at 800°C.

without any horizontal plateau in the beginning as in the latter's case.

3.0 Nodular Cast Iron

During the year under review, work on nodular cast iron was pursued with a view to study the effect of various elements on the formation and growth of spheroidal graphite in cast iron. A number of synthetic pig irons were made by melting mild steel in indirect and direct arc furnaces with additions of graphite and ferro-silicon, in order to keep Mn, S and P on the lower side. The molten base irons were treated with various inoculants given below:

Calcium — Calcium addition was made as metallic calcium, calcium hydride, calcium silicide, calcium-copper and calcium metal with CCl₄ vapours. Except in the case of certain silicide inoculations and where Ca and CCl₄ together were used as inoculants, all the calcium additions were followed by a treatment of the molten metal with ferro-silicon also.

Micro-examinations revealed that only a few nodules were formed; some fine flakes were also present. The treatment produced copious loose granular slag with considerable chilling of the molten iron. Higher melt temperatures for inoculation and higher pouring temperatures might ensure a more spherulitic graphite in the cast iron.

Zirconium — Zirconium additions were tried out as ferro-zirconium containing 60 per cent zirconium with subsequent inoculation of the treated iron by zirconium treatment. The chilling action was more pronounced than in the case of calcium additions. In view of the difficulty in getting very high molten melt temperatures necessary for successful inoculation, in some heats, ferro-zirconium was added to the molten cast iron in the indirect electric arc furnace after switching off the arc but with the rocking arrangement switched on. The metal was finally tapped into a red hot ladle and treated with ferro-silicon. Under

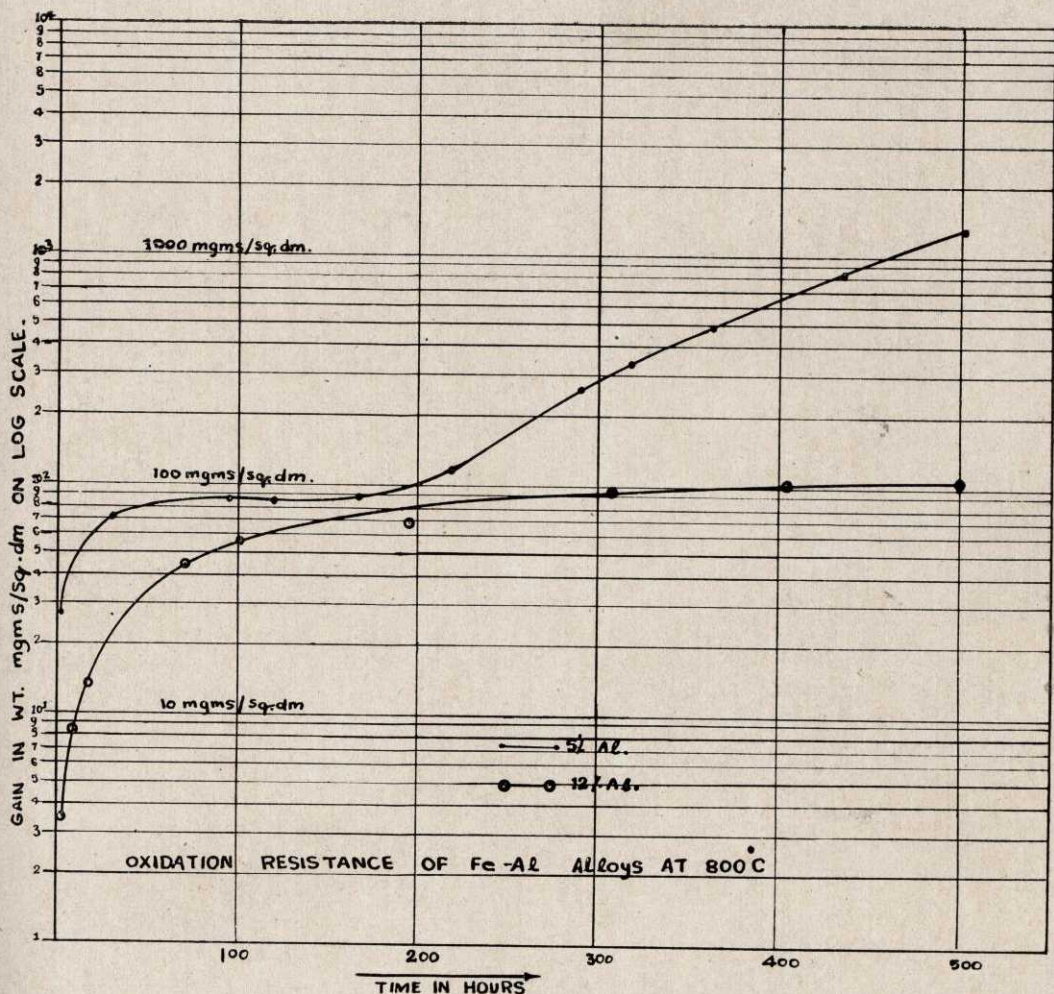


FIG. 6 — SCALING RESISTANCE OF Fe-Al ALLOYS CONTAINING 12 AND 5 PER CENT AL AT 800°C.

these circumstances, small nodules were observed in the solid iron; coarse flakes were also present. The slag volume was not so much as in the case of calcium treatment.

Double Addition of Calcium and Zirconium — Simultaneous additions of calcium and zirconium to a suitable composition of base iron were attempted in a few heats. Combinations tried were ferro-zirconium plus calcium hydride, ferro-zirconium plus Ca-metal, and ferro-zirconium plus calcium silicide. In all cases, the melt was finally deoxidized with ferro-silicon. This double treatment with calcium and zirconium was

slightly more effective than inoculation with ferro-zirconium alone. The microstructure showed quasi-flakes. There was slight increase in the hardness of the cast iron.

Boron — Boron was added as ferro-boron containing 12.39 per cent boron and the molten iron was subsequently treated with ferro-silicon. Micro-structure showed graphite as quasi-nodules and flakes.

Zinc — The addition of zinc was tried both as zinc metal and brass with subsequent inoculation with ferro-silicon. There was no improvement in the graphite shape as revealed by the micro-structure.

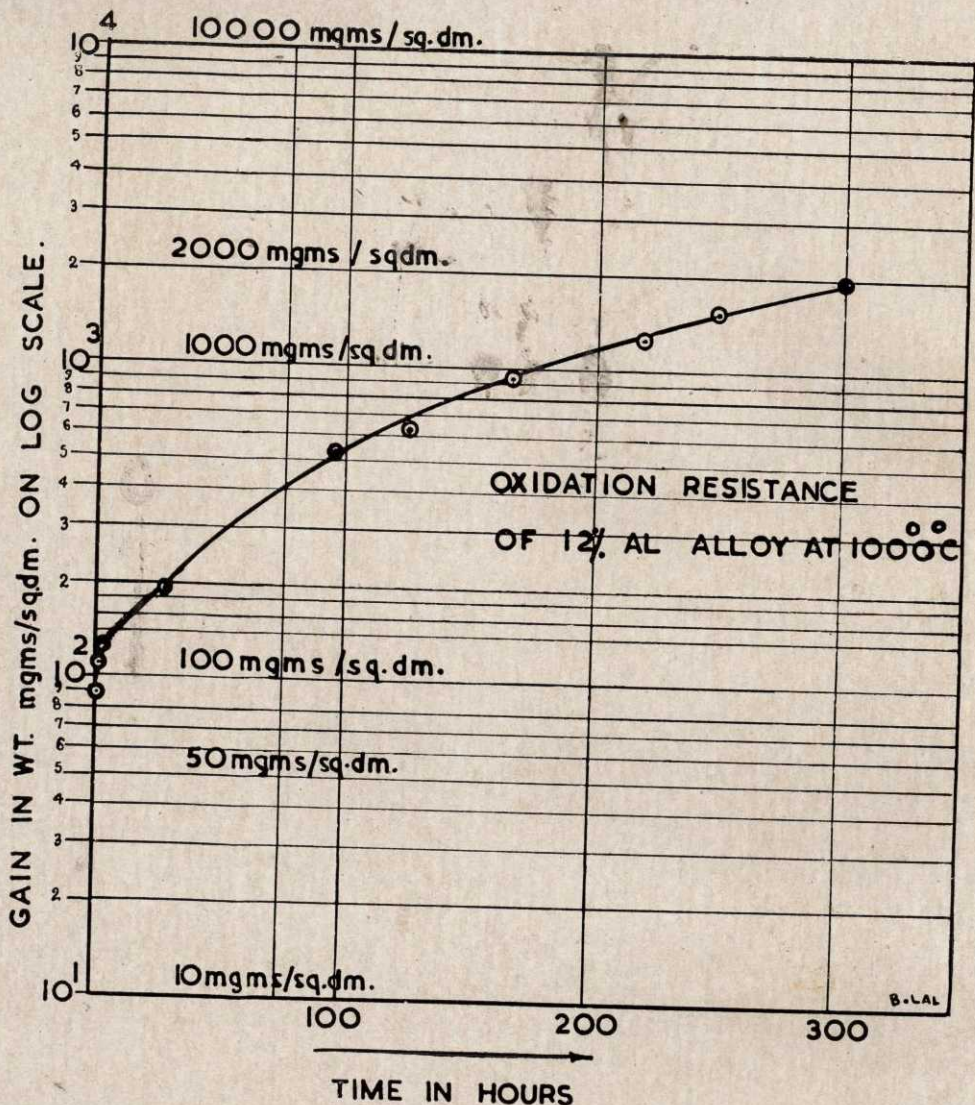


FIG. 7 — SCALING RESISTANCE OF Fe-Al ALLOY CONTAINING 12 PER CENT AL AT 1000°C.

Sodium — Sodium additions to cast iron were made as metallic sodium and as fluxes comprising of mixtures of NaF, NaCl in different proportions. All the treated irons were finally deoxidized with ferro-silicon. There was agglomeration of graphite in the cast iron resulting in graphite clusters. Graphite flakes were not present in the samples.

Magnesium — The reduction of $MgCO_3$, MgO and $MgCl_2$ with metallic calcium, calcium silicide, calcium carbide and cal-

cium-copper was attempted in a few melts to examine if the Mg produced would nodularize the graphite in the cast iron, on an economical basis. $MgCO_3$, MgO and $MgCl_2$ together with Ca, calcium silicide, calcium carbide and calcium-copper was added to the molten cast iron of a suitable composition and subsequently inoculated with ferro-silicon. In one heat where $MgCl_2$ and calcium-copper were added, the micro-structure showed good nodules.

Silicon — Inoculation with silicon in the form of silicon carbide did not show much improvement in the graphite shape. The graphite was present mostly as quasi-flakes.

The matrices in all these cases consisted of ferrite and pearlite. The effects of vibrations on grey cast iron before and after inoculation with ferro-silicon or silicon carbide were also studied with a view to ascertain if this treatment improved the shape of the graphite in the solidified cast iron. There was some improvement but the vibrations resulted in quasi-flakes and fine graphite dispersed unevenly throughout the matrix.

In the next phase of this study, attention is being diverted to high sulphurous cast irons to see if there is any effect on the shape of the primary graphite, keeping in view the high degree of nucleation obtained by the presence of increased sulphur contents of the cast irons.

4.0 Study of the Properties of Indigenous Foundry Moulding Materials

This project was taken up with a view to assess the moulding characteristics of Indian foundry sands and bonding clays and to determine their suitability for various types of castings. A large number of samples had already been studied, and during the period under review, twelve sand samples were investigated. A summary of the work done is given below:

(i) *River Sand from Southern Rly., Golden Rock, Madras* — This sand sample was found suitable for non-ferrous castings, such as aluminium, brass and other light metals. By using suitable wash of high refractoriness, this sand can be used for cast iron jobs.

(ii) *Yellow Sand from Batala Engineering Co. Ltd., Batala* — The sample was acceptable for brass and bronze castings. Poor surface finish was obtained when cast iron was poured into these sand moulds.

(iii) *Core Sand from Batala Engineering Co. Ltd.* — The sand proved suitable for making small and medium sized cores for cast iron jobs. Application of suitable core wash was recommended for getting improved surface finish.

(iv) *River Sand from Batala Engineering Co. Ltd.* — The sand was found unsuitable for cast iron job.

(v) *Green Facing Sand from Southern Rly., Golden Rock, Madras* — The application of this sand up to 2 in. thickness as facing sand was recommended for cast iron work.

(vi) *Dry Facing Sand from Southern Rly., Golden Rock, Madras* — Up to 1-2 in. thickness of this sand as facing sand was recommended for cast iron jobs.

(vii) *Backing Sand from Southern Rly., Golden Rock, Madras* — This sand after tempering to 5-6 per cent moisture content was recommended for applications in green sand and dry sand moulds respectively. This sand was also satisfactory for the two facing sands mentioned under items (v) and (vi).

(viii) *Moulding Sand from Southern Rly., Mysore* — The sand was found suitable for cast iron and non-ferrous foundry jobs. It was characterized by high strength and low permeability.

(ix) *Moulding Sand from Britannia Engineering Co. Ltd., Titaghur, West Bengal* — The sand possessed a grain fineness No. of 162 and 53 per cent of A.F.S. clay. The silica content was 60 per cent in the as-received state. The sintering range was 1110°-1150°C. This sand should be regarded as a loam rather than a moulding sand.

(x) *Silica Sand from Britannia Engineering Co. Ltd., Titaghur, West Bengal* — This sand had 1.7 per cent A.F.S. clay and grain fineness number of 47.8. It was composed mainly of quartz and feldspar. Percentage of silica present was nearly 72. Approximate sintering range was 1200°-1250°C. This sand was useful for cast iron jobs.

(xi) *Bonding Clays* — Investigation work on the bentonite sample 'Hathi-ki-Dhani' sent by Messrs Mining Engineers, Jodhpur,

was completed. 3-5 per cent of this bentonite was found suitable for binding high silica sand at the moisture range of 1.5-3.3 per cent. Cast iron castings had good surface finish when poured in moulds made with high silica sand bonded with the above bentonite, but in the case of steel, mould wash was required for obtaining good surface finish.

Bentonite sample received from Messrs Britannia Engineering Co. Ltd., Titāghur (West Bengal), was tested to specifications laid down for its use.

pH values and total-base exchange capacity determination gel, swelling index, wet and dry fineness tests and chemical analyses were conducted for the 6 samples of sodium-base-bentonites, supplied by Messrs Industrial Minerals & Chemical Co., Bombay, on behalf of the Indian Standards Institution. Petrographic and differential thermal analyses of these bentonites are now under progress.

4.1 CO₂ Process

The object of the study is to investigate the possibility of reducing the consumption of CO₂ by mixing it with other diluents for the purpose of hardening moulds and cores.

Experiments were continued on the CO₂ process of hardening moulds and cores. As a result of an extensive study carried out on the hardening characteristics of (sodium) silicate-bonded sands with/or without CO₂, with gaseous mixtures of CO₂ and air or N₂ or O₂, following conclusions were arrived at.

(i) Zircon sand with a grain fineness number of 124.5 required considerably lower percentages of sodium silicate for giving high strength values as compared to fine grained Rajmahal sand with a GFN of 92.3. Increased additions of sodium silicate to zircon sand imparted high plasticity to the sand.

(ii) It was observed that the finer the grain size of the sand, higher was the compressive strength. Further, the coarse grained sand develop high strength with prolonged

gassing time whereas the fine grained sand developed high strength when the gassing time was decreased.

(iii) From a study of gaseous mixtures on silicate bonded sand, it was concluded that any attempt to dilute the CO₂ by air, or N₂ resulted in lowering the strength values of the sand mixtures. When air or O₂ was passed alone, the sand mixture did not develop any strength. Further the comparative effect of air, O₂ or N₂ as diluents showed that CO₂ when diluted with air gave better strength as compared to the values obtained with CO₂ and N₂ or CO₂ and O₂. A progressive decline in strength was observed with decrease in the CO₂: air or CO₂: N₂ or CO₂: O₂ ratio. In these experiments, a gassing pressure of 30 mm. Hg and a gassing time of 30 seconds were found optimum to give compressive strength of the order of 100-150 lb./sq. in.

(iv) On hardening the silicate bonded sand by the application of heat alone, it was found that the binder requirement was more than when the sands were hardened by CO₂ or by a mixture of CO₂ with other gases. Optimum drying temperature and time were found to be 200°C. and 5 min., respectively. Further, a little water additions helped to improve the strength characteristics of the sands on hardening. In the case of natural moulding sands, additions of even appreciable quantities of water along with sodium silicate did not impart adequate strength to the sand mixture.

A comparative study of the strength values obtained by various methods showed that the strength values obtainable with silicate bonded and dried specimens were comparable to, and in fact higher than, the values obtainable in the case of dried oil-sand specimens. Additions of sucrose along with the sodium silicate to the sand mixture imparted still higher values of compressive strength.

(v) The established objection of using a moulding sand containing clay for the silicate bonding was sustained.

5.0 Aluminium-bronzes (Railway Electrification Project)

Super-duty aluminium-bronze castings of tensile strengths of the order of 60 kg./mm². and minimum elongation 20 per cent are required for 25 kV. traction overhead equipment in Indian Railways Electrification Project. At the instance of Research, Design and Standards Organization, Government of India, Ministry of Railways, Chittaranjan, the work was taken up at the National Metallurgical Laboratory, Jamshedpur, in order to standardize the method of melting and casting in detail so as to satisfy the specifications laid down by the French Consultants for the Railway Electrification Project (R.E.P.). This was necessary in order to enable the railway workshops to undertake the casting of such alloys.

R.E.P. specification covers alloys having aluminium 8-12 per cent, iron plus manganese 10 per cent max., balance copper, nickel addition being made only in special alloys. The investigation work was carried out on nickel-free aluminium-bronzes and comprehensive work was done to find a compositional range which would meet the specified properties. Aluminium-bronze alloys within the following range of chemical composition were found to possess the desired mechanical properties, provided proper care was taken during melting and casting.

Aluminium	9.5-10.0 per cent
Iron	1.5- 2.0 „
Manganese	0.5- 0.8 „
Copper	Balance

The procedure employed for melting and casting to get the properties within specification is summarized below:

1. The charge was melted as quickly as possible in a slightly oxidizing atmosphere.
2. Master alloys of copper-aluminium, copper-manganese and iron-aluminium to minimize oxidation and dross formation was used.
3. Stirring was minimized to avoid oxidation and entrapment of oxides.

4. Pouring temperatures were maintained between the following ranges:

For gravity die casting 1080°-1120°C.

For sand castings 1050°-1070°C.

5. In gravity die castings, the mould was preheated to about 150°-200°C. and 1 ml. of CCl₄ per 5 kg. of charge was used prior to pouring to ensure an inclusion-free casting with close tolerance and good surface finish.
6. In sand castings, 'Inverted horn-gate' system with choke was employed in order to avoid oxide inclusions.

This development work undertaken for the Indian Railways was accepted by them as valuable for applications in overhead electrical fittings in their electrification projects.

6.0 Tinless Bearing Alloys

The project referred to this Laboratory by the Indian Railways for the development of a bearing alloy free from tin to serve as a substitute for the lead-based bearing alloy containing 6 per cent tin, currently in use on the Railways, has been completed. A comprehensive report on different aspects of copper-lead and zinc-aluminium-copper bearing alloys has been made. The salient features of the various compositions developed are furnished below.

(i) *Copper-lead Alloys* — The composition selected for study was a 70:30 copper-lead alloy. Lead segregation was reduced to a minimum by the use of a water-cooled copper mould; use of small amounts of manganese or nickel also reduced lead segregation.

The structure of the alloy was found quite favourable, the soft phase of lead embedded in the copper matrix imparted antifrictional property to the alloy.

The bonding characteristics of copper-lead alloy to steel and gunmetal base were also studied, and it was found that adequate care and attention were needed to get a bonded layer free of blow holes.

(ii) *Zinc-aluminium-copper Alloy* — The alloy studied had a composition of 34 per cent Al, 6 per cent Cu balance zinc. Considerable skill was required in making the alloy in view of the high solubility of hydrogen in aluminium.

The alloy exhibited good casting properties and could be sand cast to give a smooth surface.

The cast structure of the alloy needed modification by annealing at 300°-400°C. whereby a hard phase of CuAl_2 embedded in a soft matrix of zinc and aluminium solid solution could be obtained.

The alloy had a strength of 17-18 tons/sq. in. with poor ductility. The wear tests were performed on the alloy under a load of 382 lb./sq. in. of the projected area at a speed of 200 r.p.m. in lubricated condition. Its wear-resistance and antifrictional properties were found to be superior to other common bronzes, e.g. gunmetal, leaded bronze and phosphor bronze (Table 6).

The thermal conductivity of the alloy was found to be superior to gunmetal and steel. Compression tests were also undertaken to study the compressibility of the alloy up to a load of 3500 lb./sq. in.

Comprehensive experiments were carried out to study the bonding characteristics of the alloy to gunmetal and steel. Different conditioning treatments and annealing of

the bonded specimens were tried, but they all resulted in the formation of a brittle intermediate layer.

As zinc-aluminium-copper alloys possess good wear-resistance and antifriction property and thermal conductivity, they may be suitable for bearing purposes in the as-cast condition. These alloys have, however, the disadvantage that they are difficult to be bonded to the gunmetal or steel backing.

A bearing of Zn-Al-Cu alloy was made and put up for service trials on a half-ton Side-blown converter being fabricated at the National Metallurgical Laboratory.

7.0 Aluminium-silicon Alloys

In view of the remarkable range of properties associated with the aluminium alloys containing silicon and their growing demand in various engineering fields, work was undertaken to develop new methods of effecting modifications in these alloys.

The influence of phosphorus in controlling the coarse and heterogeneous solidification of primary silicon particles in hyper-eutectic aluminium-silicon alloys has long been established, but its effects in refining the eutectic cell size have been rather poor, especially when the alloys are sand-cast. No single modifying agent has been so far suggested to be effective on both the primary silicon particles and on the eutectic. So effort was made to develop a new modifying agent which would refine both the eutectic and primary silicon. Additions of titanium, boron, zirconium and vanadium were tried separately and in conjunction with phosphorus. Carbon tetrachloride vapours were introduced into the melt containing these elements, using nitrogen as a carrier gas. The structure and mechanical properties of these alloys after this treatment were studied. The effect of chlorine gas on these alloys was also studied. During the course of research a new modifying agent was discovered which could refine both primary silicon particles as well as the eutectic.

TABLE 6 — COMPARISON OF WEAR AND ANTIFRICTIONAL PROPERTIES

BEARING ALLOY	WEIGHT LOSS IN 40 hr. gm.	WEIGHT LOSS IN 80 hr. gm.	INTE- GRATED FRIC- TIONAL WORK DONE IN kg. (80 hr.)
Zn-Al-Cu Alloy (TB. 4)	0.0018	0.0030	81050
Zn-Al-Cu (TB. 5)	0.0015	0.0025	95890
Silicon-bronze	0.0015	0.0018	99750
Gunmetal	0.0032	0.0142	110850
Leaded-bronze	0.0018	0.0038	106200
Phosphor-bronze	0.0074	0.0252	132300

It was also observed that phosphorus, if used in conjunction with the new modifying agent, enhanced the effect of the latter in improving the micro-structure, tensile strength, machinability and fluidity of these alloys.

The single spiral fluidity casting of a 23 per cent Si-aluminium alloy modified with phosphorus and that of the same alloy when treated with the modifying agent developed in this Laboratory are shown in the figs. 8 and 9.

Steps are being taken to patent the process of modification.

The effects of this nucleant on certain commercial aluminium alloys, such as 5 and 7 per cent Mg-Al alloys, 4 per cent Cu-Al alloys and on their mechanical properties are under study. The fluidity was remarkably improved after the addition of the new nucleant.

8.0 Powder Metallurgy

The work was taken-up with a view to (i) produce copper powder continuously by electrodeposition; (ii) make metal powders

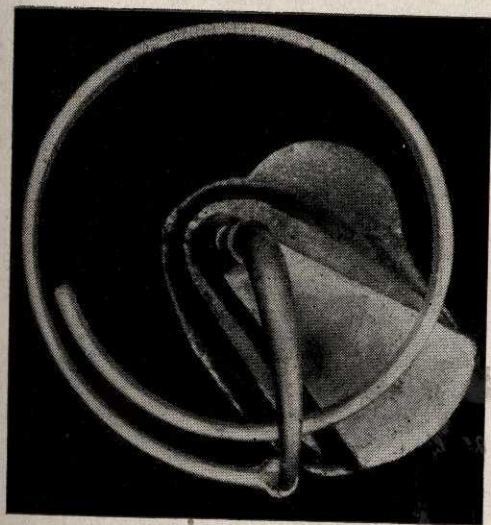


FIG. 8 — ALUMINIUM-SILICON ALLOY FLUIDITY SPIRAL WITHOUT THE USE OF MODIFYING AGENT

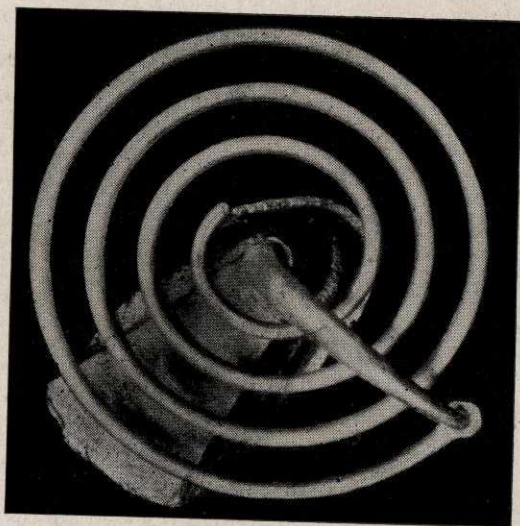


FIG. 9 — ALUMINIUM-SILICON ALLOY FLUIDITY SPIRAL WITH THE ADDITION OF MODIFYING AGENT DEVELOPED AT THE NATIONAL METALLURGICAL LABORATORY

of aluminium and tin by atomizing; and (iii) make porous bronze bearings.

Production of copper powder by continuous electrodeposition could be achieved by simple means of control. Copper powder is made by electrolysing acidified aqueous solution of CuSO_4 . In this instance, the problem consisted of favourably modifying the deposition characteristics of the electrolyte and of securing the removal of the powder deposits from the cathodes. Thus, the powder deposited on the cathodes had to be removed at the appropriate time; otherwise the process was liable to break down because of the deposit becoming coarse and adherent. As a solution to this problem, mechanical contrivances are reported to be in use for removal of powder deposit from the cathodes. Such a procedure, however, involves the use of complicated equipment as the number of cathodes used in cells for the copper powder production are many. Moreover, the timely synchronizing of the formation of the required grade of powder and its removal by mechanical means is difficult in practice to achieve; these factors render the mechanical means

of controlling a difficult and undependable process for making electro-deposited copper powder. The method that has been developed at the National Metallurgical Laboratory consists of treating the acidified CuSO_4 electrolyte with an organic chemical substance, which is found to modify the deposition characteristics of the electrolyte so as to prevent any possibility of conversion of the useful grades of powder into unwanted coarsening types. The chemical reagent used also permits wide variations in operating conditions thereby causing easy removal of the powder deposits. Table 7 furnishes data of manufacturing conditions and the results obtained in this method of making copper powder. Application for patenting this method of making copper powder was filed during the period under review.

For making aluminium and tin powders, certain improvements were done to the atomizing equipment developed at the National Metallurgical Laboratory, such as the use of detachable nipples containing holes. Because of this improvement, the atomizing equipment could be used over much longer periods than before. The atomizing equipment now in use consists of an annular nozzle formed by an inner truncated cone telescoping into an outer truncated cone. The nipple is fixed into the inner cone and the molten metal poured into the inner cone issues out of the hole of

the nipple for atomization by the air blast blowing through the annular nozzle. The hole of the nipple can be adjusted to secure the degree of atomization desired. It is believed that the use of the nipple helps to direct the air blast properly to prevent premature blocking up of the metal passage. The capacity of the equipment is limited by the pressure and quantity of air available for use as air blast. At a time, 1 lb. of metal could be treated. The following are the data regarding efficiency of atomization attained in these experiments:

Metal	Sieve analysis of powder made			
Tin	{	-200 mesh USS	= 55 per cent	
		-270 mesh USS	= 40 per cent	
Aluminium	{	-30 mesh USS	= 85 per cent	
		-100 mesh USS	= 45 per cent	

In continuation of the work already under way on porous bronze bearings through powder metallurgy techniques, progress was made on the development of the sizing operations. Sizing is a finishing operation done to correct the non-uniformity in dimensions, wrong dimensions of the sintered bearings and for correcting to the necessary extent the shape of the sintered bearings. Tools were designed for sizing of spherical shape porous bronze bearings in the press currently in use.

Further work is envisaged on the (i) making of copper powder on a larger scale; (ii) making

TABLE 7 — DATA RELATING TO PRODUCTION OF COPPER POWDER

COMPOSITION OF THE ELECTROLYTE				TEMPERA- TURE OF ELECTRO- LYSIS °C.	CATHODIC CURRENT DENSITY amp./sq. meter	SIEVE ANALYSIS OF THE POWDER OBTAINED				APPARENT DENSITY OF THE POWDER gm./cc.
Copper g/l.		H ₂ SO ₄ g/l.	Organic sub- stance g/l.			U.S.S. SIEVE FRACTION % by wt.				
Initial	Final					-200 140	-140 200	-200* 270	-270	
7.5	14.5	190	2.0	25	1200	1.3	15.8	32.0	50.9	2.20
10.0	17.5	160	1.0	25	3000	3.7	18.5	28.6	49.2	2.60
11.0	13.0	130	1.0	26	3000	0.0	12.5	33.9	53.6	2.70
12.0	14.0	130	1.0	30	3000	0.0	20.8	30.1	49.1	3.07
12.5	14.0	130	3.0	30	3000	0.0	19.9	27.3	52.8	2.20
15.0	17.5	160	3.5	25	3000	0.7	14.0	34.7	50.6	3.00
15.0	17.5	190	3.0	28	3000	4.2	27.5	34.2	34.1	3.40
14.0	16.0	160	3.0	30	3000	8.6	36.4	30.1	19.3	3.60

of iron based porous bearings by powder metallurgy methods and making of porous bronze parts to customer's requirements; (iii) making of tungsten powder and use of molybdenum sintering furnace for sintering carbide powder compacts; (iv) vacuum sintering of carbides.

8.1 Iron Powder

The objective of the project was to produce pure iron powder for the use of flame cutting and scarfing with autogenous torch. Fine iron powder is supplied through a specially designed cutting nozzle which is furnished with separate passage for air-iron powder stream in addition to the passages for oxygen and oxyacetylene gases. The iron powder burns in oxygen stream and thus produces over a localized area exceedingly high temperature and also produces slag particles which act as fusion agents. This process is adopted for surface dressing ingots of high chromium and stainless steel and for cutting large sized sections of highly alloyed steels.

A typical sieve analysis of the iron powder of Swedish origin is given below:

<i>Mesh size</i>	<i>Percentage</i>
-40	0.4
+100	
-100	21
+170	
-170	41.3
+270	
-270	37.3

The powder analyses Fe, 93.18; FeO, 4.76; SiO₂, 0.32; Al₂O₃, 0.54 and C, 0.11 per cent.

At present the requirements of this iron powder in India are fulfilled by importing annually about 100 tons of the powder at a price of over Rs 2000 per ton.

The pure iron powder can also be used in the manufacture of small parts using powder metallurgy techniques. This process for making high purity iron powder is based on the direct reduction of iron ore fines, with charcoal powder as the reducing agent.

Iron ore fines from the mines were used for the study of the production of the iron powder of same grade and sieve analyses as that of the foreign origin.

Iron ore fines and charcoal powders were dried at about 150°C. for about 1 hour before reduction process was carried out. The ore fines were suitably packed and subjected to solid state reduction with optimum soaking at high temperature. The temperatures were chosen so as to reduce the ore in short lengths of time and without sintering the reduced ore to a dense compact mass. When the reduction temperatures were on the slightly low side, the reduced mass could readily be crushed to powder.

The iron ore in the form of very fine powder, analysing Fe, 63.8; SiO₂, 4.88; Al₂O₃ 1.92 per cent, was used for the production of iron powder. The iron powder had the analysis of Fe, 85.7; FeO, 4.3 per cent.

Slime from iron ore beneficiation treatment, 1 in. Noamundi grade II containing 60.7 per cent Fe was reduced at high temperatures for different lengths of time. Best result yielded the maximum of 78.8 per cent Fe.

Likewise slime from 2 in. Noamundi grade I containing 49.5 per cent Fe yielded 68.5 per cent Fe pure iron powder.

Iron ore in the form of very fine blue powder exists in Indian iron ore mines as what is termed as 'Blue Dust'. This is of very fragile nature and would crumble down to fine powder on application of slight pressures. 'Blue Dust' on similar treatment to solid state reduction cycles yielded pure iron powders of over 97.5 per cent iron content. This Blue Dust was thereafter used for making iron powder on a much larger scale. The dried iron ore fines after high temperature solid stage reduction yielded a reduced mass which was crushed in a steel mortar to $-\frac{1}{4}$ in. size to facilitate milling. After milling, about 5 lb. of iron powder was obtained in one batch. The powder had the analysis of 93.5 per cent Fe and 5.0 per cent FeO.

The sieve analysis of the powder was

$\left. \begin{array}{l} -140 \\ +270 \end{array} \right\} \sim 25 \text{ per cent}$
 $-270 \sim 75 \text{ per cent}$

About 50 lb. of this iron powder was thus made and was then dried at 150°C. for $\frac{1}{2}$ hour. It was thereafter used for cutting of heavy casting of 15 per cent Cr steel using an auto-genous gas torch most successfully showing equivalent performance as the imported iron powder for the purpose.

9.0 Light Metals and Their Alloys

This project was taken up with a view to develop suitable aluminium-based alloys having high strength to weight ratio from indigenous raw materials including the use of rare earth group of elements.

Binary Al-Mg alloys containing high magnesium (7-10 per cent), not readily hot workable, were prepared and the effects of adding up to 5 per cent misch-metal (51 per cent Ce) to these alloys were investigated. The alloys were prepared by melting together the calculated quantities of aluminium, magnesium and misch-metal. Production conditions were standardized for obtaining pore-free melts, by the use of suitable fluxes and degassers. The melts in each case were cast into cylindrical and square moulds for tensile test specimens and assessment of hot-working properties respectively. As-cast microstructures of the untreated Al-Mg (7-10 per cent) alloys were identical in all cases; the only phases present being Al_3Mg_2 and Mg_2Si . When misch-metal was added, new phases made their appearance along with two other phases. With increase in the misch-metal content from 1 to 5 per cent, Al_3Mg_2 phase gradually decreased, and there was a gradual increase in the quantity of the new phase till, at 5 per cent misch-metal level, practically no Al_3Mg_2 phase could be observed. It was thereby assumed that the new phase essentially contained cerium and may be Mg, Ce, Al_4Ce or its mixture with ternary Al-Mg-Ce. It has not been possible to identify

these phases by metallographic examination only and their identification by X-ray and electron diffraction technique is now under way. The microstructure of the wrought specimens (made from rolled sheets) followed the same pattern as that in the cast specimens, with the exception that in this case the grain size had become smaller, possibly due to the homogenization treatment followed by mechanical working.

During preliminary work carried out in the solid solution range of these alloys (350°-500°C.), it was established that a suitable homogenization temperature for the alloys studied was around 450°C. The ingots were, therefore, soaked at about 460°C. for four hours before being worked. Forging and rolling were both carried out in the range 460°-400°C. The material was kept in the furnace again for a minimum period of about 20 minutes before working was continued. Hot forging was carried out to reduce the ingot thickness to about 0.5 to 0.8 in., which were then further reduced to about $\frac{1}{8}$ in. thick sheet by rolling. The results of these workability tests are summarized below.

(i) *Al-7 per cent Mg Alloy*—The untreated as well as the misch-metal treated ingots could be forged and finally rolled down to a thickness of about 0.125 in. It was observed that the workability of the alloy improved with increase in the misch-metal (or cerium) content.

(ii) *Al-8 per cent Mg Alloy*—The untreated Al-8 per cent Mg alloy ingots could be hot forged to slabs of about 0.7 in. thickness, but further forging resulted in the appearance of longitudinal cracks along with the entire length of the slab. Further forging was, therefore, discontinued. The alloy containing 1 per cent misch-metal could be hot forged to a thickness of 0.5 in., after which rolling of the slab was undertaken which failed resulting in a crocodile type of crack. This effect was noticed in several slabs of this composition range.

Ingots of Al-8 per cent Mg alloy with additions of 2.5 per cent misch-metal could

be successfully hot forged and rolled down to about $\frac{1}{8}$ in. thick sheet. The amount of reduction attainable per pass was found to increase with increase in the misch-metal content.

(iii) *Al-9 per cent Mg Alloy*—The untreated ingots of this composition could be forged to a thickness of about 1 in., but trials for further reduction in size resulted in development of cracks in the slab. The ingots containing 1 per cent added misch-metal could be forged to 0.67 per cent, but it failed in the very first pass in rolling, showing crocodile cracks. With misch-metal content at 2 and 3 per cent, the ingots could be hot forged to about 0.6 in. thickness, and could also be further rolled to about 0.4 in. thickness. In the next pass in rolling, however, the slabs in both cases crumbled to pieces. The cause of this failure was investigated. In the case of the alloy containing 4 per cent misch-metal, although the ingot could ultimately be hot worked down to about 0.125 in. thickness, edge cracks developed in the slab during rolling, and the cracked portions had to be discarded before rolling was renewed. With misch-metal content of 5 per cent, the ingots could be successfully forged and rolled without failure.

(iv) *Al-10 per cent Mg Alloy*—The untreated alloy could be hot worked to about 0.75 in. when the forging was done continuously. Further rolling caused appearance of cracks on the edges; continued rolling, after discarding the cracked portions, resulted in a crocodile failure in the slab. Ingots with 1 per cent added misch-metal did not stand hot forging at all; cracks developed at several places during forging without attaining any reduction in the section of the ingot. When the misch-metal content was raised to 2 and 3 per cent, the ingots could be hot forged to about 0.8 in. thickness and further hot-rolled to about 0.2 in. thick sheet with considerable difficulty. Extensive edge cracks appeared in the alloy containing 2 per cent misch-metal after rolling to 0.5 in. thickness and further rolling was

done only on the small uncracked portion. In the case of the 3 per cent alloy, cracks appeared in the very first pass in hot-rolling from 0.8 in. thickness, but an unaffected portion could be rolled down to 0.2 in. when handled more carefully. At this stage, a part failed showing a fracture somewhat similar to alligatoring. When the misch-metal content was raised to 4 and 5 per cent, the ingot could be reduced from the initial 2×2 in. section to only about 1 in. thickness, but the slabs showed severe edge cracks, rendering further working impossible. Further work on the above subject is actively being pursued.

Tensile test specimens were made from both as-cast rods and rolled sheet to B.S.S. 18: 1950 and tensile strengths were measured in Avery tensile-testing machine for cylindrical specimens and in Amsler tensile-testing machine for the flat specimens. It was observed that the tensile strength of all the cast alloys showed an initial decrease with misch-metal additions up to 2.3 per cent beyond which there was an increase. This lowering of tensile strength in this region might have been caused by the decrease in the quantity of Al_3Mg_2 and the formation of cerium containing phase and may be in some way responsible for the improved workability of the alloys. The exact mechanism of this change in the behaviour of Al-Mg alloys, brought about by the addition of misch-metal whilst not clearly understood, is under close study. The tensile strength of the wrought alloys increased with the increase in the quantity of misch-metal added.

Hardness values of both as cast and wrought specimens were measured after giving the specimens a micro polish. It was observed that hardness of the cast as well as wrought alloys increased with increase in the amount of misch-metal added, possibly due to the higher hardness of the cerium containing phases. As was reasonably expected, average hardness was higher in the case of the wrought specimens. Work was initiated on the effects of different heat treatment

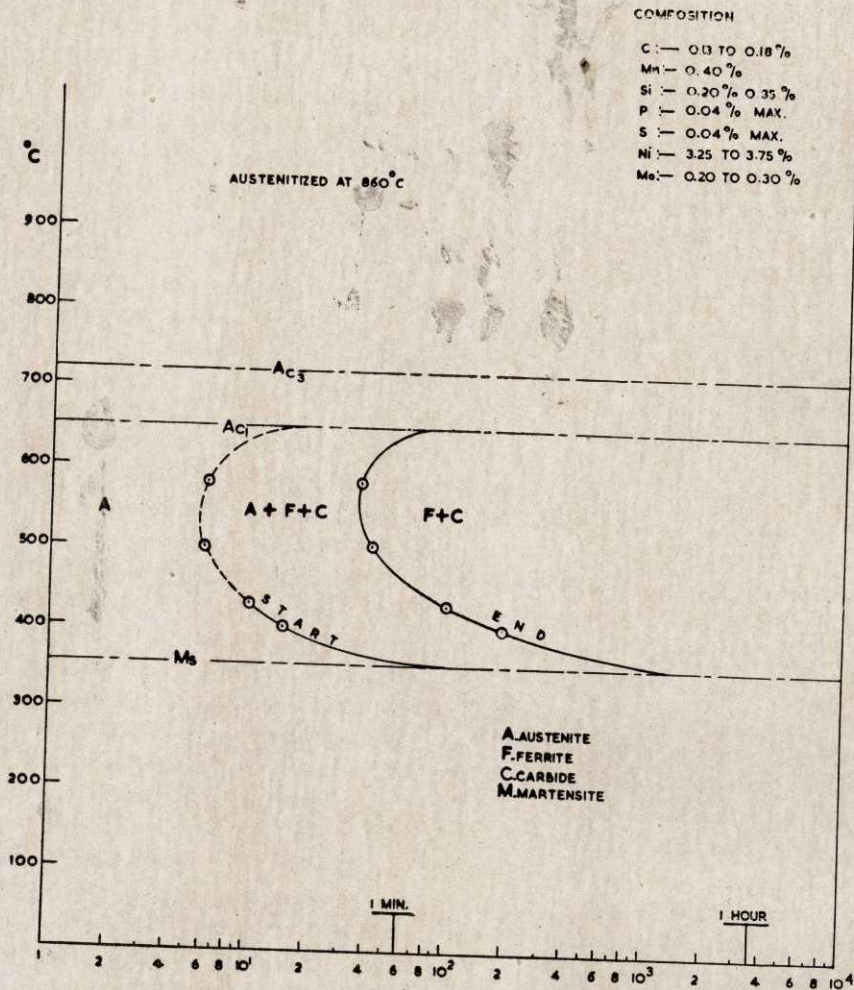


FIG. 10 — T.T.T. CURVE OF NICKEL STEEL

cycles on the various misch-metal treated Al-Mg (9-10 per cent) alloys, to determine suitable solution treatment temperature for each composition. Study of ageing of these alloys at different temperatures in the as-cast and wrought specimens was also concurrently taken up.

10.0 Continuous Casting

The project was taken up with a view to design and fabricate in the Laboratory a continuous casting plant on pilot plant scale

for light metals and alloys in the first instance. A double-walled water-cooled copper die ($1\frac{1}{4}$ in. i.d. $\times$ 2 in. long) was designed and fabricated with the exit water falling directly on the cast rod in the form of a spray. With water circulating at a rate of 3500 c.c./mm., and a drawing rate of nearly 18 in./min. only 2 in. length of the rod was obtained when aluminium was poured in the graphite pot preheated to 350°C. Because of the interrupted drawing of the rolls the surface finish of the continuously cast rod was not altogether satisfactory. The pilot

plant for continuous casting is now in the design and fabrication stages.

11.0 Study of Isothermal Transformation Characteristics of Indian Steels

This project was taken up with a view to prepare an atlas on the isothermal transformation characteristics of Indian plain carbon and alloy steels. The dilatometric, metallographic and hardness techniques are being followed to study the isothermal transformation characteristics of alloy steels. The curves of far more rapidly transforming steels are determined by microscopic methods only, while for slowly transforming steels, dilatometric as well as microscopic methods are adopted.

T.T.T. curves of the following steels were complete during the year under review.

	<i>Si-Mn Spring Steel</i>	<i>Mn-Steel</i>	<i>N-i-Steel</i>
C %	0.55	0.32-0.39	0.13-0.18
Si %	1.99	0.1-0.39	0.2-0.35
Mn %	0.75	1.35-1.65	0.40
P %	0.026	0.04	0.04
S %	0.018	0.04	0.04
Ni %	—	—	3.25-3.75
Mo %	—	—	0.2-0.3

The curves for such steels are shown in Figs.10-12

Hardenability Curve — Jominy End Quench Tests were performed to assess the hardenability of alloy steels under development. The Jominy test involves austenitizing a standard specimen 4 in. long and 1 in. in diameter and quenching it by water jet, which impinges on one end of the specimen. The specimen cools very rapidly at the quenched end and progressively less rapidly towards the opposite end of the test piece.

The hardness of the specimen at every $\frac{1}{16}$ in. is taken. The hardness value so obtained are plotted against length to obtain the Jominy curve. Jominy End Quench Curves of the following steels are shown in Figs. 13-15.

	<i>Ni-Gun Steel</i>	<i>Laminated Spring Steel</i>	<i>Si-Mn Spring Steel</i>
C %	0.31	0.85	0.55
Si %	0.20	0.19	1.99
Mn %	0.70	0.60	0.026
P %	0.036	0.04	0.018
S %	0.033	0.015	—
Ni %	3.57	—	—

At present the following steels are under study

	<i>Mo-Steel</i>	<i>Ni-Cr-Mo Steel</i>	<i>C Tool Steel</i>
C %	0.60	0.40	1.32
Si %	—	0.19	0.34
Mn %	0.7-1.0	0.59	0.42
S %	0.04	0.04	0.013
P %	0.04	0.04	0.018
Ni %	—	1.65	—
Cr %	—	1.21	—
Mo %	0.25	0.44	—

12.0 Study of Strain-ageing and Quench-ageing Embrittlement in Steel

The project was taken up with a view to study the possible causes, effects and prevention of quench and strain-ageing embrittlement in steels.

The effect of boron on the quench-ageing and strain-ageing characteristics was studied during the last year on nine specimens. The charge consisted of Low-moore iron, ferro-boron and ferro-silicon for deoxidization in

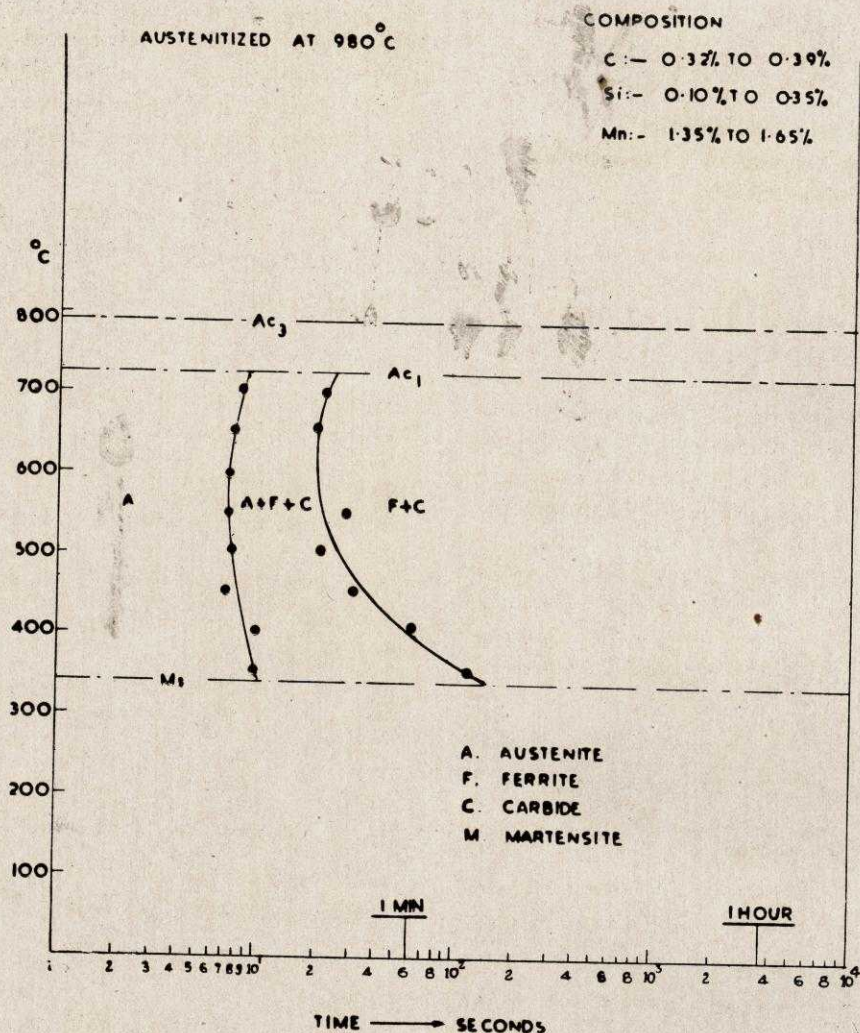


FIG. 11 — T.T.T. CURVE OF 15 PER CENT MN-STEEL

the first five heats, while in the next four heats, the boron contents were kept constant and carbon contents of the steel were varied by the addition of petroleum coke. In the first five heats, the hardness, electrical conductivity and maximum stress progressively increased with longer ageing time. The same increasing trend was observed in the other four specimens except in one, where hardness and electrical conductivity did not show any rise after one month's ageing. During this year the study of strain-ageing and quench-ageing behaviour of boron-treated low-carbon

steel was continued. After forging and rolling, the cast ingot specimens were taken for tensile strength, hardness and electrical conductivity tests. Quench-ageing behaviour was studied by noting the changes in hardness values of the specimen after quenching and subsequent ageing, whilst strain-ageing was observed by change in tensile strength and electrical conductivity after prestraining and subsequent ageing. Side by side, metallographic examination of the aged specimens was also studied.

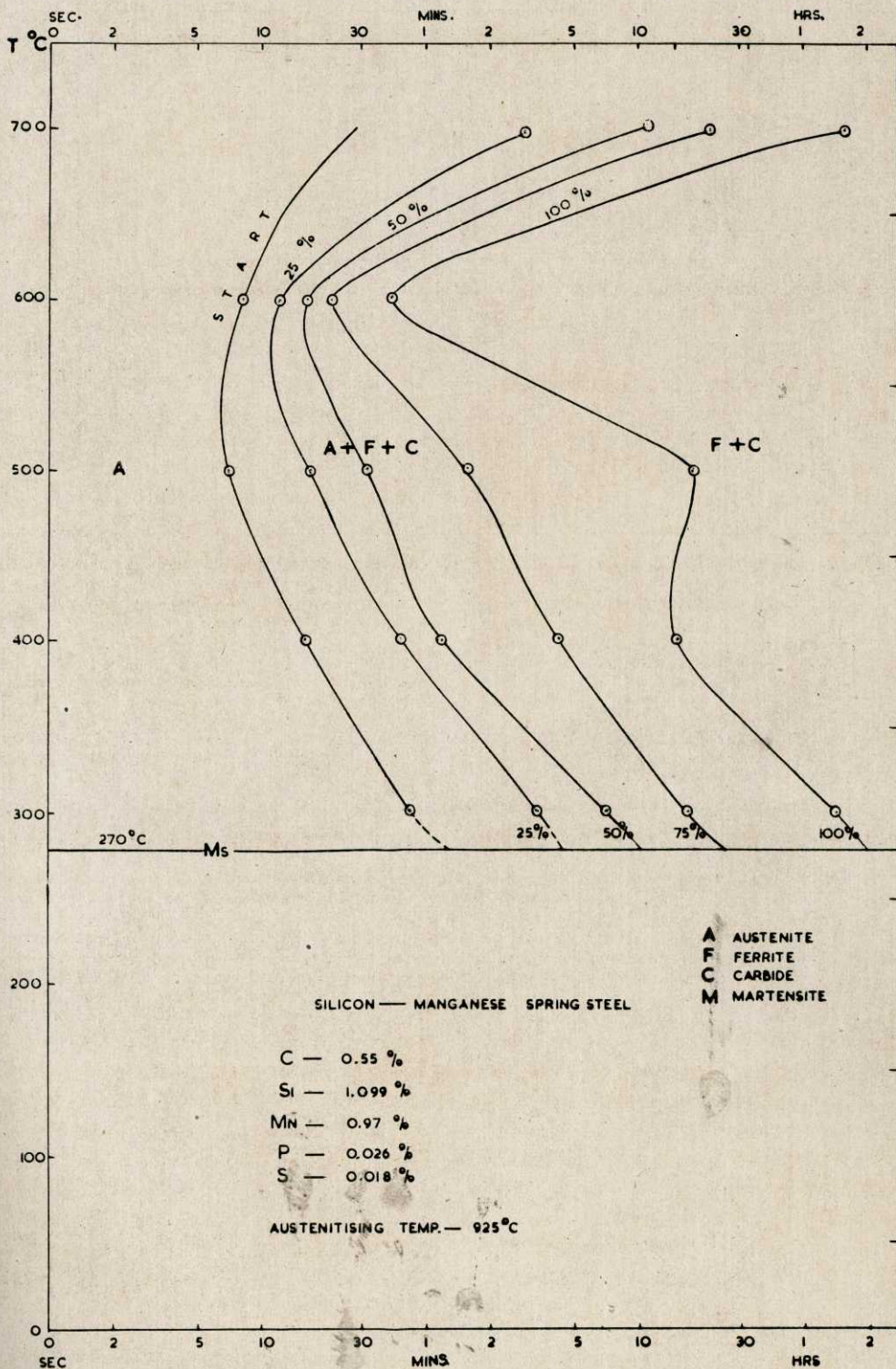


FIG. 12 — T.T.T. CURVE OF SILICO-MANGANESE SPRING STEEL

TABLE 8 — CHEMICAL COMPOSITION OF HEATS STUDIED

	C %	N %	B %	Mn %	Al %	Si %
B-11	0.04	0.0076	0.0012	0.009	0.0017	0.01
B-12	0.04	0.0078	*	0.014	0.08	0.08
B-13	0.03	0.016	*	0.022	*	0.24
B-14	0.04	0.017	*	0.028	*	0.22

*Not yet analysed.

TABLE 9 — HARDNESS VALUES OF THE QUENCHED AND AGED SPECIMENS

No. of SPECI- MEN	HARDNESS VALUES (V.P.H.) AFTER SOLUTION TREATMENT AND QUENCHING AND SUBSEQUENT AGEING FOR								
	1 hr.	4 hr.	24 hr.	48 hr.	1 week	1 month	2 months	3 months	4 months
B-11	127	130	132	133	135	130	126	126	126
B-12	120	123	124	125	125	122	120	120	120
B-13	128	129	126	124	124	125	123	121	121
B-14	116	123	118	118	119	120	119	115	118

TABLE 10 — ELECTRICAL CONDUCTIVITY OF BORON TREATED LOW-CARBON STEEL

ELECT. CONDUCTIVITY IN ohm/cm. AFTER SOLUTION TREATMENT AND PRESTRAINING TO 5% REDUCTION IN DIAMETER AND AGED FOR

	1 hr.	4 hr.	24 hr.	48 hr.	1 week	1 month	2 months	3 months	6 months
B-11	52610	52610	52480	—	53440	52810	53370	53430	53800
B-12	55520	55250	55280	—	55860	54670	55400	55520	55540
B-13	73310	64220	65670	68530	69020	70080	72800	72610	73580
B-14	78450	76400	72300	77780	77820	78630	78750	79000	79110

TABLE 11 — TENSILE STRENGTH VALUES OF BORON TREATED LOW-CARBON STEEL

SP. No.	MAX. AND YIELD STRESS OF SOLUTION TREATED AND PRESTRAINED (TO 5% ELONGATION) TENSILE TEST SPECIMENS AFTER AGEING FOR									
	Immediately after straining		After 24 hr.		After one week		After one month		After two months	
	Yield stress T.S.I.	Max. stress T.S.I.	Yield stress T.S.I.	Max. stress T.S.I.	Yield stress T.S.I.	Max. stress T.S.I.	Yield stress T.S.I.	Max. stress T.S.I.	Yield stress T.S.I.	Max. stress T.S.I.
B-11 (1)	UY 21.49	24.67	UY 23.83	27.85	UY 24.53	29.43	UY 25.36	30.09		
	LY 22.02		LY 23.44		LY 24.17		LY 24.04			
(2)	UY 22.48	25.43								
	LY 23.02									
B-12 (1)	UY 24.06	27.28	UY 25.67	29.66	UY 29.81	35.18	UY 25.71	30.24		
	LY 24.06		LY 24.79		LY 29.32		LY 25.08			
(2)	UY 28.85	32.09								
	LY 28.36									
B-13	UY 22.43	24.90	UY 23.49	27.05	—	—	UY 22.79	27.83	UY 24.53	28.53
	LY 22.18		LY 23.46				LY 22.55		LY 23.57	
B-14	UY 21.13	23.35	UY 23.69	26.73	—	—	UY 22.74	26.77	UY 22.69	27.19
	LY 20.46		LY 23.31				LY 22.14		LY 22.17	

UY and LY indicate upper and lower yield stresses respectively.

Few more heats have been made by the addition of aluminium as deoxidizer in place of ferro-silicon which was previously added before the addition of boron.

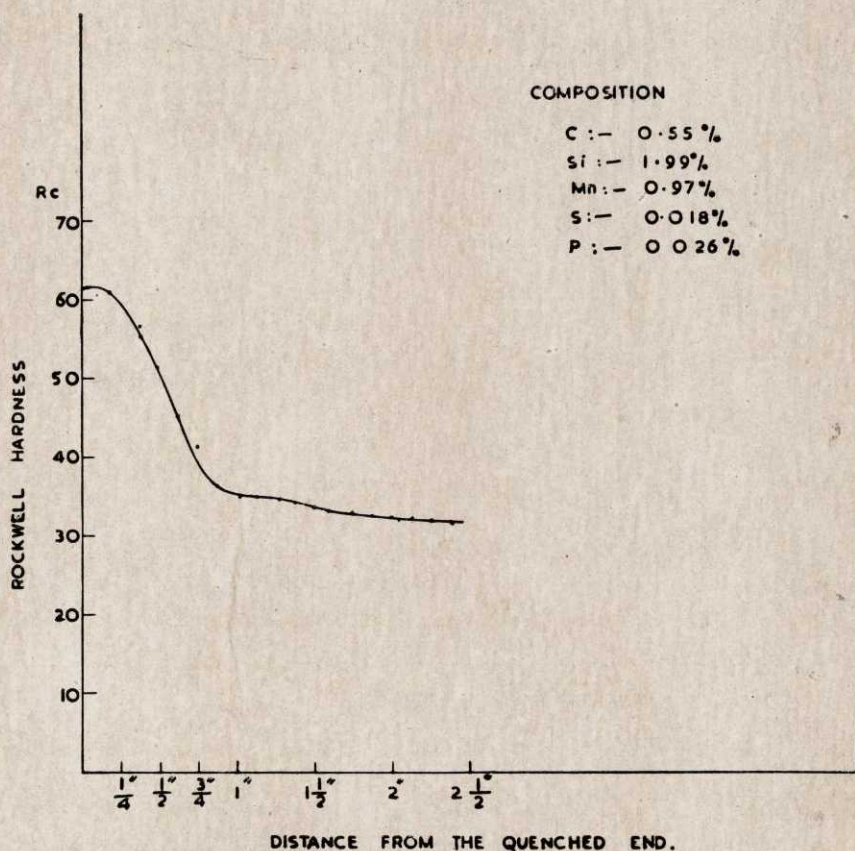


FIG. 13 — JOMINY END QUENCH HARDENABILITY CURVE OF SILICO-MANGANESE SPRING STEEL

Chemical composition of the heats on which work was done is given in Table 8.

These specimens after ageing for six months did not show any non-ageing characteristics. Hardness, electrical conductivity and maximum stress progressively increased with longer ageing time. However, increase of these values were not much in case of specimen No. B-13 and B-14. Further, micro-study also revealed very little precipitation, on the grain-boundary. These results are summarized in the Tables 9, 10 and 11.

13.0 Development of Nickel-free Coinage Alloy

Although nickel is eminently suitable for coinage alloys, it does not exist in India,

thereby posing a real problem for use in coinage alloys. Work was, therefore, taken up to develop suitable coinage alloys containing manganese as one of the constituents and having identical metallurgical and physical properties with the present cupro-nickel coinage alloy.

In continuation of the studies on the working behaviour of alloys developed at National Metallurgical Laboratory, further production trials were made at the Government of India Mint, Alipore. Both hot and cold rolling were carried out on ingots cast from 60 per cent scrap, 40 per cent virgin metal made in arc furnace and satisfactory results were obtained. In order to find the suitability of 100 per cent scrap melting in oil fired and arc furnaces, melts were made

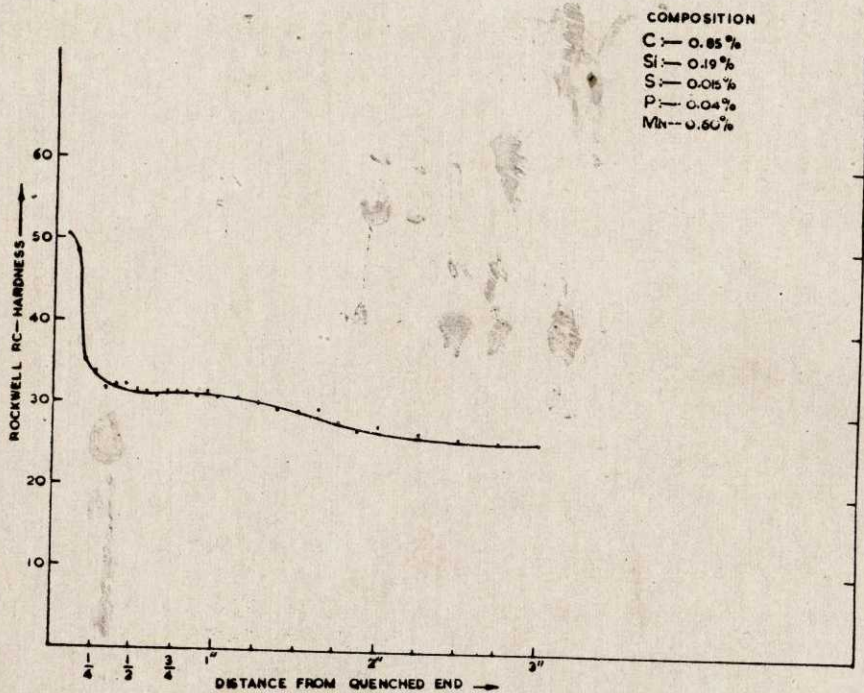


FIG. 14 — JOMINY END QUENCH HARDENABILITY CURVE OF LAMINATED SPRING STEEL

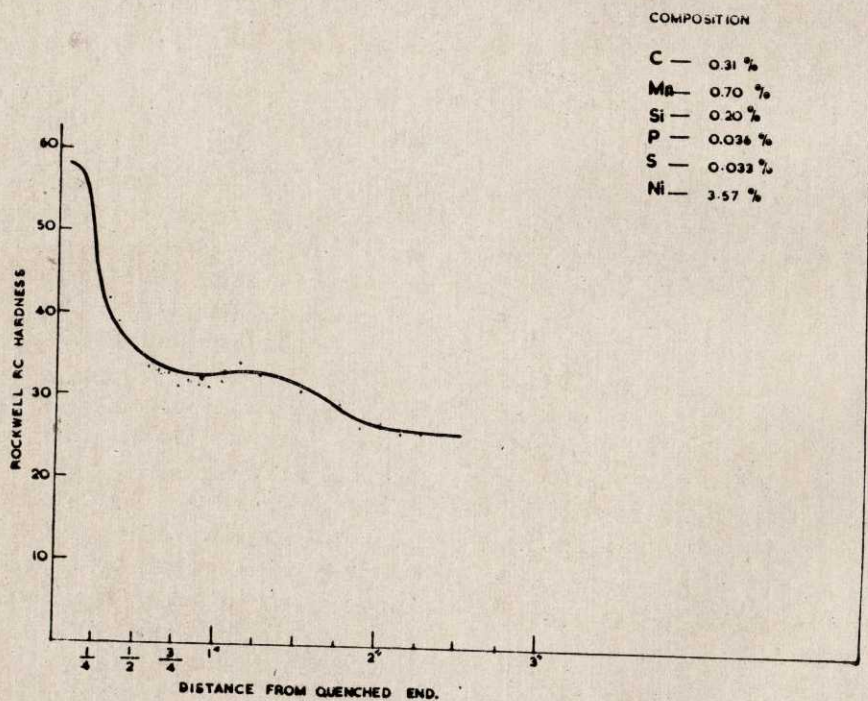


FIG. 15 — JOMINY END QUENCH HARDENABILITY CURVE OF NICKEL GUN STEEL

with varying scrap additions (100-60 per cent) and virgin metal (0-40 per cent). These melts were cast in $4 \times 4 \times 30$ in. ingots and $\frac{1}{2} \times \frac{1}{2}$ ft. $\times 30$ in. slabs. From the studies carried out till now, it was observed that use of scrap in the charge material did not yield very sound ingots. However, the hot and cold rolling characteristics of the ingots made under the above conditions were satisfactory. An interim report covering the progress of the work so far done in collaboration with India Government Mint was prepared.

13.1 Production of Manganese Bearing Brasses

Brass is extensively used in India for utensil making. Due to the shortage of production of copper in India and import of the entire requirement of zinc, this project was taken up with a view to reduce the copper and zinc contents in brass by their partial substitute by manganese.

In continuation of the studies on the various properties of manganese-bearing brasses, additional melts were made in the oil fired furnace, on selected composition to determine the effects of manganese, zinc and nickel in binary and ternary copper base alloys in greater detail. These melts were, as usual, cast in $6 \times 8 \times 1$ in. slabs and were hot and cold rolled to sheets. The effects of cold working and temperature of annealing on the various mechanical properties, i.e. hardness, tensile strength, per cent elongation, etc., were studied. Investigations on the determination of thermal conductivity of these alloys included a few binary compositions, containing manganese below 10 per cent or above 30 per cent. Electrical conductivity of the above compositions were also determined to find the relationship between thermal and electrical conductivities of these manganese-bearing copper alloys. An experimental set-up was made to study the changes in electrical conductivity with temperature to formulate the temperature coefficient of electrical resistivity.

14.0 Production of Magnesium by Electrolytic Method from Magnesium Chloride

Messrs Tata Chemicals, Mithapur, produce a large quantity of magnesium chloride as a by product in their salt industries which has a very limited market. Investigation was taken up on the possibility of production of magnesium metal from this product.

Conditions were standardized whereby dehydration of the hydrated salt with or without additions of alkali chloride and fused bath electrolysis of the dry salt could be carried out satisfactorily. With the addition of alkali chlorides, the dried salt obtained was subjected to electrolytic trials. Attempts made to prepare cell feed to the required specification, viz. MgCl_2 , 1.5 H_2O in the absence of any alkali chloride containing not more than 2 per cent MgO were successful. To achieve it, the whole drying technique was suitably modified.

250 kg. of hydrated hard lumps of the salt were crushed to small size and dried in the modified dryer with hot air circulation and mechanical stirring under controlled humidity. A capacity dryer was designed and fabricated based on the data obtained from present scale drying oven. Mild steel vessel was used with centrally placed graphite, covered by a ceramic diaphragm, as anode and body of the vessel served as a cathode for electrolytic trial experiments. The pot was externally heated in a gas-fired furnace fitted with controls for adjusting the temperature. Experiments were conducted with this type of cell using alkali-free cell feed and factors involved for best operation of the cell, viz. composition of the bath temperature, current density, inter-polar gap, current and energy efficiency, etc., were evaluated; current difficulty relates to the diaphragm. Development work was taken up to find out a suitable composition for the diaphragm. Continuous electrolytic run will be attempted incorporating the new diaphragm in the cell.

14.1 Electrolytic Production of Al-Mg Alloy

Binary alloys of aluminium with varying magnesium contents from 3, 5, 7 and 10 per cent are useful light metal alloys, but are difficult to prepare by conventional methods. It was considered highly probable on theoretical grounds, that with molten aluminium cathode at the bottom, fused electrolysis of a suitable magnesium bath in a graphite pot with graphite anode, might produce aluminium-magnesium alloys. With the object of preparing aluminium-magnesium alloys electrolytically, this investigation was taken up. A suitable cell was designed and fabricated. Cathode pot, anode holder, ceramic diaphragms and other electrical accessories were procured and a suitable salt mixture was also prepared. Bench scale experiments were done with molten aluminium as cathode and the cell was externally heated in a hot muffle furnace. When electrolysis was started, the ohmic resistance of bath produced sufficient heat which supplemented by controlled external heating could keep the bath in a molten conditions. The electrolyte was fed into the cell from a second fusion pot. After running the electrolysis for the specified period of 2 hours the alloy was agitated with argon and ladled out. The alloy obtained with varying quantities of Mg depended upon various factors, viz. period of run, current density, temperature, etc. Not more than 7 per cent of Mg in the alloy could be obtained under the existing conditions of operations. These preliminary trials were designed to establish optimum conditions of electrolysis. Results so far obtained were encouraging to warrant certain essential modification in engineering design of the cell and furnace.

15.0 Electrolytic Production of Chromium and Carbon-free Iron-Chromium Alloy

Work on electrolytic preparation of chromium and carbon-free iron-chromium alloys

from Indian chromite ores was completed, based on experiments to define optimum conditions of electrolysis, such as cathode current density, acidity of the electrolyte, temperature, composition of the anolyte, duration of electrolysis, concentration of ammonium sulphate in the electrolyte and the ratio of iron to chromium.

15.1 Electrolytic Production of High-purity Chromium

Good deposits of pure chromium can be obtained by the electrolysis of chromic acid solutions containing small percentages of sulphate, but the cost of this operation due to low current efficiency rendered the metal expensive.

Preliminary experiments carried out in the Laboratory from solutions containing fluorides gave higher current efficiencies than the conventional solutions. Experiments were carried out in 500 ml. beakers with 300 ml. solution. The top of the cell was covered with a glass lid having holes to allow the cathode and anode to pass through. A 12 in. condenser was attached to the glass lid which helped to maintain the composition of the electrolyte constant. Lead-tin alloy was used as anode and copper tubes as cathode. The composition of the electrolyte was as follows:

Chromium acid	300 gm./l.
Sulphuric acid	1 gm./l.

Varying amounts of hydrofluoric acid were added to the above solution, and electrolysis was done at various temperatures (40°-60°C.). Maximum current efficiency obtained was 22 per cent. With higher current densities and higher temperatures and with only hydrofluoric acid as the catalytic agent, the current efficiency rose to 27 per cent. Further work with varying conditions is in progress.

16.0 Studies on Corrosion of Metals and Alloys

In order to carry out long-term studies on various aspects of corrosion under Indian

tropical weather conditions, the corrosion section of the Laboratory is under heavy expansion. Additionally, long-range studies on marine corrosion and its causes and effects, which will considerably help in reducing corrosion in damages and losses in rapidly expanding industries being established in Indian coastal areas have been projected apart from marine corrosion effects in naval and merchant sea-going vessels. For this purpose regional marine corrosion research station is under establishment at Digba on a plot of land measuring 5 acres which has been kindly donated by the Government of West Bengal.

Under the auspices of the Metals Committee of the Council of Scientific & Industrial Research, Corrosion Committees have been constituted for channelizing the corrosion research in India. For this purpose, ten different committees, covering various aspects of corrosion studies, have been formed, with a representative from National Metallurgical Laboratory on each of the committee. Two of these committees have been constituted under the convenorship of Dr. B. R. Nijhawan, Director, and Dr. T. Banerjee, Deputy Director, National Metallurgical Laboratory.

(i) *Studies on Atmospheric Corrosion of Metals and Alloys* — Further work was continued on the studies on atmospheric corrosion of various metals and alloys exposed to industrial atmosphere of Jamshedpur. Samples of mild steel and Tiscor exposed horizontally, at 45° and vertically in the month of July 1959, to determine the effect of angle of exposure on corrosion, were removed after completion of one year of exposure. Determination of weight loss indicated that samples exposed horizontally corrode at rates 2-3 times more than those exposed vertically.

To study the behaviour of coupled metals and alloys in atmosphere, a detailed and long range programme of work was taken up. For preliminary studies, samples of aluminium, zinc, copper, etc., in galvanic

contact with mild steel, brass and stainless steel were exposed during July 1960. The samples were bolt-wire type designed on the basis of work carried out at the Bell Telephone Laboratory in the U.S.A. For comparative tests, samples of aluminium, zinc mild steel, stainless steel, copper, etc., were also exposed in wire form. Three sets of these samples were removed after 1½, 3 and 6 months' exposure. Tests were continued on atmospheric corrosion resistance of low-alloy high tensile structural steels, under active development at the National Metallurgical Laboratory. Tests carried out under various conditions of exposure in this Laboratory indicated the importance of properties of rust formed on mild steel and low-alloy steel in increasing their resistance to corrosion. A detailed programme to study the properties of 'rust' was, therefore, drawn up. For these studies samples of mild steel and Tiscor were exposed in the month of January 1961. The samples, to be removed after different exposure periods, will be studied, in addition to loss in weight, in relation to the following properties of rust:

1. Soluble and insoluble sulphate
2. Total iron
3. Moisture content
4. Ferrous iron
5. Porosity
6. Structure

For a proper assessment of the effects of climatic and pollution factors on corrosion resistance, lead peroxide candle is exposed every month to determine sulphur pollution of the atmosphere. Continuous record of temperature and humidity is also being kept.

(ii) *Development of Substitute Metallic Container Suitable for Packing and Marketing of Kerosene Oil* — Tin-coated steel sheets are used for fabricating containers to market kerosene oil. Tin being a costly imported metal involves a considerable amount of foreign exchange. The present investigation was taken up to develop a suitable substitute container for kerosene oil. Preliminary

studies indicated the suitability of using zinc-coated steel treated in acid sodium dichromate solution. On the basis of these findings, comprehensive tests were started for studying the optimum conditions for chromate treatment by varying the time of immersion and acid content of the solution. The acid content was varied between 5 and 40 c.c. while the time of immersion ranged between 5 and 40 sec. The treated specimens were immersed in oil-water mixture and tests continued for a long time. From the data obtained till now, it appeared that both acid content and time of immersion control the protective nature of the film formed. Current work relates to tests undertaken with chromate treated galvanized steel containers filled with kerosene oil, under varied exposure conditions, e.g. with fluctuation of temperature and humidity of the empty space above the oil level in the container.

(iii) *Mechanism of Chromate Passivation of Zinc* — Treatment of zinc in acid chromate solution improves its resistance to corrosion in stagnant layer of water. The mechanism of film formation is under scrutiny, from the point of view of ascertaining the relations between the thickness of the chromate film formed, amount of zinc attacked, composition of film and its resistance to corrosion.

For the present series of investigations, galvanized steel, rolled zinc and zinc plated steels were used. The samples were first treated in 200 gm./l. of sodium dichromate solutions containing 5-40 c.c. of sulphuric acid for 5-60 sec. The film formed was then stripped in 20 per cent chromic acid solution held at 80°C. From the various results obtained, it was observed that the attack on zinc increased with increase in acid concentration but the amount of film formed decreased after initial increase with rise in acid concentration. The acid concentration, beyond which the amount of film formed decreased, was found to vary for galvanized steel, rolled zinc and plated zinc. Further work on this problem is in progress.

(iv) Suggestions regarding causes of corrosion and remedial measures to be adopted were given to various industrial problems on corrosion referred to the Laboratory. Of the various problems detailed investigations were carried out on the following two problems encountered in the caustic soda industry:

(a) *Corrosion of Nickel Tubes in the Steam Chest of Caustic Soda Evaporator* — Localized corrosion had occurred in steam side of the expanded nickel tubes in steam chest of caustic soda evaporator. Such corrosion led to perforation within 1-2 years of service. Investigation was, therefore, taken up to determine relevant factors responsible for this type of attack and to formulate remedial measures. Tests were carried out by passing steam containing CO_2 and air in different rates over nickel tubes to determine the effect of CO_2 : air ratio on corrosion of nickel. Long term tests, extending over 30 days, were carried out at room temperature in solutions containing different amounts of CO_2 and air. The results indicated that corrosion rate was maximum when the ratio of CO_2 : air was between the range of 40: 60 and 80: 20. This type of attack can be minimized by the following methods:

- (i) to admit air inside the steam chest so as to reduce the CO_2 : air ratio below 40: 60.
- (ii) replacing nickel by inconel tubes which is immune to attack by CO_2 : air mixture dissolved in the steam condensate.

(b) *Corrosion of Brine Storage Tank* — Mild steel tanks used for handling saturated brine solution in the caustic soda industry were found to be severely pitted at areas intermittently exposed to the solution. Investigation was taken up with a view to find out the remedial measure to be adopted for this type of attack and to minimize premature failure of the tanks. After conducting at-site study, further laboratory tests under total and intermittent immersion conditions were carried out with mild steel and aluminized steel samples. Saturated brine

solution was prepared from commercial grade of sodium chloride, and to it 5, 3, 1.5, and 1 gm. of NaOH and 2.5 gm. of Na_2SO_4 and 1 gm. of Na_2CO_3 per litre were added. The precipitated calcium and magnesium hydroxide and the filtered solution were used during these tests. Another set of experiments was also carried out with purified saturated brine solutions of pH 7.9, 10 and 11.6. For carrying out intermittent immersion test, an experimental set-up was designed in which it was possible to control the rate of rising the solution level. The results of the various tests indicated that though it was possible to reduce the total corrosion by increasing the pH, it was not practicable to stop it completely even at a pH of 11.6 in the intermittently exposed areas. The attack in the intermittently exposed areas was localized and formed deep broad pits specially at higher pH. As far as the protection of metallic coating on steel was concerned, aluminium and zinc were not found to be effective in solutions of pH 10 and above. Metallic containers made of high alloy steels would not be economical but use of suitable plastic coatings on steel can considerably enhance the life of the tanks.

17.0 Preparation of Plating Salts

The electroplating industry in India has so far been using imported plating salts, polishing compositions and plating equipment. Due to strict import restrictions, these proprietary plating salts are not available in the market and the electroplating industries in India are experiencing considerable difficulties for their plating materials. Some of the Indian producers have marketed plating goods including plating salts but these products have in most cases failed to produce equivalent results as the imported products gave. To assist the plating industry in India, development work on the formulation of common plating salts such as cyanide copper plating salts, dull and bright nickel plating salts, etc., was taken up at the

National Metallurgical Laboratory, based essentially on the use of raw materials and chemicals made in India. Additions of special ingredients beneficial in increasing conductivity, good throwing power, brightness and related properties for good plating were also investigated from indigenous resources.

Cyanide copper plating salts and dull nickel plating salts were formulated and tested in the laboratory with good results. Dull nickel plating salts were also tested by an industrial firm of repute, who reported the National Metallurgical Laboratory made nickel salt to be as good, if not superior, to the imported salt. Preparations were made for continuous trial of the National Metallurgical Laboratory salts for a period of at least 6 months on a production basis.

17.1 Hard Chromium-plating on Aluminium

The use of hard chromium plating on aluminium has considerably increased for many engineering applications in producing a hard, wear-resistant and oil-retaining surface for applications in components such as cylinders, pistons, bearings and brake drums.

A suitable process for hard chromium plating on aluminium having the requisite properties was worked out. Samples of aluminium were roughened by chemical etching which was observed to produce improved adhesion of the coating than the mechanical roughening. Specific etching treatments were developed to provide a mechanical key for the deposit. After such chemical treatments, the aluminium surfaces were given immersion coatings of iron or nickel from their respective solutions which were cathodic to aluminium. Comparative studies were made on the physical properties and characteristics of chromium deposits with their corresponding hardness, produced on the two layers separately. The chromium plating bath used contained a lower con-

centration of chromic acid (250 gm./l.) than the usual bath.

Investigations to electrodeposit chromium directly on aluminium after chemical etching eliminating the immersion step in the deposition processes were taken up. Different etching agents were tried out so that the subsequent deposits were adequately adherent.

18.0 Liquid Gold

A large number of firms had asked for samples of Liquid Gold for testing which were sent to them. The process was released through the National Research Development Corporation of India to a number of interested firms for commercial exploitation on payment of significant premia and royalties. To impart the requisite technical know-how to these firms, practical demonstrations of the process were held over a period of several weeks.

19.0 Determination of Gases in Metals

The presence of gases in metals is known to influence their properties and at times rather adversely. Gas analyses of different metals and alloys, therefore, form an important part in research and development work in metals and alloys. The apparatus required for this purpose was not only costly but had to be imported. Work was, therefore, taken up for designing and fabricating the apparatus for accurate gas analyses, such as of oxygen, nitrogen, hydrogen in different metals, ferrous and non-ferrous alloys.

The vacuum fusion apparatus and hot vacuum extraction apparatus which had been fabricated at the National Metallurgical Laboratory worked most successfully. Samples such as depolarized nickel, titanium metal, L.D. steel, electrolytic manganese, electrolytic nickel, copper, brass, aluminium metal, tool steel, etc., were gas analysed.

In order to study the rate of adsorption of hydrogen during pickling, pickled with the different types of inhibitors, quick and con-

tinuous analyses of hydrogen became necessary. So some modification regarding the introduction of the pickled sample in the vacuum system was made. To eliminate the risk of losing some of the hydrogen from the pickled samples before heating, the latter were introduced through a mercury lift. Inhibitors such as beta-naphthol, urea, di-o-tolyl-thiourea up till now being used. Amongst them di-o-tolyl-thiourea acted best as an inhibitor, even 0.005 gm./100 c.c. in 20 per cent sulphuric acid and pickled for 4 hours the hydrogen pick up was found to be only 1.5 c.c. per 100 gm. of the steel sample.

To avoid the complication in maintaining a steady high vacuum, a carrier gas technique was developed for the determination of hydrogen in steel and alloys. This method was first introduced by Shanhan and Cook consisting in heating the sample at 650°C. in a current of inert gas like nitrogen or argon. Hydrogen was diffused out of the sample and was carried out by the carrier gas current. Hydrogen was oxidized to water by heated copper oxide and iron oxide catalyst while passing through them. The water thus formed was determined by Karl Fisher titrator. Some experiments were carried out to determine hydrogen in metals and alloys. It was observed that the hydrogen values obtained by the carrier gas method were quite in agreement with the values obtained by the vacuum extraction method. Some deviations were observed only in cases when hydrogen content was below 1 c.c. per 100 gm.

20.0 Study of Non-metallic Inclusions in Steel

This investigation was taken up with a view to determine the distribution of oxygen amongst the oxide inclusions and also the total oxygen contents of the inclusion material.

To study the cleanliness of plain carbon steel and alloy steels, it was necessary that study should be made on the actual methods

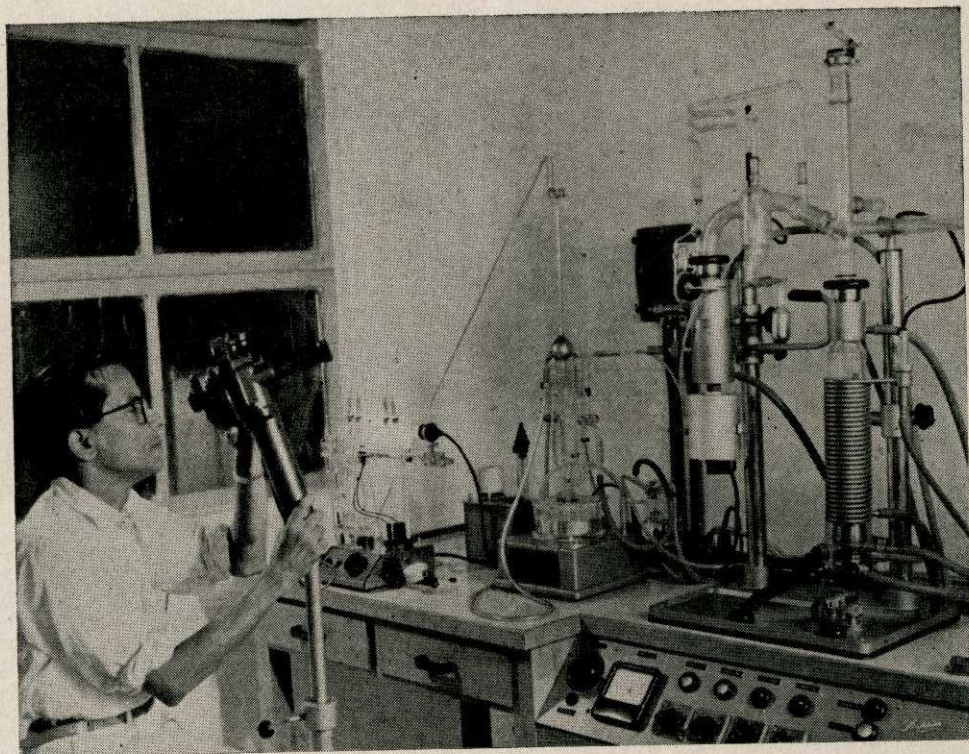


FIG. 16 — A VIEW OF THE VACUUM FUSION APPARATUS INSTALLED AT THE NATIONAL METALLURGICAL LABORATORY

for the determination of inclusion in steel. For this purpose, an apparatus was set up for estimation of oxide inclusion in steel. The method consisted in dissolving a sample of steel in a solution of alcoholic-iodine, filtering, igniting and weighing the insoluble residue. The ignited residue was examined for silica, oxides of iron, manganese and aluminium, etc. The alcoholic-iodine solution was prepared by dissolving 70 gm. of pure iodine in 600 ml. of pure methyl alcohol. This solution was deoxidized by passing purified nitrogen for 5-6 hours in a special 'boiling' flask (Fig. 17) made for this purpose and the solution was alternately boiled and cooled in the presence of nitrogen. The mild steel sample for examination was cut into specimens of size $1 \times 2 \times 5$ mm. The samples were first cleaned with carbon tetrachloride and then with dilute hydrochloric acid. It was washed and dried and about 10 gm. of

the sample was accurately weighed and placed in the reaction flask. The reaction vessel consisted of a R.B. flask (1000 ml.) fitted with a straight 8-in. Liebig condenser. The upper end of the condenser was closed by a bung carrying a long delivery tube and a shorter outlet tube. Fig. 18 shows the arrangement of the apparatus. The prepared alcoholic-iodine solution was filtered into the reaction vessel containing the sample. The condenser and delivery tube were attached to the flask which was supported on a sand tray heated on a hot plate. The flow of nitrogen was started in the reaction flask which was heated slowly to 65°C . This was continued for about 10 hours when the sample of iron dissolved leaving the inclusion residue. The flask was cooled overnight in the presence of purified nitrogen. The solution was filtered and residue washed with methyl alcohol rapidly, dried and

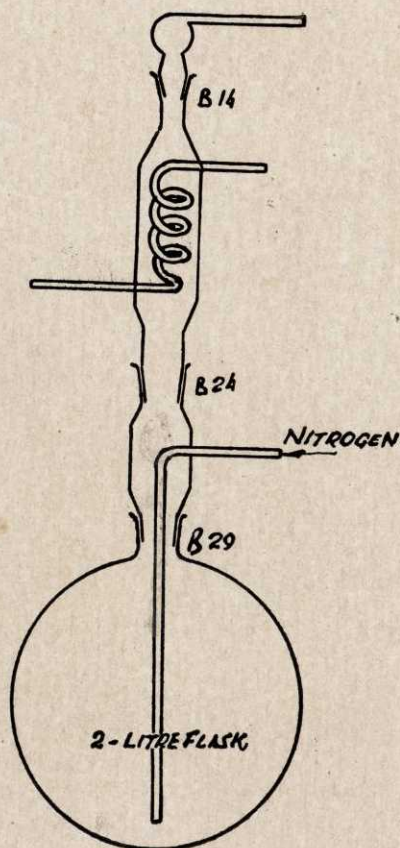


FIG. 17 — BOILING FLASK

ignited and weighed and analysed for oxides of iron, manganese, silica and aluminium. The residue of inclusion thus obtained was examined by X-ray diffraction, spectrographic and chemical methods. Total oxygen as determined by vacuum fusion gas analysis technique which compared exceedingly well with the values calculated for total oxygen from the oxide residues experimentally obtained in these studies.

In early stages, difficulty was experienced as the samples did not dissolve completely and it could be detected only while filtering. Later on, a powerful magnet was used in order to detect the presence of undissolved iron. The residue of inclusion on X-ray diffraction analysis showed requisite lines

pertaining to Fe, FeO, MnO₂, Al₂O₃ and aluminium-silicate.

After micro-chemical analysis of the inclusion residue, total oxygen was calculated from the total oxides and compared with total oxygen obtained by vacuum fusion method. Three mild steel samples were examined by this method and the results of chemical analysis of the inclusion residue, total oxygen calculated from the sum of oxides and total oxygen and vacuum fusion method are shown in Table 12.

It would be observed from Table 12 that there was good agreement between the total oxygen as calculated from the sum of oxides and its value actually obtained by the vacuum fusion analytical technique followed at the National Metallurgical Laboratory.

21.0 Preparation of Foundry Chemicals

This investigation was taken up to prepare foundry chemicals generally imported in India from foreign countries. A heat gene-

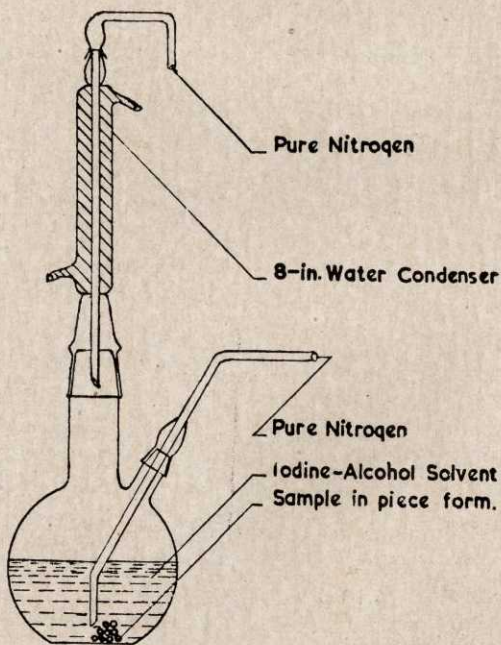


FIG. 18 — ARRANGEMENT OF THE REACTION FLASK

TABLE 12—SHOWING THE RESULTS OF CHEMICAL ANALYSIS OF THE INCLUSION RESIDUE, TOTAL OXYGEN CALCULATED FROM THE SUM OF OXIDES AND TOTAL OXYGEN BY VACUUM FUSION METHOD

CONSTITUENTS	MILD STEEL I		MILD STEEL II		MILD STEEL III	
	Test 1	Test 2	Test 1	Test 2	Test 1	Test 2
Total ignited residue, %	0.132	0.145	0.053	0.048	0.059	0.061
Silica (SiO ₂), %	0.008	0.010	0.012	0.008	0.039	0.034
Iron oxide (FeO), %	0.054	0.072	0.024	0.017	0.012	0.016
Alumina (Al ₂ O ₃), %	0.044	0.030	0.0086	0.010	0.003	0.005
Manganese oxide (MnO), %	0.017	0.027	0.0056	0.006	0.005	0.006
Total*	0.123	0.140	0.053	0.041	0.059	0.061
(a) Calculated from the sum of SiO ₂ , FeO, MnO, Al ₂ O ₃	0.040	0.041	0.014	0.013	0.026	0.025
(b) By vacuum fusion method	0.039	0.039	0.010	0.010	0.020	0.020

*The oxides FeO and MnO are changed during ignition to Fe₂O₃ and Mn₃O₄.

rating compound used in the production of sound and pressure tight casting was prepared. The material was applied on the surface of the liquid metal in the feeding heads of iron and steel castings and its properties were under evaluation based on practical trials.

22.0 Synthetic Cryolite

Cryolite is an essential mineral required in steel, ceramic and aluminium production industries. It is also used in insecticides and in the manufacture of enamels and opaque glass.

Natural cryolite is not found in India. Its requirements amounting to about 7500 tons per annum are met entirely by import chiefly from Denmark and England. Synthetic cryolite is produced on a commercial scale in the United States, Canada and several European countries. These countries are thus no longer dependent upon imported natural cryolite. The artificial cryolite is equally satisfactory and in practice it actually contains somewhat less objectionable impurities. In view of the expanding demand of this material in India, the need for its synthetic preparation has become most imperative, particularly in the context of known extensive deposit of fluorspar in

Rajasthan and Madhya Pradesh. Fluorspar is one of the chief raw materials used in the production of synthetic cryolite. These fluorspar deposits are, however, of low-grade type and have to be beneficiated in the first instance. Extensive work on laboratory and pilot plant scale has been done at the National Metallurgical Laboratory on the beneficiation of fluorspar.

Different countries have adopted different processes for the production of synthetic cryolite. In the process normally employed, fluorspar (acid-grade) is treated with sulphuric acid to produce hydrofluoric acid which is then neutralized with proper amounts of soda ash and aluminium hydrate to produce cryolite. In another process, synthetic cryolite is made by the reaction of ammonium fluoride, sodium sulphate and aluminium sulphate, ammonium fluoride being produced by heating a dry mixture of fluorspar and ammonium sulphate.

Investigations were, therefore, taken up at the National Metallurgical Laboratory to produce synthetic cryolite by fluoboric acid process. In this process, low-grade fluorspar is first beneficiated to metallurgical grade, having the composition: SiO₂, 3.8; CaF₂, 84.37; CaCO₃, 9.2; Fe₂O₃, 0.34; Al₂O₃, 1.28; MgO, 0.36; S, trace, moisture, 0.04; and L.O.I., 6.36 per cent. Synthetic leach

liquid was used and hydrated alumina and sodium carbonate were used for precipitation. Comprehensive work was conducted to determine optimum conditions of leaching and precipitation. The main reaction occurring between calcium fluoride, boric acid and sulphuric acid is given below: $2\text{CaF}_2 + \text{H}_3\text{BO}_3 + 2\text{H}_2\text{SO}_4 + \text{H}_2\text{O} = \text{HBF}_4 + 2\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$.

The leach solution was then filtered out; the residue washed with water and the total filtrate was evaporated to 500 c.c. The temperature of the solution was then raised to 90°C. Requisite amount of hydrated alumina was added and the solution digested for half-an-hour. Theoretical amount of sodium carbonate was then slowly added for a period of half an hour; the temperature was raised to 95°C. and the solution was digested for another half-an hour. The precipitated cryolite was allowed to settle and then filtered and washed. The reaction during precipitation is given below: $3\text{HBF}_4 + \text{Al}_2\text{O}_3 \cdot 3\text{H}_2\text{O} + 3\text{Na}_2\text{CO}_3 = 2\text{AlF}_3 \cdot 3\text{NaF} + 3\text{H}_3\text{BO}_3 + 3\text{CO}_2$. The precipitate was dried at 120°C. for 4 hours. Further cyclic studies of the process are actively under progress.

23.0 Spectrographic and Polarographic Studies

(i) *Spectrographic Analysis of Nickel-free Stainless Steel* — Work was taken up to develop a suitable rapid spectrographic method for conducting simultaneous analysis of high Cr and Mn contents in Ni-free austenitic stainless steel developed at the National Metallurgical Laboratory.

Spectrographic estimation of high percentage of elements present in sample often offer difficulty in getting accurate results. Ni-free stainless steel contain high Cr and Mn contents. Conventional chemical methods, though accurate, are time consuming. Efforts were, therefore, made to develop a method so that high Cr and Mn contents of Ni-free stainless steels and related alloys could rapidly be determined

with the same degree of accuracy if not more, as offered by the chemical methods. Experiments were undertaken to reduce the intensity of Cr and Mn lines by using neutral filters of various densities with large quartz spectrograph and finally filter of density D 0.99 (i.e. 10 per cent transmission of radiation) proved to be quite satisfactory to give the required analytical line pair of Cr and Mn suitable for microphotometer measurements with a considerable reduction of background also.

Porous cup sparking technique with sample in solution form was adopted. Synthetic standards of variable composition (Cr, 15-25; Mn, 3-15; Cu, 0.5-3.0 per cent) were prepared and a few exposures were taken under standardized conditions to study the intensity of the lines required for analytical purpose. The working curves for both Mn and Cr were plotted with data obtained from the above conditions. It was observed that Mn curve was quite smooth and linear without any variation whatsoever, but working curve for Cr above 20 per cent appeared to fluctuate. Further investigation to rectify such irregularities was taken up. This method gave scope for determination of Cu that may be present in this steel but in that case, other conditions being the same, the filter was not needed.

(ii) *Rapid Spectrographic Method to Analyse Slag* — In the manufacture of steel, considerable importance is attached to analytical control of the furnace slag. Sample taken at intervals throughout the process required analyses as quickly as possible. Conventional chemical methods are, however, time consuming. With a view to improve both the speed and accuracy of such analysis, attempt is being made to use spectrograph as a tool for analysing steel making and blast furnace slags. Use of untreated slag sample (sample as received without preliminary chemical treatment) has the advantage of speed but in view of possibilities of loss of accuracy in the result, due to the fact that slag structure considerably varies

and may have different excitation characteristics for similar composition, attempts were made to treat the slag samples chemically for uniformity of composition and accuracy of results.

To start with, basic open hearth steel slag was fused with a mixture of sodium carbonate and borax (all spec. pure) at about 1000°C . Finally, this mass was extracted with 25 per cent (V/V) HNO_3 and a few c.c. hydrogen peroxide solution. A very clear solution of the sample was thus obtained. Large quartz spectrograph was used. Few drops of the solution were dried on a graphite electrode. A.C. intermittent arc was used for excitation, taking graphite as counter electrode. For estimation of CaO and SiO_2 , neutral filter of density D 0.99 (i.e. 10 per cent transmission of radiation) was used. The prominent analytical lines of Si and Ca were obtained with a satisfactory reduction of intensity of the lines suitable to be measured by microphotometer. For the determination of Al_2O_3 , MnO and MgO , filter may not be used. Since, it was extremely difficult to prepare standards synthetically, where Si was to be included, actual slag with known composition (by accurate chemical analyses) was taken as the standard. Few samples of open hearth slag of different furnaces and with different composition were taken as the standards. Further work in respect of obtaining of a smooth and linear working curve, reproducibility of the method and accuracy, etc., was undertaken.

(iii) *Spectrographic Estimation of Phosphorus in Steel* — The objective of this study is to develop a method to spectrographically analyse phosphorus in steel, using Hilger Large Quartz Spectrograph. Spectro-chemical analysis of phosphorus in steel is troublesome in the near ultraviolet and visible regions. In order to overcome this trouble, new instruments like Fluorite Pdychromator-Vacuum Spectrograph were developed which avoided these difficulties by making use of the resonance lines of the elements in the far ultraviolet region. Attempts were made

to make use of the existing Hilger Large Quartz Spectrograph in estimating phosphorus in steel. In case of Large Quartz Spectrograph P 2136.2A line had to be selected for analysis. Cu 2135.98A is an interfering line with P 2136.2A line. This difficulty would not arise at all provided the steel was absolutely free from copper. A series of exposures were made with standard samples of various phosphorus and copper contents (up to 0.46 per cent Cu). Standard steel samples supplied by British Chemical Standards of known composition of phosphorus and copper were taken into solution and excited. Phosphorus lines up to 0.02 per cent phosphorus contents were well defined.

(iv) *Polarographic Work* — (a) Work was initiated on the estimation of Fe, Cu, Pb and Cd in pure zinc and zinc base die casting alloys, polarographically. A set of synthetic standards of variable composition of Pb, Cd, Fe and Cu were made so as to prepare the calibration curve. Several polarograms were taken under various conditions and finally standardized; results obtained were most satisfactory.

(b) Work was initiated on the estimation of Cu, Pb, Cd and Zn in concentrated zinc ore. 2.5 gm. of the finely powdered ore were dissolved in a mixture of HCl and HNO_3 to get a clear solution. In that 1 c.c. of H_2SO_4 was added and evaporated to almost dryness. This was then diluted with water and precipitate filtered off. Twenty c.c. of concentrated ammonia with a few c.c. of 0.1 per cent gelatine were added in this filtrate and a portion was polarographed after deaeration. Well-defined curves for copper and cadmium were obtained and the results obtained for Cu and Cd were satisfactory. But for Zn, this solution was found to be highly concentrated. The precipitate of PbSO_4 was dissolved in ammonium acetate solution and polarographed in that medium taking ammonium acetate as supporting electrolyte. Well-defined curves for Pb giving satisfactory results were thus obtained.

24.0 Effect of Hydrogen on the Physical Properties of Metals and Alloys & Mechanism of Phase Transformation in Alloys Deposited at Constant Cathode Potential

Under the Council of Scientific & Industrial Research Fellowship Scheme, work was started on two important fundamental problems relating to:

- (a) The effect of hydrogen on the physical properties of metals and alloys.
 - (b) Mechanism of phase transformation in alloys deposited at constant cathodic potential.
- (a) The mechanism of phase transformation in electrolytic alloys had been well established by earlier workers in this field. Depending on the conditions of deposition, the electrolytic metal at the cathode could crystallize in a lattice which was distorted or a completely new lattice can be formed when compared to its equilibrium phase. In addition to other factors, hydrogen during electrolysis was said to be responsible for such transformations. The work on the electrolytic composition of nickel carried out by earlier workers had shown that hydrogen content of the two allotropic forms of electrolytic nickel could vary considerably. But however, there had been no attempts for a quantitative explanation regarding the formation of various phase in electrodeposited metals nor the factors responsible for their existence had been fully explained. The

present work was undertaken with a view to study the mechanism and condition of deposition that would determine the nature of phase or phases with special reference to the role of occluded hydrogen in the electro-deposition of iron and nickel.

Electrolytic Deposition of Iron and Nickel —

In the electrolytic deposition of iron, the bath contained only pure ferrous-ammonium sulphate (B.D.H. analar quality and the pH being adjusted by pure sulphuric acid). The hydrogen content of the electrodeposited iron was determined by vacuum extraction method. Table 13 gives the structure and results obtained of electrodeposited iron. Table 14 gives the results of electrodeposition of nickel.

X-ray photograph obtained with electro-deposition of iron or nickel showed no evidence whatsoever for the existence of a phase other than the equilibrium one. Further work is in progress.

(b) Electrolytic alloys differed considerably in their physico-chemical properties from the alloys produced thermally. Elucidation of the mechanism of alloy formation at the cathode could throw some light on the properties of these alloys, vis-à-vis the electrochemical conditions existing during their production. Work in this direction could, therefore, provide a basis on which the field of applications of electrodeposited alloys could be expanded.

Hume-Rothery put forward the hypothesis that where the atomic diameters of the

TABLE 13 — ELECTRODEPOSITION OF IRON

Bath composition: ferrous ammonium sulphate = 0.893 M; pH = 3.2; Temp. = 30°C.

EXPT. No.	CATHODE POTENTIAL in volt	CURRENT Amp./0.37 dm ² .	% CURRENT EFFICIENCY	H ₂ IN C.C. per 100 gm.	X-RAY
1	-0.8295	0.985			
2	-0.7848	0.380	85.0	670.1	B.C.C.
3	-0.7448	0.177	84.1	798.7	B.C.C.
4	-0.7178	0.079	80.7	871.0	B.C.C.
5	-0.5555	0.040	81.5	867.8	B.C.C.
			—	—	B.C.C.

TABLE 14 — ELECTRODEPOSITION OF NICKEL

Bath composition: nickel chloride = M; boric acid = 30 gm./l.; pH = 2.5; temp. = 28°C.

EXPT. No.	CATHODE POTENTIAL in volt	CURRENT amp./0.37 dm. ²	% CURRENT EFFICIENCY	THICKNESS OF THE DEPOSIT	X-RAY
1	-0.5993	1.170	97	126.00 × 10 ⁻⁵ cm.	F.C.C.
2	-0.5770	0.770	98	83.80 × 10 ⁻⁵ cm.	F.C.C.
3	-0.5350	0.375	99	41.30 × 10 ⁻⁵ cm.	F.C.C.
4	-0.5993	1.150	97	40.97 × 10 ⁻⁵ cm.	F.C.C.
5	-0.5770	0.755	99	20.65 × 10 ⁻⁵ cm.	F.C.C.
6	-0.5350	0.375	97	10.02 × 10 ⁻⁵ cm.	F.C.C.
7	—	0.370	100	2.05 × 10 ⁻⁵ cm.	F.C.C.
8	—	0.370	100	0.34 × 10 ⁻⁵ cm.	F.C.C.

solvent and solute differ by more than 14-15 per cent of that of the solvent, the size factor was unfavourable and the solid solution was thereby very restricted. Work was, therefore, started on a system of this type (Ag-Cu alloys), keeping in view the possibility of the existence of metastable phase in electrodeposited alloys and their subsequent transformation on ageing/annealing to thermally stable structure as observed by Banerjee and Allmand.

Phase-transformation in Electrolytic Silver-copper Alloys — This system was systematically investigated and a research paper entitled "Phase-transformation in Electrolytic Alloys with Special Reference to Copper-zinc and silver-copper" was prepared.

Phase-transformation in Electrolytic Copper-Cadmium System — The results obtained on this system corroborated the earlier theory on the subject. Systematic work was carried out on the thermal production of the pure intermediate phase of copper cadmium alloys to be used as a reference for further study of electrolytic copper-cadmium system. Attempts were made to interpret the results in terms of the correlation of the electrochemical parameters such as overvoltage, current efficiency and hydrogen diffusion, etc., with the work of formation of the new phase nuclei at the cathode. The work further established that gamma phase was never a primary product and was formed from other metastable phases by further changes.

Electrodeposition of Nickel-Zinc Alloys — Work was undertaken on the study of the mechanism of phase transformation in electrolytic Ni-Zn alloys deposited at constant cathode potential. About fifteen alloys were deposited containing nickel contents from 85.06 to 31.1 per cent, from different baths holding suitable proportions of nickel sulphate, zinc sulphate and boric acid. No addition agents other than boric acid were tried. The deposits obtained were annealed in vacuum sealed pyrex glass tubes at 300°C. for 47 hours. The X-ray photographs of the as-deposited alloy and after annealing were obtained with the Debye-Scherrer 9 cm. powder camera.

25.0 Beneficiation and Reducibility Studies of Tisco Iron Ores

Studies were initially undertaken on the beneficiation of five samples of iron ores from Noamundi (I & II), Joda, Gorumahisani and Badampahar (amounting to 80 tons) which normally form the ore burden of the blast furnaces at the Tata Iron & Steel Co. A detailed examination of the ore samples was conducted which included petrological studies, chemical analysis, crushing and screening characteristics. The samples were subjected to washing and heavy media separation method of concentration. Sintering study was also conducted on the natural fines obtained from the ore samples after crushing

as well as on the $-\frac{3}{8}$ in. materials obtained after washing the ore.

Ore from Joda was the hardest to crush, followed in order, by Noamundi II, Noamundi I, Gorumahisani and Badampahar. Prescreening of the ores as received at -2 in. size was found to be necessary to decrease the load on crushers. Roll-grizzlies are to be preferred to vibrating screens for this purpose due to the wet and sticky nature of the ores. Screenability tests indicated that maximum screening difficulties were encountered even with as little as 5 per cent moisture in case of ores from Joda and Noamundi II, with 10 per cent in case of ores from Noamundi I and Badampahar and with 15 per cent in case of Gorumahisani ore. Drying of wet ore may solve the problem of screening to some extent but the fines would tend to ball up during drying to form hard lumps. Some of these may break up during screening but others will be carried to the blast furnace. Also the problem of 'adhered fines' would still be there. Washing of the ore (wet screening preceded by scrubbing), however, can substantially assist in the problem of handling of wet and sticky ores as indicated by the results obtained. A clean, sized and washed product free from 'adhered fines' for direct charging to the blast furnace and a properly classified product for sintering were obtained by washing. Use of sized ore and sinter in the blast furnace would reduce the metallic loss in flue dust and ensure smoother blast furnace operation. There was a preferential removal of silica over alumina when washing alone was employed, whereas heavy media separation of washed ore resulted in preferential rejection of alumina over silica. Results of washing followed by heavy media separation at -2 in. size indicated the improvement in the iron values and the reduction of silica and alumina content in the final concentrates. The loss of iron values were due to rejection of slime as well as that of float product obtained after washing and heavy media separation. Although the alumina-silica ratio

had not improved, there were substantial rejections of alumina and silica in the final concentrate. The extent of alumina rejection was such that the final alumina in the slag would fall within favourable limits due to lower alumina-silica ratio in the coke ash. Should, however, the alumina content increase beyond optimum levels, additions of quartzite or banded hematite-quartz or use of high silica flux could be resorted to, while obtaining full advantages accruing through washing and heavy media separation. The $\frac{3}{8}$ in. classifier product containing only 3-5 per cent of -200 mesh material was found to be free-flowing and as such, it should not be very difficult to handle this product in the sinter plant. Should any difficulty be experienced, this could be solved by proper design of bunkers and employing knockers. Sintering studies showed that good quality sinter could be produced from $-\frac{3}{8}$ in. classified product obtained after washing.

Investigations thus indicated that scrubbing of these ores followed by wet screening and classification would solve their ore-handling and screening problems and would yield a clean product for sintering. Rejection of 10-12 per cent by weight of slime at -2 in. size did not improve the grade of the ore by more than 1-2 per cent Fe. On the other hand, heavy media separation of the washed ore was found to eliminate practically all the laterite and shaly gangue material which constituted a large bulk of the alumina in the ore, and thus substantially improve the grade of the ore by 2-3 units. The beneficiated ore was very low in total insolubles and would be more desirable and acceptable feed for smooth blast furnace operation. The above samples were representative of the run-of-mine ore from the various mines.

Subsequent studies were undertaken to see whether or not various types of ores, which are encountered during their mechanical open-cut mining operations, are equally amenable to the beneficiation treatment schedules recommended for general run-of-

mine ore from their various mines. Eight samples, viz. four from Noamundi, two from Joda and two from Noamundi Washing Plant were received for washing and heavy media separation tests. The washing and heavy media separation tests performed on six samples of iron ore from Noamundi and Joda showed that washing would solve the problem of handling and screening of wet and sticky ores and would thus produce a clean and sized product free from adhered fines and also a properly classified $-\frac{3}{8}$ in. material most suitable for sintering. Washing by itself, however, might not yield a product with an optimum desirable alumina to silica ratio for efficient blast furnace operation. Heavy media separation process worked satisfactorily with all the ores investigated irrespective of the position whether the gangue was laterite or shale. Laterite could be rejected effectively at 3.0 sp. gr., whereas shale needed 3.1. It was found to reduce the total insoluble substantially and thus considerably improve the grade of the final product. In spite of an unfavourable alumina to silica ratio, the total insoluble contents in the final concentrates were low enough for smooth blast furnace operations. Should there exist the need to further reduce the alumina/silica ratio in the blast furnace burden, requisite additions of high silica flux could be considered, as followed at several iron and steel plants. Washing alone was found not to substantially improve the grade of iron ores investigated, but would very much facilitate their screenability and handling. Heavy media separation, on the other hand, rejected a large bulk of laterite and shaly material and thereby substantially reduced the insoluble contents in the final products and improved their grades. The beneficiation tests employing heavy media separation, jigging, Humphrey's Spiral, magnetic separation and electrostatic separation made with two samples, viz. a $-\frac{3}{8}$ in. and $+\frac{1}{8}$ in. washed product and the other a $-\frac{1}{8}$ in. classifier sand from the Noamundi Washing Plant

indicated that the washed product as well as the classifier sand could be successfully beneficiated to produce a suitable material for sinter plant, but recoveries would be somewhat low, viz. about 64 per cent in the concentrate from the washed product and about 57 per cent in the case of the classifier sand.

Studies on the reducibility of beneficiated iron ores from Joda, Badampahar, Gorumahisani and Noamundi were also carried out by passing hydrogen at the rate of 1000 ml. per minute at 800°C. on cubical samples of 1 cm. edge from -2 in. and $+1$ in. beneficiated fraction, and 25 gm. each of $-1+\frac{3}{8}$ and $\frac{3}{8}$ in. washed fractions. The results thus indicated that Joda and Noamundi II iron ore deposits possess lower reducibility characteristics than Badampahar, Gorumahisani and Noamundi I grade ores in each size gradings. The reducibility characteristics of iron ores from Noamundi I, Badampahar and Gorumahisani are more or less similar.

A detailed investigation report in three parts was furnished to Messrs Tata Iron & Steel Co. Ltd. The Chairman of the Board of Directors, Tata Industries, stated at the annual Board meeting, "In this connection, I wish to record our appreciation of the assistance we have received from the National Metallurgical Laboratory at Jamshedpur, whose investigations, undertaken at our request, have formed, to some extent, the basis of our ore beneficiation programme."

26.0 Beneficiation Studies on a Complex Low-grade Copper-Lead-Zinc Ore from Bhotang Mines, Rangpo, Sikkim

A sample of copper-lead-zinc ore received from the Indian Bureau of Mines at the instance of the Department of Mines and Fuel, for beneficiation tests, assayed Cu, 0.67; Pb, 1.48; Zn, 4.50; Fe, 15.22; S, 8.69; SiO_2 , 58.20; Al_2O_3 , 6.70 per cent and Ag, 2.1 oz./ton with traces of Co, Cd, Ni and Au. Chalcopyrite, galena, sphalerite and pyrrhotite were

the chief sulphide minerals, whereas quartz and chlorite constituted the bulk of the gangue. Though the copper, lead and zinc minerals were fairly free from the siliceous gangue at about 48 mesh size, these were in intimate association with each other as well as with pyrrhotite, thereby necessitating very fine grinding to about 400 mesh for their liberation. Bulk flotation of the copper-lead minerals followed by repeated cleanings of the rougher copper-lead floats after grinding to -325 mesh yielded good copper and lead refloat concentrate assaying Cu, 27.55 and Pb, 53.58 per cent with estimated recoveries of 75 per cent copper and 60 per cent lead respectively. Silver constituted an important byproduct in the copper and lead concentrates assaying 14 and 31.0 oz./ton respectively. Activation of sphalerite from the copper-lead rougher tailing, followed by grinding to -325 mesh size and subjecting the same to repeated cleanings yielded a zinc refloat concentrate assaying Zn, 51.2 per cent with an estimated recovery of 76.0 per cent zinc. Heavy media separation of the original -1 and +6 mesh ore at a separating density of 2.8, rejected 43.7 per cent by weight of the barren rock with a loss of only 5.9 per cent copper, 2.8 per cent lead and 3.1 per cent zinc in it. Flotation performed with the mixed zink product and the -6 mesh untreated fraction is expected to yield results similar to those obtained by straight flotation. A definite advantage in adopting the heavy media separation/flotation process is that crushing and grinding cost would thereby be considerably lower, as more than 43 per cent by weight of barren rock could be eliminated at a relatively coarse size. A report embodying the detailed recommendation on the basis of work carried out on the samples and specification of machinery and equipment with estimated cost for a Milling Plant to be installed at Bhotang mines, Sikkim, of capacity to treat hundred tons of run-of-mine ore per day, was furnished to the Ministry of Steel, Mines and Fuel, Government of India, who stated

that "... The results indicated by the test in your Laboratory are certainly comparable to some of the best achieved in other parts of the world and perhaps cannot be bettered except in conditions of commercial scale processing . . ."

27.0 Beneficiation of Low-grade Manganese Ores

(i) *From Lohandabud Group of Mines, Jharsuguda, Orissa* — Three samples of low-grade manganese ore (marked La, Lc and Ld) were received from Messrs Shivji Nathubhai of Jharsuguda, Orissa, for beneficiation studies. The mixed sample assayed Mn, 40.03; MnO₂, 34.71; Fe, 4.82; SiO₂, 19.34; Al₂O₃, 3.8; CaO, 1.1; BaO, 3.32; MgO, 0.60 and P, 0.18 per cent. Braunite was the principal manganese mineral followed by psilomelane, hausmanite, pyrolusite, jacobite, hollandite, sitaparite and vredenburtite while quartz, garnet and pyroxenes constituted the chief gangue followed by amphiboles, barytes, biotite, feldspars and hematite. Electrostatic separation of the magnetic product, obtained from high intensity magnetic separation after hydroclassification of the -35 mesh ground mixed sample, gave a concentrate assaying Mn, 45.2 and SiO₂, 9.7 per cent with a recovery of Mn, 63.4 per cent. Cationic flotation using Armeen-12D as collector for siliceous gangue at a pH range of 8-8.5 produced a manganese concentrate assaying Mn, 44.8 and SiO₂, 11.0 per cent with a recovery of Mn, 60.0 per cent. Due to the very complex nature of the mixed sample, its beneficiation to the desired extent could not be achieved. However, the sample Ld alone, when subjected to high intensity magnetic separation, yielded a concentrate assaying Mn, 46.2 and SiO₂, 7.7 per cent with a recovery of Mn, 85.8 per cent indicating that this sample could be profitably beneficiated alone, without mixing with the other samples.

(ii) *Briquetting and Sintering Studies of Manganese Concentrates from Low-grade Ore*

from *Siljora-Kalimati Mines, Orissa* — The project was taken up on behalf of Messrs Rungta & Sons, Chaibasa, for preparing manganese concentrates from low-grade manganese deposits of their mines and for conducting studies on their briquetting and sintering characteristics so as to make them suitable for charging directly in the furnace for the production of ferro-manganese.

About 5 tons of low-grade manganese ore were crushed to $-1\frac{1}{2}$ in. size and subjected to reduction roast treatment in a vertical reduction furnace for preparing manganese concentrate for briquetting and sintering studies. Dry magnetic separation, at -4 mesh, of about $3\frac{1}{2}$ tons of the reduced ore was carried out. Wet magnetic separation of the magnetic product obtained from the above test, after grinding to about 65 mesh, yielded a combined manganese concentrate assaying Mn, 49.5 and Fe, 7.9 per cent, with a manganese recovery of 69.4 per cent.

Laboratory studies were made on the sintering characteristics of the manganese concentrate. The effects of variation, such as coke, moisture and sinter fines in sinter mixture on the sintering time and on the quality of sinter produced were studied. Research results showed that the optimum amounts of coke and water contents were 6.0 and 10.0 per cent respectively for producing a sinter with good size stability. The final sinter assayed Mn, 53.46; Fe, 8.13; SiO_2 , 3.42; Al_2O_3 , 10.19 and P, 0.055 per cent.

Physical tests were carried out in the briquette-testing machine in the temperature range $450^\circ\text{--}525^\circ\text{C}$. using maximum load of 10 kg./sq. cm. The briquettes were prepared using various combinations of binders, viz. 10 per cent pitch alone; 6 per cent sulphite lye and 4 per cent pitch; 5 per cent sulphite lye and 4 per cent pitch; 4 per cent sulphite lye and 4 per cent pitch; 3 per cent tar and 4 per cent sulphite lye; 3 per cent tar and 3 per cent sulphite lye. The briquettes stood the tests in all cases, indicating good strength at this temperature. Tests at normal, as well as at elevated temperatures

with briquettes made of 2.5 per cent cement alone; 4 per cent cement and 6 per cent lime; and 3 per cent cement and 7 per cent lime also gave satisfactory results. A few tests performed with briquettes made of 1 per cent cement and 5.9 per cent lime gave green compression strengths ranging between 1700 and 1890 lb./sq. in. Based on these small-scale briquetting tests, arrangements are being made for large-scale tests.

(iii) *Reduction of Iron Content in a Sample of Manganese Ore* — A sample of manganese ore was received from Messrs National Carbon Co., Calcutta, to investigate the possibility of reducing its iron content by physical methods so that it could be employed in dry cell industry. The sample assayed Mn, 52.09; MnO_2 , 74.50; Fe, 5.9; Al_2O_3 , 2.3 and SiO_2 , 2.8 per cent. Petrological examination of the sample revealed that pyrolusite was the principal manganese mineral followed by small amounts of wad, psilomelane and manganite. Hematite, magnetite and goethite constituted the ferruginous gangue and the siliceous gangue consisted mainly of muscovite and quartz. Straight wet magnetic separation of the original sample (mostly 6 mesh) as well as after grinding to -35 mesh size yielded concentrates assaying Mn, 53.6 and Fe, 4.6; and Mn, 53.7 and Fe, 4.5 per cent with recoveries of 88.5 and 94.2 per cent Mn respectively. Dry magnetic separation after desliming of -35 mesh ground sample produced a concentrate assaying Mn, 54.9 and Fe, 3.7 per cent with a recovery of only 61.4 per cent Mn. Tabling followed by magnetic separation of the table concentrate as well as slime yielded the best results in a combined non-magnetic manganese concentrate assaying Mn, 54.55 and Fe, 3.6 per cent with a recovery of 78.2 per cent Mn.

(iv) *From Koira, Orissa* — Two types of low-grade manganese ores, one ferruginous and the other siliceous, were received from Messrs Orissa Manganese & Minerals Ltd., Koira, Orissa, to study the possibility of their upgrading in order to obtain concen-

trates suitable for the production of standard grade ferro-manganese or for use in the manufacture of dry cells.

A high grade manganese concentrate having a Mn to Fe ratio of 7:1 with a recovery of about 67 per cent Mn could be produced from the low-grade ferruginous manganese ore from Koira, assaying Mn, 31.48; Fe, 22.06; SiO_2 , 3.9; Al_2O_3 , 5.5; P, 0.09 and BaO, 0.52 per cent. About 2.8 tons of run-of-mine ore would be required to produce one ton of concentrate. The process is based on low-temperature gaseous reduction followed by magnetic separation. The process needs no external heating except to initiate the reduction process. A siliceous manganese ore from Orahuri mine, Koira, assaying Mn, 36.7; SiO_2 , 31.05; Fe, 2.52 and BaO 0.5 per cent could be beneficiated to Mn, 52.3; MnO_2 , 79.8; Fe, 3.45 and insolubles 4.15 per cent with a recovery of 80.6 per cent Mn by high intensity magnetic separation after desliming. Based on the experimental results, a project report is being prepared for setting up of beneficiation plants for treatment of the ferruginous and siliceous manganese ores from Koira.

28.0 Beneficiation of an Amorphous Gypsum Sample from Rajasthan

A sample of gypsum designated as amorphous gypsum, analysing 37.71 per cent SO_3 (equivalent to 81.08 per cent gypsum CaSO_4) was received from Messrs Bikaner Gypsums Ltd., Rajasthan, for beneficiation studies. The sample contained quartz and calcite as the principal gangue followed by minor amounts of mica, hornblende, chlorite, tourmaline, garnet, feldspar, halite and clay minerals. When the original sample was crushed to -10 mesh and screened through 150 mesh, it gave a +150 mesh fraction of purity 87.0 per cent gypsum and 5.79 per cent insolubles, with a recovery of 88.5 per cent gypsum. This product is suitable for use in the manufacture of ammonium sulphate

fertilizer. The -150 mesh rejects, when subjected to flotation, yielded a concentrate of grade 96.4 per cent gypsum and 0.54 per cent insolubles, with a gypsum recovery in the product of 87.3 per cent. This concentrate can be used in the manufacture of plaster of Paris.

29.0 Beneficiation and Briquetting Studies of Fluorspar from Thurwahi, Rajasthan

A sample of fluorspar received from the Director of Mines & Geology, Rajasthan, for beneficiation assayed CaF_2 , 22.5; SiO_2 , 64.65; CaCO_3 , 1.75; Al_2O_3 , 4.3; Fe_2O_3 , 2.7; Pb, 0.04 and S, 0.20 per cent with traces of Zn, Ba, Cu and W. Feldspar was the chief gangue with minor amounts of quartz and calcite. Fluorspar was fairly liberated from the gangue at about 100 mesh size. Flotation employing sodium oleate-oleic acid emulsion as collector and sodium silicate as gangue depressant at 9.5 pH, followed by two cleanings of the rougher float could yield a refloat concentrate of metallurgical grade assaying CaF_2 , 85.1; SiO_2 , 6.9; CaCO_3 , 3.8; Pb, 0.03 and S, 0.23 per cent with a recovery of 88.3 per cent CaF_2 . Better grades of concentrate could be obtained by more cleaning operations.

Briquetting studies on fluorspar concentrate from low-grade ore were also conducted. About 200 lb. of a sample assaying 22.5 per cent CaF_2 were ground to about 65 mesh. The ground material was subjected to flotation tests using oleic acid and sodium oleate as collectors to yield a concentrate assaying 89.4 per cent CaF_2 with a recovery of 91.0 per cent CaF_2 in the product. Batch briquetting tests were performed using different binders such as lime, bentonite, starch, cement, sulphite lye, etc. Briquettes made with 0.5, 1, 1.5 and 2 per cent starch, keeping bentonite percentage constant at 2 per cent, gave green compression strengths of 560, 828, 1082 and 892 lb./sq. in. respectively. Briquettes prepared with varying percentages

of bentonite ranging from 0.5 to 5.0 keeping starch at 0.5 per cent gave green compression strengths varying from 370 to 676 lb./sq. in. Briquettes made, using 2.5 per cent of lime keeping cement constant at 3 per cent, gave green compression strengths between 260 and 700 lb./sq. in. Use of sodium silicate with lime did not improve the results. Tests performed with 5, 4, 3, 2 and 1 per cent of sulphite lye as binder gave green compression strengths of 1750, 1275, 891, 746 and 637 lb./sq. in. respectively. Based on the results of the batch tests, about 5 tons of the sample were ground to about 100 mesh in the ball mill. Primary flotation of the ground material using oleic acid as collector for fluorspar followed by reflation of the primary concentrate employing "tannin" as depressant for calcite were completed. The final concentrate is being sampled for analysis as well as large-scale briquetting studies.

30.0 Reduction of Iron Oxide Content in a Bauxite Sample from Roadside Mine, Jabalpur District, Madhya Pradesh

A bauxite sample was received from Messrs Associated Cement Companies Ltd. for the reduction of Fe_2O_3 content to preferably less than 2.0 per cent in the raw ore equivalent to 2.8 per cent in the calcined ore. The sample assayed Al_2O_3 , 53.3; Fe_2O_3 , 5.2 and TiO_2 , 8.0 per cent. The principal gangue minerals in the sample were anatase, goethite and ilmenite with minor amounts of quartz and tourmaline. Magnetic separation of the sample at -10 mesh after desliming yielded a non-magnetic product assaying Fe_2O_3 , 3.0; TiO_2 , 7.1 and Al_2O_3 , 56.76 per cent, with an alumina recovery of 75.5 per cent. Rejection of iron oxide (Fe_2O_3) was 28.7 per cent and 26.9 per cent in the magnetic fraction and slime respectively. Reduction roast followed by magnetic separation of the sample at -3 mesh produced a non-magnetic product, assaying Fe_2O_3 , 3.47; TiO_2 , 10.5 and

Al_2O_3 , 72.5 per cent with a recovery of 79.2 per cent Al_2O_3 with a total rejection of 57.6 per cent Fe_2O_3 in the magnetic fraction. Reduction roast followed by magnetic separation of a calcined sample, assaying Al_2O_3 , 75.6; Fe_2O_3 , 6.0 and TiO_2 , 10.0 per cent gave a non-magnetic product, assaying Fe_2O_3 , 4.38; TiO_2 , 10.6 and Al_2O_3 , 77.6 per cent with a recovery of 84.1 per cent Al_2O_3 .

31.0 Screen Analysis and Chemical Analysis of Iron Ore Sample from Kiriburu

Two iron ore samples were received from the National Mineral Development Corporation, Kiriburu Project, Orissa, for screen and chemical analysis. The lump ore was of a very good grade with only 1.8 per cent total insolubles, whereas the sample of fines assaying 63.3 per cent Fe contained 4.09 per cent insolubles. The fine fractions of the latter were found gradually increasing in insoluble contents, indicating that employing suitable beneficiation technique it should be possible to partially eliminate the insolubles and improve the grade of the $-\frac{1}{2}$ in. sample.

32.0 Beneficiation of Low-grade Graphite from Doomara and Lotiyana Villages, Ajmer, Rajasthan

(i) *From Doomara* — A low-grade graphite sample was received from the Director of Mines & Geology, Rajasthan, for beneficiation tests. As received, the sample assayed C, 8.19; ash, 84.37; volatile matter, 4.92; and moisture 2.52 per cent. The gangue minerals present in the sample were mica, quartz and tourmaline with traces of calcite. Graphite was found to be intimately associated with mica even at -200 mesh and, therefore, a high grade of graphite concentrate could not be obtained by flotation methods. A concentrate obtained after one reflation assayed 56.65 per cent ash, indicating that it

was not possible to produce even a foundry grade graphite from the sample.

(ii) *From Lotiyana* — A low-grade sample was received from the Director of Mines & Geology, Rajasthan, for beneficiation tests. As received, the sample assayed C, 2.17; ash, 84.03; volatile matter, 13.52; and moisture 0.28 per cent. The sample was of a lower grade than the one from Doomara and in this also graphite was very intimately associated with mica even at —200 mesh. It was not possible to produce even a low-grade concentrate for foundry purposes.

33.0 Beneficiation of Low-grade Chromite

(i) *From Kankauli Deposits, Ratnagiri District, Maharashtra* — A low-grade chromite sample was received from the Geological Survey of India, Calcutta, for beneficiation tests. As received, the sample assayed Cr_2O_3 , 34.72; FeO, 21.5; Al_2O_3 , 14.74; SiO_2 , 9.6; MgO, 17.21; CaO, 2.0 and soluble iron, 0.76 per cent. Chlorite, talc, serpentine and orthopyroxenes were the gangue minerals present in the sample with traces of hematite. Chromite was fairly liberated at about 65 mesh size. Tabling at —48 mesh size produced concentrates assaying Cr_2O_3 , 49.61 and FeO, 28.72 per cent (Cr/Fe ratio 1.52 only) with a recovery of 91.2 per cent Cr_2O_3 . Iron content of the sample could not be appreciably lowered by magnetic separation due to its chemical combination with chromite mineral itself, indicating that a metallurgical grade of concentrate could not be produced from this sample by established ore dressing methods. Concentrates suitable for refractory and chemical industries could, however, be produced employing simple gravity methods of concentration.

(ii) *From Vagda Deposits, Maharashtra* — A low-grade sample of chromite was received from the Geological Survey of India, Calcutta, for beneficiation studies to make it suitable for metallurgical purposes. The

sample as received assayed Cr_2O_3 , 33.14; FeO, 21.51; soluble Fe, 1.03; MgO, 14.5; SiO_2 , 14.74; Al_2O_3 , 11.0 and CaO, 2.24 per cent. The predominant gangue mineral in the sample was olivine followed by chlorite-serpentine group of minerals, talc, muscovite, and traces of hypersthene, magnetite and ilmenite; chromite was fairly liberated at about 65 mesh. Tabling after hydro-classification of the sample at —35, —48 and —65 mesh sizes yielded concentrates assaying Cr_2O_3 , 43.5; FeO, 25.8; Cr_2O_3 , 46.4; FeO, 26.4; and Cr_2O_3 , 47.4; FeO, 26.2 per cent with recoveries of 66.4, 78.2 and 75.2 per cent Cr_2O_3 respectively. Magnetic separation of the sample at different sizes to eliminate iron did not produce a concentrate of satisfactory grade. Flotation using anionic and cationic collectors also did not yield satisfactory results. Iron being chemically combined with chromite mineral, a concentrate with a Cr/Fe ratio of 3:1 suitable for metallurgical purposes could not be produced by physical ore-dressing methods. This concentrate is suitable, however, for refractory purposes.

34.0 Beneficiation of Low-grade Garnet from Guda, Ajmer

Five different varieties of low-grade garnet samples were received from Assistant Mining Engineer, Government of Rajasthan, for beneficiation tests with a view to make them suitable for abrasives. As the individual samples represented small reserves, they were mixed for experimental work. The mixed garnet sample contained 51.0 per cent garnet. Petrological examination of the mixed sample showed that the gangue minerals consisted of quartz, epidote, muscovite, biotite, chlorite, and inclusions of an opaque mineral. Magnetic separation of —20 mesh ground sample yielded a magnetic concentrate of purity 60.3 per cent garnet with a recovery of 89.7 per cent garnet. Tabling, followed by dry magnetic separation of the table concentrates at —65 mesh and —100

- mesh sizes, yielded concentrates of purity 79.5 and 86.3 per cent garnet respectively, indicating that high grade concentrates cannot be produced from the sample.

35.0 Reduction of Fe_2O_3 Content in a Rutile Sample from Quilon, Travancore

A sample of rutile assaying TiO_2 , 95.06; Fe_2O_3 , 1.5 and SiO_2 , 3.7 per cent was received from Messrs Travancore Minerals Ltd., Quilon, Travancore, at the instance of Indian Bureau of Mines, for further reduction of Fe_2O_3 content. High intensity magnetic separation of the sample after sizing yielded a rutile concentrate (non-magnetic) assaying Fe_2O_3 , 0.95 and TiO_2 , 95.1 per cent with a recovery of 98.3 per cent TiO_2 . Magnetic separation, followed by electrostatic separation of the non-magnetic product, had been completed.

36.0 Beneficiation Studies on Limestone Samples from Purnapani Quarries, Sundergarh, Dt., Orissa

Four limestone samples (Nos. 1, 2, 3 and 4) were received from the Hindustan Steel Ltd. for beneficiation tests to make them suitable for metallurgical purposes by elimination of silica. Silica in the above samples ranged between 10 and 22 per cent.

(i) *Sample No. 1* — The sample, as received, assayed CaO, 46.0; CO_2 , 39.6; MgO, 4.8; SiO_2 , 7.0; Al_2O_3 , 1.7; Fe_2O_3 , 0.65 and SO_3 , 0.25 per cent. Petrological examination of the sample showed that carbonates present were both calcite and dolomite, the former being predominant. The gangue minerals in order of abundance were quartz, chlorite, muscovite, feldspar and pyrite. The gangue minerals were liberated at about 150 mesh. Flotation tests using oleic acid emulsion as collector for limestone and sodium silicate as depressant for siliceous gangue yielded concentrates assaying 49.7-51.3

per cent CaO with recoveries ranging from 98 to 99.3 per cent CaO. A feed with a fineness of 66.7 per cent —200 mesh which gave a concentrate assaying 49.7 per cent CaO and 3.3 per cent insolubles with a recovery of 99.3 per cent CaO was chosen for further studies.

(ii) *Sample No. 2* — The sample, as received, assayed CaO, 43.8; CO_2 , 36.9; MgO, 4.2; SiO_2 , 11.4; Al_2O_3 , 2.8; Fe_2O_3 , 0.99 and SO_3 , 0.29 per cent. Grindability tests indicated that a grind with 81 per cent —200 mesh feed was optimum for flotation. Flotation concentrate obtained with this grind assayed CaO, 49.67 and insolubles, 4.3 per cent, with a recovery of 97.3 per cent CaO.

(iii) *Sample No. 3* — The sample, as received, assayed CaO, 41.7; MgO, 4.0; CO_2 , 37.0; SiO_2 , 13.7; Al_2O_3 , 2.2; SO_3 , 0.26 and Fe_2O_3 , 1.0 per cent. Flotation tests with feeds, containing 87.4 and 75.9 per cent —200 mesh by weight yielded concentrates assaying 49.7; and insolubles, 4.2; and CaO, 49.8 and insolubles, 3.3 per cent with respective CaO recoveries of 93.6 and 95.0 per cent. Further work is in progress.

37.0 Flotation of Ferruginous Manganese Ores

Under the Council of Scientific & Industrial Research Fellowship Scheme, work was initiated on the separation of manganese minerals from the iron minerals by flotation. Because both the minerals behave similarly to the ordinary flotation reagents, the first step in this research scheme is to collect various manganese minerals and the commonly associated gangue minerals and to purify them. Afterwards, these minerals are to be mixed in various proportion, and their flotation behaviour is to be studied under different conditions.

A modified type of the Hallimond Tube was fabricated for flotation experiments. The advantages of this apparatus were: (i) samples of 2 or 3 gm. could be floated;

(ii) operating variables could be closely controlled; and (iii) during flotation, neither the reagent concentration nor the pulp density changed.

As part of the research scheme, technical information pertaining to the subject was collected. Purified samples of goethite, hematite, pyrolusite, garnet, etc., were prepared and the minerals psilomelane, limonite, jacobsonite, etc., are yet to be collected and purified.

38.0 Studies on the Morphology of Phosphorus in Indian Manganese Ores

Under the Council of Scientific & Industrial Research Fellowship Scheme work was initiated on morphology of phosphorus in Indian manganese ores.

Very little information is available about the forms in which phosphorus is present in Indian manganese ores. Manganese ores contain varying amounts of phosphorus, as a separate mineral or as a complex compound of manganese, iron, etc.

A systematic study was undertaken on the high phosphorus Indian manganese ores, with a view to identify the nature of phosphorus in them which will throw light on the possible methods of separation for production of manganese concentrates suitable for standard ferro-manganese.

The problem was taken up on the basis of the following

- (i) Collection of literature and information
- (ii) Collection of manganese ore samples from various parts of India, which are high in phosphorus.
- (iii) Laboratory study to include the following aspects:
 - (a) Ore-microscopy and petrological studies
 - (b) Chemical analysis
 - (c) Contact print photographic study
 - (d) X-ray study
 - (e) Ore-dressing studies.

Samples from Rajasthan, Maharashtra (Nagpur and Bhandara districts) and Madhya Pradesh (Balaghat district) were collected for studies. Samples from Orissa and Andhra Pradesh are also being collected.

39.0 Production of Basic Refractories from Indigenous Resources

(i) *Studies on Almorah Magnesite* — Magnesite bricks are indispensable in lining the basic open-hearth furnaces. The most well-known magnesite deposit in India occurs at Salem in Madras. Recently new deposits have been located in Almorah district of U.P. Laboratory scale study conducted previously on these samples indicated that this magnesite, similar to the Austrian type, could more advantageously be used for the production of magnesite bricks than that of the deposits at Salem. A two-ton sample of Almorah magnesite was crushed to —5 mesh and fired between 1450° and 1500°C. It showed signs of hydration after a few weeks. It was re-fired at 1600°C. From the grog thus obtained, test specimens were made and properties such as refractoriness under load, porosity, firing shrinkage, etc., were determined. In another trial of calcination, a two-ton sample was calcined at 1650°C. for over 6 hours. The properties of this grog and specimens made therefrom were examined. Preliminary tests indicated a high bulk density, compared to the grog fired at 1600°C. A detailed study was made of the distribution of dolomite in the magnesite bulk sample by means of differential thermal analysis. Only traces of calcite were observed. The possibilities of separating the dolomite from magnesite was studied, especially with reference to the calcination and crushing characteristics. The densification of magnesite of different size fractions at different firing temperatures was studied in detail. The sintering, densification, grain growth and recrystallization were studied.

(ii) *Chrome - magnesite Refractories* — In view of the expansion of the Indian Iron and

Steel Industry during the Second and Third Five Year Plan periods, the demand for basic refractories using chrome ore is expected to steadily increase. Recent trends in steel making and in refractory practice in all basic open-hearth furnaces with chrome-magnesite roofs have clearly indicated the latter's superiority in respect of life of the lining, operational control and increased steel production over other types of acid roofs. Typical compositions, comprising of Byrapur and Bezwada chrome ores and magnesite, were prepared in different proportions. A precalcination treatment was given and from the grog so obtained, final test specimens were made, which showed lower bursting expansions. Determination of other properties of the chrome-magnesite series of test specimens was carried out.

(iii) *Sintering of Dolomite in Shaft Kiln of Hindustan Steel Plant, Rourkela* — Investigations were taken up on sintering of dolomite since the dolomite shaft kiln at the Rourkela plant of Hindustan Steel Company was not working at all satisfactorily, and it was thought that a fully comprehensive raw material study would be helpful. Samples from Hirri were obtained. Their chemical composition and mineralogy were studied. The dolomite was fired at different temperatures, using different size fractions. In some of the samples a coating of mineralizer was also given. The densification, dusting and hydration characteristics were studied. To avoid dusting, selective mining of low silica dolomite was found imperative. In order to get a dense product capable of resisting atmospheric hydration for over a week, a firing temperature of 1680°C. using 2-4 in. lumps was recommended. Use of smaller size charge such as 1-2 in. pieces, with mineralizer addition and firing of the same at 1600°-1650°C. was found to yield the best sinter. The dolomite was found to react with the alumino-silicate lining at 1400°C. and above and hence magnesite lining was recommended above the temperature zone of 1400°C. Further bulk samples

from different mines and pits at Hirri, Hardi and Baradwar were received. The suitability of these materials was investigated.

(iv) *Studies on Tata-Chemicals Magnesite* — A sample of pea-size magnesite was sent by Tata Chemicals Ltd., Bombay, for testing and evaluation, as a refractory material. The source of this magnesite was said to be sea water. The chemical analysis showed high magnesia, low silica and slightly high amount of lime. The P.C.E. was found to be high and refractoriness under load was at 1520° and to 1540°C.

40.0 Development of Super-duty Silica Bricks

With the expansion of metallurgical and related industries during successive Five Year Plans, the demand for super-duty silica bricks from indigenous raw materials has greatly increased. A number of quartzite samples collected from different localities were studied and Jamda quartzite was selected for comprehensive investigations in the first instance. A consignment of 5 tons of quartzite from Jamda was received for carrying out large-scale trials. To overcome the drawback of low strength noticed in earlier batches of full size bricks made out of Jamda quartzite, small-scale trials were carried out by changing the gradings as well as grinding the silica rock in a dead bottom pan mill. These specimens were fired on a very slow schedule lasting over a week and cooled slowly down to the room temperature. Full size bricks made according to this process had a crushing strength in the range of 3000-4000 lb./sq. in. and the reheat expansion of these bricks were also negligible. The refractoriness under load of these specimens were at 1670°-1680°C. with a load of 50 lb./sq. in. The porosity, however, was still of the order of 24-25 per cent which is rather on the high side for modern super-duty bricks. Efforts were directed to determine whether the porosity could be reduced

any further by suitable modification in grading, etc.

41.0 Development of High Alumina Refractories

High alumina refractories were required to meet a wide variety of service requirements in metallurgical furnaces and cement kilns. The presence of high titania in many of our bauxite deposits made them unsuitable for manufacture of refractory bricks containing above 60 per cent alumina. Investigation work was, therefore, undertaken to determine the suitability of Indian bauxites containing varying amounts of impurities, specially titania and iron oxide, for the manufacture of high alumina refractory having 60 per cent Al_2O_3 and above and to develop suitable compositions thereof.

(i) *Shevaroy Bauxite* — Based on preliminary work, 85, 80 and 70 per cent alumina brick were made using Shevaroy white bauxite and Travancore China clay, which were fired at $1600^\circ\text{--}1550^\circ\text{C}$. depending upon the composition. The bricks were very well fired and found to be extremely hard. The porosity values were of the order of 10-15 per cent and some even less. One application for this type of extremely hard bricks would be as skid rails in bloom and billet reheating furnaces. Specimens from 85, 80 and 70 per cent Al_2O_3 bricks made from Shevaroy bauxite were tested with manganese-bearing slags for observing the effect of corrosion, which was found to be slight and intolerable from the service point of view.

(ii) *Katni Bauxite* — Preliminary trials, such as evaluation of temperature for firing and volume shrinkage, etc., were carried out with samples of bauxite, supplied by Dyer's Limestones Co. Ltd., which were high in titania content (7.2-9.4 per cent). 60, 65 and 70 per cent alumina compositions were made by mixing plastic fireclay with the bauxite in requisite proportions and the grog was prepared by firing briquettes made out

of the respective mixes. The grog was crushed and several grades were used for carrying out packing density trials. On the graded mixes with maximum packing density was used for pressing brick specimens which were fired at $1550^\circ\text{--}1600^\circ\text{C}$. The porosity of the specimens was found to be 22, 11 for 70, 65 and 60 per cent high alumina bricks respectively. Reheat shrinkage values were less than 1 per cent. Determination of the other physical properties is in progress.

(iii) *Development of Foamed Alumina Bricks* — This project was taken up to produce high temperature insulation brick which could be used up to $1800^\circ\text{--}1900^\circ\text{C}$. Such high temperature insulation material is not made in the country. The problem was divided into three phases: (i) sintering of alumina by prior additions of mineralizers; (ii) selection of a proper forming agent and developing optimum techniques for the formation of bubbles in the casting slip and its stabilization; and (iii) fabrication of different shapes and firing them. Starting with the first phase, different mineralizers like rutile, red mud, ilmenite, China clay, dolomite, chromite, etc., were added to aluminium trihydrate and after mixing thoroughly in ball mill, bars were pressed, which were then fired at 1600°C . Some of the mineralizers gave very encouraging results. Preliminary trials were in progress for selecting a suitable foaming agent and for standardizing a technique for obtaining evenly distributed stabilized bubbles in the casting slip.

(iv) *Development of Calcium-aluminate Cement Using Indigenous Raw Materials for the Manufacture of Castable Refractories* — Ten compositions with different lime/alumina molar ratios were made using limestone and aluminium trihydrate. The test specimens were fired to temperature ranging from 1350° to 1650°C . The mineralogical compositions were examined under the microscope and the development of different calcium aluminates in fired bodies was studied. It was found that with higher silica contents in the limestone, liquid formation occurred at lower

temperatures. The setting time, bonding characteristics and other physical properties of these cements were determined.

42.0 Studies on Carbon Refractories

This project related to long-term investigations on carbon refractories and relating carbon products for application in metallurgy. In the first stage, it was proposed to study carbonaceous materials from the point of view of their ultimate utilization as starting materials in the production of carbon products, based on an examination of their pyrolytic characteristics with and without pretreatment as well as additions in the first instance, to be followed up by a study of their graphitization potentialities. Some of the carbonaceous materials readily available in India, such as the low-ash coals of Assam, washed coals of the Jharia and Raniganj area and lignite from Neyveli and petroleum coke from Digboi were taken up for the first part of the study. In connection with this project the following apparatus and equipment were fabricated during the period.

- (i) *D.T.A. Apparatus* — This apparatus consists of drilled copper block as sample holder and a vertical furnace with attachment for controlling the ambient atmosphere inside the furnace. Calcined petroleum coke is used as inert and the pyrolytic reaction occurring in coal up to 700°C. can be followed with this set-up. The apparatus is being standardized.
- (ii) *Agylutinating Apparatus* — This is a kanthal wound vertical tube furnace capable of sliding up and down. The sample holders are hung vertically from a cross beam fitted to the vertical guides and provisions are made for atmosphere control. A dimmerstat and a sunvic provided in the heating circuit give the necessary control over the rate of rise of temperature as well as its maintenance at any specified level up to a max. of 1000°C.

- (iii) *Geissler Plastometer* follows a standard design and is under fabrication.

- (iv) *Furnace to Operate at 2000°C.* — A transformer and carbon resistance furnace were designed. The transformer is being assembled.

The experimental work was more or less of the preliminary nature. In the first instance, it was considered necessary to briquette the raw materials without using any binders so that they could be easily handled in subsequent experiments. Various grain sizes and compacting pressures were tried and optimum conditions were standardized. Similarly the prethermal treatment of coal needed standardization of the conditions under which these treatments must be conducted. These were worked out.

43.0 Development of Oxide Refractories from the Beach Sands of Kerala

Under the Council of Scientific & Industrial Research Fellowship Scheme, work was initiated on development of oxide refractories from beach sands of Kerala.

Monazite, zircon and sillimanite are some of the constituents of the mineral sands of Kerala. Monazite contains phosphate, thorium and rare earths, such as cerium, lanthanum, etc. Attempts had already been made to decompose monazite by heating it with a mixture of calcium-oxide and calcium chloride and then leaching it with dilute acids to separate phosphate. A scheme was drawn out to examine whether the rare earths could be split up into their individual components like cerium oxide, lanthanum oxide, etc., by heating the mixture with calcium oxide and chloride. It was also envisaged that each component of monazite, viz. phosphate, thorium and the different rare earth oxides, were to be tried one after another and in combination in different proportions with sillimanite and zircon to study their effect on sintering, physical properties and structure. A study of the refractory

properties of some samples of ball clay and silica sands available in large quantities in Kerala was also aimed at.

With the above objects in view, an X-ray study of the new techniques applied to decompose monazite, viz. heating with a mixture of calcium oxide and calcium chloride and then leaching with dilute acids, was started. Debye-Scherrer photographs of this powdered monazite were taken and their d -values were calculated. These values were compared with those published in standard reference cards and in literature and found to tally with them. Similar X-ray diffraction photographs were taken of the monazite heated to 800°C ., heated mixture of monazite, calcium oxide and calcium chloride. Rare earth residues were separated by the new method, and the phosphatic fraction was isolated. The d -values of these were calculated and the efficiency of the process was reviewed.

A study of the chemical and physical properties of the ball clay from Kumbalam (Kerala State) and silica sands brought from Trivandrum beach was also taken up.

44.0 Studies on Clays from South India

This study was taken up on behalf of the Director of Industries & Commerce, Madras State, and was an attempt to catalogue the characteristics as well as possible refractory applications of clays from various localities in Madras State. Five samples were received from Madras. Work on sampling and testing was taken up. The investigation would follow mainly the lines on which clays from other areas were studied including their chemical and particle size analysis plastic and green properties and behaviour on heating to various temperatures. It was also expected that correlation between their physical properties and their mineralogical analysis would be attempted. The work so far accomplished included: (i) size analysis on the sieve, and (ii) water of plasticity

determination. Chemical analysis and P.C.E. determination are in progress.

45.0 Base-exchange Study on Clays

Cation exchange on clays offered a diagnostic method of identifying clay minerals present in a particular clay, as the type of ions absorbed on the clay affects the green and fired properties of clays. Much work has been done on the effect of absorbed cations, such as Na^+ , K^+ , Li^+ , Ca^{++} , Ba^{++} , Mg^{++} , etc., on the workability of clays. It was contemplated in the present work to carry out some specific cation exchange on some Indian ceramic clays and study its influence on the green and fired properties of clays. It was also contemplated to carry out differential thermal analysis and X-ray analysis as supplementary to base exchange in identifying the clay minerals present.

Base exchange capacity (B.E.C.) of Rajmahal clay —18 BSS, —300 BSS and —2 micron fractions was determined by saturation with ammonium acetate and subsequent distillation of ammonia. X-ray study coupled with B.E.C. value of Rajmahal clay revealed that it was a poorly crystallized kaolinite. D.T.A. of the same clay confirmed the above view. D.T.A. and B.E.C. of fireclay, Kotah (Rajasthan), indicated that it was not predominantly kaolinitic. X-ray analysis did not give the characteristic kaolinite pattern; this indicated that the clay mineral present in the above clay was other than kaolinite. The examination of these two clays was carried out with a view to choose an ordered and a disordered kaolinite for further study. The determination of B.E.C. of Rajmahal China clay was carried out by a number of different methods, viz. (i) saturation with ammonium acetate; (ii) continuous potentiometric titration of the clay acid; (iii) potentiometric titration of the H-clay and allowing sufficient time for attainment of equilibrium for each addition of alkali; and (iv) saturation of the clay acid with neutral 1N KCl and titration of the acid

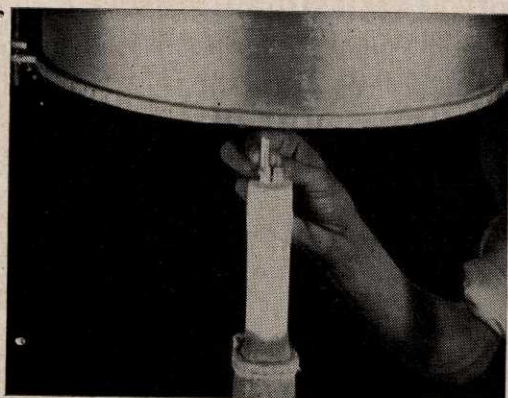


FIG. 19 — PHOTOGRAPH SHOWING THE SAMPLE HOLDER AND THE FURNACE USED IN THE HIGH TEMPERATURE D.T.A. THE SAMPLE HOLDER CONSISTS OF AN ALUMINA BLOCK CEMENTED TO AN ALUNDUM TUBE. TWO ALUMINA THERMOCOUPLE TUBES PASS THROUGH TWO HOLES DRILLED IN THE ALUMINA BLOCK AND CEMENTED TO IT; THESE SERVE TO CARRY THE THERMOCOUPLES AND ALSO SUPPORT THE PLATINUM FOIL CUP CONTAINERS FOR SAMPLE AND INERT MATERIAL. IN THE PHOTOGRAPH, THE PLACING OF A PLATINUM FOIL CUP OVER ONE OF THE THERMOCOUPLE TUBES IS INDICATED

liberated. Of the above methods continuous potentiometric titration yielded a very low value for B.E.C. indicating that sufficient time should be allowed for attainment of equilibrium between clay and alkali. The values obtained by other methods agreed closely. Mono-cationic forms of Rajmahal clay containing Li, Na, K, NH_4 , Ca, Ba, Mg

were prepared with a view to study the effect of these cations on thermal behaviour of kaolinite. The thermal reactions of kaolinite were studied up to 1350°C . by differential thermal analysis using a specially designed sample holder for this purpose. Even though the temperatures of dehydroxylation, primary mullitization and secondary mullitization peaks were not appreciably altered, the amplitudes of the peaks were very much changed with different cations.

46.0 Thermal Conductivity of Insulation Refractories

Insulating bricks from different Indian firms as well as one sample from a foreign source were collected for the determination of their thermal conductivity values using A.S.T.M. thermal conductivity apparatus. Physical properties, such as cold crushing strength, linear change on heat, apparent porosity and bulk density, etc., and thermal conductivity of five different samples were determined. The values are given in Table 15.

47.0 Suitability of Amphibole Type of Asbestos for Industrial Purposes

Five samples of asbestos from Singhbhum District were received from Geological Survey

TABLE 15 — PHYSICAL PROPERTIES OF INSULATION REFRACTORIES

REF. No. OF BRICKS	TYPE OF INSULATING BRICK	A.P. %	B.D. (C.G.S. UNIT)	COLD CRUSHING STRENGTH lb./sq. in.	THERMAL CONDUCTIVITY VALUE B.T.U.	MEAN-TEMPERATURE $^\circ\text{F}$.	% LINEAR CHANGE AT 850°C . (24 hr.)	% LINEAR CHANGE AT 1500°C . FOR	
								3 hr.	24 hr.
1.	Hot face (imported)	72	0.75	252.2	3.66	729	0.20	—	3.34
2.	Backing (Indian)	73	0.72	528.6	3.00	586	0.30	13.59	—
3.	do	77	0.61	98.37	1.87	544	0.37	25.75	—
4.	do	71	0.78	250.2	5.47	746	1.25	—	—
5.	do	79	0.58	158.0	0.84	532	0.91	—	—
6.	do	66	0.90	223.1	—	—	0.08	20.5	—
7.	do	65	0.94	321.0	—	—	0.17	21.48	—
8.	Hot face (Indian)	59	0.96	536.4	—	—	0.03	0.43	Vitrified and shrunk badly
9.	Backing (Indian)	65	0.96	130.5	—	—	0.043	5.12	—

of India to study their suitability for industrial uses. Samples were identified as tremolite and anthophyllite of amphibole group. Due to their brittleness and low tensile strength none of these samples could be spun into yarn. These samples were tried for making asbestos cement board with calcium aluminate cement, Portland cement and Sorel cement as binders. Specimens were prepared using different percentage of binders and pressing discs and bars. The properties of these specimens, as-made, as well as after firing them at different temperatures ranging from 500° to 1100°C., were studied. The specimens made with calcium aluminate cement as binders showed maximum crushing strength. Asbestos insulation brick specimens were also prepared by adding 30 per cent of combustible material like saw dust, petroleum coke or charcoal to mixes containing asbestos and clay. Their strength and porosity were determined after firing them at 700° and 1100°C. From this preliminary study it was concluded that though none of the specimens could be used for spinning into yarn, these could be successfully used for making different kinds of asbestos cement boards, etc., which have wide applications both in industries as well as in the building trade. Samples were collected from different localities for further investigations.

48.0 Plasticizing and Colloidizing of Non-plastic Refractory Materials

An important and major advance in ceramic technology is that of producing artefacts from high temperature refractory raw materials which do not have binding properties in the raw state without the additions of binders which alter chemical composition and more or less adversely affect their high temperature properties. Pure oxides like alumina, magnesia and thoria are samples of such refractories growing in importance. Of a lesser importance but of great commercial potential

are materials like sillimanite and kyanite. Experiments were initiated with sillimanite from the beach sands of Travancore and its grindability under certain predetermined conditions was assessed.

The size analyses (as determined by the Andreasen Pippette) of the various grinds is given in Table 16. Experiments with other variables such as weight ratio of the fluid medium to the material ground and the pH of the fluid medium were undertaken and Andreasen Pippette determinations thereof were made. Side by side with the variation in fineness, the actual effect of increasing fineness on the various physico-chemical properties of the ground material was also under close study. The properties chosen were: (i) sedimentation volumes; (ii) base exchange capacity (if any); (iii) pH of the suspension of known concentrations; and (iv) variation of the fluidity of the suspension with variation in pH. The experimental procedures for all these determination were standardized with the 72 hours. ground sillimanite as the base material. The standardization experiments were, however, of a preliminary and exploratory nature. Some interesting data obtained in this connection are given in Table 17.

49.0 Correlation of Chemical and Physical Properties of Blast Furnace Refractories with Their Resistance to Carbon Monoxide Disintegration

This investigation was taken up in order to collect data on blast furnace refractories from different sources, both indigenous and foreign and to analyse them to arrive at optimum figures for desirable physical properties. These data would be used in collaboration with Indian Standards Institution and other research laboratories in formulating suitable specifications for blast furnace refractories. A standard type of carbon monoxide disintegration apparatus for testing blast furnace refractories was set up and

TABLE 16 — SIZE ANALYSIS OF POT MILL GRINDS, GROUND FOR

PARTICLE SIZE OF MATERIAL AS RECEIVED		FINER THAN μ	72 HR. %	96 HR. %	120 HR. %	144 HR. %	168 HR. %	192 HR. %
44 mesh	2.25	26.00	78.9	87.3	100.0	100.0	90.2	83.4
—44+52	7.75	8.00	62.6	68.1	99.2	97.9	85.7	82.8
—52+72	42.75	6.00	54.6	62.2	86.5	90.6	86.4	82.0
—72+100	37.50	4.00	45.0	50.1	68.1	79.2	77.7	78.8
—100+150	8.25	3.30	38.5	41.2	58.8	71.1	70.8	75.3
—150	1.50	2.50	31.6	33.8	47.7	59.9	61.1	68.3
		1.15	15.4	15.9	22.4	29.5	31.4	39.1
	100.00	0.80	10.6	8.8	15.2	20.1	21.9	26.3
		0.60	8.6	8.3	10.0	14.0	14.0	18.5

TABLE 17 — pH FLUIDITY RELATIONSHIP OF 72 HR. GROUND SILLIMANITE — 50% SUSPENSION

pH	FLUIDITY Sec.	ACID SIDE	
		pH	Fluidity Sec.
8.45	46.0	7.90	56.5
8.95	42.0	7.80	100.0
9.20	34.0	7.60	112.0
9.45	31.5	7.25	120.5
9.58	28.0	7.35	135.0
9.70	27.0	6.35	75.0
9.95	25.5	4.20	26.0
10.00	24.5	4.05	20.9
10.10	22.5	3.92	20.1
10.15	20.0	3.85	19.5
10.25	20.0	3.96	20.1
10.27	18.6	3.92	20.2
10.30	18.9	3.50	20.8
10.36	18.0	3.10	—
10.40	26.0	—	—
10.45	25.5	—	—
10.50	20.5	—	—
	23.0		

50.0 Studies on Refractory Brick Forming

This project was based on the National Metallurgical Laboratory's patent on 'Improvements in, or relating to, the manufacture of bricks from refractory mixes'; and stage 1 comprised of a comparative study of the hydraulic press and the adapted pneumatic hammer (Fig. 20) as brick forming machines. The object of this investigation was to collect data on the performance of the hammer from the point of view of the product that it yielded and the economics of its working. These data will assist in giving a clear idea of the correlation of impact pressing with single stage toggle or hydraulic pressing. In dry press brick manufacture, the properties of the products depended mainly on the following factors:

1. Size, shape and grading of the base material particles
2. Nature and quantity of the binding material used
3. Quantity of water added
4. Plasticizer additions
5. Mixing
6. Nature of compaction
7. Drying schedule
8. Firing schedule

In the study so far all the variables except the nature of compaction were kept constant. This factor had two sub-variables, namely (a) compaction under one stage pressing, and (b) compaction under a number of impacts.

tests were carried out on one set of blast furnace bricks from one indigenous and one foreign source. Other physical properties like porosity, permeability, cold crushing strength, permanent linear change on reheating, etc., as well as chemical analysis of the bricks were done. Efforts would be made to correlate the different properties when samples from different sources would be studied. Data of physical properties for the two sets of blast furnace refractories: (a) indigenous; (b) imported, are given in Table 18.

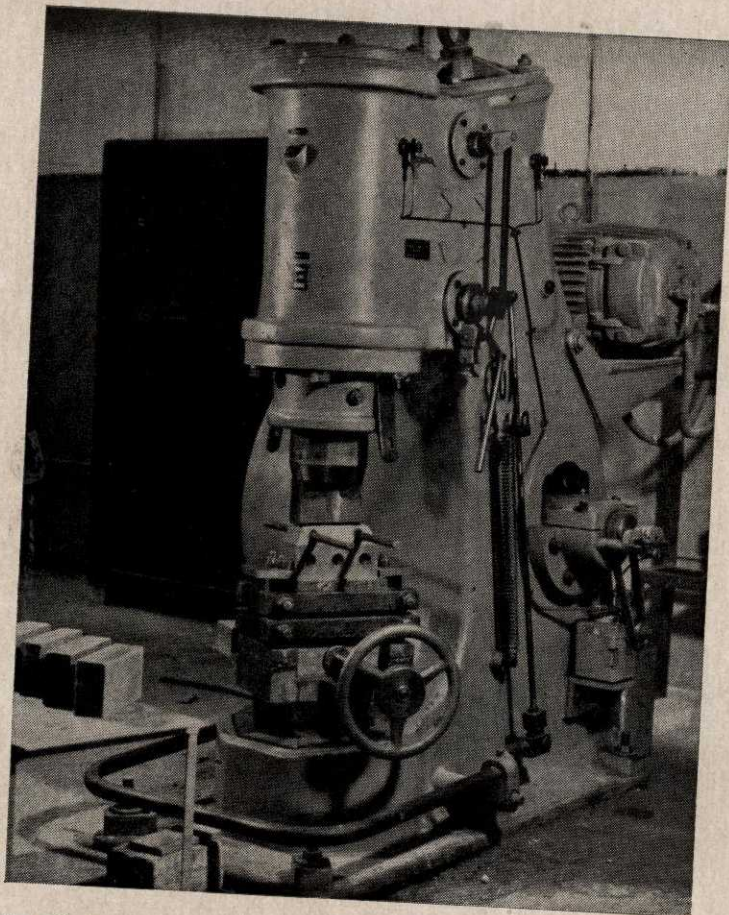


FIG. 20 — ADAPTED PNEUMATIC HAMMER AS BRICK FORMING MACHINE

TABLE 18 — PROPERTIES OF BLAST FURNACE REFRACTORIES

No.	TESTS	HEARTH AND BOSH		INWALL	
		Indigenous	Imported	Indigenous	Imported
1.	Porosity	24.89%	20.0%	25.53%	14.0%
2.	Porosity after reheat test	17.46%	n.d.	16.95%	n.d.
3.	Permeability C.G.S. Unit	2.55	1.3 }	2.40	0.25
4.	Permanent linear change (reheat) (2 hr. at 1450°C.)	0.87%	—	1.30%	0.0 to 0.5%
5.	Cold crushing strength	499 kg/sq. cm.	n.d.	386.2 kg/sq. cm.	n.d.
6.	Carbon monoxide disintegration	—	—	Withstood 100 hr. test	Out of 5 samples two cracked and fell apart after passage of CO for 80 hr. while 3 others with- stood the test for 100 hr.

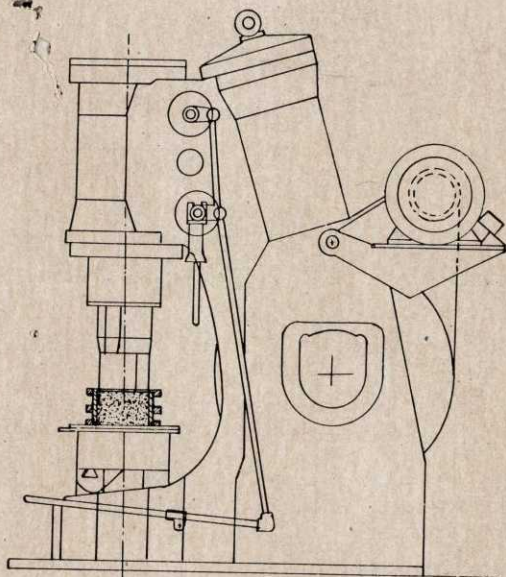


FIG. 21 — DIAGRAMMATIC SKETCH OF THE ADAPTED PNEUMATIC HAMMER FOR BRICK MAKING

Under the first sub-variable, the pressure adopted and the dwell time varied and in the second, the intensity of the impact and the number of impact varied. These aspects were studied in detail; their effects could be expected to be twofold, namely (a) the particle grading of the material and hence its natural packing density may be altered due to the crushing action of the pressure of impact, and (b) the mix may be compressed to a minimum volume depending on the magnitude of the compacting action. An assessment of these two actions under various pressure/dwell relationships in the case of one stage pressing and under various intensity/number relationship of the impacts in the case of impact compaction was made by means of suitable quantitative tests on the resultant products obtained. A fired clay grog was prepared and crushed to a known granulometric composition in order to keep its dry packing density constant. Its other physical properties were also kept constant by keeping chemical composition, method of fabrication and firing schedules constant.

After a number of experiments, a standard clay-grog-water mix was arrived at and this was used as base material. A 2 in. dia. steel die had been used in these experiments.

Compaction of this mix under the following conditions were studied:

1. Single stage hydraulic pressing at various pressures ranging up to 20,000 lb./sq. in. and in varying height.
2. Under blows on AFS rammer with increasing number of blow and varying the heights of compacts
3. Under the mixes' own weight by an increasing number of bumps in a modified Westman Hugil apparatus.
4. Under blows from a pneumatic hammer of controlled intensities.

An extremely interesting result was that under all methods of compaction, packing followed a parabolic equation of the type ax^b .

The results of the hammer experiments are still under analysis. It is expected that these results will yield data on the actual efficiencies of the various methods utilized. In the next stage, it is proposed to project the work on larger-sized specimens in order to evaluate the effect of size of the specimens on compactions. Table 19 and Fig. 22 give some relevant data.

TABLE 19 — GRAPHIC SOLUTIONS FOR THE TREND OF PACKING UNDER VARIOUS METHODS OF COMPACTION

General equation $y = ax^b$ where y = bulk density of compact, a and b are constants and ' x ' is the specific compaction variable

Wt. OF COMPACT	NATURE OF 'X'	VALUE OF CONSTANT	
		'a'	'b'
A. Ramming under AFA Rammer			
100 gm.	No. of rammings	1.406	0.047
150 gm.		1.320	0.057
200 gm.		1.321	0.054
250 gm.		1.310	0.055
B. Packing under Its Weight by Bumping			
100 gm.	No. of bumps by a factor of 29	1.512	0.027
150 gm.		1.486	0.0309
200 gm.		1.440	0.035
250 gm.		1.427	0.0363

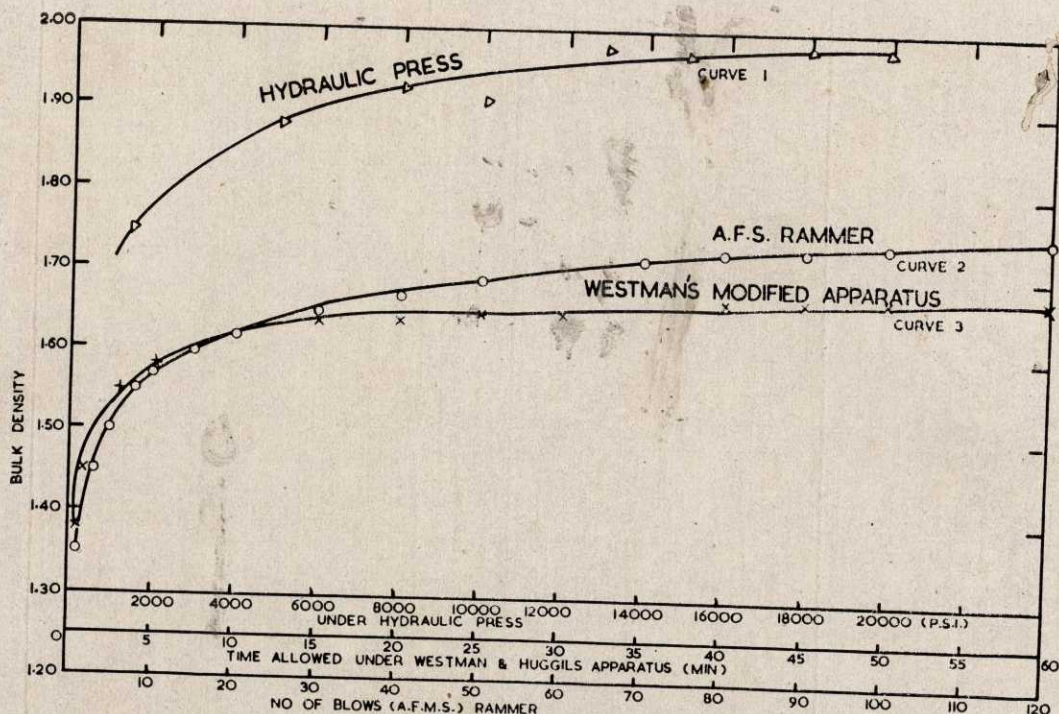


FIG. 22 — VARIOUS COMPACTION CURVES FOR 100 GM. COMPACT

51.0 Development of Magnetic Materials

The demand for magnetic materials is multiplying in India following establishment of telephone, electronics and other industries. At present, the quantity needed is mostly met by import. This project was, therefore, taken up with a view to obtain detailed technical knowledge regarding their manufacture and to develop, if possible, permanent magnets and other magnetic materials from indigenous sources. The work is initially concerned with the preparation and study of alloys of the well-known Alni, Alnico and Alcomax types.

Encouraging low values of the hysteresis loss were obtained for a low-aluminium low-manganese steel containing about 2-3 per cent of each of these elements. Attempts were made initially to prepare a few heats of 20 lb. each, as a preliminary to make a much larger heat to standardize the procedure of

melting, working and heat treatment of the alloy. It was, however, found that the material could be cold rolled with considerable freedom from edge cracks, even when rolled to as much as 80 per cent reduction of thickness. Arrangements are now being made to prepare larger heats so that requisite large-scale service tests could be arranged for in due course.

With regard to the investigations on cast Alni, Alnico and Alcomax type permanent magnets, attempts were made to prepare about 20 lb. of Alcomax type of alloy with about 8 per cent aluminium, so that the exact heat treatment operations in a magnetic field to yield desired anisotropic magnetic properties could be determined, which, however, did not meet with much success. While preparation of this alloy was kept in mind, considerable work was carried out to systematically study the effects of the time and temperature of tempering upon the magnetic properties of two alloys of the Alni type,

prepared earlier. Tempering was carried out in the range 500°-700°C. and for duration up to 400 hours after each tempering treatment, the samples were air cooled and tested for magnetic properties. It was found that in both of these alloys, the coercive force and remnance increased in the first 2-4 hours of tempering at 600°C., then slightly decreased, remaining almost constant for duration of tempering extending up to about 50 hours and then decreased further gradually. In one of these alloys which contained small amount of copper, the effects were somewhat clearer. These results indicated that considerable resistance to deterioration of magnetic properties, when over-heated, would be exhibited by these tempered alloys provided slightly lower magnetic properties could be tolerated.

The development of permanent magnets of the Alni, Alnico and Alcomax types starting from the powders of the constituent metals and using the techniques of powder metallurgy was undertaken. Several preliminary experiments were carried out, using powders of aluminium, iron and nickel, which had been obtained several years earlier. Sintering of compacts made from these powders indicated their unsuitability. Pending receipt of fresh powders, small quantities of the requisite powders, particularly iron powder, were prepared by pulverizing flakes obtained by electrolysing a bath of ferrous ammonium sulphate at room temperature, using mild steel and/or armco iron anodes and a stainless steel cathode. At a current density of approximately 20 amp./sq. ft., satisfactory deposition of iron in the form of flakes took place. These flakes could be removed from the cathode by tapping it gently, and after washing and drying, they were powdered to -200 mesh for subsequent use.

To avoid the direct use of aluminium powder, as sintering difficulties would be experienced on account of adherent surface layers of oxide, aluminium was used in alloyed form with iron. A master alloy of iron and aluminium, in approximately equal

proportions, was first prepared by melting Armco iron and commercially pure aluminium, in a high-frequency furnace and chill casting the molten alloy into an iron mould. The ingot, so obtained, was quite brittle and could be broken and pulverized to yield suitable alloy powder of -200 mesh. The nickel powder, previously used, could also be reduced in a stream of pure dry hydrogen prior to mixing it with the other powders. These powders were thoroughly mixed in the proportion of approximately 60 per cent iron, 27 per cent nickel and 13 per cent aluminium. Suitable quantities of the mixture were pressed in a rectangular die at a pressure of 30 tons/sq. in. to yield compacts approximately 2 in. long to $\frac{1}{2}$ and $\frac{1}{4}$ in. high. It was found that the green compacts were very fragile and developed cracks on storage. Several of them split into wedge-shaped pieces. The pieces were found to sinter well when sintered at 1300°C. for 2 hours in hydrogen, which was carefully purified and dried. The density of the sintered material was very nearly equal to the theoretical density of the alloy indicating virtually perfect sintering. However, as the green compacts tended to break up into smaller pieces, no extensive test for magnetic properties was carried out.

It was considered that the poor strength of the green compacts might be due to brittle and hard nature of the iron powder, obtained by the electrolytic method. The iron powder was, therefore, annealed, initially at 500°C. and later at 800°C., for 2 hours in each case, in a stream of pure dry hydrogen. It was found that annealing at the latter temperature softened the iron powder to an extent, sufficient to give a satisfactory compact. The iron powder, as prepared, was found to retain about 10 c.c. of hydrogen per gram while after annealing for 2 hours at 500°C., the powder had hydrogen to the extent of 0.1 c.c./gm. and 0.02 c.c./gm. respectively. The general trend revealed by these sintering experiments was that the specific gravity increased while the electrical resistivity decreased with increasing time and temperature

of sintering. These sintered compacts are being examined for their magnetic properties as well as for their internal structure by X-ray.

52.0 Transformation Studies of Austenite

This project was taken up to elucidate certain aspects of the transformation characteristics of austenite and its retention. Some of the aspects were thermal stabilization of the austenite above and below the M_s temperature, the effect of interrupting the quench, the effect of isothermal hold and the effect of specific heat-treatment operations.

One aspect of this investigation was the quantitative assessment of the extent of transformation of austenite to martensite by X-ray methods. Previously, specimens of a high-carbon, high-chromium (2.3 per cent and 13.6 per cent chromium) die steel, heat treated to retain varying percentages of austenite, were examined by a beam of X-ray from a cobalt target, suitably monochromatized. It was reported that qualitatively satisfactory pictures were obtained but the background radiation due to the presence of considerable quantities of chromium did not permit accurate assessment of the relative integrated intensities of the diffraction lines due to austenite and martensite. As no chromium target was available, one of the existing copper targets was chromium-plated and the radiations from this target were used for examining the specimens, after suitably monochromatizing them using a quartz bent-crystal monochromator. By further limiting the monochromatized beam by suitable stops and recording the pattern obtained, when the beam was incident on the suitably electropolished surface of the specimen at an angle of about 60° , considerably better pictures were obtained. It was, however, found that these pictures showed generally a spottiness, due to the relatively coarse grains of the austenite crystals. An arrangement was, therefore, designed and

constructed, whereby the specimen could be rotated without changing the angle of incidence of the X-ray beam on it, but by varying the eccentricity of the rotation, a much greater number of austenite crystals could be irradiated. The picture obtained by so mounting the specimen and rotating it showed a surprising degree of improvement and it was possible to estimate quantitatively the relative proportions of austenite and martensite.

A low-carbon, low-chromium tool steel, whose austenite stabilization had been the subject of extensive investigations, was examined in two heat-treated conditions and the relative proportions of austenite and martensite were determined. When the steel was ultrasonically activated at 98°C ., in boiling water and quenched to room temperature, the austenite martensite ratio was about 1:7 with an overall hardness of about 785 VPN. In the hydrogen-stabilized condition, the relative proportions were about 1:5, with a hardness of about 710 VPN. Assessment under other heat-treated conditions is being taken up. For the above assessment, considerable numerical data were required with regard to the Lorentz and polarization factor, Debye-Waller temperature factor, structure factor, multiplicity factor, sample absorption factor and lattice parameters of the austenite and compiled for enabling the conversion to be made of measured integrated intensities to proportions of these two phases.

Another aspect of this investigation was the study of the effect of various factors on the reactivity of austenite and the extent to which austenite transformed to martensite; considerable new information was obtained and some new concepts emerged therefrom such as the following:

The stabilization of austenite by hydrogen was examined in considerable detail. On the basis of a mechanism for the stabilization of austenite where austenitic strain embryos were deactivated by interstitial carbon atoms, the ineffectual role of hydrogen in austenite stabilization in the amounts present

in normally heat-treated steels had been established. It was also experimentally established that if hydrogen could be so introduced and retained in a steel when in austenitic condition, that its migration towards and capture by the strain embryos were assured, then stabilization would result. By cathodically impregnating a low-carbon, low-chromium tool steel (M_s approximately 93°C.) at 98°C. and a low-carbon unstabilized 18:8 stainless steel at room temperature considerable stabilization of the austenites was observed in both cases. The stabilization obtained in the first steel by cathodic impregnation was of the same order as that obtained in this steel by a prolonged isothermal hold in boiling water and confirms conclusively an a priori consequence of Cohen's original theory of stabilization, viz. that any interstitial solute atom could cause stabilization.

Activation of the austenite in these two steels was also experimentally detected by bombarding them with ultrasonic waves, under cavitating conditions. Specimens of these steels, in their austenitic states, were suspended in boiling water, through which ultrasonic waves were allowed to pass and be incident on them. Activation was detected on bombardment with both low-frequency as well as high-frequency waves, for very short as well as longer durations and also with different intensities of the ultrasonic beam. The low-frequency waves obtained from a magnetostriction transducer, driven by a Mullard oscillator and the high-frequency waves from a X-cut quartz plate, excited into resonant vibration by the tuned output of a Hartley valve oscillator, used earlier for the determination of the elastic constants of metals by an ultrasonic method.

It was also experimentally established that ultrasonically activated austenite could be stabilized by cathodic impregnation and vice versa. The effects of simultaneous bombardment and impregnation are being examined. The activation had been ascribed to the generation of strained regions in the austenitic

lattice, due to the passage of shock-waves, which were generated in the boiling water as well at the surfaces of the suspended specimens, when cavitation in the water was produced by the passage of ultrasonic waves.

The combined effects of impregnation and cooling were also examined with reference to the low-carbon, low-chromium tool steel. A very considerable amount of stabilization appeared to result, and that was examined in detail.

53.0 Lattice Parameters of Iron-chromium Alloys and Study of the Sigma Phase in Alloy Steels

Work was taken up with a view to determine the nature and structure of sigma phase arising in alloy systems of the transition elements. The importance of this investigation can be appreciated from the fact that the presence of sigma phase generally renders the steel unsuitable for fabrication and cold deformation. The plan of work is to prepare various alloys of the transition elements from metals of as high a degree of purity as is possible to obtain and to examine their metallographic structures as well as lattice parameters.

Several trial runs were made with the zone refining furnace, which is being constructed for refining commercial aluminium, as a preliminary to the construction of a suitable apparatus for adequate refining of such of the transition elements as cannot be obtained by ordinary methods to the requisite specification. It was found that several modifications were necessary and these were attended to. Modifications were made in the arrangement for the relative motion of the specimen and furnace. In the modified arrangement, the furnace ascended or descended vertically at a slow rate, the specimen, passing at its centre, remaining stationary. Some modifications are to be made to the mounting of the electric motor, which, through a set of reduction gears and

couplings, raises and lowers the electric furnace. Commercial aluminium was subjected to zone refining but it was found by spectroscopic analysis that the refining was quite inappreciable. As zone refining by this method of the transition metals, which have considerably higher melting points than aluminium, will undoubtedly present greater difficulties and when several pure metals were received later on, it was decided to suspend further work on zone refining.

54.0 Preferred Orientation in Rolled Sheets

The usual method of determining the preferred orientation produced on cold-rolled sheet is based on the study of the nature and intensity of X-ray reflections. This method could advantageously be replaced, if possible, by methods based on the value of the electrical conductivity and the velocity of propagation of stress waves in rolled sheet as a function of the angle to the direction of rolling.

The electrical resistivities of cold rolled 60: 40 brass parallel and perpendicular to the

direction of rolling as well as at angles inclined variously to the direction of rolling had been determined by the ballistic throw method as well as the Kelvin Double Bridge method. The value of electrical resistivity obtained by these methods for a 98 per cent cold rolled 60: 40 brass are given below as a function of the angle with the rolling direction:

ANGLE WITH ROLLING DIRECTION IN DEGREE	ELECTRICAL RESISTIVITY BY BALLISTIC METHOD $\times 10^6$ ohm/cm.	ELECTRICAL RESISTIVITY BY KELVIN BRIDGE METHOD $\times 10^6$ ohm/cm.
0	8.58	8.57
11	9.01	9.03
22½	8.10	8.11
34	8.06	8.03
45	8.49	8.49
56	8.11	8.14
67½	7.71	7.69
79	7.79	7.76
90	8.03	8.04

The percentage increase in electrical resistivity of the cold-rolled brass in various directions, using the values of resistivity determined by the ballistic method is given below:

Angle with rolling direction	0°	11°	22½°	34°	45°	56°	67½°	79°	90°
Per cent increase in electrical resistivity	25.4	31.8	18.5	17.4	24.1	18.6	12.8	14.0	17.4

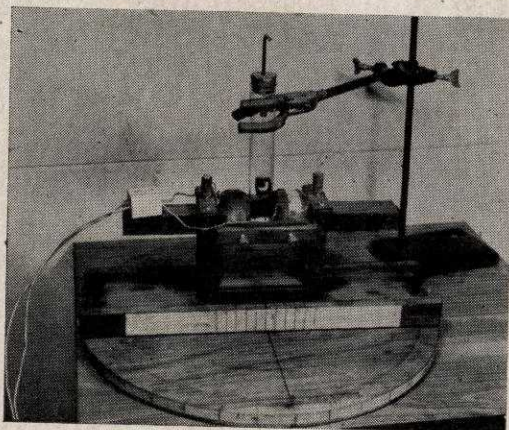


FIG. 23 — EXPERIMENTAL SET-UP OF BALLISTIC DETERMINATION OF ELECTRICAL RESISTIVITY

As the deformation processes in a crystal, even though of cubic symmetry, are not isotropic, the tendency for the generation of lattice defects, when it is cold worked, cannot also be taken to be isotropic. Anisotropy of electrical resistivity in cold-rolled cubic metals and alloys should, therefore, be no longer considered incongruous, except in ideal, perfect and defect-free material, even though theoretically, no anisotropy in electrical resistivity can be expected to arise. Anisotropy, as has been observed above has, in recent years, been clearly demonstrated by several investigators in copper, nickel and some brasses and is clearly consistent with recent theories of the generation of non-random dislocation arrays by cold work,

leading to anisotropic scattering of the conduction electrons.

From the increase in the resistivity, rough estimates can be made of the number of dislocation pairs generated by the cold rolling. Using values for effective electron mass, nuclear charge, volume of unit cell, etc., appropriate to 60:40 brass, the increase in resistivity appeared to correspond to generation of dislocation pairs of the order of 5×10^{13} per sq. cm., the exact number in any given direction depending on the actual increase in the resistivity. These values appeared to be somewhat on the high side when compared with values obtained from the energy stored during cold work. The discrepancy was undoubtedly due to the estimates being rather approximate. Apart from errors arising in the estimated values of effective electron mass, nuclear charge, etc., only edge-type dislocations had been considered, other types of imperfections as well as anisotropy in the elastic constants being ignored in those calculations. The pile-up of dislocations or their further dissociations into partial dislocations and stacking faults had also been not considered in making those estimates.

The observed anisotropy in electrical resistivity appeared to be capable of being satisfactorily explained qualitatively on the basis of the increase in the resistivity in any crystallographic direction as related to the case of slip in this direction. In the case of the cold-rolled 60:40 brass, the principal texture may be taken as [110] [112]. The angular separations between the [112] direction (the direction of rolling) and various crystallographic directions in the rolled sheet was determined by reference to the standard [110] projection for a face-centred crystal as given below:

Slip along the directions with low indices was assumed to be easier than in directions of high indices and consequently, the increase in electrical resistivity was considered to be rather low in these directions of relatively easier slip. The anisotropy of resistivity would, therefore, be determined by the angular relationships between the various crystallographic directions in the textured sheet. A very satisfactory qualitative explanation could, therefore, be given on this basis of the observed anisotropy in the electrical resistivity.

55.0 Direct Evaluation of the Effective Camera Radius in a Debye-Scherrer Photograph

A method was developed for determining the value of the effective camera radius to be used in a Debye-Scherrer powder photograph for calculation of the Bragg angles and lattice parameter. Measurement of the linear distances between three lines of the Debye photograph appeared to be sufficient for evaluating the effective radius. The limiting assumption, common to the conventional methods, that the film shrinkage is uniform over the entire film length is not required to be made in this method of evaluation.

56.0 Utilization of Vanadium-bearing Titaniferous Magnetite Deposits

The vanadium-bearing deposits in India are located at Singhbhum and Mayurbhanj and contain about 1.5-2.5 per cent vanadium pentoxide with 10-16 per cent titanium dioxide. The conservative estimate puts the reserves to about 20-22 million tons. Investigations were taken up for the utilization

TABLE 20 — ANGLES BETWEEN ROLLING DIRECTION [112] AND SPECIFIED CRYSTALLOGRAPHIC DIRECTIONS IN COLD-ROLLED 60:40 BRASS

Direction	[335]	[315]	[114]	[221]	[331]	[551]	[110]	[331]	[221]	[111]
Angle with [112]	5°	15°	19°20'	35°20'	41°30'	46°40'	54°40'	67°50'	74°	90°

of these deposits for the production of vanadium pentoxide and subsequently ferro-vanadium. The investigations on the recovery of vanadium by salt roasting was completed and a pilot plant is being installed. It was observed earlier that chlorination at lower temperatures removed 80-90 per cent of vanadium. Systematic studies on the chlorination of vanadium ores using 50 gm. of material for each experiment were started in a vertical tubular furnace. The effect of temperature on the rate of removal of vanadium and iron was studied.

57.0 Thermal Beneficiation of Low-grade Chrome Ores

The large reserves of low-grade Indian chromites having Cr:Fe ratio less than 3 cannot be directly used for the production of high grade ferro-chrome. The conventional ore-dressing methods fail to improve this ratio as FeO and Cr_2O_3 are chemically bound in the chrome spinel. Attempts were made to upgrade the ore by preferential reduction of FeO and its subsequent removal by acid leaching.

Earlier observations revealed that preferential reduction of low-grade Tagadur chrome ore with an initial Cr:Fe ratio of 1.04 could be carried out at 1300°C . by using 10 per cent of coke. The rate of reduction studied ranged from 30 minutes to 4 hours. It was observed that Cr:Fe ratio increased progressively with time up to 3 hours and decreased thereafter with a simultaneous decrease in Cr-recovery. A systematic study on the effect of particle sizes of ore and coke was undertaken, and it was observed that the optimum sizes of ore and coke are $-100+150$ and $-120+170$ mesh (B.S.S.) respectively. A series of tests using briquettes pressed at 500-6000 p.s.i. indicated that at least 1000 p.s.i. was needed for briquetting for greater reduction efficiency. A microscopic examination of the reduced ore revealed that the reduction proceeded along the grain boundaries, vein and cracks in the

ore indicating that the iron in the reduced ore could be removed easily by acid leaching.

The effects of various factors on the leaching of the reduced chrome ores by sulphuric acid were completed and the variable factors studied included the effect of particle size, strength of acid, ore: acid ratio and temperature of leaching. It had been observed that the leaching was fast when the reduced ore was crushed to -100 mesh B.S.S. sieve and heated to boiling. The strength of acid from 1-10 per cent had no effect of leaching whereas the dissolution of chromium was completely eliminated by controlled ore: acid ratio. Cr:Fe ratio of 3 per cent could be obtained with almost 100 per cent Cr recovery by using decreased amount of acid.

58.0 Production of Ferro-alloys

Excepting ferro-manganese and ferro-silicon there is at present no commercial scale production of other ferro-alloys in India including ferro-chrome. With the establishment of Alloy, Tool and Special Steel Plant in the Third Five Year Plan, there will be a great demand for the different types of ferro-alloys. A broad-based investigation was, therefore, taken up for the development of manufacturing techniques of important ferro-alloys, followed up by their production on pilot plant scale.

(i) *Production of Carbon-free Ferro-chrome* — Based on the systematic studies on the production of carbon-free ferro-chrome by aluminothermic reactions, a further quantity of about 400 kg. of the alloy was produced. The earlier experiments were carried out by hand charging and a bunker was subsequently designed for mechanical charging during the reaction. This enabled only two persons to attend to the production, whereas formerly about half a dozen people were required for continuous charging of the reaction mixture.

Systematic studies of the various factors involved in the production of carbon-free

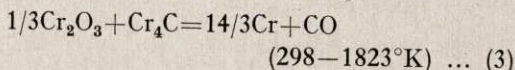
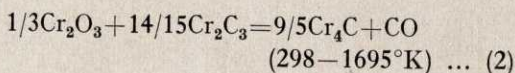
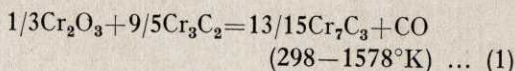
ferro-chrome were completed. The factors studied included the effect of particle size, rate of feed, preheating temperature, quantity of reducing agent, energizers, fluxes, size and shape of vessel, etc. It was observed that a minimum quantity of 15 per cent of energizers was required for the chromite having Cr:Fe ratio as 2.1:1 with 54.5 per cent Cr_2O_3 content. The metallic recovery increased more with increasing quantity of aluminium powder than that theoretically required for producing metal to desired specifications without any aluminium. Preheating the charge improved the recovery of metallic values. The maximum recovery was obtained when chromite ore crushed to -40 mesh B.S.S. sieve was used. The addition of 10 per cent lime was found to result in better separation and slag. Conditions for the production of carbon-free ferro-chrome were standardized and an alloy containing Cr, 75-77; Fe, 20-25; Al less than 0.5; Si, 1.2-5 and C, traces was produced on large scale. The investigation was completed.

(ii) *Studies on Production of Ferro-titanium* — Systematic studies on the production of ferro-titanium by aluminothermic reactions were undertaken and the effects of particle size, different energizers, rate of feeding were studied. It was observed that preheating of the charge and vessel was essential for good recoveries. In experiments, so far carried out, the recovery of titanium was only 25-32 per cent while the recovery of iron was more than 90 per cent. Further experiments are in progress to increase the recovery of titanium and to produce the alloy to the desired I.S.I. specification containing 40 per cent titanium.

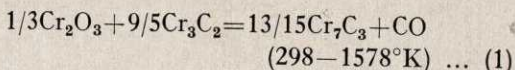
(iii) *Production of Carbon-free Ferro-manganese* — Preliminary experiments on the standardization for the production of carbon-free ferro-manganese were carried out and about 27 kg. of alloy was produced.

(iv) *Production of Low-carbon Ferro-chrome from High Carbon Ferro-chrome; Free Energy Changes in the Interaction of Carbides and Oxides* — In the production of

low-carbon ferro-chrome from high-carbon ferro-chrome it has been observed that the carbon cannot be decreased to below 1.5-2.00 per cent under normal working temperature. Free energy changes of the interaction of chromium carbides and oxides for the following reactions have been determined.



The free energy changes from the specific heat changes and the heat content changes for the temperature range for respective reactions are as follows:



$$\Delta C_p = -7.41 + 2.81 \times 10^{-3} T + 5.69 \times 10^5 T^{-2}$$

$$\Delta H^\circ = +68603 - 7.41T + 1.4 \times 10^{-3} T^2 - 5.69 \times 10^5 T^{-1}$$

$$\Delta F^\circ = +68603 + 17.06T \log T - 1.41 \times 10^{-3} T^2 - 2.84 \times 10^5 T^{-1} - 73.81T$$

$$1/3\text{Cr}_2\text{O}_3 + 14/15\text{Cr}_2\text{C}_3 = 9/5\text{Cr}_4\text{C} + \text{CO} \quad (298-1695^\circ\text{K}) \dots (2)$$

$$\Delta C_p = -2.95 - 0.01 \times 10^{-3} T + 1.48 \times 10^5 T^{-2}$$

$$\Delta H^\circ = +75076 - 2.95T - 0.005 \times 10^{-3} T^2 - 1.48 \times 10^5 T^{-1}$$

$$\Delta F^\circ = +75076 + 6.794T \log T + 0.005 \times 10^{-3} T^2 - 0.74 \times 10^5 T^{-1} - 52.64T$$

$$1/3\text{Cr}_2\text{O}_3 + \text{Cr}_4\text{C} = 14/3\text{Cr} + \text{CO} \quad (298-1823^\circ\text{K}) \dots (3)$$

$$\Delta C_p = -4.77 + 3.91 \times 10^{-3} T + 2.1 \times 10^5 T^{-2}$$

$$\Delta H^\circ = +81951 - 4.77T + 1.96 \times 10^{-3} T^2 - 2.1 \times 10^5 T^{-1}$$

$$\Delta F^\circ = +81951 + 10.98T \log T - 1.95 \times 10^{-3} T^2 - 1.05 \times 10^5 T^{-1} - 58.62T$$

The calculated free energy values for the above reactions are tabulated in Tables 21 and 22.

TABLE 21 — STANDARD FREE ENERGY CHANGES FOR THE DECARBURIZATION OF CHROMIUM CARBIDES

TEMP. (°K)	REACTION (1)	REACTION (2)	REACTION (3)
1000	+44295	+42747	+54226
1100	+42479	+39845	+51757
1200	+40765	+36962	+49296
1300	+39083	+34117	+46835
1400	+37347	+31263	+44365
1500	+35711	+28457	+41894
1600	—	+25642	+39394
1700	—	+22877	+36296
1800	—	—	+34434

TABLE 22 — STANDARD FREE ENERGY CHANGES FOR THE REACTIONS (4a) AND (4b)

TEMP. (°K)	REACTION (4a)	REACTION (4b)
1900	+3720	—
2000	+530	—
2100	—2630	—
2173	—5010	—
2200	—	—6070
2273	—	—9230

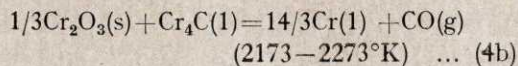
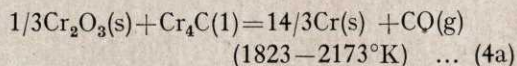
TABLE 23 — EQUILIBRIA CARBON MONOXIDE PRESSURES FROM FREE ENERGY CHANGES OF REACTIONS (1) TO (3)

CO PRESSURE IN atm.	TEMPERATURE IN (°C.)				
	0.01	0.1	0.25	0.5	1.0
Reaction I	957	1079	1136	1182	1283
Reaction II	1244	1410	1485	1545	*
Reaction III	1337	1501	1573*	†	†

*Probably above the melting point of Cr_4C .

†Probably above the melting point of $(\text{Cr}_4\text{C-C})$ eutectic.

The free energy changes for the reaction of the lowest carbide of chromium at temperatures higher than 1823°K as are tabulated below:



The equilibria CO pressures derived from free energy change of the reactions (1) to (3) are indicated in Table. 23

It will thus be observed that under normal oxidizing fluxes, and normal working temperatures, in an arc furnace, it was not possible to remove the carbon below 1.5-2 per cent unless the partial pressure of CO was reduced either by flushing with an inert gas or the reaction was carried under reduced pressures.

59.0 Production of Magnesium from Dolomite

This investigation was taken up to produce magnesium by silico-thermal reduction of dolomite. The pure magnesium so obtained would find varied applications especially in structural light alloys.

A small 2 in. dia. tubular furnace was set up and laboratory-scale trials were undertaken. A mechanism was incorporated for introduction and withdrawal of the briquetted sample into and away from the hot zone under vacuum. Experiments were carried out at 1100°C . and it was observed that 85 per cent of the magnesium could be recovered in a period of less than 2 hours. Various factors so far studied included particle size of reactants, use of catalysts, briquetting pressure, addition of calcium oxide, use of different grades of ferro-silicon and addition of alkalis and their effect on the deposit. The rate of reaction at different pressures was also studied. It was observed that the optimum particle size for ferro-silicon and calcined dolomite are —170 and —100 mesh B.S.S. respectively. The use of catalysts up to 1 per cent was beneficial for the reaction and ferro-silicon in quantities greater than equimolecular proportion was found necessary for the better recoveries. High recoveries were obtained when a better grade of ferro-silicon was used. A few experiments of one pound assembly were carried out and the details of the pilot plant are being worked out.

59.1 Production of Magnesium Powder

The investigation was taken up to produce and supply magnesium powder to the High Explosives Factory at Kirkee of the Ministry of Defence, Government of India, for their use in defence requirements.

The magnesium powder produced by milling the ingot turnings was found to conform to the bulk density requirements and the specification for the particle size. It was observed that during milling, some of the magnesium was always oxidized. The powder so produced thus could not conform to the specification so far as magnesium content is concerned. The possibilities of grinding larger quantities and the use of inert atmosphere were examined and the specification for the unit has been worked out.

60.0 Solubility of Nitrogen in Plain Carbon and Alloy Steels

A study was taken up with a view to determine the solubility of nitrogen in nickel-free austenitic chromium-nitrogen-manganese-copper stainless steel in solid and liquid state.

The set-up for the determination of the solubility of nitrogen was further modified to decrease the errors in the system, and volume of the unit was decreased and the gas burette was enclosed in a constant-temperature water bath thus avoiding any fluctuations in the volume of gas introduced in the system. In order to check the accuracy of apparatus and the reproducibility of the results, several experiments were carried out with samples of mild steel, 18-8 stainless steel in the form of thin sheets of 0.014 in. thickness and pure iron wire at 1000°C. It was found that the purity of the commercial nitrogen after its purification for the removal of oxygen and moisture by familiar laboratory methods was not sufficient since the samples were coated with oxide film, thereby preventing the complete nitrogen absorption.

The purification assembly for the removal of oxygen from nitrogen was modified and the gas was lastly passed over powdered chromium at 900°C. Further experiments on the solubility of nitrogen in 18-8 stainless steel were undertaken.

61.0 Studies on the Production and Recovery of Alumina from Indian Bauxite

Systematic investigations were taken up on the studies of Indian bauxites for their suitability for alumina by Bayer's process. The properties under study included relative grinding characteristics, mineralogical and chemical composition and recovery of alumina by caustic soda leaching. Three samples from Lohardaga, Kaira and Katni were received. The Lohardaga sample was found to contain 90 per cent of total alumina as gibbsite, 2.5 per cent as boehmite, and 4.8 per cent as kaolinite and 3.0 per cent as diasporite. Kaira and Katni deposits contained 67-60.5 per cent of total alumina as gibbsite. The percentage composition of the other minerals of the deposits were investigated. The ball mill grindability rate tests indicated that Lohardaga sample was relatively easier to grind than the Kaira and Katni deposit. The leaching characteristics of these samples would also be studied.

62.0 Utilization of Red Mud for the Recovery of Metallic Values

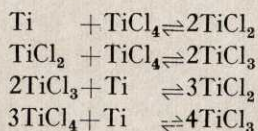
The red mud containing Fe_2O_3 , TiO_2 and unrecovered alumina is a waste product of the aluminium industry and considerable quantities are discarded every year. Investigations were taken up for the utilization of red mud for recovering aluminium and other metallic values.

For the recovery of alumina, investigations were taken up by roasting with soda ash and limestone at 1050°-1100°C. Roasted product was leached and the alumina subsequently precipitated from the solution. It was

found possible to recover about 90-92 per cent of alumina. The effects of various factors such as the quantity of reactant, temperature of roasting, particle size, etc., were under investigation.

63.0 Catalytic Distillation of Titanium from Titanium-bearing Alloys

Aluminium-titanium alloys containing 55-60 per cent titanium can be made by aluminothermic reactions in the presence of energizers. It is proposed to study the recovery of titanium from the aluminium-titanium alloys by chlorination with TiCl_4 and AlCl_3 . The reaction of TiCl_4 with titanium and the disproportionation of subhalides produced may be represented as follows:



It is proposed to study initially the reaction between titanium and titanium tetrachloride and suppress the side reactions. The partial pressure of TiCl_4 at various temperature required to suppress the formation of TiCl_3 was calculated from thermodynamic data. An assembly for the reaction of a gas mixture containing titanium tetrachloride at different vapour pressures with titanium turnings was set up. Argon gas was used as a carrier for titanium tetrachloride vapours. An assembly for the purification of titanium tetrachloride and for transferring the same to the reacting unit without contamination with moisture was designed and fabricated. The crude titanium tetrachloride was purified and distilled to a colourless liquid. Fifteen trials were carried out to study the effects of temperature and of the rate of flow titanium tetrachloride vapours on the reaction rate. At higher temperatures, the refractory tube (silica) was found to be attacked and cor-

dierite, a magnesium-aluminium silicate, is being tried.

64.0 Production of Aluminium Powder

For the production of ferro-alloys by aluminothermic reactions, 45 mesh (B.S.S. sieve) aluminium powder serves the requirements. Systematic studies on production of aluminium powder to desired specifications using either aluminium ingots or scrap was undertaken.

Preliminary experiments were carried out to produce powder to requisite specifications either by agitation or blowing compressed air on a molten stream of aluminium. Agitation of molten aluminium did not yield very satisfactory results as during the solidification, violent agitation was required to prevent coalescing of particles of aluminium. Further experiments were carried out by atomizing the molten aluminium. The molten aluminium was allowed to flow and was atomized by compressed air. The powder was collected in a water tank, dried and used for ferro-alloy production in small scale. It was observed that the outlet tube got clogged due to cold air blast and further work was undertaken to eliminate this handicap.

65.0 Measurement of Carbon Activity in Iron and Its Alloys

Under the Council of Scientific & Industrial Research Fellowship Scheme, work was initiated on equilibrium study between carbon dissolved in iron and definite mixtures of carbon monoxide with carbon dioxide which is of considerable importance in order to gain an exact knowledge of the phase boundaries that existed in Fe-C system, activity of carbon in different regions of the system, heat of solution of carbon in different solid solutions, heat of transfer of carbon from the gas mixture to the iron, heat of transformation of the different forms of iron as well as the nature of the crystal structure, etc.

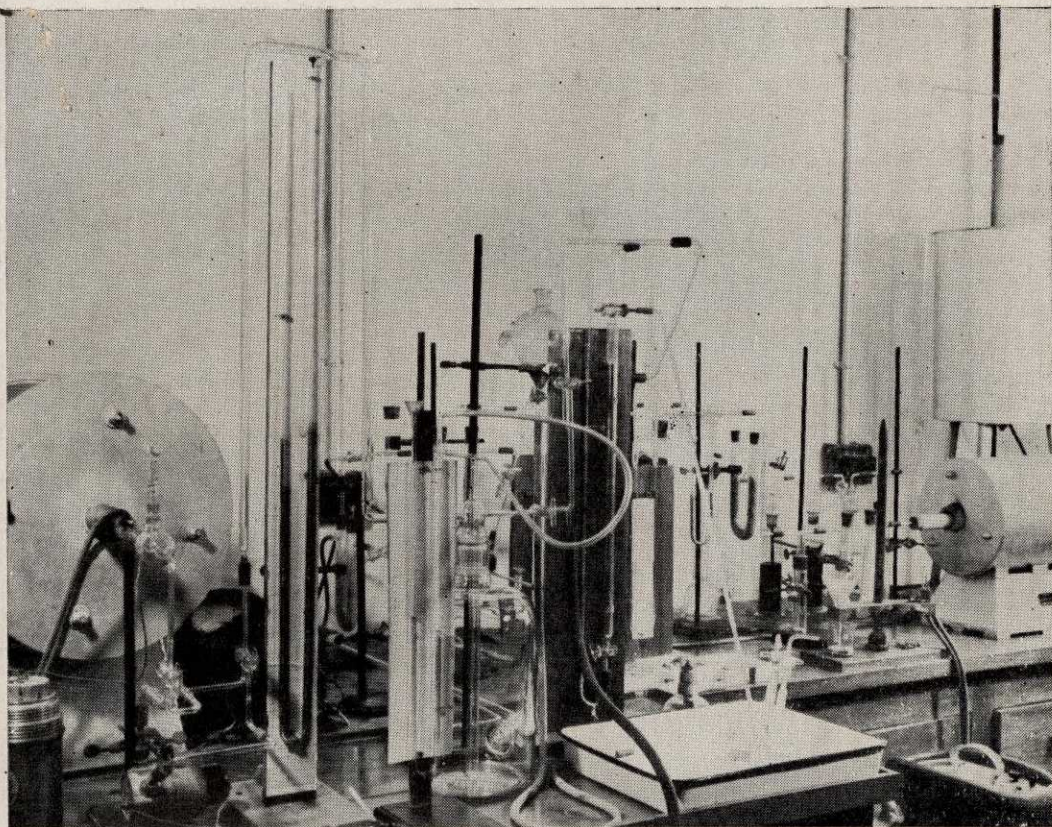
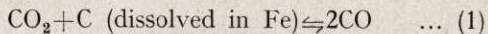


FIG. 24 — SET-UP FOR MEASUREMENT OF CARBON ACTIVITY IN IRON AND ITS ALLOYS

The fundamental equation involving equilibrium is



If graphite is chosen as the standard state for carbon, then

$$a_c = \frac{P^2_{\text{CO}}}{P_{\text{CO}_2}} K_2 \quad \dots (2)$$

where a_c is the activity of carbon dissolved in iron at the temperature of experiment, $P^2_{\text{CO}}/P_{\text{CO}_2}$ is the ratio of the gas mixtures at equilibrium and K_2 is a constant derived from the equilibrium of gas mixtures existing with graphite at that temperature.

A knowledge of the ratio of the two gas mixtures at equilibrium and the constant K_2 would lead to evaluate the activity of carbon dissolved in iron or its alloys.

In the field of binary solution involving the solid phase and the gas mixtures of methane-hydrogen or carbon monoxide-carbon dioxide, H. Schenda, H. Holtey and later on R. P. Smith came forward to investigate. Procedures adopted to investigate the equilibrium by them were almost the same. Recently H. Schenda and H. Keiser in Aachen adopted a slightly different procedure than the previous one. The method chosen for studying the equilibrium in the present work basically tallied with that of earlier workers.

A closed vacuum tight circuit system was used for determining the equilibrium. The assembly consisted of a platinum wound furnace, circulating pump, flow meter, a reservoir of capacity about 5000 c.c. and a

gas-measuring burette for sampling the gas samples. The temperature of the furnace was accurately maintained to $\pm 5^{\circ}\text{C}$. with the help of a chopper bar type temperature regulator and is recorded by a temperature recorder. Gas mixture was analysed by an Ambler's gas analyser. The iron sample was kept over a previously weighed porcelain boat and placed inside the reaction tube held at a constant temperature through which circulated a stream of the fixed gas mixture. The sample was kept inside the furnace until equilibrium was attained with a particular gas mixture; the sample was then quenched and its carbon content was determined. Sampling of the gas mixtures for analyses (about 5 c.c.) from the vacuum tight circulation system was done while the experiment was in progress.

(i) *Iron Samples* — For initial experiments, the iron wire used was G.R. piano wire and subsequently BISRA'S pure iron of about 5 mm. thickness was used. The approximate size of each specimen was 1.50 cm. and 1 cm.

(ii) *Preparation of Constant Gas Mixture* — The carbon monoxide was prepared by passing CO_2 over heated graphite. The purified $\text{CO} + \text{CO}_2$ were separately passed through calibrated flow-meters and mixed in a reservoir. The gas mixture was circulated to ensure homogeneous mixture.

(iii) *Temperature Control* — Temperature of the furnace was controlled with the help of a chopper bar type temperature regulator. The temperature of the specimen was noted from the thermocouple placed exactly above the middle of the sample in a porcelain boat. The temperature was automatically recorded on a temperature recorder.

(iv) *Period of Equilibrium* — The period of equilibrium depended on many factors like rate of circulation of the gas mixture, diffusion of carbon from the gas phase to the solid phase and the rate of diffusion of carbon through iron, etc. Initial experiments were carried out to determine the approximate equilibrium period and it was observed that

equilibrium was reached within 72-96 hours, depending on the carbon content.

(v) *Analysis of the Iron Samples* — Samples after equilibrium were analysed for iron by the combustion method. From the preliminary experiments, it was observed that either the circulating pump did not work efficiently or carbon deposition had taken place. All gas circulating pump was designed and fabricated. Attempts were made to use a fuel pump for the circulation of gases over the system which was found to work efficiently. The results of the equilibrium carbon obtained for varying gas mixtures are indicated in Table 24.

TABLE 24 — EQUILIBRIUM CARBON CONTENT OF IRON SAMPLE

	WEIGHT OF SAMPLE	$\text{P}^2\text{CO}/$ PCO_2	FINAL CARBON CONTENT % by wt	DURATION OF EQUI- LIBRIUM hr.
1.	0.4206	2.200	0.04	72
2.	1.1986	1.177	Trace	48
3.	1.0178	1.938	0.01	48
4.	1.0092	5.780	0.07	48
5.	1.0948	7.452	0.10	48
6.	1.1248	40.740	0.96	48

Further experiments would be carried out using varying gas mixtures up to the carbon saturation in austenite for the temperature 1000° , 1100° , 1200°C . and above. The activity of carbon in chromium alloy would also be studied after the activity of carbon in iron had been completed.

66.0 Development of Electrical Resistance Alloys for Heating Elements

Conventional types of heating elements used for domestic or industrial heating appliances contain high percentages of nickel and some cobalt. Resources of nickel and cobalt hardly exist in India. There is considerable consumption of nickel and cobalt containing heat-resistance elements in India for domestic and industrial heating application. It is

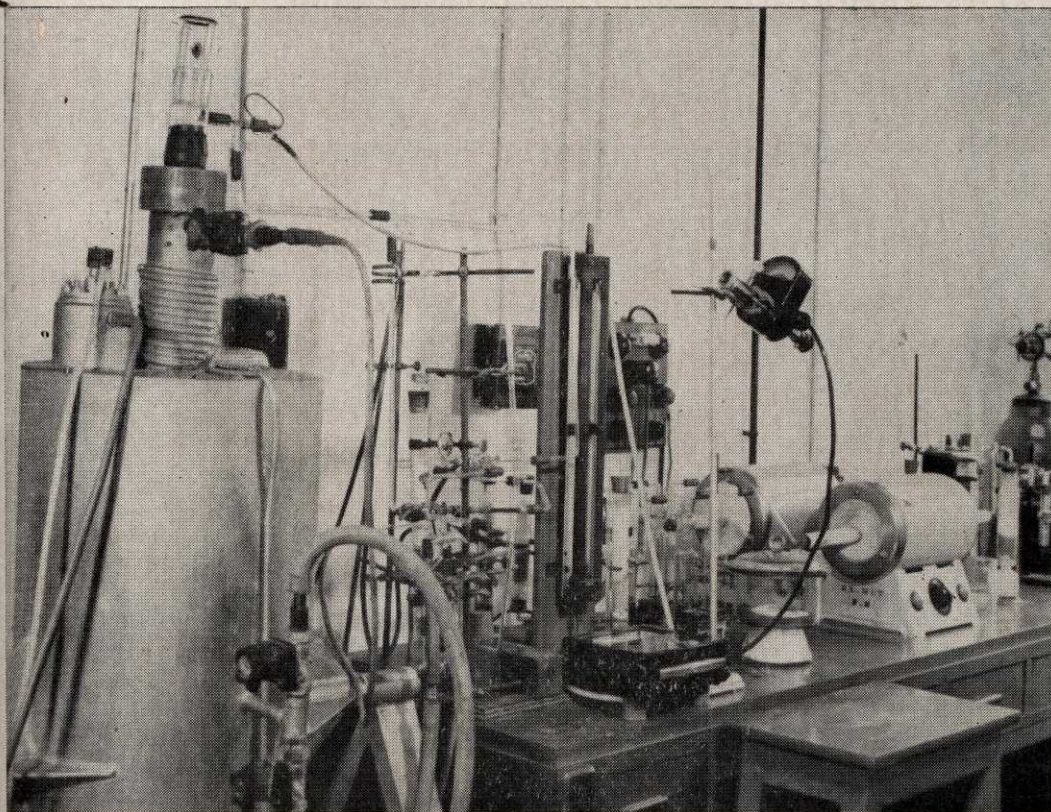


FIG. 25 — SET-UP FOR DETERMINATION OF CARBON SOLUBILITY IN IRON-CHROMIUM-SILICON ALLOYS

also obvious that with increase in availability of electric power and industrial growth, the demand for such heat-resistance electrical elements will substantially increase. With a view to develop electrical heat-resistance elements containing no nickel or cobalt, intensive research and development work was taken up at the National Metallurgical Laboratory leading to the development of indigenous based electrical heating elements.

Desirable characteristics that are needed for the finished product of electrical heating element are the following:

- (i) High electrical resistivity and low temperature coefficient of resistivity.
- (ii) Good resistance to scaling and oxidation above the red heat temperatures.
- (iii) Low coefficient of thermal expansion.

- (iv) Comparatively high melting point and low melting range.
- (v) High temperature strength and stability of structure at elevated temperatures.
- (vi) Low specific gravity.
- (vii) Good cold drawability of the material to enable final cold drawing operation to produce fine gauges of the wire.

It has been established that Cr and Al, when used as alloying additions to Fe, increase both the scaling resistance as well as its electrical resistivity of the heat-resistance elements. Al has been found to exert a more pronounced effect than Cr. These considerations led to the adoption of Fe-Cr-Al based heating elements in various countries. One of the chief difficulties with

this type of alloy is their strong tendency towards grain growth during use at elevated temperatures, rendering the material thereby weak and brittle. So at each stage of hot working, extreme care has to be exercised in controlling the temperature and amount of working closely following predetermined mechanical working and heat-treatment cycles. Furthermore, extremely deleterious effects of calcium and other impurities and inclusions on scaling resistance and workability of these special alloys necessitated their exclusion and proper control by using selected base melting charge and taking recourse to precision melting and casting techniques. Pouring temperature exercised considerable influence on the hot workability of the alloy ingots.

Use of manganese within certain amount has been found to considerably improve working without deleterious effect on scaling resistance. Titanium and zirconium in small amounts also help to inhibit grain growth during high temperature service of the heating elements.

Fe-Cr-Al based heating elements suffer from the following known defects which need rectification:

- (i) Very low strength at high temperature.
- (ii) Brittleness due to undue grain growth.
- (iii) Poor creep characteristics leading to sagging of the heating elements in service when used in unsupported condition.
- (iv) Poor workability.

Successful development of Fe-Cr-Al alloys of optimum composition based essentially on Indian alloying elements aims at eliminating or otherwise reducing the above-mentioned defects by suitable additions of minor indigenous alloying elements besides improving and standardizing the melting, casting and fabrication techniques. Cobalt additions up to 3.5 per cent improves high temperature properties of these electrical heat-resistance alloys. Cobalt, however, is non-existent in India. Zirconium and titanium,

on the other hand, are also known to improve high temperature properties by reducing grain growth. Resources of the ores of zirconium and titanium are abundant in India. Manganese is known to improve fabrication properties though slightly impairing high temperature oxidation resistance.

The present work is aimed at studying the effects of various indigenous alloying elements in optimum combination to obtain maximum ease in fabrication and final requisite high temperature properties and service life. Detailed study was also made to formulate optimum melting and casting techniques. Chromium and aluminium additions were made either as low-carbon ferro-chromium and aluminium metal lumps or a master alloy of chromium and aluminium. Melting, finishing and pouring temperature, forging, hot and cold workability are some of the factors exhaustively examined during these comprehensive investigations. Over thirty experimental heats were made with chromium contents varying from 10 to 25 per cent, aluminium 3 to 8 per cent and balance iron besides making minor additions of indigenous alloying elements such, as manganese, silicon, zircon, vanadium, titanium, boron, etc., with a view to improve mechanical workability, high temperature strength and reduce brittleness due to grain growth of these alloys.

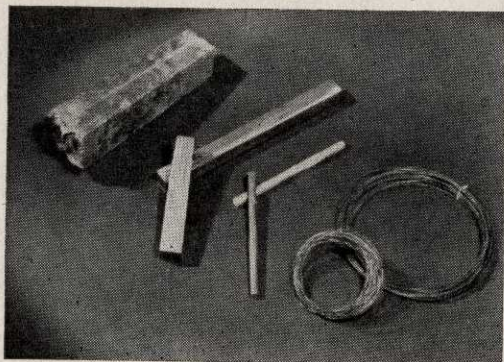


FIG. 26 — STAGES OF PRODUCTION OF HEATING ELEMENTS

During wire drawing, it was noticed that lubrication and the coating given on the wire rod exercised considerable influence on the drawability of these materials. Suitable precoat as well as lubricant in drawing iron-chromium-aluminium alloys were worked out. It was noticed that under suitable drawing conditions, the wire could be drawn 40-50 per cent prior to annealing operation. Successive stages of work from ingot to finished work are illustrated in Fig. 26.

Electrical resistivity of these elements was found to be much higher than that of nichrome. Comparative figures are given in the following table:

TABLE 25 — COMPARATIVE DATA OF ELECTRICAL RESISTIVITY

MATERIAL	ELECTRICAL RESISTIVITY micro-ohm/cm.
E 4 (Experimental alloy)	141.1
E 20 "	128.5
E 28 "	128.5
E 31 "	136.0
Nichrome	128.4
Kanthal D	131.1

The temperature coefficient of resistivity was also found to be not high, particularly in high temperature regions. For conducting accelerated life tests on these heat resistance elements, equipment was designed and fabricated at the National Metallurgical Laboratory for testing the heating elements to standard A.S.T.M. rigid specifications.

Accelerated life tests of new alloys developed at the National Metallurgical Laboratory as well as conventional resistance alloys such as nichrome and kanthal were carried out at various temperatures. The results of life tests based on some preliminary experiments are given in Table 26.

The results in Table 26 indicated that some of the experimental heats possessed excellent properties for use as heating elements and could successfully replace some imported alloys such as nichrome and kanthal alloys.

TABLE 26 — RESULTS OF ACCELERATED LIFE TEST

ALLOY	TEST TEMP. °C.	ACCELERATED LIFE TEST hr.
Nichrome	1200	67
Experimental heat No. 6	1200	94
Experimental heat No. 31	1200	80
Experimental heat No. 31	1150	200 (still continuing)

67.0 Development of Low-alloy High-tensile Structural Steel

This broad-based investigation was taken up at the National Metallurgical Laboratory with the object of developing suitable low-alloy structural steels possessing good mechanical properties, working characteristics, corrosion resistance and weldability. The main aim of the project is to find out suitable compositions for low-alloy high-tensile steel utilizing mainly indigenous alloying elements.

In the first phase, the work was concentrated in making laboratory scale heats of existing low-alloy high-tensile steel with slight modification of the percentages of alloying elements. This was done with a view to standardize the conditions of the laboratory-scale production of high-tensile steels. Main alloying elements of the first series were manganese, chromium, silicon and copper.

In the next series, additions of small percentage of vanadium and titanium were made to harden the ferrite as well as to improve the grain structure. Additions of aluminium and zirconium were also made with the idea of getting finer grain size. In this series basic composition was kept within the following limits, viz. C, 0.1-0.15; Mn, 1.1-0.8; Cr, 0.6-1; Cu, 0.25-0.4 and Si, 0.3 per cent max. Addition of vanadium of the order of 0.1-0.2 per cent was found to strengthen the steel considerably. Almost similar strengthening was obtained by addition of titanium.

A number of heats were made in the high frequency furnace as given in Table 27. The total charge was 10,000 gm. in all the cases and every time two 5 kg. ingots were made out of the charge. The ingots were hot forged at 1150°C. to $\frac{1}{2}$ in. square bars for making tensile and Charpy impact test pieces and hot rolled to $\frac{1}{4}$ in. and $\frac{1}{16}$ in. thick plates for making weldability and corrosion test pieces respectively. The hot worked pieces were given normalizing treatments before making the test pieces. Tensile tests, hardness measurement after various heat treatments, impact properties at room as well as at sub-zero temperatures were conducted on all the heats and the results are given in Table 28.

One of the heats of low-alloy steel showed excellent mechanical properties. Weldability test based on Reeve's method (miniature) were also made with a number of steels. Other weldability tests are in progress. The corrosion resistance of this steel is expected to be good as the copper and chromium content is quite high. The corrosion tests were comprehensively planned and started.

68.0 Development of Thermostatic Bi-metals

Thermostatic bi-metals are temperature sensitive elements that are widely used in a

great variety of ways for indicating, regulating and controlling temperature, and as thermal relays, circuit breaker and overload protection devices. With the increase of electrical industry in India, need for bi-metal is increasingly felt but still the entire requirement is met by import. This project was, therefore, undertaken to develop suitable manufacturing technique for different types of bi-metals.

Stress was laid on the following aspects of the project: (i) development of high expansion Mn-Cu-Ni alloys; (ii) improvement in working technique of the invar-group alloys; (iii) bonding of brass with invar; (iv) bonding of Mn alloy with invar; and (v) improvement in the bonding of all ferrous bi-metals.

(i) *Development of High-expansion Mn-Cu-Ni Alloys* — Mn-Cu-Ni alloys containing 70-80 per cent Mn are well known for possessing extraordinary high thermal expansion combined with good elastic properties and high electric resistivity. Coefficients of thermal expansion of five alloys of this group were determined at different temperatures up to 400°C. A sudden rise in the value of the coefficient at 250°-300°C. was observed and the change was reversible. On the basis of the thermal expansion characteristic, the alloys Mn, 75; Ni, 15; Cu, 10 per cent and Mn, 72; Ni, 10; Cu, 18 per cent were selected. Further work was carried out to evaluate the

TABLE 27 — CHEMICAL COMPOSITION OF HEATS MADE

HEAT No.	C %	Mn %	Cr %	Si %	S %	P %	Cu %	V %	Ti %	Ni %
28	0.04	0.46	0.55	0.043	0.019	0.014	0.560	—	—	—
29	0.06	0.62	0.72	0.007	0.021	0.013	0.012	—	—	—
21/6	0.06	0.97	1.06	0.048	0.013	0.015	0.460	—	—	—
21/7	0.04	1.40	1.06	0.085	0.016	0.016	0.400	—	—	—
21/6R	0.08	1.10	0.81	0.040	0.040	0.026	0.370	Trace	0.14	—
21/6R2	0.15	2.40	1.50	1.800	—	—	0.150	0.10	—	—
21/6R3	0.06	2.21	1.08	0.400	0.028	0.028	0.460	0.20	—	0.22
21/7R	0.05	2.27	1.02	0.400	0.028	0.030	0.320	—	—	—
30	0.33	2.40	1.49	1.860	—	—	0.120	0.15	0.06	—
31	0.08	1.20	0.95	0.020	—	—	0.310	0.10	—	—
32	0.11	1.98	1.09	0.260	—	—	0.320	—	0.06	—
33	0.33	2.08	1.13	0.940	—	—	0.140	0.15	—	—
32/1	—	—	—	—	—	—	—	—	—	—
32/2	—	—	—	—	—	—	—	—	—	—

TABLE 28 — MECHANICAL PROPERTIES OF HEATS MADE

HEAT No.	TENSILE TEST RESULTS				HARDNESS VALUES (VPH) AFTER				CHARPY IMPACT VALUES (ft./lb.) AT				BEND TEST RESULTS AND REMARKS
	Max. S T.S.I.	Y.P.T. S.I.	R.A. %	Elong. %	Anneal- ing	Normal- izing	Oil Q	Water Q	Room temp.	0°C.	-20°C.	-40°C.	
28	29.16	27.36	70.4	40.6	103	163	216	197	140	152	130	52	1/8 in. thick sheet withstood 3 reversed bend over 2.5 mm. radius and withstood 130° flat bend.
29	27.50	21.38	74.0	37.5	93	171	223	223	152	102	110	36	
21/6	31.31	28.08	75.2	39.0	119	216	232	241	—	—	—	—	
21/7	32.65	—	70.8	37.5	130	212	285	286	100	76	50	43	do
21/6R	30.40	—	65.9	37.5	—	—	—	—	106	76	46	—	
21/6R ₂	92.00	—	36.0	10.0	400	348	459	470	—	—	—	—	
21/6R ₃	68.50	—	64.0	21.0	241	309	—	—	52	42	40	61	1/8 in. thick sheet withstood the bending up to 90° with 5 mm. radius, when the bend was fur- ther increased to 130° fine crack occurred.
21/7R	—	—	—	—	—	—	—	—	56	—	—	—	
30	Test pieces could not be made	—	—	—	266	635	661	—	42	58	38	51	
31	49.95	—	48.7	21.9	143	227	295	349	—	—	—	—	0.055 in. thick sheet withstood the bending up to 90° with 2.5 mm. radius, when the bend was further increased to 130° fine crack occurred.
*32	69.69	—	54.4	15.6	264	331	383	393	—	—	—	—	
33	Test pieces could not be made	—	—	—	381	429	487	610	—	—	—	—	
32/1	—	—	—	—	140	282	286	329	—	—	—	—	*0.055 in. thick sheet withstood the bending up to 90° with 5 mm. radius, when the bend was further increased to 130° fine crack occurred.
32/2	—	—	—	—	159	271	314	337	—	—	—	—	
34	—	—	—	—	200	247	362	375	—	—	—	—	

mechanical properties of these alloys at different temperatures.

Improvement was made as regards the casting and working technique of these alloys. Extraordinary high casting shrinkage, high viscosity of the molten metal, formation of thick scum on the surface are the troublesome factors. Large hot top was used to avoid piping. Various deoxidizers were tried, and Al and Ca was found to give good result. Silicon was to be rejected as it induces hot-shortness. Hot working temperature of these alloys is in the range of 850°-900°C.

(ii) *Improvement on Hot Working of Low-expansion Alloys* — As the invar group of alloys has very low expansion at low temperature and as their expansivity increase very rapidly with temperature, the ingots were heated very slowly in order to avoid very high thermal stresses. Soaking was given for a considerable period in the temperature 800°-900°C., and hot-worked at temperature 1100°-1000°C. Extreme care was necessary during the forging of these alloys to avoid cracking.

(iii) *Bonding of Brass with Invar* — Several attempts were made to bond brass with invar. A few attempts were made to cast brass on invar with no apparent success. However, brass was successfully bonded by hot pressing (at 840°C. followed by hot rolling at

800°C.) a 4-ply pack. Much more work is to be done to improve the technique.

(iv) *Improvement in bonding of Mn-Alloys with Invar* — Extensive trials were made to bond Mn-alloy with invar. Initially low power of the rolling mill was a handicap. Much improvement could be made by using the hydraulic press and the new rolling mill. Excessive oxidation of the bonding surface during peripheral welding was responsible for the earlier failures. By using slabs of larger dimensions and improving the welding technique, cent per cent bonding could be achieved. Bonding was effected by 4-ply method by pressing at 920°C. followed by hot rolling at 900°-850°C.

(v) *Improvement in the Bonding of All-Ferrous Bi-metals* — Bonding of invar alloys with high expansion Fe-Ni-Mn alloys or Fe-Ni-Cr-Mn alloys is comparatively easy. Much improvement was attained by pressing the composite at 1200°C. followed by hot rolling at 1100°-1000°C. Both 4-ply method appeared to be economic and simple. After hot rolling sheets were pickled in 20 per cent HCl solution at about 70°C. In the case of high sensitive bi-metals no pickling could be done as the Mn-alloy was vigorously attacked even by dilute acids. At present oxide was removed by mechanical rubbing with sand and grinding. By covering high expansion side with wax, invar side may be pickled with HCl solution. After pickling, sheets were given about 50 per cent cold reduction (or more) and recrystallized at 650°-700°C. Cold reduction and annealing was repeated until final thickness was obtained. Ordinarily, grain size number obtained was greater than 6. Except for special cases, bi-metal sheets were given 30-40 per cent cold reduction followed by stress relief annealing at 350°C. Stress was, therefore, laid on the following types of bi-metals: (i) general purpose bi-metal used up to (a) 200°C., (b) 350°C., and (ii) high sensitive bi-metals. Further work under way related to the study of mechanical properties of these alloys at higher temperature.

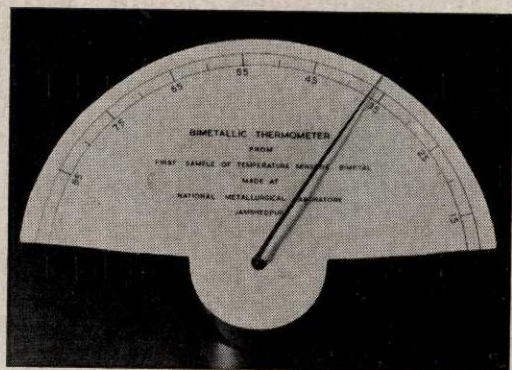


FIG. 27 — BI-METALLIC THERMOMETER

69.0 Development of Controlled Friction Materials

The investigation was taken up at the instance of the Ministry of Railways to develop a suitable combination of material which will give a coefficient of friction not more than 0.16 in long service under repeated sliding condition.

It was observed previously that the stearic acid soaked process of fabric liner versus phosphor-bronze gave higher coefficient of friction than that in service test though in laboratory test the result was satisfactory. The failure of the material was attributed to the high ambient temperature in service causing melting and breakdown of the lubricating film, giving thereby high friction. Therefore, the work was channelized in another direction of using a solid lubricant stable under these conditions. Two lines of attack were followed:

- (i) Use of graphite or P.T.F.E. powder mixed in various proportions from 10 to 30 per cent by volume with bakelite powder with and without asbestos and sintering the compact mass under pressure; and
- (ii) use of $\text{PbO} + \text{SiO}_2 + \text{FeO}$ coating on mild steel base bonded by sintering at temperature of 900°F . under a neutral atmosphere.

In the first method using the compound buttons with grey cast iron, experiments at 300 lb./sq. in. pressure showed that friction did not rise beyond 0.16 μ even after long time of run. The buttons were found to possess sufficient compressive strengths. Graphited buttons showed higher friction of 0.2 μ initially rising to 0.26 μ or more after only a short run. Further experiments were conducted to standardize the process of making the compact and also to try with only surface coating. In the second method of attack, initially prepared specimens showed poor bonding of the coating which peeled off during test. Experiments were carried out to exclude FeO and bond only $\text{PbO} + \text{SiO}_2$

to the mild steel base. It was thought that required FeO would form during the bonding operation itself and may result in sound bonding.

70.0 Internal Friction Studies

An apparatus was designed and fabricated to study the phenomena such as strain ageing, temper brittleness, diffusion of interstitial solutes like carbon and nitrogen in α -iron through the measurement of internal friction.

The apparatus consisted of the specimen in the form of a wire. The upper end of the wire was held by a pin-chuck made into extension rod which was clamped rigidly with the main body of the frame and from the lower end of the specimen was suspended the inertia bar and discs. The design of the pin-chucks and the inertia system were made in such a way as to minimize friction loss of the grips and air friction. The whole system was enclosed in a cylindrical container made of stainless steel. The upper end of the container was sealed air tight to the top extension rod holding the specimen. The lower and wider end of the container that projected down the furnace housed the inertia system and electromagnet for giving initial torque to the specimen and was fixed on the box table through a rubber ring to ensure air tight joint. The inside of the container communicated through a bottom hole to a vacuum pump to ensure the required pressure in the container. A window was provided in the container for making observations with a lamp and scale arrangement. Heating of the specimen was done by an electric furnace which was held in place with the help of a suitable frame. Temperature around the specimen was controlled by a high temperature thermostat and Sunvic regulator and the temperature of the specimen was measured by means of a thermocouple potentiometer. The frequency of vibration could be adjusted to some extent by the change of diameter of the inertia disc,

or also by changing the mass for which there is limitations; it was also conveniently adjusted by the length and diameter of the specimen itself. Cooling arrangements were provided for cooling the projecting parts of the container above and below the furnace by providing a continuous water flow through a water jacket. For setting the specimen into vibration an electromagnetic system was used. The deflecting rod made of soft iron, which was actuated by an electromagnet from the outside, is engaged initially so as to give a deflection to the specimen. On switching on the current, the electromagnet below attacked the rod and set the system into vibration.

With the help of this apparatus studies were started on the effects of cold work on damping capacity of Armco iron. In this connection, experiments were conducted using Armco iron wire specimen of 22 swg size and 6 in. length between the grips. The frequency of oscillation was 0.92 cps. Torsional strain was of the order of 10^{-5} to avoid amplitude dependent damping. Experiments were conducted at various temperatures with different amounts of cold work given to the initially annealed specimen.

71.0 Development of Clad Metals

The process of cladding aluminium on mild steel and the optimum conditions required for obtaining good bond strength was described in earlier National Metallurgical Laboratory Annual Reports. Work was carried out during the period under review on the development of bonding technique for aluminium base bearing alloys that were bonded to other suitable backing alloys for use as bi-metallic bearing. In this connection, attempts were made, in the first instance, to make a suitable aluminium-base bearing material with improved bearing properties. A large number of heats were made in the gas-fired furnace to get a suitable aluminium-base bearing metal of improved bearing properties suitable for roll cladding to be used as over-layer on hard backing material or as-cast or heat-treated condition. Various mechanical tests, viz. tensile, compression, antiseizure, etc., were carried out to determine the frictional and other properties of the alloys. Test results are given in Table 29.

The alloys made were subjected to metallographic examination which revealed hetero-

TABLE 29 — MECHANICAL PROPERTIES OF THE ALLOYS MADE

HEAT No.	ULTIMATE TENSILE STRENGTH tons/sq. in.	ELONGATION %	YOUNG'S MODULUS $\times 10^6$ lb./sq. in.	HARDNESS IN B.H.N.	COEFF. OF FRICTION (UNDER LUBRICATION)	SP. GR.	WEAR	TEST
							* †	
SBI	22.21	2.00	—	42.4	0.046	—	—	—
SB4	7.00	23.43	6.5	23.5	0.018	—	—	—
SBSI	25.70	1.56	11.4	136.0	0.040	3.6	*	†
SBS4	10.10	6.25	—	43.5	0.028	—	—	—
SB5	22.16	nil	—	129.0	0.060	—	—	—
SB6	16.00	6.25	10.3	58.6	0.060	—	—	—
SB7	6.00	nil	8.3	49.2	0.066	—	—	—
SB8	9.36	nil	—	68.0	0.100	—	—	—
SB9*	—	—	—	117.0	0.023	+	+	+
SB10	22.00	3.12	11.4	118.0	0.036	+	+	+
(SBSI-R)	—	—	—	—	—	+	+	+
SB11	10.80	3.00	11.0	82.1	0.029	+	+	+
					0.057			
SB12	19.50	1.56	9.0	136.0	—	—	—	—
SB13	23.94	1.56	11.9	126.0	—	—	—	—

*Weight loss in 80 hr..... 0.0022 gm.

†Integrated work done in kg. 80 hr..... 57690.

‡No other test could be done as the material was full of blow holes.



FIG. 28 — PHOTOELECTRIC TEMPERATURE CONTROLLER DESIGNED AND FABRICATED AT THE NATIONAL METALLURGICAL LABORATORY

geneous structures. Metallographic structure of the alloys SBS 1 and SB 4 showed improved bearing properties. The alloy SBSI revealed dendritic structure showing as-cast condition of the specimen. The structure consisted of deep etching black patches (zinc and probably lead) over a white matrix consisting of a solid solution of Cu, Mg and zinc in aluminium. Irregular flow of deep-etched black lines on the grain boundary of light-etched white matrix was observed for the alloy SB 4. The alloys SB 4 and SBSI satisfied the properties required for a good bearing flow. The alloy SB4 could also be rolled, forged and bonded with hard backing material as mild steel, following the technique developed for bonding aluminium with steel.

72.0 Photoelastic Stress Analysis

Study of distribution of stresses in engineering components is necessary from the point of view of understanding the service behaviour of the components as well as for improving the design. With this view the work on photoelastic stress analysis was started at the National Metallurgical Laboratory.

Key-hole type of notches were used in various machine parts. Quite a number of service failures were known to have originated from the root of key-hole notches. With a view to understand the behaviour of various types of key-hole notches, analysis of stresses by photoelastic method was made with notches of various geometry having different combination of depth and width in

the centre of rectangular beam of photoelastic model. The shear stress along the vertical line of symmetry for various notches was plotted against the distance from the notch from end, and maximum stress at the root of the notch was determined. In the case of narrow notches, stress distribution at the root of the notch was found to be little affected by the sharpness of the corner of the notch, and the stress at the centre of the notch was almost the same as it was in the sharp corner; but in the case of wider notches, the stress at the middle of the notch was much less than that at the sharp corner, and the distribution near the root of the notch depended upon the radius of curvature at the corner of the notch.

73.0 Wearing Characteristics of Indigenous and Foreign Grinding Media

An investigation was taken up on behalf of Messrs Dalmia Iron & Steel Company to assess the comparative wear characteristics of indigenous and foreign grinding media (Kordt & Rosch, Germany) usually used in cement mills.

Detailed studies were made in respect of their chemical analysis, visual and micro-structure examinations, hardness survey,

repeated impact tests, and actual service wear tests in an experimental ball mill using gypsum and cement clinker as ground minerals.

74.0 Development of Photoelectric Temperature-controller

Under the Council of Scientific & Industrial Research Fellowship Scheme work was carried out on the design of various types of temperature controllers. A photoelectric temperature controller was developed in this Laboratory for controlling the temperature of electric resistance furnace to an accuracy of $\pm 1.5^{\circ}\text{C}$., at temperatures ranging from to 300° to 1300°C . This controller can replace temperature controllers of foreign make with resultant economy of foreign exchange. The prototype fabricated in this Laboratory worked satisfactorily. A chromel-alumel thermocouple sensed the change in temperature in a potentiometric circuit. The change in thermo-e.m.f. deflected a mirror galvanometer. The light from the galvanometer mirror fell on a photocell and the resultant photoelectric current was amplified by a differential cathode follower direct-coupled amplifier and then was applied to a P.O. relay. This relay switched 'on' and 'off' the furnace current.

PILOT PLANTS

75.0 Low-shaft Furnace Pilot Plant Project

The smelting in the Low-shaft Furnace is normally based on the carbonization of the self-fluxing briquettes on the upper regions of the furnace, followed by their reduction in the bosh and hearth regions. The process necessitates making of the briquettes of iron ore, non-coking coal, and limestone in requisite proportions with the addition of binders like coal-tar pitch and sulphite lye. Besides, technological difficulties associated with making briquettes suitable for smelting in the furnace without the generation of prohibitive amount of dust involve additional expenditure for the binders employed. The production of 15 tons/day of pig iron in the Low-shaft Furnace Pilot Plant requires 100 tons of raw materials, which consume 8 tons of binders. In other words, 15 tons of pig iron require 8 tons of binders. The process, therefore, is uneconomical, and the smelting operation, based on such techniques, cannot obviously be exploited under commercial conditions, particularly in India. Furthermore, for the development of high temperature crushing strength, a certain amount of coking coal has to be used; the process thereby becomes dependent on coking coal and partly fulfils its objective. Attempts were, therefore, made to smelt acceptable foundry grades of pig iron with sub-standard grades of raw materials in the bedded form of the charge by suitable variation of the grain size of these ingredients and an adjustment of operational conditions such as blast volume, blast pressure, velocity of the blast, etc. It has been possible to utilize large varieties of non-coking coals

by this technique, and it would be no exaggeration to mention that by and large tremendous strides were made during the period under review to achieve the objective of attaining pig iron with non-coking coals.

During the period under review, extensive trials were conducted round the clock on three-shift basis and valuable and interesting results were obtained. Iron ores from Orissa and Bihar were smelted with large varieties of non-coking coals from Raniganj or Disergarh coalfields either as blended charge or by briquetting. The possibility of using iron ore from Chanda District with non-coking coals from Ballarpur, Kampti and Wardha (Maharashtra State) was thoroughly investigated. Besides, extensive investigations were conducted with self-fluxing briquettes made from iron ore fines, limestone and slack coals primarily with a view to utilize fine-grained run-of-mine materials. These investigations will be useful in the establishment of small regional iron and steel plants.

Smelting trials were conducted with the following objectives in view:

- (i) Variation in the fuel consumption, slag volume, daily output with gradual increase in briquettes in the burden in stage from 25 to 75 per cent.
- (ii) Variation in the fuel consumption, output and a study of the operational characteristics on smelting exclusively with briquettes. The gradual increase in blast volume blown in by increasing the effective diameter of tuyeres on the smelting characteristics.
- (iii) To assess the maximum daily production and related fuel rate with lumpy iron ore, limestone and nut coke.

- (iv) Exclusive use of non-coking coals containing high volatile and ash contents.
 (v) Smelting of iron ores from Chanda District with non-coking coals from Ballarpur, Kampti and Wardha Valley (Maharashtra State).

The analyses of the raw materials are as follows:

Analysis of Nut Coke

	%
Moisture	3.0
Ash	20.1
Volatile matter	1.8
F.C.	75.1

Analysis of Limestone

(Bisra Stone & Lime Co. Ltd., Birmitrapur, Orissa)

CaO	44.80%
MgO	3.72%
SiO ₂	6.96%

Analysis of Dolomite

(Assam/West Bengal)

CaO	31.30%
MgO	20.70%
Al ₂ O ₃	0.40%
SiO ₂	0.63%

(i) The briquettes were bonded with 4 per cent coal-tar pitch and 4 per cent sulphite lye and contained the following main ingredients:

Analysis of Coke Ash

	%
SiO ₂	52.08
CaO	3.80
Al ₂ O ₃	33.00
MgO	2.12
P ₂ O ₅	1.58
Fe	6.00

	%
Washed coking coal (Bokaro, Kargali)	41.5
Non-coking coal (Bankola, Raniganj)	20.5
Tisco dust iron ore (Noamundi)	25.0
Limestone (Bisra)	13.0

Analyses of Non-coking Coals and Coking Coals used in Briquettes

LOCATION	PROXIMATE ANALYSIS ON AIR-DRIED BASIS			
	Moisture %	V.M. %	F.C. %	Ash %
1. Bankola, Raniganj (non-coking coal)	4.0	32.00	44.56	19.44
2. Kargali, Bokaro (washed coking coal)	1.2	29.96	56.60	12.20
3. New Sitalpur (Disergarh washed coking coal)	1.5	36.30	49.30	12.90
4. Sirka (non-coking coal)	2.5	30.80	49.52	19.68

Ash Analyses of Coals

COLLIERY	SiO ₂ %	Al ₂ O ₃ %	CaO %	MgO %	Fe %	P %
1. Bankola	56.3	24.46	10.42	0.39	4.59	0.37
2. Kargali	46.3	29.70	3.92	3.35	5.88	0.33
3. New Sitalpur	54.4	25.30	6.60	2.50	4.10	0.90
4. Sirka	54.3	27.26	2.70	1.50	4.50	0.50

Analyses of Iron Ores

LOCATION	Fe %	SiO ₂ %	Al ₂ O ₃ %
1. Barbil (Orissa)	58.0		
2. Noamundi (Tisco dust ore)	63.0	4.00	7.0
3. (M/s Orissa Mineral Ltd.) Barajamda	65.5	3.50 1.54	6.0 2.0

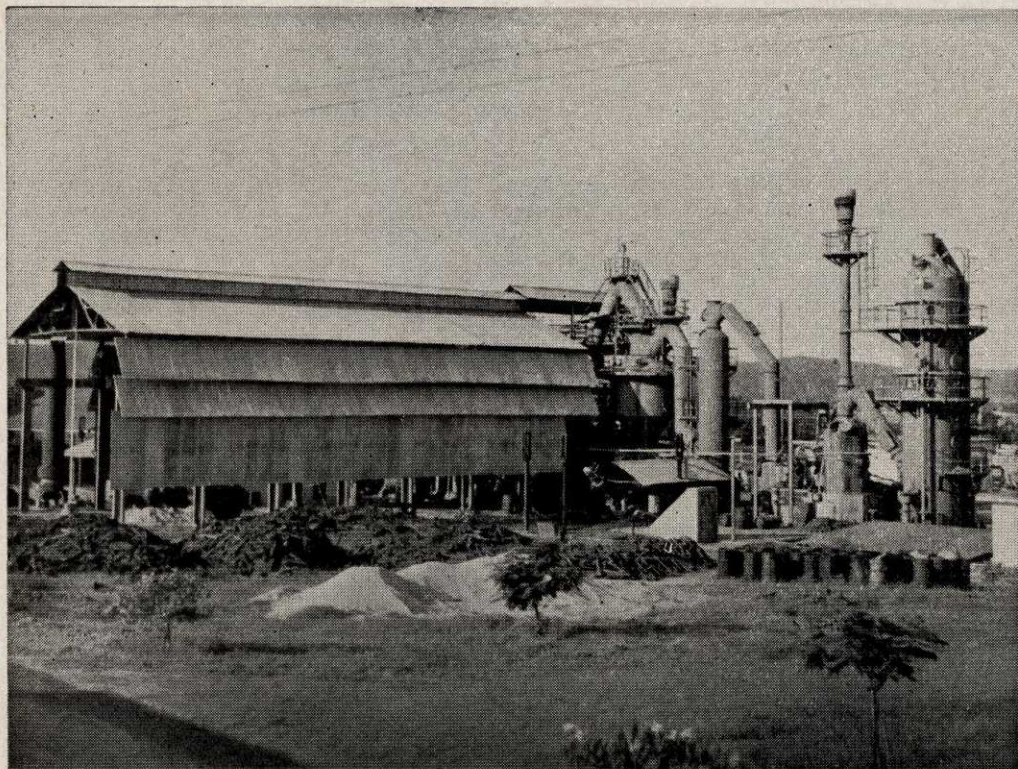


FIG. 29 — A VIEW OF THE LOW-SHAFT FURNACE WITH THE NEW SHED FOR INSTALLING STEEL-SMELTING PILOT PLANT UNITS

As the room temperature crushing strength of green or carbonized briquettes did not disclose their behaviour during smelting, comprehensive tests were conducted on briquettes by the simultaneous application of load with rise in temperature from 400° to 600°C . The Shatter Index values of the briquettes, both before and after carbonization, were obtained to determine their crushing characteristics. It was found that while room temperature strength of the briquettes originated from the binders, the high temperature crushing strength depended on the addition of a small amount of coking coal. The non-coking coals for briquetting should also possess some binding characteristics.

With the progressive increase of briquettes in the burden from 25 to 75 per cent, the furnace was blown in through the 60 mm.

tuyeres under the following operational conditions:

OPERATIONAL VARIABLES	BRIQUETTES IN THE BURDEN		
	25%	50%	75%
Hot blast volume, Nm^3/hr .	2000	2250	2200
Hot blast pressure, mm. WC.	1600	2700	2550
Hot blast temperature, $^{\circ}\text{C}$.	575	580	575
Top gas temperature, $^{\circ}\text{C}$.	380	475	450
Fuel consumption/ton of pig iron	1.8	1.68	1.63
Slag volume/ton of pig iron	1.1	0.94	0.92

The operation with briquettes led to the generation and accumulation of dust particularly at the furnace top which formed a kind of fluidized bed, thereby reducing the porosity of the burden column. The penetration of the blast, therefore, became

increasingly difficult and furnace operations were interrupted. Due to deposition of tar and dust, the charging sluice mechanism did not always work smoothly and had to be cleaned frequently which also interrupted the continuity of furnace operation.

The composition of the gas did not change conspicuously with progressive increase in the quantity of briquettes. The amount of dust loss progressively increased from 4 per cent on 25 per cent briquettes to 7 per cent of the raw materials charged at 75 per cent briquettes in the burden.

(ii) The effective tuyere diameter was changed from 60 to 75 mm. and finally to 100 mm. in three steps with consequent increase in the volume of the air blast blow in. The furnace was operated under the following operational conditions:

VARIABLES	TUYERE DIAMETER		
	60 mm.	75 mm.	100 mm.
Volume of hot blast, Nm ³ /hr.,	1600-2500	2200-3000	2700-3000
Pressure of blast, mm. WC.	1800-3000	2200-2800	1900-2600
Temperature of hot blast, °C.	580-600	575-595	595
Temperature of top gas, °C.	350-400	350-450	350-500

While the composition of the pig iron and the slag did not change conspicuously with the alteration in the rate of blowing, the productivity increased from 6.9 tons with 60 mm. tuyere diameter to 8.77 with 75 mm. tuyere diameter and ultimately 11.2 tons with 100 mm. tuyere diameter. The fuel consumption was around 2 tons of fixed carbon and the slag volume was about 1 ton per ton of pig iron, irrespective of output. The higher fuel consumption for melting exclusively with briquettes in comparison with burden consisting of lumpy raw materials and nut coke can be attributed to the heat requirement for the carbonization of the briquettes at the upper stack level. The smelting operation with the briquettes was characterized by a heavy accumulation of dust at the top of the furnace which, from time to time, forced to open the explosion doors with consequent ejection of the dust and release of pressure. The deposition of

the dust on the top of burden column, causing interruption of blowing, led to the conclusion that the floating dust at the top was not carried on to the dust catcher by entrainment in the top gas. The average analysis of pig iron was:

	%
C	2.5-3.5
Si	2.5-4.5
Mn	0.25-0.75
S	0.03-0.09
P	0.6

while the corresponding analysis of the slag was:

	%
CaO	35-40
SiO ₂	30-35
Fe	0.2-0.7
Al ₂ O ₃	22-24

The top gas analysed:

	%
CO	23
CO ₂	3.5
H ₂ O	2
CH ₄	8

(iii) In order to assess the maximum daily production, the furnace was operated with lumpy iron ore, limestone and nut coke. The furnace was blown through 100 mm. tuyere diameter with a hot blast volume of 3700-4200 Nm³/hr. at a pressure of 1700-1800 mm. WC., when a daily output of 14 tons was attained with a fuel rate of 1.8 tons fixed carbon per ton of pig iron. As the particle size of the iron ore and limestone was not optimum, further reduction in fuel rate can be achieved by suitable adjustment of the grain sizes.

(iv) In view of the technical difficulties of making self-fluxing briquettes coupled with applicational hazards and unfavourable eco-

nomics due to additional cost of binders, attempts were made to utilize various non-coking coals in lumpy form directly in bedded form of burdening. The analyses of coals and their ash are given below:

ture and the fuel consumption were lowered. The CO/CO₂ ratio was also lowered indicating an increased indirect reduction due to the closer contact of the ore particles with the reducing gases. The bridging, hanging and

Analyses of Totally Non-coking Coals Used

LOCATION	PROXIMATE ANALYSIS ON AIR-DRIED BASIS			
	Moisture %	V.M. %	F.C. %	Ash %
1. Saunda (Bokaro Ramgarh Karan-pura Coalfield)	7.7	35.9	45.2	12.3
2. Ghugus (Ballarpur Colliery, Wardha Valley Coalfield)	9.7	34.4	42.8	13.1
3. Real Jambad (Raniganj Coalfield)	4.7	33.6	43.9	17.8

Ash Analyses of Non-coking Coals Used

COLLIERY	SiO ₂ %	Al ₂ O ₃ %	CaO %	MgO %	Fe %	P %
1. Saunda	61.2	31.90	2.7	3.1	2.7	N.A.
2. Ghugus	31.7	24.76	4.3	0.8	—	N.A.
3. Real Jambad	61.5	27.80	2.1	2.2	4.4	N.A.

In the operation with non-coking coal (Saunda Colliery) it was observed that while no significant difficulty had been experienced with 25, 50 and 75 per cent non-coking coal in the fuel burden, the exclusive 100 per cent use of Saunda coal was accompanied by bridge formation in the stack of the furnace which obstructed regular descent of the burden. It also involved irregularities due to the accumulation of dust in upper regions of the furnace. Despite these difficulties, foundry grades of pig were obtained.

The non-coking coal from Real Jambad Colliery was received from the mine in big lumps, approximately 10-12 in. in size. These were hand crushed to —3 in. The employment of bigger particle size of coal increased the porosity of the burden column with simultaneous rise in top temperature. Despite the larger particle size of fuel and other raw materials, the high dust production of 8-12 per cent of the raw materials was attributed to the channelling of the burden column with consequent entrainment of comparatively large particles in the gas stream. It was observed that by the addition of fine-grained iron ore (— $\frac{1}{8}$ in.), the top tempera-

difficulties of blast penetration limit the employment of fine-grained ingredients for smelting.

For the utilization of raw materials from Maharashtra State, several combinations of burdening materials like replacement of a part of nut coke by non-coking coal from Ghugus Colliery, employment of big size coke and non-coking coals and the effects of changes in blast volume and pressure of smelting conditions were attempted. The operation with 50 per cent non-coking coal (Ghugus Colliery) in the fuel burden was associated with the major difficulty of high dust generation. The inadequate permeability of the burden column developed pressure inside the furnace which was released by opening the explosion door at frequent intervals. A change in the blast velocity of penetration of blast did not materially help to regularize the furnace operation. In view of the large volume of dust generated from Wardha's tertiary sub-bituminous friable coal, the porosity of the burden column was increased by 50 per cent admixture of blast furnace coke (3.4 in. in size). The blast velocity was decreased using 100 mm. tuyere

diameter. Although the operation became comparatively regular, the generation of the dust was still abnormally high, often reaching 15 per cent of the weight of raw materials charged. Although further attempts are under way, from these trials it was considered that friability of this coal and its chemical characteristics prevented its direct utilization for iron smelting in the low-shaft furnace.

Chemical Analysis of Pig Iron — Associated with higher silicon in the metal, it was further noted that the carbon content of the iron was relatively low. Several measures were tried to overcome this difficulty. A piece of large size coke (approximately 4-6 in.) was added per charge with the idea of retaining a part of it in the hearth area of the furnace with which the molten pig iron will come into contact and thereby absorb additional carbon; although it helped slightly in bringing up the carbon, it was not, in general, successful for raising the carbon, appreciably. Following the partial success of the addition of large pieces of coke, it was considered that small additions of coke breeze to the charge might be beneficial for the absorption of carbon by the metal on account of its fine particle size. The addition of small amount of coke breeze in the charge, however, resulted in smelting difficulties referred to earlier such as increased dust production, hanging, scaffolding, whilst, on the other hand, it did not result in increased carbon content of the iron made. Both these measures were, therefore, abandoned. It may not be out of place to mention here that our observations in this context were somewhat contrary to the observations made at the International Low-shaft Furnace where the carbon content of iron was found to increase with decrease in the coke size of the burden.

75.1 Investigations on Briquetting

Extensive investigations were undertaken either to improve the quality of briquettes or to utilize the byproducts obtained during the

smelting operation. For this purpose the following experiments were conducted:

- (i) Employment of powder sulphite lye as binder in place of liquid sulphite lye.
- (ii) Utilization of flue-dust in the briquettes.
- (iii) Addition of coke breeze in the briquettes to reduce the swelling index.
- (iv) Study of the briquetting characteristics with perfectly non-coking coal received from the Government of Maharashtra.
- (v) Utilization of byproducts of tar as binder.

(i) For this purpose, briquettes were made of the same base compositions, e.g. washed coking coal 39.8 per cent, non-coking coal (Bankola) 24.7 per cent, iron ore 24 per cent, limestone 14.5 per cent and powder sulphite lye to the extent of 4, 6 and 8 per cent was added as binder respectively. A limiting load of 10 kg. during carbonization between 400° and 600°C. was obtained with 8 per cent sulphite lye in the briquettes. The experiment was repeated with non-coking coals from Samla and Joypuria collieries with identical results. However, the use of sulphite lye powder was associated with difficulties in handling due to its hygroscopic nature and it often became pasty before mixing. In another set of experiments the powder sulphite lye was diluted with water to the same consistency, viz. specific gravity, to that of liquid sulphite lye (1.25) and its quantity was increased from 4 to 6 per cent and then 8 per cent as binder. In the latter case, the limiting load for the briquette was found to be 10 kg. The Shatter Index values of the briquettes made with different percentages of solid sulphite lye were comprehensively studied. The Shatter Index, both in the green stage as well as after carbonization, was noticed to improve with increase in quantity of the binder.

(ii) During the smelting operation of the low-shaft furnace with briquettes, a large amount of flue-dust was evolved. It contained a very high amount of fixed carbon

with other ingredients such as Fe_2O_3 , SiO_2 and CaO . In order to improve the overall economics of smelting in the low-shaft furnace, the major portion of the flue-dust needs to be recycled. As such fine particles cannot be charged directly in the furnace, its incorporation in the briquettes was, therefore, attempted. With 5 per cent flue-dust in the briquettes having base compositions of 39.8 per cent washed coal, 29.7 per cent non-coking coal (Bankola), 14 per cent limestone and 25 per cent iron ore, the sintering property did not deteriorate and the briquettes did not swell on carbonization. The limiting load during carbonization between 400° and 600°C . was 7 kg. acceptable for the purpose of smelting in the furnace. The quantity of flue-dust was then increased to 10 per cent with practically no alteration in either sintering characteristics or the limiting test load of the briquettes. With 15 per cent flue-dust, however, the binding became rather poor. It was, therefore, considered that up to a maximum of 10 per cent flue-dust can be incorporated in the briquettes. The smelting characteristics and generation of flue-dust from such briquettes were assessed by actual furnace trials.

(iii) The briquettes usually underwent considerable swelling during carbonization due to swelling characteristics of coal. In order to reduce the swelling characteristics, coke breeze to the extent of 10 and 15 per cent replacement of fixed carbon was added to the briquettes of following compositions:

	%
Coke breeze	3.22
Washed coking coal	37.79
Non-coking coal (Bankola)	19.74
Iron ore	24.75
Limestone	14.5
Coke breeze	9.3
Washed coking coal	33.3
Washed coking coal (Bankola)	18.6
Iron ore	24.0
Limestone	14.3

In both cases 8 per cent sulphite lye was used as binder. The swelling characteristics of the briquettes were reduced on the addition of coke breeze but more than 10 per cent of it could not be added as it caused deterioration, at room temperature, of the physical properties of the briquettes.

(iv) The crushed non-coking coal received from the Government of Maharashtra (Ghugus Colliery, Maharashtra) was carbonized at 700° - 800°C ., but no coke was obtained. As no briquettes could be made with different percentages of this non-coking coal with washed coking coal, this coal cannot be employed for smelting in the form of briquettes.

(v) For making briquettes, 4 per cent coal-tar pitch (softening point 65°C .) was employed as binder. In order to improve the overall economics, utilization of byproduct tar obtained from the pre-cooler basin of the gas-cleaning plant was tried after the removal of its moisture contents. This tar, however, was not free from dust particles. Briquettes were made with 39.8 per cent washed coking coal, 21.7 per cent non-coking coal from Bankola, 14.5 per cent iron ore and 24 per cent limestone to which 4 per cent sulphite lye and 4 per cent byproduct tar were added as binders. The limiting load on carbonization between 400° and 600°C . showed a poor value of only 2 kg. The quantity of byproduct tar was then increased in stages to 6 and 8 per cent when the limiting load increased to 4 kg. with 6 per cent tar. The addition of 8 per cent tar, however, did not further improve the limiting load. The Shatter Index values of these briquettes both in the green state as well as after carbonization did not disclose any drawback for the utilization of the byproduct tar. However, it is felt that the tar dust mixture should be subjected to distillation before it can be usefully employed for briquetting. It was concluded that solid sulphite lye had no special advantage over the liquid sulphite lye; 10 per cent flue-dust can be added to the briquettes without any deterioration

of its properties but actual usage will depend on further furnace-smelting trials; the addition of 10 per cent coke breeze reduces the swelling behaviour of the briquettes and was, therefore, expected to generate less dust during smelting; the utilization of by-product tar could be possible after its distillation to free it from its dust contents.

75.2 Investigations on the Injection of Gaseous Fuels

The high fuel rate of low-shaft furnace smelting had been mentioned previously. It was considered that utilization of the furnace gas containing high percentage of methane and carbon monoxide by injection through the tuyere may reduce the fuel rate for iron smelting. With this aim in view, preliminary trials were conducted in a basic lined small cupola, employing cold air blast suitably enriched with oxygen and coke oven gas. The burden consisted of iron ore, coke or non-coking coals and limestone or exclusively of self-fluxing briquettes. The degrees of oxygen enrichment and introduction of coke oven gas were varied from 8 to 17.5 per cent and 3 to 6.5 per cent respectively. The blast volume was 25-30 cu. ft./min. at a pressure of 2.7 lb./sq. in. It was found that the gaseous fuel should be introduced at the level of the tuyeres, as introduction at a higher level shifted the high temperature

zone upwards in the shaft while introduction at lower than tuyere level chilled the hearth area. In general, foundry grades of pig iron of desired compositions were obtained, but the carbon pick-up was somewhat low. The operation was generally smooth. It was noticed that intermittent introduction of the gaseous fuel was not hazardous. Investigations on iron smelting were also conducted in an one-ton cupola either with non-coking coal or with self-fluxing briquettes employing cold air blast enriched with oxygen. The descent of the burden was irregular and the metal did not attain the requisite temperature. Based on these preliminary investigations, arrangements were made for feeding the top gas from the low-shaft furnace into a basic lined cupola, which is being redesigned and revamped for conversion into a modified low-shaft furnace smelting unit at the National Metallurgical Laboratory, for the smelting of pig iron.

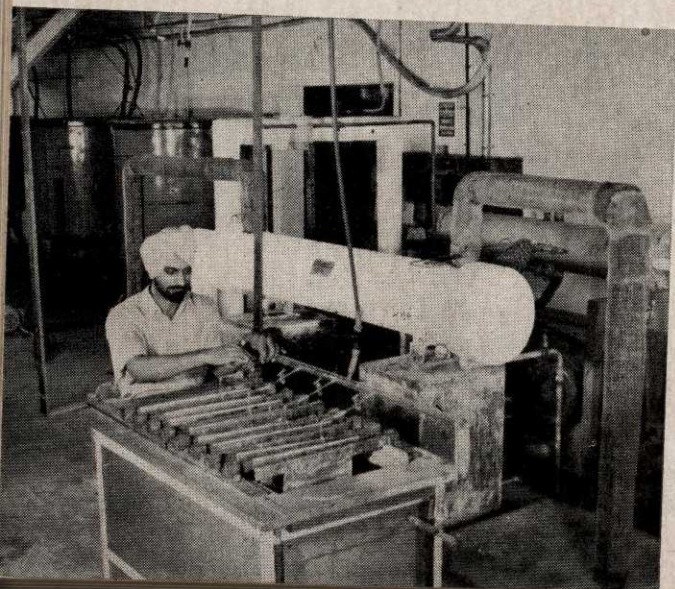
76.0 Pilot Plant for Production of Electrolytic Manganese Metal

Regular production of electrolytic manganese metal from low-grade manganese ores by the method developed and patented by the National Metallurgical Laboratory was continued in the existing pilot plant. The manganese metal thus produced found considerable application in the development of nickel-free stainless steel, coinage alloy, manganese bearing brasses, bimetallic elements and many other alloys.

More than 1500 lb. of manganese metal were produced in the existing pilot plant, out of which a considerable portion was utilized for various projects currently under progress in the National Metallurgical Laboratory. Over 500 lb. of manganese metal were sent to the Government of India Mint for experimental heats on coinage alloy.

The erection of a 100 lb. per day electrolytic manganese pilot plant to meet the increased demand for manganese metal

FIG. 30 — A VIEW OF THE 100 LB. PER DAY PILOT PLANT FOR ELECTROLYTIC MANGANESE METAL



made considerable progress. The installation of all heavy equipment and the laying of acid-resisting pipelines were completed. The rotary kiln for reduction of crushed low-grade manganese ore was remodelled taking into consideration the easier control and convenience of operation and the safety of the operators. The lead-lined reactor vessel with stirring arrangements for leaching of spent liquor with reduced manganese ore was fabricated and installed as well as other lead-lined settling, purification and storage tanks.

Considering the extremely satisfactory performance of the 32 lb. per day manganese metal cell, design for a 100 lb. manganese metal cell with 16 cathodes with the submerged area of $1\frac{1}{2} \times 2\frac{1}{2}$ in. for each cathode, capable of taking 3000 amp., was completed and its fabrication was taken up at the National Metallurgical Laboratory. This 100 lb./day manganese cell was expected to go into full operation in the very near future.

76.1 Pilot Plant for Production of Electrolytic Manganese Dioxide

Work was continued on the existing small pilot plant for production of electrolytic manganese dioxide from low-grade manganese ores by the method developed and patented by the National Metallurgical Laboratory.

A sample of electrolytic manganese dioxide produced in the National Metallurgical Laboratory was sent to Messrs Ray-O-Vac Co., U.S.A., for evaluating its battery activity. An extract from the first report on the quality of product made in the National Metallurgical Laboratory furnished by the firm is reproduced below:

"The sample and control material (the cells in the control group were assembled with electrolytic manganese dioxide by American Potash & Chemical Co.) were processed into depolarizer mix using the existing Company formulation for 100 per cent

electrolytic manganese dioxide and assembled into cells giving the full data:

Voltage and Amperage

(a) Open Circuit Voltage at 21°C.

	MAX. VOLTS	MIN. VOLTS	AVE. VOLTS
Control material	1.750	1.750	1.750
NML electro-MnO ₂	1.850	1.830	1.840

(b) Short Circuit Amperage at 21°C.

	MAX. AMP.	MIN. AMP.	AVE. AMP.
Control material	9.1	8.4	8.75
NML Electro-MnO ₂	9.8	9.0	9.33

The National Metallurgical Laboratory made electrolytic manganese dioxide, when made into cells with the Leclanché system, performed satisfactorily in flashlight type drains *initially*.

Initial and three-month flashlight performance ranged from 93 to 119 per cent of control which is considered good."

The erection of the 100 lb./day electrolytic manganese dioxide pilot plant made good progress during the period under review. In the regeneration side the 100 lb./day manganese metal plant was used for manganese dioxide plant also.

A 100 lb./day manganese dioxide cell with arrangements for passing steam through attached coils to keep the electrolyte at 90°C. was fabricated at the National Metallurgical Laboratory and installed. The cathodes and anodes for the cell were also made at the National Metallurgical Laboratory. The steam to the cell was supplied from an electrode steam boiler.

This plant is expected to go into operation in the very near future.

76.2 Battery Activity of Electrolytic Manganese Dioxide

A number of cells were prepared with electrolytic and natural manganese dioxides and their shelf life and discharge character-

istics were studied after various periods of ageing.

Discharge tests were carried out with one year-old cells prepared with National Metallurgical Laboratory electrolytic MnO_2 , West African ore, Tirodi ore and Shivrajpur ore. Their voltages were also studied during the period of storage. The test results for National Metallurgical Laboratory electrolytic MnO_2 conform to the U.S. Standard specifications for the '4 ohm intermittent test'.

76.3 Effect of Trace Elements on the Battery Activity of Electrolytic Manganese Dioxide

Several batches of MnO_2 samples were deposited from baths containing manganese sulphate and Cr, Fe, Pb, Ni, Th, etc., as trace elements with the intention of introducing these ions into the crystal lattice of MnO_2 and studying the effect of the battery active properties. The effect of addition agents on the current efficiency of MnO_2 deposition was also studied. A utilization test unit was designed and fabricated to evaluate battery activity by an electrochemical method, thus avoiding the time-consuming method of preparing actual cells and testing them. Preliminary utilization test experiments were carried out using the samples containing trace elements. The $p\text{H}$ -potential relationships of the various samples were studied by suspending them in electrolytes of $p\text{H}$ varying from 2 to 12 to determine the samples best suited for battery use. A study of the X-ray structure of the samples was also made to determine the effects of the addition of trace elements on the formation of γMnO_2 .

77.0 Pilot Plant on Hot-dip Aluminizing of Steel Wire and Sheet

The importance of aluminizing in preference to galvanizing has now been well

established apart from the basic consideration that in India resources of zinc are considerably less abundant than aluminium and also the country's entire requirement of zinc is at present imported. With this objective in view, the research project was initiated to develop the aluminizing process basically differing in the use of flux such as molten salt, aqueous and organic fluxes. Based on the laboratory scale results, an integrated pilot plant was set up with a view to determine the commercial feasibility and economic aspects of the process developed by this Laboratory, the details of which were reported earlier.

Samples of aluminized wire produced at the pilot plant were sent to the British Iron & Steel Research Association, U.K., for their detailed study of the structure and properties. Their detailed report on this subject was highly encouraging as can be seen from their report: "Despite the more extensive sampling, there is no evidence of failure of the alloy layer. This is most gratifying, especially since it is understood that no active flux but a protective film was used. The sample behaved quite well, especially since it was slightly thicker. Sample A 110 wire was exceptional in quality and sets a very high standard." It was most gratifying to note that BISRA had also recorded that there was no evidence of failure of the alloy layer. The microphotographs taken on the longitudinal section of the wires had shown a more representative picture of the coating as compared to those obtained in the transverse section. It was also indicated that

- (i) the National Metallurgical Laboratory's process of preparation and prefluxing seemed to be effective as there was no sign of failure of the alloy layer;
- (ii) the wiping left thin coating without decreasing lustre;
- (iii) the coating thickness obtained was 0.6-1.6 mils.; and
- (iv) good wrapping properties have been observed.

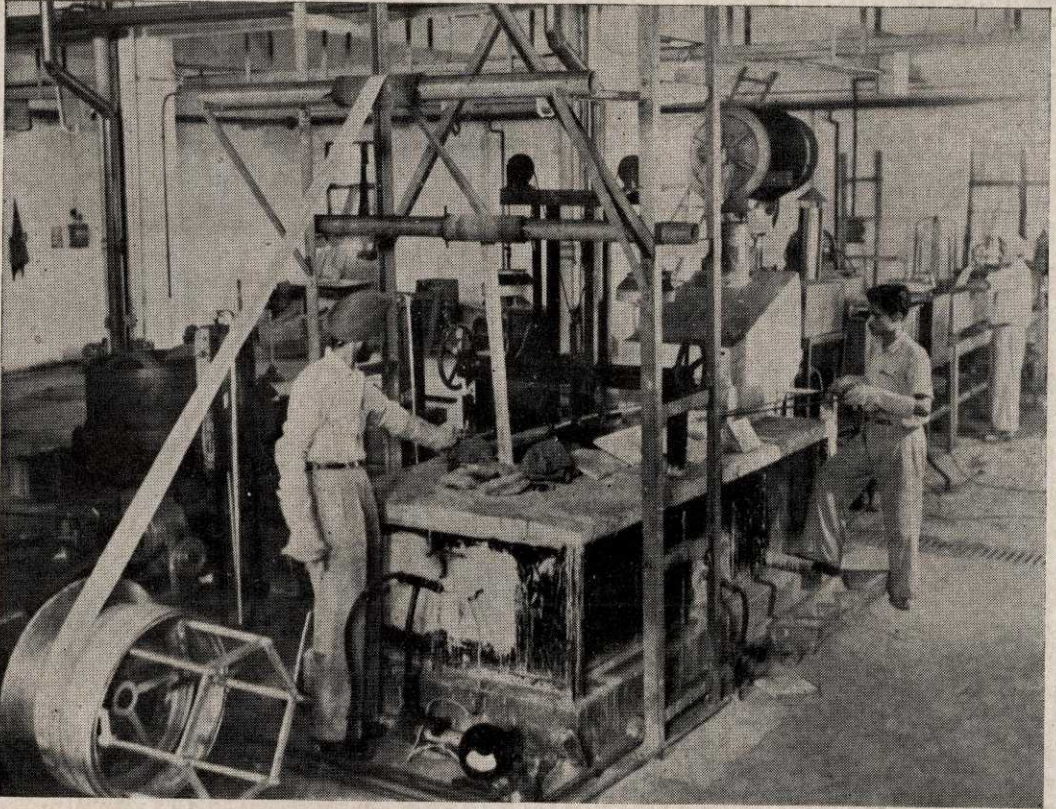


FIG. 31 — PILOT PLANT FOR HOT-DIP ALUMINIZING OF STRIPS, DESIGNED AND FABRICATED AT THE NATIONAL METALLURGICAL LABORATORY

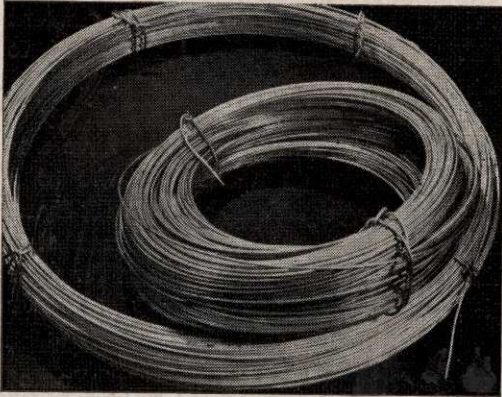


FIG. 32 — COILS OF WIRE ALUMINIZED AT THE PILOT PLANT SET UP AT THE NATIONAL METALLURGICAL LABORATORY

During the year under review, several miles length of steel wires were aluminized and supplied to the General Manager,

Post & Telegraphs Workshops, Calcutta, for field trial on telephone lines. Comprehensive investigations were also conducted on the evaluation of properties of aluminized wire supplied to the Post & Telegraphs Workshops as given below:

Coating Weight — Representative 12 in. samples were tested for coating weights as per ASTM A428-58T stripping test wherein a substantial weight of coating is dissolved in 20 per cent sodium hydroxide solution at 90°C. and finally in hydrochloric acid in one to three cycles. Wiped coatings had 220-350 mgm./ft. of aluminium on 10 SWG wire. Coatings obtained by use of sizing die had a coating weight of 300-400 mgm./ft.

Salt Spray Corrosion Test — Accelerated corrosion test as per ASTM B117-54T showed that an increase in coating weight from 220

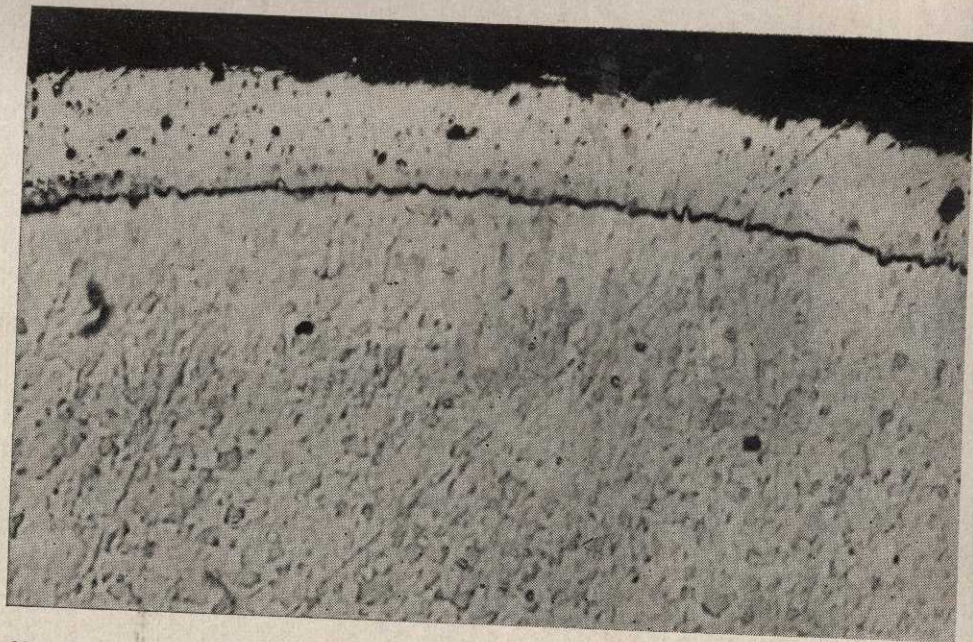


FIG. 33 — TRANSVERSE SECTION OF HOT-DIP ALUMINIZED M.S. WIRE SHOWING A CONCENTRIC COATING. THE OUTER ALUMINIUM LAYER AND THE ALLOY LAYER HAVE OPTIMUM THICKNESS. $\times 250$

mgm./ft. to 350 mgm./ft. in wiped coatings decreases corrosion rate from 65 mgm./ft./120 hours to 15 mgm./ft./120 hours. Corrosion rate of coatings obtained by use of sizing die is only around 10 mgm./ft./120 hours.

Soundness Tests — Coatings behaved well in Preece test withstanding five dips and in 15 per cent HNO_3 test wherein very little gas evolved in 5-6 hours.

Electrical Resistance — Good aluminized steel wire had an electrical resistance around 24 ohms./mile and will be suitable for telephone lines.

Mechanical Properties — Tensile test results on M.S. wire in the uncoated, coated and drawn conditions are given below:

SAMPLE	DIA. in.	MAX. STRESS T.S.I.	ELON- GATION %
Uncoated annealed M.S. wire	0.125	30.93	18.75
Hot-dip aluminized wire	0.123	33.82	12.50
Cold-drawn alu- minized wire	0.116	46.46	6.25

Jointing and Soldering — Wrapped joints like Brittanica, wrapped tap, cross tap, knotted tap, Bell Hunger's plain tap and twist joints can be made as practised in telephone and telegraph lines. These joints can be soldered by (i) Pb, 58; Sn, 40 and Zn, 2 per cent or (ii) Zn, 90; Al, 7 and Cu, 3 per cent solders. Aluminized wire lends itself to 'electric resistance butt welding' also.

The Ministry of Defence and Ministry of Railways, Government of India, have also evinced interest on aluminized wire as a substitute for galvanized steel wire for signal and other use. Several aluminized steel wire coils were also sent to them for their trial.

78.0 Pilot Plant for the Beneficiation of Low-grade Manganese, Chrome, Iron and Other Similar Ores

The National Metallurgical Laboratory, right from its inception, had recognized the importance of beneficiation of low-grade ores of India in the conservation of her

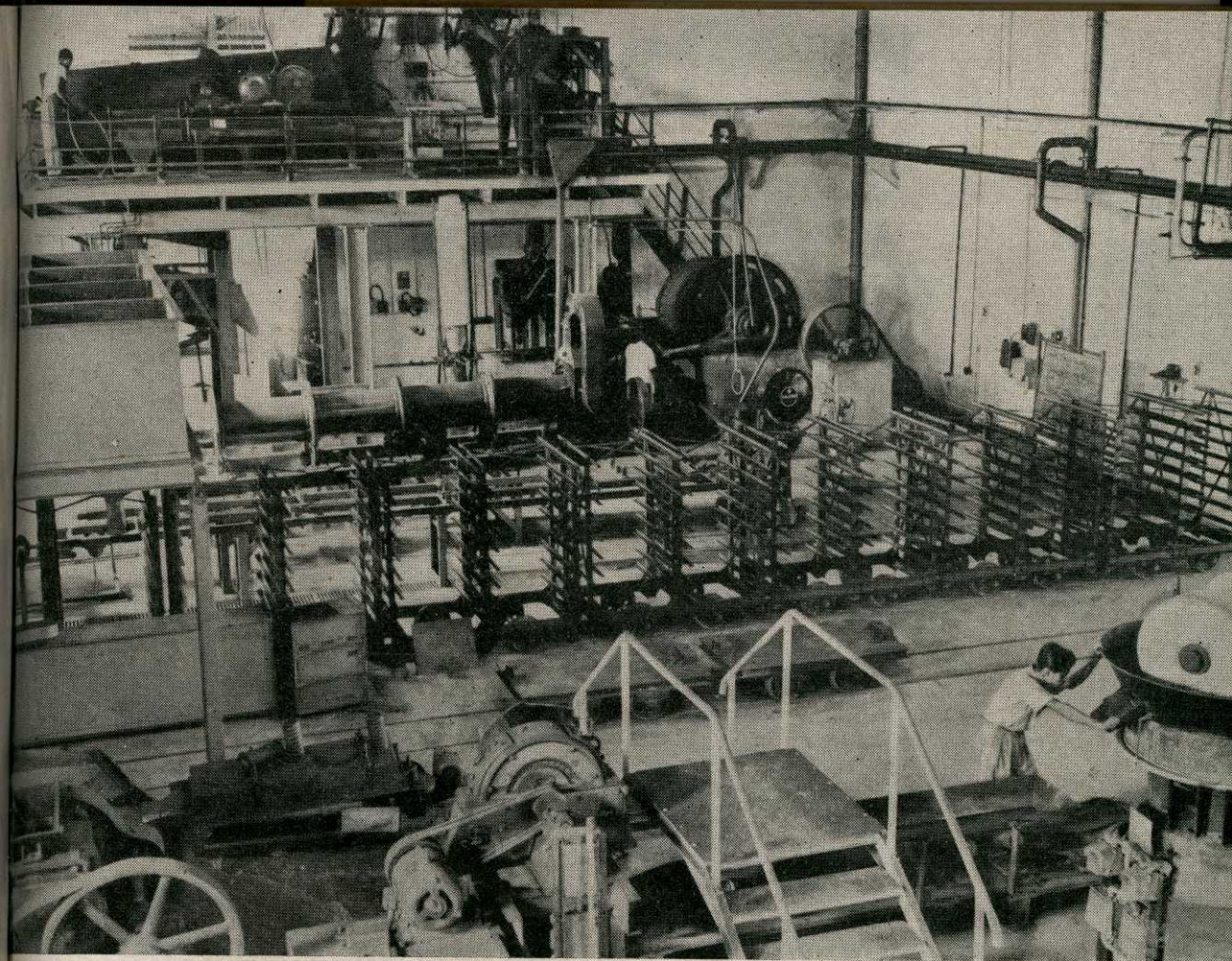


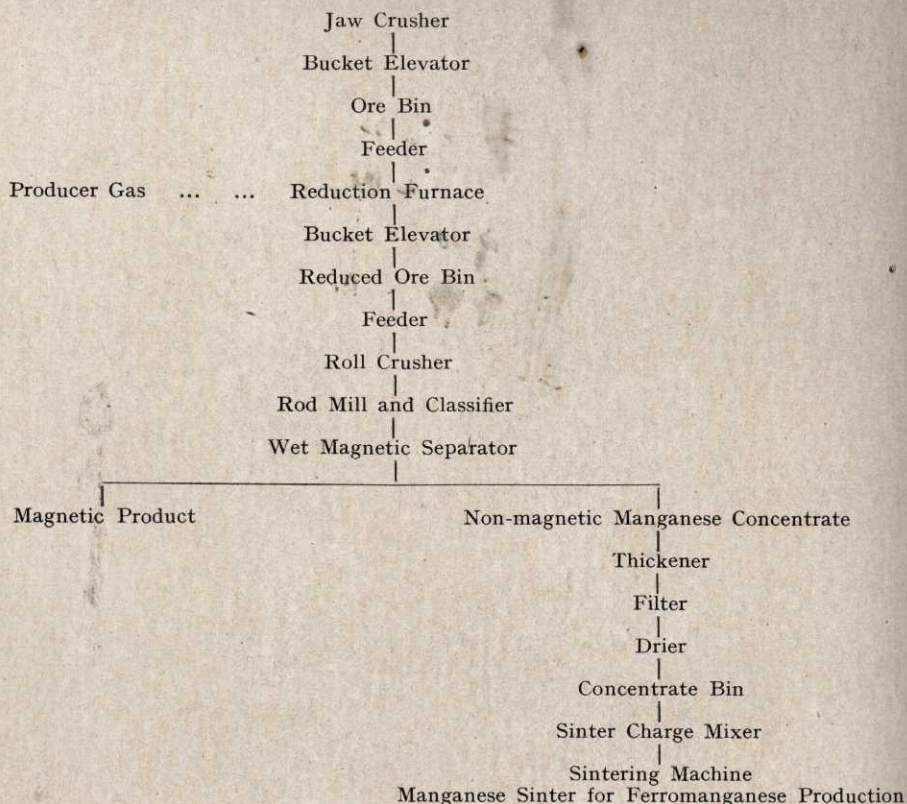
FIG. 34 — CORNER OF THE NEW PILOT PLANT TECHNOLOGICAL BAY SHOWING THE ORE-DRESSING PILOT PLANT AND THE TUNNEL DRIER

mineral wealth. Since the Laboratory's inception, systematic investigations were undertaken into the beneficiation of low-grade manganese, chrome, iron and other similar ores. It was also realized at an early stage that it was necessary to translate the results of laboratory work to a pilot plant scale before commercial scale upgrading plants could be set up for the beneficiation of indigenous low-grade ores. With a view to achieve this objective a pilot plant of $\frac{1}{2}$ -ton capacity per hour has been set up for the treatment of some specific types of manganese, iron and other ores. The layout of the plant has been so designed that many types of ores can be treated by suitable adjustment in the flow of materials.

The plant has been equipped with jaw and roll crushers for crushing, ball mill in closed circuit with a classifier for grinding, blade washer, vibrating screens for screening, a vertical reduction furnace and a producer gas unit for carrying out reduction trials, dry and wet magnetic separators, electrostatic separator, heavy media unit, jigs, shaking tables, thickener, filter, drier, briquetting press with mixer, continuous sintering unit and auxiliary equipment such as belt conveyors, bucket elevators, vibrating feeders, ore bins, etc.

Two typical flowsheets, one for the beneficiation of low-grade ferruginous manganese ores and the other for iron ores, are given in the following pages.

FLOWSHEET FOR THE BENEFICIATION OF LOW-GRADE FERRUGINOUS MANGANESE ORES

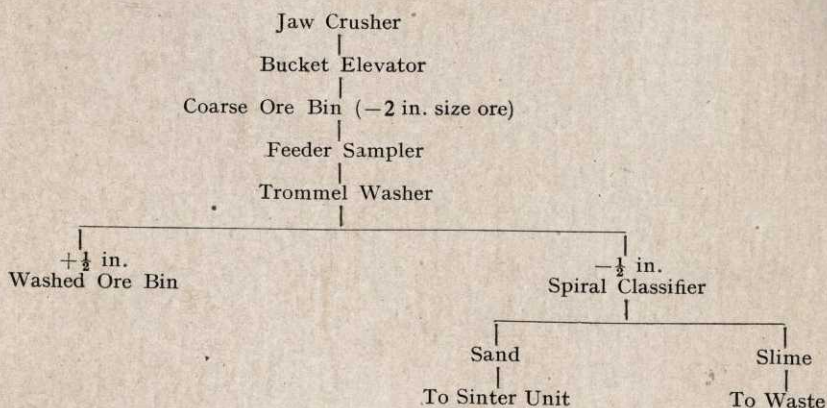


The raw ore of 5-6 in. maximum size is crushed to 1-1½ in. size in a jaw crusher and elevated to an ore bin. The crushed ore is delivered to a reduction furnace through a vibrating feeder. Reducing gas from a producer gas unit, designed and fabricated at the National Metallurgical Laboratory, enters the vertical reduction furnace at the bottom and reduces iron oxides of the descending charge of the ore in the furnace to magnetite and oxides of manganese to manganoous oxide. The reduced ore is stored in a bin from where it is fed to a roll crusher which crushes the material to about 10 mesh. The crushed material is then subjected to wet magnetic separation to yield a non-magnetic manganese concentrate. If finer grinding is necessary, the roll-crushed material is ground in a rod mill in closed circuit

with a classifier. The fine manganese concentrate is either sintered or briquetted. The above flowsheet is suitable for the treatment of ferruginous manganese ores low in silica and alumina. But the layout of the plant is so designed that other types of low-grade ores can also be treated, though some of the operations may have to be done on a batch scale.

Beneficiation of iron ores in the metallurgical sense relates not merely to the enrichment of the iron content of the ore by rejection of gangue but also is a step towards ore and burden preparation; the latter aims at physical improvement of the ore by crushing, sizing, agglomeration of resultant fines, etc., for uniform operation of the free blast furnace to ensure minimum fuel consumption consistent with maximum productivity of

FLOWSHEET FOR THE BENEFICIATION OF IRON ORE



quality output. India possesses vast reserves of high-grade iron ores. However, the question of their beneficiation has to be examined in the light of several basic considerations, such as flow of materials during screening, handling and preparation under Indian dry and wet weather conditions, amenability of ore fines to sintering including self-fluxing sinter production and upgrading of metallic values by the elimination of gangue and of the extreme fine fractions of the ores containing bulk of the gangue.

With the above in view, the pilot plant has been designed to beneficiate Indian iron ores also.

The raw iron ore (5-6 in. maximum size) is crushed to about 2 in. size in a jaw crusher and elevated to a coarse ore bin. The crushed ore is then delivered through a Syntron vibrating feeder to a Trommel washer to disintegrate the loosely adhering particles of clay and other extraneous materials. Water-sprays are provided inside the washer. The discharge end of the washer is provided with $\frac{1}{2}$ in. screen for screening the washed ore. The washer discharge containing $+\frac{1}{2}$ in. material forms the feed either for further beneficiation by heavy media process or for direct charging to the blast furnace. The $-\frac{1}{2}$ in. material is treated in a spiral classifier for the elimination of very fine gangue mate-

rial and excess water and the classifier sand can be used for producing blast furnace sinter.

During the period under review, the following items of equipment were received and installed at the National Metallurgical Laboratory:

- (i) Mixer machine for briquetting press
- (ii) One chain type bucket elevator
- (iii) One 9 ft. dia. thickener
- (iv) One gravel washer
- (v) One 6 ft. dia. hydroclassifier
- (vi) One 8×12 in. duplex jig
- (vii) One 4×4 in. hydroclassifier
- (viii) One 4×4 ft. ball mill

The crushing section consists of a belt conveyor, jaw crusher, vibrating feeder, 16 ft. bucket elevator, double roll crusher, 35 ft. bucket elevator and two ore bins. The washing section consists of a gravel washer and a crossflow classifier. A trial washing test with iron ore was successfully carried out, employing the crushing and washing sections. The ball mill has also been put into commission.

78.1 Pilot Plant for Beneficiation of Low-grade Manganese Ores

Realizing the importance of large-scale plant trials for beneficiation of low-grade manganese and other ores for drawing up

production flowsheets for large-scale commercial plants, a pilot plant capable of treating one ton of low-grade ore per hour is being set up. This Laboratory has already carried out systematic investigations on the beneficiation of more than thirty indigenous low-grade manganese ores from different parts of the country and this work has clearly established that many of the ores can be upgraded to the required rigid specification. The projected pilot plant, apart from yielding data on the technical feasibility of the laboratory processes on a pilot plant scale, would also provide equally valuable economic and design data for setting up commercial upgrading plants, and will thereby serve to fill up the gap between the processes developed in this Laboratory and their ultimate industrial scale application. The projected plant will be of the custom mill type, capable of employing a large variety of flowsheets depending on the ore characteristics.

A plot of land covering 5-6 acres adjacent to the Low-shaft Furnace Pilot Plant of the National Metallurgical Laboratory has been taken over from the Tata Iron & Steel Co. Ltd. for setting up the pilot plant. Action has also been initiated for the procurement of capital equipment worth over Rs. 22 lakhs under U.S. Exim Bank Credit Scheme from the United States. The first shipment of imported equipment under the above project scheme has already arrived in Calcutta. Detailed layout of the plant is now being prepared. Arrangements are also in progress for procurement of electrical, civil and other equipment required for the above plant from Indian sources.

79.0 Pilot Plant for Refractories

The development of metallurgical refractories based on indigenous resources of raw materials is vital to the growth of heavy metallurgical industries in India. In industrial scale implementation of research results in diverse fields of refractories technology,

extrapolation of laboratory scale bench results to industrial scale is impracticable. It is necessary to emphasize that in the case of refractories technology, pilot scale investigations will ensure the production of new or improved refractories on a scale that can rationally evaluate the quality of bulk scale output. With this object in view, a comprehensive pilot plant for the production of the various types of refractories is being established in the National Metallurgical Laboratory to produce refractory materials on a quantitative output basis related to the following projects, on which laboratory scale researches have yielded fruitful and highly promising results:

- (i) Dense mullite and hot face insulation refractories from bladed kyanite
- (ii) Zircon and sillimanite refractories from Travancore beach sands
- (iii) Magnesite refractories from Almorah magnesite
- (iv) Forsterite refractories from magnesium-silicate rocks
- (v) Chemically bonded and metal clad mixed basic refractories
- (vi) Stabilized dolomite refractories
- (vii) Chrome-magnesite refractories from low-grade chromites
- (viii) Carbon refractories from petroleum coke and low-ash coals
- (ix) Dense carbon aggregates and Soderberg paste from indigenous raw materials
- (x) Carbon-bonded and clay-bonded graphite crucibles

This pilot plant has been so designed by the National Metallurgical Laboratory that various stages implicit in the production of refractories can be translated on a semi-industrial scale either on unit basis or on a continuous "raw material to finished product" flow basis. The National Metallurgical Laboratory pilot plant has been designed to handle a little over one ton of raw material per day of eight hours. The pilot plant consists of a crushing and grinding section, a mixing and forming section and a drying

and firing section. A casting section has also been included. Almost all the items of equipment will be fully instrumented so that collection and coordination of resultant process data can effectively be accomplished.

During the period under review the following plants and equipment were installed.

(i) *Crushing, Grinding and Mixing Machines* — The crushing and grinding machinery were installed and commissioned early this year. They have been in regular service ever since.

(ii) *Adaptation of a 500-ton Hydraulic Press for Making Bricks and Blocks* — This adaptation, completely automatic in action, will, with the addition of roller conveyor to carry the bricks off the plate, need only one attendant to operate the press. The adaptation does not need any separate motor for

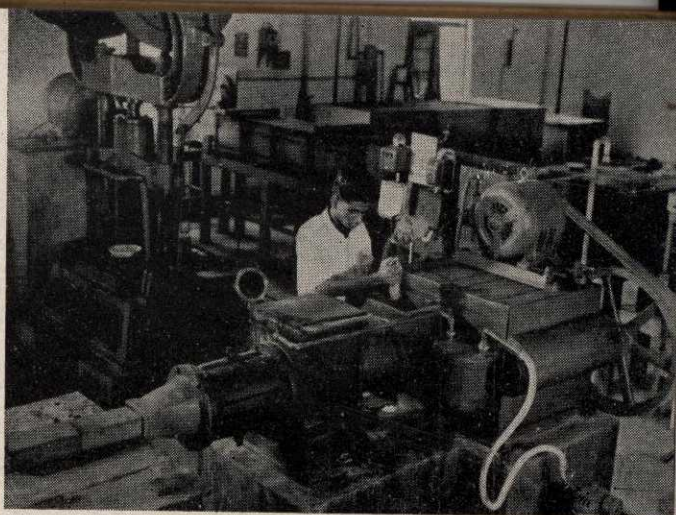


FIG. 35 — DE-AIRING EXTRUSION MACHINE FOR REFRACTORY PRODUCTS

the movements of the various involved parts as it would utilize the vertical up and down motion of the top platen of the press for actuating such movements.

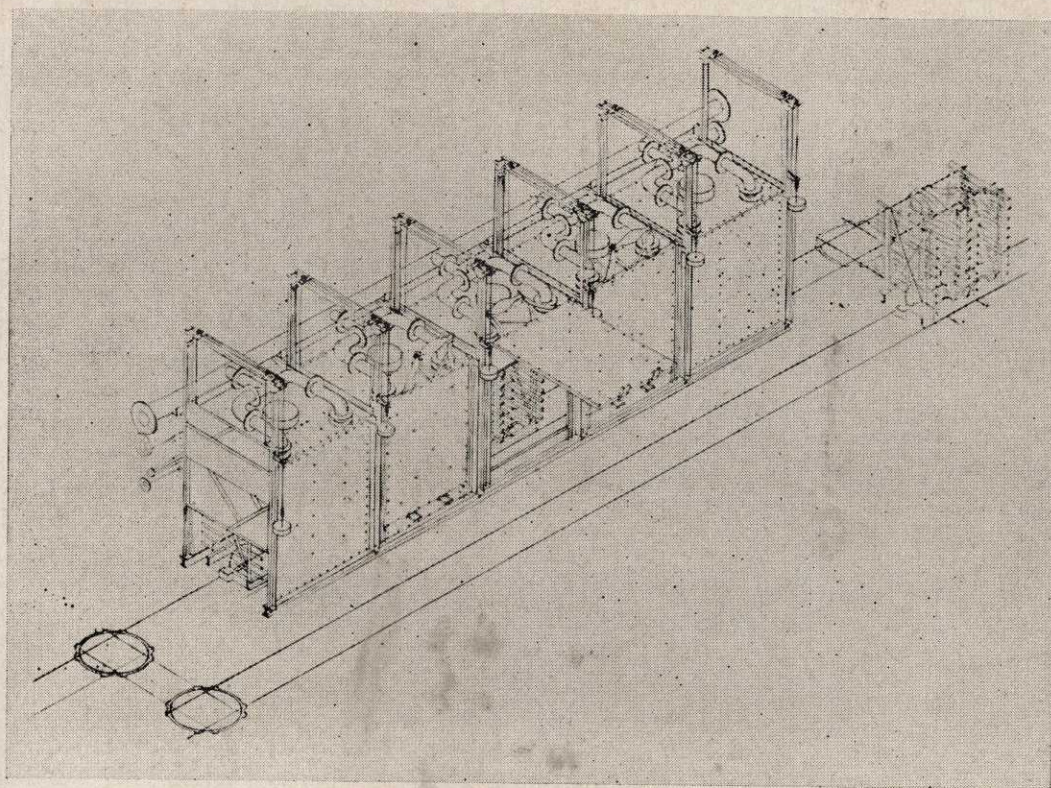


FIG. 36 — SCHEMATIC DIAGRAM OF THE CONTINUOUS TUNNEL DRIER, DESIGNED AND UNDER FABRICATION AT THE NATIONAL METALLURGICAL LABORATORY

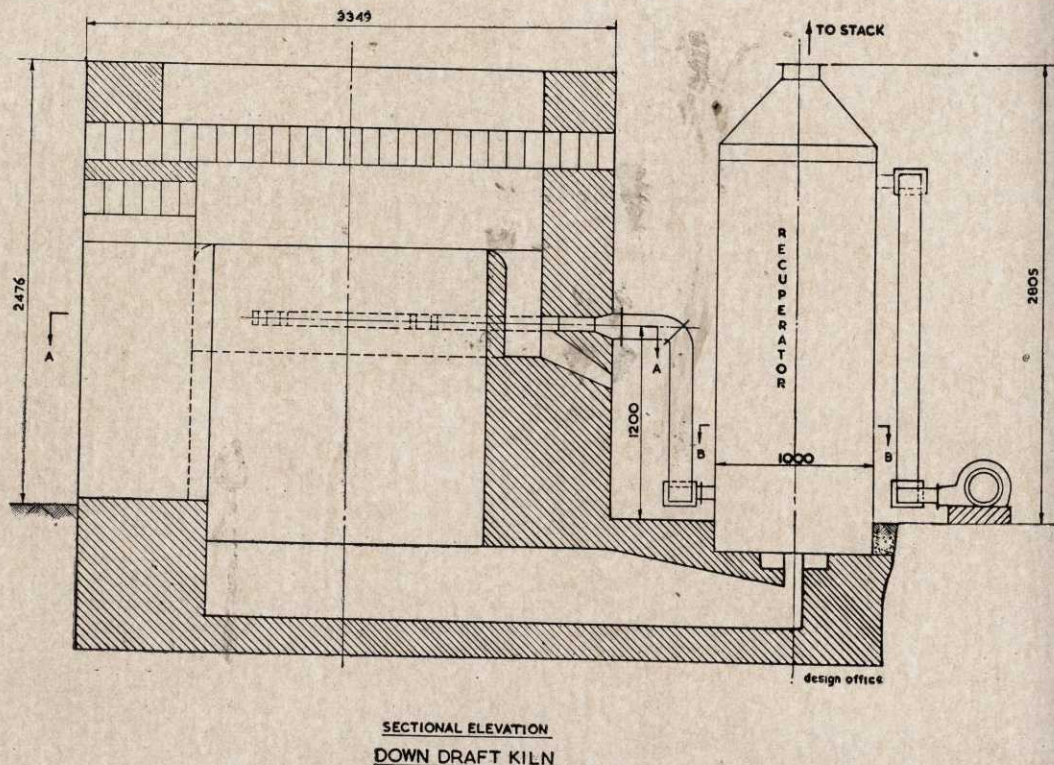


FIG. 37 — SCHEMATIC DIAGRAM OF THE DOWN DRAFT KILN, DESIGNED AND UNDER FABRICATION AT THE NATIONAL METALLURGICAL LABORATORY

(iii) *Continuous Tunnel Drier and Down Draft Kiln* — Designs for continuous tunnel drier and down draft kiln have been completed and the equipment is under fabrication. Figs. 36 and 37 are the schematic layouts of the continuous tunnel drier and down draft kiln as designed at the National Metallurgical Laboratory.

80.0 Pilot Plant for the Recovery of Vanadium Pentoxide from Vanadiferous Magnetite Ores

Vanadium-containing titaniferous magnetites occur in Bihar and Orissa and their reserves have been estimated at about 22 million tons. The ore contains an average of Fe, 55-65; TiO_2 , 8-12 and V, 0.8-1.8 per cent. These deposits owe their importance primarily to their vanadium contents. A systematic research study for recovery of

vanadium pentoxide was undertaken. Based on the laboratory scale research investigation, a pilot plant for the treatment of one ton of the ore per day has been set up.

Installation of the major units and their auxiliaries was completed. Pilot plant studies for the recovery of vanadium from vanadiferous ores containing 0.8-1.8 per cent vanadium were taken up. Amongst the units installed and commissioned during the period under review were leaching tanks fitted with steam coils and agitators, settling tanks, commissioning of filter press and fixing steam heating lead-coated foils for the precipitating tanks. All pipeline work for interconnecting the tanks for counter-current leaching and circulation of the enriched liquor for precipitation of vanadium pentoxide and its subsequent separation was completed.

Twenty tons of vanadiferous ore from Rairangpur were collected and crushed. A systematic study on standardizing the roasting conditions was taken up as it was observed that unlike the laboratory scale studies, where batch roasting had been carried out, recovery in the kiln was much less and could be attributed to the short retention time of the ore in the kiln. The retention period of the ore in the kiln was, therefore, further increased by modifying the pulleys and gear system of the kiln. Two tons of ore were roasted and leached for the recovery of vanadium pentoxide. The details of centrifuge, evaporator and vacuum crystallizer were worked out.

81.0 Pilot Plant for Thermal Beneficiation of Low-grade Chrome Ores

Suitability of chromite in the production of standard grade ferro-chrome limits the Cr: Fe ratio at 3:1 with a minimum of 48 per cent Cr_2O_3 ; MgO and alumina should not exceed 5 per cent. The Indian chrome ores are characterized by their high iron contents occurring in chemical combination with chromite. Conventional methods of ore dressing such as conventional roast reduction followed by magnetic separation have not been found to improve the Cr: Fe ratio, even though a substantial increase in the Cr_2O_3 content of the ore was achieved. The thermal beneficiation process investigated in this Laboratory consisted in selective reduction of iron in the chrome ore with solid reducing agents and its subsequent removal by acid leaching.

The chrome ore along with coke is fed in a rotary kiln. The maximum temperature at the firing zone will be about 1300°C . and the inclination of the kiln is so adjusted that a retention period of 2-2½ hours is obtained in the high temperature zone of the kiln. The reduced ore is fed into a cooler and cooled in neutral atmosphere. The cooled reduced mass is crushed to -10 mesh and is fed into a

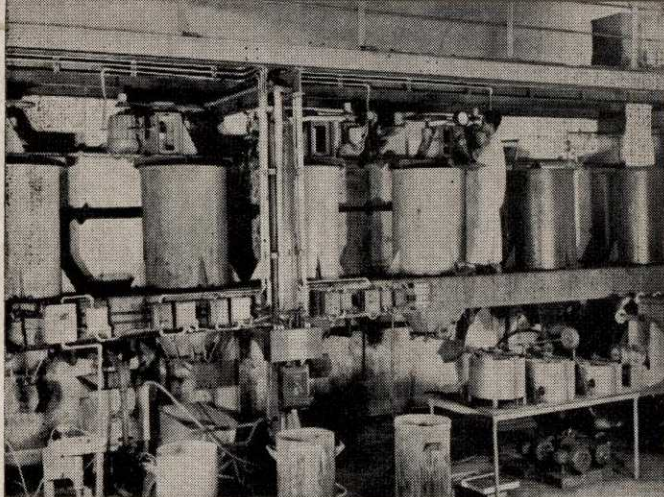


FIG. 38 — PILOT PLANT FOR THE RECOVERY OF VANADIUM PENTOXIDE FROM INDIAN VANADIFEROUS MAGNETITE ORE, SET UP AT THE NATIONAL METALLURGICAL LABORATORY

lead-lined, steam-heated tank, 2½ ft. in dia. and 5½ ft. in height, for leaching. The leached mass will be taken into a thickener and a residue enriched in Cr: Fe ratio will be obtained. The liquor containing ferrous sulphate and dissolved salts is used for the recovery of ferrous sulphate and subsequently for production of ferric oxide by roasting. The details of the rotary kiln for roasting of chrome ores on pilot-plant scale have been worked out. Meanwhile roasting of the ore in the smaller kiln designed and fabricated at the National Metallurgical Laboratory was taken up. Lead-lining of the leaching tanks for the removal of soluble iron part was completed and the tanks were installed in position.

82.0 Pilot Plant Studies for Thermal Beneficiation of Low-grade Manganese Ores

The specification for manganese ores for metallurgical purpose requires Mn: Fe ratio to be as high as possible and preferably not below 7:1. The phosphorus content should also not exceed 0.1-0.13 per cent and the silica and alumina contents of the ore should not be more than 8 and 10 per cent respectively. Thermal beneficiation employed for the treatment of low-grade manga-

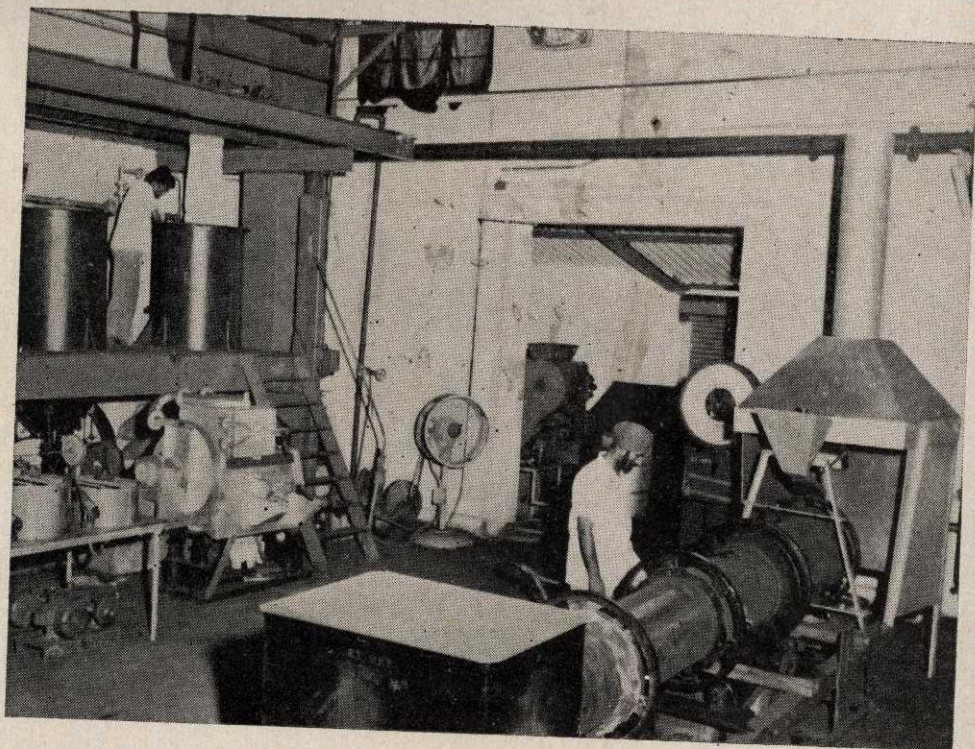


FIG. 39 — PILOT PLANT FOR THERMAL BENEFICIATION OF LOW-GRADE CHROME ORE SHOWING THE ROTARY KILN, DESIGNED AND FABRICATED AT THE NATIONAL METALLURGICAL LABORATORY

nese ore is undertaken in a two-stage process, some work on which has already been carried out at the National Metallurgical Laboratory. The process consists in treatment of low-grade manganese ore through suitable thermal techniques aiming at the production of an enriched manganese slag and iron as a by-product in the first stage, followed by the smelting of enriched manganese slag. On commercial scale, the possibility of a combination of acid Krupp-Renn rotary kiln during the first stage to produce an enriched slag followed by the use of basic Stuzelberg rotary furnace to yield a manganese rich ferro-alloy in a second stage will be taken up. Alternatively, electric smelting of enriched manganese slag to produce ferro-alloy may also be considered. The method is based on studies of free energy values of the oxides of manganese, iron and phosphorus and thermodynamic factors involved in their reduction under optimum conditions of temperature

and basicity. A furnace with a capacity of 100 lb. charge was designed at the National Metallurgical Laboratory.

83.0 Pilot Plant for Pyrometallurgical Production of Magnesium from Dolomite

A shed, 80×30 ft., for the installation of magnesium pilot plant is under erection. Detailed specifications for the equipment required for the plant have been worked out. Preliminary large-scale work using one retort was undertaken during the period under review most successfully.

84.0 Pilot Plant for the Production of Ferro-alloys

Based on the successful laboratory scale production of various types of ferro-alloys, a

pilot plant is being set up for the production of ferro-alloys on a large scale. A one-ton submerged arc furnace is being installed. It is hoped that useful results will flow out from the pilot operation of the furnace with direct benefits to the industry, especially in the context of the demand for basic and special ferro-alloys for the Alloy and Tool Steel Plant now being set up in the public and private sectors. This furnace will be available for the first time in India for pilot smelting of ferro-alloys. Customer's Indian raw materials would be accepted in trial batches for investigating their amenability to electrothermal smelting *vis-à-vis* the products aimed at and ferro-alloys actually smelted. Valuable information would be gained on important factors such as electric power and electrode consumption, recovery yield data, etc. This data can yield accurate appraisal of what is to be expected on a large-scale industrial plant. At the end

of each contract tests, a full report will be prepared giving recommendation on proper metallurgical practice, electrical ratings and size of installed plant needed for a given tonnage output.

Pending the installation of the submerged arc furnace, systematic studies for the production of ferro-alloys by alumino-thermic reactions were continued. The entire set-up has been so mechanized that the reactions can be controlled by two men. 400 kg. of carbon-free ferro-chrome were produced for the different projects under way in the Laboratory. Simultaneously, studies are also being taken up for the production of carbon-free ferro-manganese and 25 kg. of the alloy have since been produced. The ferro-alloy output of this pilot plant is utilized at the National Metallurgical Laboratory and also considerable quantities thereof have been supplied to the Ministry of Defence, Government of India.

ELECTRIC FURNACE COMMITTEE

An Electric Furnace-making Committee was constituted in November 1960 with a view to evolve suitable refractory compositions used in electric furnace and to design and fabricate different complex types of furnaces tailored specifically for research jobs, using entirely indigenous materials.

During this period, work was conducted on (i) the development of cements, pastes and refractory mixes of suitable chemical and mechanical characteristics; and (ii) the development of internally wound tube furnaces, pot furnaces, high temperature muffle and gradientless furnaces.

A brief review of this work undertaken at the National Metallurgical Laboratory is presented below:

(i) *Development of Refractories, Cements, etc.*—The electrical resistance elements readily available in this country are nichrome, the Kanthal series of alloys and the platinum and platinum-rhodium alloys. Depending on the temperature of use, each one of these requires refractory supports of particular chemical compositions whilst the presence of certain other ingredients is deleterious and adversely affects the life of the furnace. For example, Kanthal should not come into contact with silica at high temperature while a 45 per cent Al_2O_3 body is very favourable for the former's long life. Additionally, the cements and pastes should have adequate dry and baked strengths, should sinter well and not crack on heating with embedded elements.

The Furnace Committee have developed three refractory pastes and cement compositions from materials that are readily available in the country. Of this, one is a 50 per cent alumina body consisting of kyanite and

clay (the grain size of kyanite is controlled) which forms a workable paste with water, adheres well to the elements on drying and sinters hard at 1000°C . Experience with a number of furnaces has shown that it has no deleterious effect on Kanthal.

The second is a aluminous composition and a calcium aluminate cement developed in the Laboratory forms the binder. This mix has hydraulic setting properties and once set and cured can be heated to operating temperatures safely. This cement is also quite strong when fired and has good binding strength.

The third cement, which is still in the process of development, depends for its strength and binding properties on phosphoric and chromic acids. A kyanite-based cement is under trial. Uses of alumina, zircon, zirconia and other bases have also been aimed at.

(ii) *Development of Internally Wound Furnace, Pot Furnaces, etc.*—The development of these furnaces, their design, and technique of fabrication of internally wound furnaces have occupied considerable time of the Furnace Committee. In the case of the internally wound furnaces, the crux of the question was to get the raw pliable refractory mix and the elements into the shape needed by the geometry of the furnaces. This fabrication technique is now mastered and several complicated designs of tube furnaces of various diameters and lengths are running most successfully in the National Metallurgical Laboratory. In the case of the pot furnaces, the main problem is embedding of the required length of resistance wire within a limited space and imparting rigidity to it while it is being covered by the refrac-

tory. This has also been successfully accomplished.

A gradientless furnace, or in other words, a furnace in which a predetermined length remains at a constant temperature is necessary for many metallurgical problems. This means generating a known amount of heat in a particular zone and retaining it in that zone or, in other words, preventing and providing compensation for heat losses occurring through radiation and convection. In the design now developed compensation for the

heat loss is provided by installing a secondary winding at the ends. This winding is isolated from the main heating winding and is controlled separately.

The future programme of the Committee is to design furnaces operating in the range of 1600° - 2500° C., to develop accurate temperature controllers, as far as possible, with indigenously available components and to study diverse factors affecting the life and thermal efficiency of electric furnaces in general.

INDUSTRIAL LIAISON, OPERATIONAL RESEARCH AND PROJECT REPORTS

As metallurgical industries are becoming increasingly conscious of research, numerous technical operational problems are being referred to this Laboratory in an ever increasing measure which are being effectively handled to increase productivity consistent with quality output of products made. Technical liaison is also actively maintained with industry through publications brought out by this Laboratory, such as the *NML Technical Journal*, *Proceedings of Symposia*, Monographs embodying the results of long-term projects, commercial exploitation of our patents, liaison with industries and scientific organizations, collection and dissemination of statistical data, through organization of international symposia on metallurgical subjects of topical value, bringing out popular technical brochures, translation service and, last but not the least, through field investigation by operational research and other technical staff of difficult operational technical subjects sponsored by the industries themselves as also on our own initiative.

During the year under review, several operational research problems were undertaken to find effective means for solving the problems posed by the industry. One foundry in Calcutta area was experiencing considerable difficulties in the production of castings for flame-proof switch gears for use in mining equipment. The requirements for such castings are that they should be free from blow holes and other defects especially on the machined faces so that a tight joint can be secured to prevent the flame and combustion products from escaping out in the

event of an explosion within the switch gear, which would otherwise cause fire hazards inside the mine. The detailed set-up of the foundry, the raw materials used, equipment available, moulding techniques adopted, including on-the-spot study of the practice followed in the foundry, were fully investigated. The key factors responsible for the successful production of the above castings were formulated and in the light of the actual plant conditions, suitable recommendations were made relating to the cupola practice, processing of raw materials, technique of moulding and casting.

Plant Visits

During the year under review, several visits were paid to the foundries in connection with their respective production problems and suitable recommendations were made to overcome the difficulties encountered in plant practice. Such technical advice and on-the-spot study also related to various types of casting defects encountered in the production of quality castings and in some cases required suggestions for a modified layout to suit the development plans ahead.

Utilization of Low-shaft Furnace Slag

A project has also been initiated to investigate the use of low-shaft furnace slag for slag cement and as light weight aggregates

for insulating concrete, concrete masonry units and structural concrete. With the licensing of Low-shaft Furnace Plant for the production of 3 lakh tons of pig iron per year, the disposal of equal quantity of slag produced from such plants will be a problem for consideration at this stage. The slag produced in the Low-shaft Furnace Pilot Plant installed at the National Metallurgical Laboratory has been taken up for investigation in two directions: (i) for use as slag-cement and (ii) for use as light weight aggregates.

(i) *Slag-cement* — Slag-cement can be prepared in two ways. In one, the slag is used as a raw material and in ordinary manufacture of Portland cement from limestone and clay, it is used in place of clay. Using of slag in place of clay brings down the operational expenses, lowering the fuel and current consumption. In the second method, the granulated slag in a chemically unchanged form is mixed with Portland cement clinker and ground together along with a little gypsum. In the present investigation the second process has been studied starting with low-shaft furnace slag.

For preparing cement, the slag was granulated by rapidly chilling the molten mass as it is used from the furnace in excess of cold water. By rapid chilling, crystallization was inhibited and the mass solidified as glass. The granulated slag was first dried and crushed to —8 mesh. Iron particles in the slag were then magnetically separated. The slag was then ground to —200 mesh in a ball mill. The ground slag was evaluated for hydraulicity by correlating its chemical composition to its hydraulic property. The values obtained indicated that the slag was suitable for slag-cement manufacture.

The hydraulicity was then numerically evaluated in a mixture with Portland cement by determining its compressive strength and tensile strength. A series of samples were made by mixing Portland cement with the slag as follows:

% by wt. of Portland cement		% by wt. of ground slag
70	+	30
60	+	40
50	+	50
40	+	60
30	+	70

Similar series of mixtures were made with ground quartz sand instead of slag. Ground quartz sand being non-hydraulic, the hydraulic properties of the slag were easily determined by difference in strength (compressive and tensile) of the two series. The compressive and tensile strengths of the two series and pure Portland cement without any addition were determined according to IS: 455-1953. The tests covered ageing for 7, 28 and 90 days in the case of compressive strength and 3, 7, 28 and 90 days in the case of tensile strength, the test specimens being stored in water. The results obtained indicated that the granulated low-shaft furnace slag possessed good hydraulic properties. After determining the hydraulic properties of the slag, the slag-cement was prepared by mixing granulated slag and Portland cement as activator in different proportions. The chemical composition, and compressive strength of the prepared slag-cement were determined according to the requirements of IS: 455-1953. Further work is in progress.

(ii) *Light Weight Aggregate* — Light weight aggregate produced from the blast furnace slag is becoming increasingly important to the steel, slag and construction industries. The scope for utilization of slag can broadly be classified into the following categories:

- (i) For insulating concrete
- (ii) For concrete masonry units
- (iii) For structural concrete

Slags for utilization as aggregates should have the following major characteristics:

- (i) Light weight
- (ii) High compressive strength
- (iii) Proper size gradation

(iv) Sufficiently dry for economical transportation and efficient mixing

(v) Free from objectionable colouration

To achieve these properties, it is essential that the slag should be treated in the first instance by applying limited quantity of water contrary to the practice for granulated slags where relatively far larger quantities of water are used. This gives rise to a cellular type of structure to the solidified mass with low density. Slags when processed as above can be used for the preparation of light weight aggregate. They have relatively high structural strength, good insulating and acoustical properties. Such slags also find extensive application as a building material in the form of moulded concrete bricks and blocks. They can be made into bricks of larger size than those made from clay.

Available work in this field shows that slag of the following composition can be used for the production of expanded form of slag for its ultimate use as light weight aggregate (Table 30).

Realizing the importance of the subject, this project has been taken up for studies at the National Metallurgical Laboratory, for the utilization of low-shaft furnace slag for use as light weight aggregate as discussed above. The slag from the low-shaft furnace was carefully treated with controlled amount of water and nearly a ton of expanded slag

was processed and crushed in a jaw crusher and subsequently in a roll crusher and sieved to the following sizes: $+\frac{3}{4}$ in., $-\frac{3}{4}$ in., $+\frac{1}{2}$ in., $-\frac{1}{2}+\frac{3}{8}$ in., $-\frac{3}{8}$ in. + 4 mesh, -4 + 8 mesh, -8 + 12 mesh, -12 + 40 mesh, -40 mesh.

The unit weight of the processed slag has been assessed as per A.S.T.M. specifications and it has been found that the aggregates produced from the low-shaft furnace possess lower unit weight as compared to the specifications prescribed for such purposes. A few samples of bricks and slabs were also cast from this light weight aggregate and further trials are under way to assess the scope of low-shaft furnace slag as light weight aggregate for insulating concrete in the first instance.

Investigation on Failures of Metals in Services

Investigations undertaken during the year under this group include an investigation into the cause of failure of steel members which form part of a 132 kV. double circuit tower which had collapsed in service. A detailed investigation was made and the circumstances under which the failure of the tower had taken place were indicated. Another interesting case investigated in this Laboratory pertained to failure of crank

TABLE 30 — CHEMICAL ANALYSIS OF SLAGS FOR USE AS LIGHT WEIGHT AGGREGATES

FURNACE SLAG	SiO ₂ %	Al ₂ O ₃ %	FeO %	CaO %	MgO %	S %	MnO %
1	38.64	13.86	0.65	43.76	1.96	1.26	0.30
2	37.26	14.84	0.65	43.51	2.32	1.28	0.58
3	38.04	14.40	0.65	43.58	2.18	1.26	0.54
4	38.04	12.61	0.18	44.54	2.06	1.07	0.54
5	35.96	11.88	0.58	41.94	8.51	1.60	0.39
6	36.90	12.29	0.39	38.26	10.66	1.95	0.52
7	34.28	12.24	0.52	42.41	9.30	1.78	0.36
8	35.60	14.65	0.16	40.87	7.42	0.92	0.36
9	36.60	11.00	0.50	42.00	8.00	2.00	1.00
10	42.20	7.51	0.39	42.05	4.65	1.14	1.89
11	38.54	8.14	0.26	45.90	4.66	1.11	1.08
12	35.78	8.77	0.39	48.65	4.63	1.09	0.65
13	39.20	10.00	0.33	44.54	5.10	1.28	0.72
14	35.98	9.76	—	43.98	6.93	1.71	—

shaft of a Perkin P-6 engine. A comprehensive investigation with data obtained through chemical analysis, magnetic crack detection, hardness survey, mechanical tests, and metallographic examination showed that while the chemical composition and mechanical properties of the steel could be considered satisfactory, severe de-carburization had taken place in areas adjacent to fractured places and due to such severe de-carburization, endurance limit of the materials was seriously affected. Non-metallic inclusions embedded in this region appeared to have acted as potential 'stress risers', thus providing the points of origin for the start of fatigue cracks. This theory was confirmed by the microscopic evidence which clearly indicated that fine cracks had emanated from pointed edges of elongated non-metallic inclusions. Most of such investigations carried out called for on-the-spot study from the research staff of the Laboratory and the results of such study had greatly helped the industries to know the causes of such failures in service and thus had

greatly helped them to avoid recurrences of such failures in future, during operation.

Project Reports

Project reports were also prepared at the specific request of industries. During the period under review a project report for the establishment of a 600 ton/annum malleable iron foundry was prepared. In the project report, general consideration and scope of application of malleable iron castings, the present level of production and estimated demand of malleable iron castings were furnished. The manufacturing technology of malleable iron castings was fully discussed in the light of the raw materials available in this country and a detailed project report was furnished with full details of plant and equipment including capital expenditure, recurring expenditure, cost analysis, profit and loss account and annual turnover. Another project report is under preparation for the establishment of 3000 ton/annum malleable iron castings.

ENGINEERING SECTIONS

Design Engineering

During the year, eleven major development and design projects were planned and executed for different pilot plants and laboratory requirements of the National Metallurgical Laboratory as per details furnished below.

(i) *Side-blown Converters* — Two basic lined converters of capacities 500 kg. and 1500 kg. were developed and designed for the processing of indigenous high phosphorus pig irons. The salient features of the converters are two-tier tuyeres with independent wind feeding pipes, a D-shaped melt well, a refractory lining contoured to minimize erosion, quick open inspection door at the tuyeres, oxygen enrichment line, butterfly valves, solid shafts with an eccentric wind receiver, etc. Fabrication work of both the converters was undertaken in the workshop of the Laboratory.

(ii) *Continuous Vapour Degreaser* — A vapour degreaser for processing steel wire continuously was developed, designed and detailed for inclusion in the pretreatment line of the wire hot-dip aluminizing plant, also designed and fabricated earlier at this Laboratory.

(iii) *Tunnel Dryer* — A tunnel dryer, 10 m. long and 1.6 m. wide, capable of accommodating five rail cars carrying a gross brick load of 5000 kg., has been fully designed and detailed for fabrication. The tunnel has been designed for continuous batchwise drying of green bricks in five chambers which may be isolated from each other by means of special insulated drop doors. A finned tube air heater has also been designed for delivering up to 2000 cfm. at temperatures up to

120°C. to the dryer. The operation is expected to be fully instrumented.

(iv) *Vertical Reduction Kiln* — A novel type of vertical furnace for processing granular materials continuously and counter-currently to the flow of processing fluid was developed for particular application to low-grade ores. The furnace, 6 m. tall and 1 m. in dia., has been provided with a special type of water seal and a rotating bottom trough equipped with a new type of elevating arrangement for the continuous discharge of the processed solids. Thermocouple wells have been provided along the entire length of the kiln for an exhaustive evaluation of the reactions. A special gas distributor was developed for use in the kiln to minimize channelling and the formation of gas pockets (hanging).

(v) *Manganese Dioxide Electrolytic Cell* — A jacketed lead-lined prototype with heating coils was designed and detailed for the production of 50 kg. electrodeposited manganese dioxide per run.

(vi) *Down-draft Kiln* — A high temperature kiln with a design temperature of 1650°C. was detailed for the firing of dried green refractory bricks.

(vii) *Ceramic-cum-metallic Recuperator* — For preheating the secondary air in the down-draft kiln, a metallic recuperator utilizing ceramic tubes for conducting the exhaust kiln gases through counter-current and cross-flowing air was developed and designed for a maximum thermal output of 60,000 k/cals per hour to yield up to 300 Nm³ per hour of hot secondary air at 600°C.

(viii) *Cyclonic Dust Catcher* — A new type of dust catcher working on the conventional cyclonic action of such equipment was

designed for in-line use with the vertical reduction kiln. The novel feature of this catcher was a device for entrapping entrained particles in the 'clean gas'.

(ix) *Hi-temperature Furnace* — A cylindrical cavity furnace utilizing globar elements was detailed. A special carrier permitting both horizontal and vertical operation of the furnace was also designed.

(x) *Strip Aluminizing Plant* — A complete package plant design is on the boards for the aluminizing of steel strip up to 400 mm. wide continuously by the hot-dip process. The project includes skelp handling equipment; a full pretreatment line containing slack pits, vapour degreaser, pickler, swillers, scrubbers, aqueous and oil fluxers; a dryer and preheater utilizing waste furnace gas; and an aluminizing bath with melting furnace. Pinch rolls, drag bridles, jockey rolls, guide rolls, etc., have been specially designed to ensure smooth operation to design specifications. A novel feature of the hot-dip aluminizing bath consists of two non-rotating immersed rolls oriented to 'finish' both sides of the strip while still in molten aluminium to yield an even coat of uniform thickness, free of blemishes.

In addition to the steel strip aluminizing project, which is on the drawing board and scheduled for completion in September of this year, a finishing centrifuge designed for hot aluminized hardware and a one-ton open-hearth furnace design are projected in the immediate future.

Electronics Engineering

During the year under review, considerable research and development work were done in evolving design and fabrication of special apparatus and equipment required for research purposes for the various divisions in this Laboratory. A hot-stage microscope was designed and fabricated for high temperature studies on the phase equilibria of refractory materials and slags. In this setup, a thermocouple was used as the melting

element which also served as the high temperature measurement device. A high temperature controller was also designed and fabricated for a Pt-Rh wound furnace for studies on phase equilibria. The furnace winding itself was utilized as one arm of the Wheatstone Bridge, the unbalanced current being amplified by servo-amplifier and fed to a two-phase servomotor driving a variac. A potentiostat was developed for indicating the potential variation between metal samples and standard electrodes in corrosion studies. For investigations on refractory materials, a thermal balance was designed and fabricated using a D.C. amplifier.

Research work was undertaken for the development of a Photoelectric Temperature Controller using a differential D.C. amplifier. During this period, considerable work was also done in connection with the modification and calibration of the Wigen-Muirhead Decade oscillator, Intervalometer, Differential Temperature analyser, Kelvin Highest Temperature controller, etc.

Electrical Engineering

(i) *Assessment of Power Requirement and Design of Power Substation* — An exhaustive electrical load survey was carried out to assess the power requirement of the National Metallurgical Laboratory up to the end of the Third Five Year Plan. The peak load up to the end of 1959 was found to be 415 kVA. while the connected load was approximately 1100 kVA. Taking into account the anticipated high power consuming loads, it was estimated that the total connected load of the Laboratory will be 5000 kVA. including the equipment projected for installation during the Third Five Year Plan. After considering several factors *inter alia* electric tariff and agreement with power supply company and the economics of utilization of electric power, the peak load was assessed at 3000 kVA. and taking into account the progressive increase in the load, the peak load was finally fixed at 3500 kVA.

Similar survey was carried out for the pilot plant for the beneficiation of low-grade manganese ores and other plants to be installed in the new area by the side of the Low-shaft Furnace Plant such as graphitization furnace pilot plant, oxygen plant, etc. Taking also the extra power needed into account, it was felt that a H.T. transformer of 1000 kVA., 6600 volts would meet the demand adequately. The high voltage power will be taken from the low-shaft furnace project. A detailed estimate for Rs. 2.77 lakhs was prepared for the substation, H.T. and L.T. power distribution and water supply in the plant.

(ii) *Design and Fabrication of Graphitization Furnace* — For fabrication of the high temperature graphitization furnace which is required to attain a temperature of 2500°C. a special electrical transformer, which is not available indigenously, was designed at this Laboratory and fabrication work is under way. Considerable progress has already been made in this direction. Following are the salient features of this transformer:

- (a) Very low variable voltage in secondary in the range of 6-12 V.
- (b) High secondary current of the order of 7000 amps.

(iii) *Design and Fabrication of Equipment for Laboratory Experiments*

- (a) Life test apparatus was designed and fabricated for testing the life of heating elements developed in the National Metallurgical Laboratory.
- (b) Thermal conductivity apparatus was designed, erected and commissioned.
- (c) Magnetic vibrating feeder was designed and its fabrication is under progress.
- (d) Several high temperature tubeless furnaces were designed, fabricated and tested.

(iv) *Electrical Power Distribution to Pilot Plant Equipment* — Complete power distribution design, erection and commissioning of various electrical equipment were done in connection with 0.8 ton arc furnace, 450 kW. electrode boiler, 3000 and 2000 amp.,

0-10 V. D.C. rectifiers and transformers, etc for the various pilot plants.

(v) *Electrical Installation of Colombo Plan Equipment* — Complete design of power cables, installation and commissioning of the fully automatic controlled 500-ton extrusion press and two high/four high rolling mills were also carried out during the year.

During the year under review, repairs of diverse research equipment were also attended.

Mechanical Engineering

During the year under review, several items of heavy equipment for the pilot plants under the research projects under way at the National Metallurgical Laboratory were designed and fabricated. In most of the equipment, the gears, the drivers and all other mechanical parts were designed and fabricated at the National Metallurgical Laboratory. An autoclave for use in extractive metallurgy technique was so designed so as to be capable of rotating in horizontal, vertical or inclined position at a slow rate inside the electric tubular furnace. A 0.5 ton side-blown converter was also fabricated at the National Metallurgical Laboratory. The shell of the converter was fabricated with a 10-gauge steel sheet, and considerable reinforcement of the shell was introduced.

In connection with the study of the isothermal transformation characteristics of Indian steels, equipment for austenitizing and isothermal holding was designed and fabricated. The apparatus was so designed that three specimens could be quenched at a time in three quenching baths provided, after austenitizing in the three different electrical heating soaking furnaces kept at different temperatures. The mechanical set-up was so designed that the furnaces and the quenching baths could move in a vertical plane, so that the specimen can be quenched without any disturbance. A number of mechanical gadgets and handling contrivances were also designed for forging operation with the 500-ton hydraulic press.

INFORMATION SERVICE

In the field of dissemination of research results to industries, technical notes of the processes developed in the Laboratory were prepared and circulated for their commercial exploitation. Practical demonstrations were held to appraise the industries of the actual know-how of the processes released free of royalty and also on premia and royalty basis such as liquid gold. Popular brochures were brought out periodically highlighting the achievements of the Laboratory and press releases were made from time to time for important announcements regarding the technical developments.

Publications

NML Technical Journal — The *NML Technical Journal*, a quarterly, has stepped into the third year of publication. It is gratifying to state that this journal has attained a set-up at par with other leading technical journals in India and abroad and several technical journals have been acquired to the library on exchange basis. It will not be out of place to mention that the *NML Technical Journal* has not only been well received by all the research and industrial organizations both at home and abroad, but also the technical articles published in the journal are being abstracted by leading abstracting organizations and scientific journals all over the world.

Monograph — A monograph on *Foundry Moulding Sands of India* embodying results of several years of exhaustive investigation work carried out by the Laboratory was also brought out. With the growth of foundry industry in India and increased demand for castings of close tolerance limits, the choice

of the right type of foundry sands and other moulding materials is of paramount importance. Until a few years ago, the foundries in India did not have at their disposal adequate technical data on the indigenous resources of moulding sands and considerable difficulties were experienced by the foundries in proper planning and rational utilization of the indigenous sands. In the context of the rapid developments taking place in the foundry industry, it is hoped that this monograph will fill the lacuna experienced by the foundries and will enable the foundries to formulate their sand practice on scientific lines based on the available resources of indigenous sands and bonding clays. It is needless to add that the research and development work on such a subject can never be deemed as complete as additional sand deposits are being explored and located by the Geological Survey of India. The results of further studies will be published in similar monographs from time to time.

Proceedings of the Symposium — During the year under review, the Proceedings of the Symposium on *Pilot Plants in Metallurgical Research and Development*, which was held under the auspices of the National Metallurgical Laboratory in February 1960, was brought out within a record time of four months. The publication, containing 27 authoritative technical papers, brought out with a full account of the discussions during the Symposium, was widely appreciated by all the delegates and was also well reviewed in many of the technical journals at home and abroad. The Symposium on "Light Metal Industry in India" was organized during the year under review which was attended by leading technologists from all

over the world. The condensed versions of all the papers presented during the Symposium were circulated in the form of booklet to all the delegates much in advance of the Symposium to stimulate discussions during the technical session.

Besides the above nearly four hundred papers have been published up to date in Indian and foreign journals. The details of the publications during the year under review are furnished in Appendix I.

Colloquia

Regular colloquia were arranged in which the research workers and other staff members of the Laboratory were given an opportunity to give an account of the work done by them for the benefit of others. During the period colloquia on the following subjects were held:

initiated earlier was further strengthened to keep the research workers posted with the processes, techniques, apparatus and material developed and patented in diverse metallurgical fields. Such a patent survey has been found to be of invaluable assistance in the commercial applicability of the Laboratory's inventions and will also help in the active pursuit of research work in fruitful directions. Patents thus surveyed cover all the patents taken in India and those reported overseas that have a bearing on the research and development work in the National Metallurgical Laboratory both on short and long-term basis. Weekly technical bulletins and handouts are given to different divisions of the National Metallurgical Laboratory giving highlights of the metallurgical research and development themes at home and abroad with a particular bias for the research and development work under way at the National

<i>Subject</i>	<i>Speaker</i>
1. Some application on X-ray powder diffraction methods in refractories research	Mr. M. R. K. Rao
2. Stability of retained austenite on ageing	Mr. C. Dasarathy
3. Studies on transformations of steels	Dr. R. C. Karnatak
4. Corrosion research in U.K.	Mr. A. K. Lahiri
5. Formulation of alkaline copper-plating salt	Mr. S. K. Ray
6. Electric steel melting	Mr. R. D. Gupta

Translation Service

Articles in French, German and Russian of direct interest to the research work were translated into English for the reference of all concerned. Oral translations were also rendered to give the research workers a quick and approximate idea of the scope and objectives of some technical publications and to exactly locate the portion of the text of direct interest to him. Besides, many other routine translation work were also attended.

Patents and Technical Information

The survey service of published patent literature on metallurgical and related topic

Metallurgical Laboratory and that projected on short and long-term basis; these services have proved to be of invaluable help to the National Metallurgical Laboratory research workers.

Library and Documentation Service

The library of the National Metallurgical Laboratory occupies a central place in its activities and has been organized on most modern lines. The entire holdings of the library — books, bound volumes of periodicals, pamphlets, and patent specifications — as on March 1961 are about 19,000, exclusively on metallurgy and allied subjects. They

are accessioned, classified according to the latest Universal Decimal Classification schedules issued by the British Standards Institution, and thoroughly catalogued in complete sets of authors, titles, and classified on cards with cross-references. The library affords all its readers open access to its shelves, all bays of which are clearly labelled with subject and classification headings. The library subscribes to 280 technical journals on all technical subjects allied to metallurgy, and receives complimentary and exchange copies of a further 125 periodicals.

The library has also developed an elaborate documentation service. Card abstracts (in printed 5×3 in. cards), issued by the Iron & Steel Institute of U.K. which have practically a worldwide coverage in ferrous metallurgy, are being subscribed. A weekly index of technical articles is also compiled by the library which, in addition to non-ferrous metallurgy not covered by the Iron & Steel Institute cards, supplements the ferrous metallurgical information given in it by including and highlighting information of specific importance to Indian metallurgy and projects in hand at the National Metallurgical Laboratory. Together, the Iron & Steel Institute abstracts cards and the cards (6×4 in. typed) from the IOTA (Index of Technical Articles) constitute the most advanced documentation service rendered by the library. The IOTA card file provides the most appropriate and up-to-date coverage much in advance of subscribed abstract services, such as Metallurgical abstracts and Chemical abstracts, and gives a more specific and complete coverage than any of these.

Technical Aid to Industries

Technical advice rendered to various industrial organizations and Government bodies has increased several folds both in the quantum of technical advice rendered as well as in the number of short-term investigations and specification tests undertaken. Two hundred and twentythree technical enquiries

were attended and forty short-term investigations and specification tests on behalf of the industries were conducted during the period under review. Details of some of the enquiries and short-term investigations undertaken during the year are furnished below, which will give an idea about the wide range of subjects dealt with.

(i) Technical Enquiries

1. Moulding of non-ferrous metals by using plaster of Paris along with the sand.
2. Sands suitable for mild steel castings.
3. Alloys for the manufacture of magnets for miniature toy motors.
4. Composition and mode of heat treatment of wire-drawing dies and cutting tools.
5. Process of plating on zinc base die casting.
6. Process of bluing the steel parts.
7. Availability of sand, plumbago powder, limestone and silica chips for use in foundry.
8. Grades of sand and resins suitable for castings.
9. Technical advice on selection of site for setting up a refractory plant in Gujarat.
10. Selection of suitable aluminium alloys for structural purposes.
11. Details of temperature range, type of crucible for melting ferro-cerium alloys.
12. Technical data on CO₂ process in foundry production of moulds and cores.
13. Technical know-how for plating on nibs.
14. Production technique for sintered permanent magnets of Alni and Alnico types.
15. Technical advice on the selection of aluminium alloys for collapsible huts.
16. Specification of steels used for different wire products.

17. Manufacturing process of artificial corundum from bauxite.
 18. Manufacture of dental alloys.
 19. Manufacturing technique for ferromanganese and copper alloys.
 20. Alloys for manufacturing high pressure gas cylinder to stand test pressure of 3500 lb./sq. in.
 21. Process for pickling of iron and steel.
 22. Technique for improving the electrical conductivity of commercial aluminium.
 23. Details of the process of manufacturing nickel salts.
 24. Application of forsterite and dolomite refractories, and availability of raw material for their production under Indian conditions.
 25. Production of corrosion resistant cast irons.
 26. Electrolytic refining of silver.
 27. Re-melting of stainless steel scrap.
 28. Manufacturing technique for chill rolls.
 29. Manufacturing technique of Fe-Mn.
 30. Corrosive effects of chlorides on lead lining and pipe.
 31. Manufacturing technique of german-silver wire and brass rods.
 32. Manufacturing details of brass and bronze powders.
 33. Nitriding of cast iron cylinder liners.
 34. Manufacturing details of hard bright wire.
 35. Manufacturing process of aluminium shots and foils.
 36. The working diagram and details of photoelastic bench and other allied equipment.
 37. Aluminium alloy castings for automobile pumps.
 38. Corrosion resistant stainless steel for fatty acid plant.
 39. Manufacture of abrasives from molten bauxite.
 40. Resistivity and tensile strength of aluminium wire for cables.
 41. Hardening of cores and moulds with sodium silicate and CO_2 .
 42. (i) Scaling loss in furnaces and their causes.
(ii) Excess fuel consumption and their remedial measures.
 43. Technique of rolling nickel-silver alloys.
 44. Development programme in the field of industry regarding possibility of utilization of quartz for production of silicon carbide.
 45. Direct reduction of iron ore for the production of sponge iron.
 46. Recovery of zinc from galvanizing dross.
 47. Detailed scheme for the manufacture of zinc dust.
 48. Recovery of gold from goldsmith's sweepings.
 49. Stainless steel suitable for the reactor tube used in activation of coke.
 50. Compounds used for compounded steel tape for use in armoured cable.
 51. Details of the process of production of electrolytic chromium.
 52. Manufacturing technique of Ni-Cr wire for heaters.
 53. The manufacturing process of alum and alumina from bauxite.
 54. Technique of boring the diamond for the manufacture of diamond dies for wire drawing.
 55. Causes of failure of thermocouples in atmosphere of hydrogen, nitrogen and ammonia gases.
 56. Details of the process of fusing magnesia.
 57. Technique for welding steel sections horizontally.
- (ii) *Short-term Investigations and Specification Tests for the Industry*
1. Suitability of Ramming mixes.
 2. Analysis of scales present inside the broken tube.
 3. Determination of oxygen content in nickel casting.
 4. Recovery of zinc metal from zinc-copper slime.

5. Examination of the wear and tear properties of grinding media.
6. Metallurgical testing of the material of bobbin flange.
7. Investigation on
 - (a) The samples of steel ingots and slabs.
 - (b) Failure of ingot mould.
 - (c) Quality of steel slabs in respect of tensile test, impact test.
8. Causes of failure of fallen steel tower.
9. Method of tempering of hosiery needles.
10. Investigation on "Failure of Big End Bolts for Air Compressor".
11. Nitrogen estimation of 3 samples of steel.
12. Causes of
 - (a) Corrosion of brine storage tank.
 - (b) Corrosion of nickel tubes in steam chest of caustic soda evaporator.
13. Low temperature Charpy impact tests of steel specimens.
14. Failure of a crank-shaft of Perkins P-6 engine.
15. Failure of boiler tube.
16. Estimation of hydrogen in 6 samples of steel.

Processes Patented

During the year under review consolidated technical notes of the following patented processes developed in this Laboratory were prepared and circulated:

1. (a) A process for the hot-dip aluminizing of ferrous materials — Indian Patent No. 55289.
- (b) Aluminizing of iron and steel — Indian Patent No. 57938.
- (c) Improvements in or relating to hot-dip aluminizing of steel — Indian Patent No. 65230.
2. (a) A process to produce dense carbon aggregates from carbonaceous materials of varied volatile content — Indian Patent No. 62938.
- (b) Production of carbon pastes for use in the continuous Soderberg

electrodes — Indian Patent No. 65696.

3. An improved method for producing nitrided manganese — Indian Patent No. 62338.

A large number of industries have expressed keen desire to utilize the above processes.

The following patents were accepted during the period under review:

1. Improvements in or relating to hot-dip aluminizing steel — Indian Patent No. 65230.
2. A process for electrolytic production of iron-chromium alloys from chromite ore — Indian Patent No. 65556.
3. Improvements in or relating to the production of chemically bonded metal clad or unclad basic refractories — Indian Patent No. 65610.
4. Production of carbon pastes for use in the continuous Soderberg electrodes — Indian Patent No. 65696.
5. Refractory compositions containing non-refractory chrome ore and refractory products made therefrom — Indian Patent No. 68174.
6. Compositions and method of making welding flux — Indian Patent No. 68171.
7. A pneumatic or such other forge hammer adapted for the manufacture of bricks or blocks of ceramic mixes — Indian Patent No. 68401.

The following patents were sealed during the period under review:

1. Refractory compositions comprising graphite and alumino-silicate materials and glazes to render such compositions resistant to oxidation — Indian Patent No. 62352.
2. A process for recovering zirconium dioxide from zircon — Indian Patent No. 63904.
3. Improvements in or relating to hot-dip aluminizing steel — Indian Patent No. 65230.
4. An improved method for the production of chromium-manganese alloys

by aluminothermic reaction — Indian Patent No. 65231.

5. A process for electrolytic production of iron-chromium alloys from chromite ore — Indian Patent No. 65556.
6. Production of carbon pastes for use in the continuous Soderberg electrodes — Indian Patent No. 65696.
7. New stainless steels and methods of preparing them — French Patent No. 1, 208, 393 and U.K. Patent No. 833308.

The following patents were released to industries on royalty and premium basis:

1. Liquid Gold (Non-patented Process).
2. Refractory compositions comprising graphite and silicon-carbide — Indian Patent No. 58869.
3. Refractory compositions comprising graphite and aluminosilicate materials and glazes to render such compositions resistant to oxidation — Indian Patent No. 62352.
4. An improved process for the production of electrolytic manganese metal — Indian Patent No. 49355.
5. An improved process for electrolytic production of high purity manganese dioxide — Indian Patent No. 47982.

Practical Demonstration

Practical demonstration of the process on "Liquid Gold", released on royalty and premium basis, was arranged for the benefit of representatives of the firms who have entered into final agreement for its commercial exploitation.

Symposium

A Symposium on "Light Metal Industry in India" was organized by the National Metallurgical Laboratory at Jamshedpur from February 14 to 17, 1961, to focus attention on the latest research and industrial development in the field of light metals and their alloys and with a view to exchange technical know-how with the leading scientists

and metallurgists from different parts of the world. The Symposium was inaugurated by Professor M. S. Thacker, Director-General, Scientific & Industrial Research. Sir J. J. Ghandy, Kt., C.I.E., Director-in-Charge, Tata Iron & Steel Co. Ltd., and Chairman of the Executive Council of the National Metallurgical Laboratory, presided.

Sir Jehangir Ghandy, Chairman, Executive Council of the National Metallurgical Laboratory, in his presidential address, stated that research on metals is to continue to ensure progress for the development of light metals and their alloys with superior structural stability at sub-zero and elevated temperature uses. In this context, he commended the good work done by the National Metallurgical Laboratory in the metallurgy of light metals and formulation of light metals and their alloys based on indigenous alloying elements such as aluminium, magnesium, rare earth groups of metals, etc. He extended, on behalf of the Executive Council of the National Metallurgical Laboratory, their sincere thanks and deep appreciation to Dr. B. R. Nijhawan, Director, National Metallurgical Laboratory, and his colleagues for valuable research and development work being done by them at the Laboratory and also for the adequate arrangements made for the Symposium.

Professor M. S. Thacker, in his inaugural address, stressed the need for comprehensive planning to raise the economy and the standards and pleaded that serious thought should be given to integrate the survey of all the resources available in India. The progress should be continuous and not spasmodic, for which, Professor Thacker said, some organization has to be created to integrate, co-ordinate and continuously plan for the effective development of the indigenous resources. In this context, Professor Thacker expressed great satisfaction and paid a warm tribute to Dr. Nijhawan and his staff for the excellent work they were doing towards the development of metal and mineral industries of the country. Referring to the technical



FIG. 40 — PROF. M. S. THACKER, DIRECTOR-GENERAL, SCIENTIFIC & INDUSTRIAL RESEARCH, DELIVERING THE INAUGURAL ADDRESS AT THE SYMPOSIUM ON "LIGHT METAL INDUSTRY IN INDIA"

papers presented at the Symposium, Professor Thacker expressed the hope that contributions and technical exchange of views arising out of the deliberations of the Symposium will be very valuable for the development of the light alloy industry in India.

Dr. B. R. Nijhawan, Director, National Metallurgical Laboratory, in welcoming the distinguished delegates from India and abroad, referred to this Symposium as a 'metallurgical summit' in having attracted valuable technical contributions from specialists in their fields and also in having gathered together distinguished scientists, metallurgists and technologists from many parts of the world. Research and development in light metals and their family of alloys,

Dr. Nijhawan said, presented a wide spectrum ranging from experiments on laboratory scale based on speculations arising out of applied or fundamental hypotheses and from study of odd-looking samples to organized pilot plant scale investigations and heavily financed industrial trials. Between the two ends of the spectrum, Dr. Nijhawan said, the Industrial Research Laboratory, like the National Metallurgical Laboratory, has been playing an useful role in diverse fields of research and development in light metal and their family of alloys, providing a vital link with the planned growth of Indian minerals and metal industries.

The Symposium drew a large gathering of top-ranking scientists and technologists from all over the world like Mr. W. E. Bardgett,



FIG. 41 — SIR JEHANGIR GHANDY, CHAIRMAN, EXECUTIVE COUNCIL OF THE NATIONAL METALLURGICAL LABORATORY AND DIRECTOR-IN-CHARGE, THE TATA IRON & STEEL CO. LTD., DELIVERING THE PRESIDENTIAL ADDRESS AT THE SYMPOSIUM ON "LIGHT METAL INDUSTRY IN INDIA"

President, Institution of Metallurgists, U.K. and Research Manager, Research and Development Department, Swinden Laboratories, Moorgate, Rotherham, U.K.; Mr. T. Benson-Gyles, W. S. Atkins & Partners, London; Mr. J. Blakiston, Engineering & Foundry Consultant, Halifax — representing the Institute of British Foundrymen; Mr. Thomas G. Digges, Chief, Thermal Metallurgy Section, National Bureau of Standards, Washington, U.S.A. — also representing the American Society of Metals, U.S.A.; Mr. Bengt R. F. Kjellgren, Chairman of the Board, The Brush Beryllium Company, Cleveland, Ohio, U.S.A.; Mr. Vijay K.

Chandhok, Staff Research Metallurgist, Crucible Steel Company of America, Pittsburgh, U.S.A.; Mr. D. MacAskill, Dorr-Oliver Incorporated, Stamford, Connecticut, U.S.A.; Dipl. Ing. F. Ostler, Otto Junker GmbH., Lammersdorf, W. Germany; Prof. G. Reginald Bashforth, Unesco Expert in Ferrous Production Metallurgy and Professor of Metallurgy, College of Mining and Metallurgy, Banaras Hindu University, Varanasi.

Twenty-seven technical papers covering the various aspects of research and development work on light metals and alloys and their production and properties were presented and discussed in six technical sessions.

The first day's technical session had four papers on Scope for Research and Development in Light Metals. The first paper entitled "Status of Beryllium Industry in the U.S.A." by Mr. Bengt R. F. Kjellgren, Chairman, The Brush Beryllium Co., Cleveland, U.S.A., dealt with the growth of the beryllium industry in U.S.A., with particular stress on the raw materials position, extraction techniques and fabrication processes adopted.

Dr. B. R. Nijhawan, Director, National Metallurgical Laboratory, in his comprehensive paper on "The Role of the National Metallurgical Laboratory in the Research and Development of Light Metal Industry in India" reviewed, in the background of the present position of light metal industry in India and the current expansion programme projected during the Third Five Year Plan, research and development work under way

at the National Metallurgical Laboratory on light metals and alloys such as production of titanium sponge by Kroll's method, silico-thermal reduction of dolomite for production of magnesium, certain aspects of electro-metallurgy of zirconium and beryllium ranging on to secondary subjects, such as production of modified aluminium-silicon alloys by alumino-thermal reduction of quartz, aluminium-titanium alloys, aluminium-magnesium alloys and passing on to fields, such as aluminizing of steel wire and sheets, production of suitable carbon paste for Soderberg electrodes from indigenous raw materials, etc. Dr. Nijhawan stated that considerable stress has been laid by the National Metallurgical Laboratory on the development of light alloys based on indigenous alloying elements. Concluding, he stressed that although in most of the im-



FIG. 42 — DR. B. R. NIJHAWAN, DIRECTOR, NATIONAL METALLURGICAL LABORATORY, WELCOMING THE DELEGATES TO THE SYMPOSIUM ON "LIGHT METAL INDUSTRY IN INDIA" ON THE INAUGURATION DAY

portant light metals and their alloys Indian resources can, by any metallurgical standard, be regarded as classic, yet the same cannot be claimed concerning their past or projected industrial growth despite the tempo of the successive Five Year Plans. It is in this context that due emphasis on the development of light metal alloy industry in India has to be rightly accorded wherein the role in research and development work is supreme, to which the National Metallurgical Laboratory is wholly dedicated.

"The Status of Titanium in 1961 — A New Structural Metal Starting Its Second Decade" was the topic of another paper by Mr. Vijay K. Chandhok, Staff Research Metallurgist of the Crucible Steel International, Pittsburgh, U.S.A. The paper

also described a new "Isoplastic strain" diagram for comparing the time-temperature-load bearing properties of titanium with other structural metals.

A comprehensive review of the research programme of the Metallurgy Division of the National Bureau of Standards was presented in a paper by Mr. Thomas G. Digges, Chief, Thermal Metallurgy Section, National Bureau of Standards, Washington, U.S.A. A number of projects on fundamental and applied aspects of light metals like evaluation of the effect of notch geometry on the flow, fracture and ductility of titanium and single crystals of aluminium, stress corrosion of magnesium and underground corrosion of aluminium alloys, fatigue of aluminium, nitriding titanium, etc., were described.



FIG. 43 — DELEGATES AT THE SYMPOSIUM ON "LIGHT METAL INDUSTRY IN INDIA" HELD FROM 14 TO 17 FEBRUARY 1961 AT THE NATIONAL METALLURGICAL LABORATORY



FIG. 44 — A GROUP PHOTOGRAPH OF THE DELEGATES WHO ATTENDED THE SYMPOSIUM ON "LIGHT METAL INDUSTRY IN INDIA"

The second day of the Symposium had ten papers on the studies and development in reduction and working of aluminium and its alloys. In a paper on "The Prospects of Aluminium in India", Dr. P. Dayal, Development Officer, Metals Directorate, Government of India, surveyed the Indian aluminium industry with regard to production, consumption and further growth, against the background of the world position. As the ore reserves for other non-ferrous metals are very inadequate, stress has to be laid to develop the aluminium industry to self-sufficiency as well as to attain some surplus capacity for export purposes.

The amenability of Kolhapur bauxite by Bayer's Process was the subject-matter of a paper by Mr. G. G. Mathad and Dr. V. A. Altekar of the Dept. of Chemical Technology,

University of Bombay. Investigations on digestion and sedimentation studies have shown that Kolhapur bauxite is one of the best ores suited for Bayer's Process.

Latest developments in aluminium reduction cells were the theme of a paper by Dr. Paul Muller and Mr. Roland E. Oehler of Aluminium-Industrie-Aktien-Gesellschaft, Zurich, Switzerland. Two types of cells are used for reduction purposes. Different types of arrangements of cells and various improvements made in cell design have also been described.

The assessment of raw materials employed and types of Soderberg paste produced abroad in relation to Indian resources and the research work conducted in this aspect at the National Metallurgical Laboratory were the subject-matter of a paper entitled

"A Study of the Production of the Soderberg Paste from Indian Raw Materials" by Messrs P. Prabhakaram and H. P. S. Murthy of the National Metallurgical Laboratory who have shown that calcined petroleum coke used normally for Soderberg paste, if converted first into dense carbon aggregate, despite any increased cost incurred thereby, will yield stronger electrodes that are more wear-resistant, possessing better conductivity.

The hazards from frictional sparks of certain light alloys were reviewed by Dr. G. N. Badami of Central Mining Research Station, Dhanbad, in which the development of light alloys free from these hazards has been advocated.

Mr. W. S. Warriar, Sales Manager of Alcan Asia Ltd., presented a paper on the use of aluminium in substitution for other materials. Aluminium-cladded sheets in various forms are being used for public, private and industrial buildings from the arctics to the tropics due to their light weight, high durability, ease of handling, and heat insulating properties, and, due to high electrical conductivity, find important use in electrical field. Aluminium has proved an ideal material for cans and offers many advantages over tinplate.

Dr. H. J. Latiere and Mr. R. Michaud of Centre de Recherches Scientifiques Industrielles et Maritimes, CNRS., Marseille, France, discussed the slip bands obtained by tension on aluminium specimens. A new concept of wet-tensile strength recently developed in West Germany and the various influencing factors were discussed by Dr. Ing. S. S. Khanna and Dr. D. Swarup of College of Mining & Metallurgy, Banaras Hindu University. The present trends in the methods of production of aluminium alloy slabs and billets for working were described in a paper by Mr. M. R. Reeve, Senior Research Metallurgist, Foseco International Ltd., U.K.

The third day of the Symposium had eight papers on development and applications of light metals. A critical review of the

aluminium industry in India with special reference to the raw material position and expansion programme was discussed by Messrs P. K. Gupta and K. N. Srivastava in their paper entitled "Development of Light Metal Industry in India". The possibilities for production and application of magnesium, titanium, etc., were also dealt with. The scope for use of light alloys, especially aluminium alloys, in railways with the present level of production and anticipated expansion was discussed by Mr. K. C. Choudhuri, Jt. Director, Research, Designs & Standards Organization, Ministry of Railways, Government of India. Discussing the relative merits of the different materials, Mr. M. K. Asundi of Atomic Energy Establishment, Bombay, stated that the demands of nuclear industry for materials of low neutron absorption, high strength and ductility, chemical compatibility and good heat transfer properties have opened a new avenue for the use of light metals. The examples cited were use of aluminium as a canning material in research reactors; magnesium alloys, such as magnax series, in power reactors; beryllium as a moderator and having high neutron scattering cross-section being used as reflector in high flux reactors.

Various methods of joining light metals, such as gas welding and brazing, metal arc welding, resistance welding, TIG and MIG, automatic arc welding and inert gas shocked spot welding along with the various developments in the respective fields, were discussed by Mr. J. A. Muliyl, Assistant Technical Sales Manager, Indian Oxygen Ltd., Calcutta. The important types of modern furnaces used in light metal industry were described along with illustration by Dipl. Ing. F. Ostler of Otto-Junker GmbH., West Germany.

The resources of magnesium minerals in India and the necessity of establishing indigenous magnesium industry in India were stressed by Messrs R. N. Misra, V. S. Sampath and P. P. Bhatnagar of the National Metallurgical Laboratory, in their paper on "Scope for Development of Magnesium

Industry in India". The work carried out in this direction in the National Metallurgical Laboratory was described and the details of layout and flowsheet of the proposed pilot plant for production of magnesium metal were given. The techniques of electrolytic production of magnesium and its alloys from its halides were reviewed by Messrs P. K. Som, P. B. Chakravarti and Dr. T. Banerjee of the National Metallurgical Laboratory, discussing in detail the difficulties experienced in the electrolytic extraction of magnesium.

The effect of misch-metal additions on the structure and workability of Al-Mg (7-10 per cent) alloys was the object of a paper presented by Dr. Y. N. Trehan, Mr. P. K. Gupte and Dr. B. R. Nijhawan. The research and development work undertaken at the National Metallurgical Laboratory have shown that misch-metal displaces the aluminium-magnesium phase by one essentially containing cerium. The hot-workability of these high magnesium alloys containing misch-metal has been correlated with the properties obtained. The structural and physical characteristics of these alloys have been studied in the cast and wrought conditions in relation to the optimum misch-metal additions. It is considered that in the background of Indian raw material conditions, the development of these aluminium based high magnesium binary alloys of aluminium, containing optimum additions of rare earth group of metals, offers good scope for applications in diverse metallurgical and engineering fields. The composition, physical properties and application of well-known cast and wrought aluminium-magnesium alloys were reviewed by Dr. Y. N. Trehan and Mr. S. P. Bhadra.

On the last day of the Symposium, four papers mainly on the study of certain fundamental aspects of light alloys were presented and discussed.

The effects of composition and thermal treatment on the natural ageing of Al-Zn-Cu alloys were discussed by Mr. J. Wood of

International Alloys Ltd., U.K. The results obtained on the structure of liquid Al-Cu alloys were presented by Dr. Rajendra Kumar and Mr. Manjit Singh of the College of Mining and Metallurgy, Banaras Hindu University. The results obtained indicated that the strength of Cu-Al bond depends upon (i) temperature and (ii) composition. A study of the changes caused by absorbed gases on the flow properties of molten metals, particularly aluminium, was reported by Mr. Kazuo Hori, Asst. Professor of Metallurgy, Faculty of Engineering, Kumamoto University, Japan. Experiments have shown that these properties are mainly dominated by the melting atmosphere on account of gas absorption in that stage. Experimental work on the determination of the corrosion rates for aircraft aluminium-copper alloys in hydrochloric acid solution was reported by Messrs K. G. Sheth, J. Sundararajan and Dr. T. L. Rama Char of the Indian Institute of Science, Bangalore. The effects of various inhibitors were studied and the data obtained were compared with pure and 92 per cent aluminium.

During the Symposium, an illuminating lecture on "The Future of Electric Furnaces in the Metal Industries of India" was delivered by Professor G. Reginald Bashforth. Film shows on "Aluminium matchplates", made by Foundry Services Ltd., and on "Hot-stage Microscopy" based on research results of the National Metallurgical Laboratory were arranged. Last, but of course not the least, the delegates were entertained, on the opening night of the Symposium, to a lively show of Indian dance and music. The Symposium concluded by visits to the National Metallurgical Laboratory and its pilot plants including the Low-shaft Furnace Project.

The Symposium Supplement — On the inaugural day of the Symposium, a special supplement was brought out in a masterly fashion by the *Amrita Bazar Patrika* — a leading daily of Calcutta. The supplement carried leading papers from several

visiting scientists from abroad, including articles contributed specially for the Symposium, which served to focus the need for development of light metals and their family of alloys, valuable work done in different centres in India on the light metals and their alloys, future trends and development of light

metal alloy industries in India. The management of the *Amrita Bazar Patrika* received adequate commendations for the excellent coverage given to the Symposium as well as to the day-to-day proceedings of the technical sessions in their newspaper.

AWARDS, DEPUTATIONS AND NOMINATIONS

AWARDS

1. Shri S. M. Arora, J.S.O., has been awarded the 'Kamani Gold Medal' for 1958 by the Indian Institute of Metals.

2. Dr. B. R. Nijhawan, Director and Mr. P. K. Gupte, Assistant Director, were awarded 'Certificate of Merit' by the Indian Institute of Metals as Co-authors of the Kamani Award, for the best paper presented at the Institute during the year 1958-59.

DEPUTATIONS

Mr. R. M. Krishnan, Assistant Director, was deputed to Sweden, U.S.A., and Japan as Member-Secretary of the Foundry Productivity Team of the National Productivity Council under T.C.M. Programme. He also attended the International Foundry Congress at Zurich and took part in the International Committee's meetings as a representative of India.

Mr. A. P. Chowdhury, S.S.O., was deputed to U.K. under the Colombo Plan for training in the design and development of electronic instruments for metallurgical research industry.

NOMINATIONS

Dr. B. R. Nijhawan, Director, has been nominated to serve as

1. Member of the Committee constituted by the Union Ministry of Steel, Mines and Fuel to consider problems in the production of iron ores and fundamental studies of various processes

2. Member of the Metallurgy Advisory Committee of the Department of Atomic Energy

3. Member of the Council of Mining, Geological and Metallurgical Institute of India

4. Chairman of the Methods of Physical Tests Sectional Committee, SMDC-3 of the Indian Standards Institution

5. Member of the Alloy Steels and Special Steel Sectional Committee SMDC-19 of the Indian Standards Institution

6. Honorary member of the Editorial Advisory Board of the Commonwealth Library of Science, Technology and Engineering, Oxford

7. Elected Vice-President of the Institute of Metals

8. Re-elected President of the Institute of the Indian Foundrymen

Dr. T. Banerjee, Deputy Director, has been nominated to serve as

1. Member of Panel of Experts in the trade of electroplater for each Engineering and Building & Non-Engineering Trade Constituted by the National Council for Training in Vocational Trades under the Directorate-General of Resettlement and Employment, Ministry of Labour and Employment, Government of India, New Delhi

2. Chairman of the reconstituted Methods of Chemical Analysis Sectional Committee of SMDC-2 of the Indian Standards Institution

Shri P. I. A. Narayanan, Officer-in-Charge, Ore Dressing, has been nominated to work as Coordinator in India for newly constituted Commonwealth Committee on Mineral Processing

Dr. E. G. Ramachandran, Assistant Director, has been nominated to serve as

1. Member of the Copper and Copper

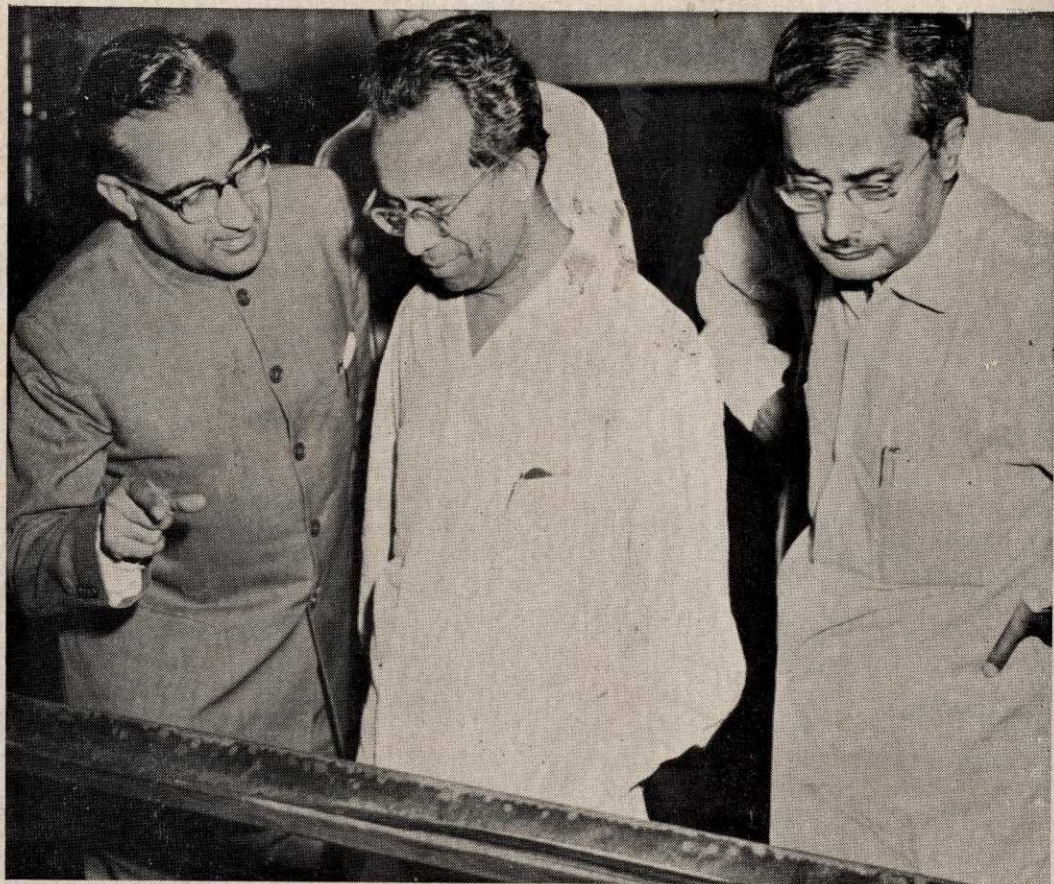


FIG. 45 — PROF. HUMAYUN KABIR, MINISTER FOR SCIENTIFIC RESEARCH AND CULTURAL AFFAIRS, GOVT. OF INDIA, AND VICE-PRESIDENT, C.S.I.R., DURING HIS VISIT TO THE NATIONAL METALLURGICAL LABORATORY. DR. B. R. NIJHAWAN, DIRECTOR, EXPLAINING TO PROF. HUMAYUN KABIR, THE VARIOUS PRODUCTS AND PROCESSES DEVELOPED IN THE LABORATORY. DR. T. BANERJEE, DY. DIRECTOR, IS SEEN IN THE PICTURE

Alloys Sectional Committee, SMDC-11, of the Indian Standards Institution

2. Member of the Development Council for Non-ferrous Metals & Alloys of Government of India

3. Member of Structural Steel Sectional Committee, BD-7 Steel Economy of the Indian Standards Institution

Dr. A. B. Chatterjea, Assistant Director, has been nominated as Member of the Light Metals and Alloys Sectional Committee, SMDC-10, of the Indian Standards Institution

Shri P. P. Bhatnagar, Assistant Director, has been nominated to serve as

1. Member of the Wire Ropes & Its Accessories Subcommittee, EDC-32: 2, of the Indian Standards Institution

2. Member of the Pig Iron & Ferro-alloys Sectional Committee, SMDC-8, of the Indian Standards Institution

Shri P. K. Gupte, Assistant Director, has been nominated as Member of the Steel Sectional Committee, SMDC-5, of the Indian Standards Institution

Shri B. N. Das, Assistant Director, has been nominated to serve as

1. Member of Metals Standards Sectional Committee, SMDC-1, of the Indian Standards Institution

2. Member of Welding General Sectional Committee, SMDC-14, of the Indian Standards Institution

Shri H. V. Bhaskar Rao, Assistant Director, has been elected to serve as an alternate Member of Refractories Sectional Committee, SMDC-18, of the Indian Standards Institution

Shri S. S. Bhatnagar, S.S.O., has been elected to serve as Member of Ferrous Metals for Automobiles Subcommittee, SMDC-19: 2, of the Indian Standards Institution

DISTINGUISHED VISITORS

During the year under review, the following distinguished visitors visited the Laboratory:

1. Professor Humayun Kabir, Minister for Scientific Research & Cultural Affairs, Government of India, and Vice-President, Council of Scientific & Industrial Research
2. Dr. B. V. Keskar, Minister for Information & Broadcasting, Government of India
3. Shri K. Kamaraj Nadar, Chief Minister, accompanied by Shri C. Subramaniam, Minister for Finance & Education, Madras State
4. Professor M. S. Thacker, Director-General, Council of Scientific & Industrial Research and Secretary to the Ministry of Scientific Research & Cultural Affairs, Government of India
5. Dr. A. G. White and Mr. Ivar S.V.N. Nilson, Leader and Member respectively, of the Expert Team for U.N.T.A.O. for substitution of indi-

genous raw materials in selected industries in India

6. Dr. J. H. Chesters, Assistant Director, Research, and M. A. Jackson, Technical Director, United Steel Companies Ltd., Sheffield, U.K.
7. Messrs William W. Buechner, Norman C. Dahl and Louis D. Smullen, from Massachusetts Institute of Technology, U.S.A.
8. Mr. Allan Danielsson, U.T.A.O. Expert in Burma and his counterpart, Mr. U. Kyaw
9. Mr. N. Nakamura, Manager, Blast Furnace in Toleata, Yawata Iron & Steel Co. Ltd., and Mr. K. Sasai, Kinoshita, Shoten Ltd., Japan
10. Mr. D. L. Bryant of Commonwealth Industrial Gases Ltd., Australia
11. Mr. Clarence Thom, Metallurgist-in-Charge of Denver's Ore Testing Laboratory, Denver Equipment Co., U.S.A.
12. Mr. K. Enozawa & Mr. K. Harada of the Tokushu Seiko Ltd., Japan, and Mr. R. Yuil & Mr. Y. Iamnishi of the Nissho Co. Ltd., Japan
13. Professor Gogala and Dr. C. Raker, Yugoslavian Expert in Iron and Steel
14. A team of ECAFE delegates
15. A team of 15 scientists from France, United Kingdom, Denmark, Indonesia, Australia, China, Czechoslovakia, Hungary, Switzerland, Israel, Italy and Japan
16. A two-member team consisting of Shri P. B. Malla, Director, and Shri M. N. Rana, Metallurgist, of Nepal Bureau of Mines, Kathmandu, Nepal

GENERAL

The Main Building of the National Metallurgical Laboratory was constructed in 1948 after elimination of the two side wings due to paucity of funds although these were initially included in the original approved plan of the National Metallurgical Laboratory. In view of the expanding activities of the National Metallurgical Laboratory and projects projected under the Third Five Year Plan, necessary steps have been taken for the construction of the 'wings' of the Main Building of the National Metallurgical Laboratory.

This will accommodate heavy equipment and other sections of the National Metallurgical Laboratory including vacuum melting units, heat-treatment units, battery of creep-resistant testing equipment, radioactive isotope tracer technique section, research and investigation section for studies on corrosion and protection of metals and alloys, extractive metallurgy division dealing with the vacuum metallurgical and hydro-metallurgical including pressure leaching sections, besides other units of the general metallurgy division dealing with electron diffraction, X-ray diffraction sections, etc. The tenders for the new extensions were invited. Actual construction work has been initiated.

Periodical additions to the staff strength of this Laboratory have been made keeping also in view the interest of general economy. During the Third Five Year Plan period, work on research and development projects that have already been approved by the Executive Council and the Planning Commission will be initiated. In addition to the expansion programme in respect of staff, adequate strengthening of staff for the various divisions already existing was under-

taken to cope up with the increased activities of the Laboratory in diverse fields. It may be mentioned here that almost one-third of the Laboratory's research activities are related to meeting the needs of industrial development and project studies and formulation, etc., for the heavy iron and steel and non-ferrous industries in the private and public sectors. It will not be out of place to mention here that the recognition of the National Metallurgical Laboratory as an effective scientific tool to assist the giant public and private metallurgical and industrial complexes has now been fully established and consolidation thereof has taken place during the last couple of years. The result is that quite a substantial number of staff members has to go to the heavy industrial complexes plant to render effective technical aid to them on a massive scale. The total staff strength of the Laboratory now exceeds 800; this includes research and development staff in the various divisions, auxiliary technical and service engineering staff, besides those on administration and accounts.

The Governing Body of the Council of Scientific & Industrial Research at the meeting held on the 15th October 1958, had approved the establishment of 4 Regional Stations for development of foundries at Howrah, Batala, Bombay and Madras.

With the phenomenal increase in research, development and pilot plant activities of the National Metallurgical Laboratory, proportionately heavy pressure was put on the Purchase and Stores Section. The National Metallurgical Laboratory, in keeping the different technical divisions and sections, fed for their day-to-day needs, supplying breakdown spares, heavy capital equipment and

annual recurring and consumable stores in the context that bulk of these needs has to be imported; then imports have accordingly to be obtained on long-term planning. Since 1956-57 to date, there has been a 400 per cent rise in the quantum of purchase and stores work and during the year under review roughly 25,000 items valuing Rs. 23 lakhs approximately have been handled and procured. The task has been all the more arduous and heavy due to severe restrictions on imports, lack of suitable indigenous substitutes and the need, imperative and inescapable of improvisation at every level. The whole work had, therefore, to be planned effectively and projected well in advance which needed both imagination and efficiency in its effective execution.

The senior technical staff members of the National Metallurgical Laboratory met once a week under the Chairmanship of the Director to discuss the progress of research, development and pilot plant work of the Laboratory to thrash out any bottlenecks encountered, exchange technical views, and above all, to ensure adequate group and teamwork, vital to the growth of research activities anywhere.

The administrative working of the Laboratory was maintained at peak efficiency including the heavy and integrated team-work of several hundreds of the staff members. The heavy budgetary accounts of the Laboratory were handled with equal efficiency and speed as shown by the Reports of the Accountant-General, Central Revenues, of

the Government of India, relating to the National Metallurgical Laboratory.

Due to the acute shortage of residential accommodation in the steel town of Jamshedpur, the Council of Scientific & Industrial Research have drawn a programme in consultation with the Building Subcommittee for providing an adequate number of quarters for the staff of the National Metallurgical Laboratory. The housing programme has been so planned that ultimately 80 per cent of the staff can be provided with residential accommodation facilities. Total expenditure covered by the above-phased construction work of housing for National Metallurgical Laboratory staff exceeds Rs. 25 lakhs.

It is gratifying to report that since taking over the construction of the staff quarters by the Council of Scientific & Industrial Research, the progress of construction of staff quarters for the National Metallurgical Laboratory has been significant as can be seen from the fact that 2 'C' type quarters have already been completed. The construction of 10 numbers of (multi-storied) 'D' type and 13 numbers of (multi-storied) 'E' type flats, 3 numbers of 'E' type quarters, has also been taken up and it is expected that these quarters will be ready within the next financial year. Tenders have also been invited for 6 numbers of 'H' type quarters. Preparation of drawings, detailed estimates, etc., are also in progress for 105 numbers of 'H' and 'E' type multi-storied quarters at the Tuiladungri area.

APPENDIX I

PAPERS PUBLISHED

The full text of the Proceedings of the Symposium on "Pilot Plants in Metallurgical Research and Development" organized by the National Metallurgical Laboratory containing all the papers presented was published during this year, a monograph on Foundry Moulding Sands embodying the results of research done in the Laboratory was also brought out. Four issues of the *NML Technical Journal* were brought out during the year under review. Besides the above, the following papers and technical articles were published in leading Indian and foreign journals.

Research Papers

1. TREHAN, Y. N., BHADRA, S. P. & GUPTA, P. K., "Electrolytic Polishing of Stainless Steels", *NML Tech. J.*, 2(2) (1960), 8-17.
2. RAMAN, P. V. & NARAYANAN, P. I. A., "Fluorspar and Its Beneficiation", *NML Tech. J.*, 2(2) (1960), 18-22; *Blast Furnace and Steel Plant* 48(10) (1960), 1045-1050.
3. MUKHERJEE, K. P., LAHIRI, A. K. & BANERJEE, T., "Brown Spots on Aluminium Circles Developed During Storage", *NML Tech. J.*, 2(2) (1960), 23-26.
4. MRINAL ROY & BHATNAGAR, P. P., "Studies on the Extraction of Vanadium Pentoxide from Vanadium Bearing Titaniferous Magnetite Ore of Bihar and Orissa", *NML Tech. J.*, 2(2) (1960), 27-31.
5. MUKHERJEE, K. P., LAHIRI, A. K. & BANERJEE, T., "Erosion of Induced Draft Fan in Thermal Power Station", *NML Tech. J.*, 2(3) (1960), 7-11.
6. GHOSE, M. K., PAIT, S. & KAR, B. C., "Spectrographic Determination of Boron in Steel by Rotating Electrode Method", *NML Tech. J.*, 2(3) (1960), 12-14.
7. PRABHAKARAM, P. & MURTHY, H. P. S., "Thermogravimetric Studies on the Low Temperature Pyrolysis of Bituminous Coal", *NML Tech. J.*, 2(3) (1960), 15-19.
8. CHATTERJEE, A. B. & NIJHAWAN, B. R., "Stress-corrosion Failure on Austenitic Stainless Steel", *NML Tech. J.*, 2(3) (1960), 20-25.
9. ADDANKI, S. RAO, LAHIRI, A. K. & BANERJEE, T., "Corrosion of Cast Iron Pots Used for Fusing Caustic Soda", *NML Tech. J.*, 2(4) (1960), 6-9.
10. DAS, B. N., CHOUBEY, R. & GOSWAMI, K. C., "Wear and Friction under Rolling with Slip", *NML Tech. J.*, 2(4) (1960), 15-21.

11. MAJI, K. D. & RAMACHANDRAN, E. G., "The Direct Evaluation of the Effective Camera Radius in a Debye-Scherrer Photograph", *NML Tech. J.*, **2**(4) (1960), 22, 23.
12. RAMACHANDRAN, E. G. & DASARATHY, C., "Stabilization of Austenite by Hydrogen", *Nature*, **187** (4738) (1960), 684, 685.
13. PRASAD, T. V. & MURTHY, H. P. S., "Development of Graphite Crucibles from Indian Raw Materials", *Trans. Indian Ceramic Society*, **19**(2) (1960), 59-66.
14. SUBRAMANIAM, M. & BHATNAGAR, P. P., "Reducibility of Salem Magnetite Ore", *J. Sci. Ind. Res.*, **19A**(12) (1960), 620-624.
15. SHARMA, R. A. & BHATNAGAR, P. P., "Beneficiation of Ores by Chlorination", *J. Mines, Metals & Fuel*, **VIII**(7) (1960), 121-128.

Technical Papers

1. BANERJEE, N. G., "Fabrication and Working of the Vacuum Fusion Apparatus for the Determination of Gases in Metals and Alloys", *NML Tech. J.*, **2**(4) (1960), 10-14.
2. NIJHAWAN, B. R., "The Role of the National Metallurgical Laboratory in Research and Development of Light Metal Industry in India", *NML Tech. J.*, **3**(1) (1961), 56-72.
- *3. NIJHAWAN, B. R., "Scope of Pilot Plant Research and Development at the National Metallurgical Laboratory", *Proc. of Symp. on Pilot Plants in Metallurgical Research and Development*, 36-49; *NML Tech. J.*, **2**(1) (1960), 11-23.
4. SHAHANI, M. J. & BANERJEE, T., "Metallurgical Pilot Plants — Theory and Practice", *Proc. of Symp. on Pilot Plants in Metallurgical Research and Development*, 66-73.
- *5. CHATTERJEA, A. B., "Pyrometallurgical Pilot Plants" *Proc. of Symp. on Pilot Plants in Metallurgical Research and Development*, 97-105.
6. NIJHAWAN, B. R., "Some Aspects of Pilot Plant Research and Development in Iron & Steel Industry", *Proc. of Symp. on Pilot Plants in Metallurgical Research and Development*, 106-112.
7. AGARWAL, B. K., MENON, M. P. & BHATNAGAR, P. P., "Recovery of Vanadium Pentoxide from Vanadium Bearing Titaniferous Magnetites — Projected Pilot Plant Studies", *Proc. of Symp. on Pilot Plants in Metallurgical Research and Development*, 182-187.
- *8. KAPOOR, A. N., GUPTA, P. K. & NIJHAWAN, B. R., "Hot-dip Aluminizing of Steel Wire — Laboratory Scale Investigations and Pilot Plant Studies", *Proc. of Symp. on Pilot Plants in Metallurgical Research and Development*, 188-196.

*Published in the special issue of the *Statesman* Supplement dated 15th February 1960, entitled "Pilot Plants in Metallurgical Research & Development" issued on the occasion of the inauguration of the Symposium organized by the National Metallurgical Laboratory.

- *9. PRASAD, T. V. & BHASKAR RAO, H. V., "Development of Metallurgical Refractories with Indian Raw Materials — Pilot Plant Studies", *Proc. of Symp. on Pilot Plants in Metallurgical Research and Development*, 197-203.
- *10. DHANANJAYAN, N., CHAKRABARTI, H. K. & BANERJEE, T., "Production of Manganese Dioxide — Pilot Plant Investigations", *Proc. of Symp. on Pilot Plants in Metallurgical Research and Development*, 204-212.
11. KRISHNAN, R. M., "Specification for High Silica Sand for Use in Foundries", *I.S.I. Bulletin*, **12** (1960), 236, 237.
12. NIJHAWAN, B. R., "Service Behaviour of Metals in Transport", *Tisco Tech. J.*, **8**(1) (1961), 1-14.
13. CHATTERJEA, A. B., "Metals and Alloys in Automobiles", *Tisco Tech. J.*, **7**(4) (1960), 329-340.
14. VED PRAKASH, "Metals and Alloys in Air Transport", *Tisco Tech. J.*, **8**(1) (1961), 35-44.
15. RAMACHANDRAN, E. G., "The Non-destructive Testing of Metals Components used in Transport", *Tisco Tech. J.*, **8**(1) (1961), 57-65.
16. KRISHNAN, R. M., "Foundry Industry in Great Britain and Some General Trends of Development", *Iron, Steel & Hardware J.*, **4**(4) (1960), 250-253.
17. BHATNAGAR, P. P. & NIJHAWAN, B. R., "Metallurgy of Newer Metals", *The Eastern Metals Review: Independence Number*, 1960, 1-13.
18. BANERJEE, T., "Producing Electrolytic Manganese", *Bri. Chem. Engg. J.*, **5**(4) (1960), 278.
19. "Electrical Resistance Alloys", *Research & Industry*, **6** (1), (1961), 1-32.
20. "Recovery of Vanadium Pentoxide from Vanadium-bearing Titaniferous Magnetites", *Research & Industry*, **5** (9), (1960), 272-274.
21. "Production of Ferro-alloys", *Research & Industry*, **5** (7), (1960), 195-199.
22. "Research Institute — Our Pride", *J. of Indian Inst. of Chemical Engineer*, **2** (3), 1960.

The following articles were published in the special issue of the *Amrita Bazar Patrika* Supplement dated 14th February, 1961 entitled "Light Metal Industry in India" issued on the occasion of the inauguration of the Symposium organized by the National Metallurgical Laboratory:

1. NIJHAWAN, B. R., "The Role of National Metallurgical Laboratory in Light Metal Industry Research and Development"
2. SOM, P. K., CHAKRAVARTI, P. B. & BANERJEE, T., "Electrolytic Production of Magnesium and Magnesium Alloys"
3. MISRA, R. N., SAMPATH, V. S. & BHATNAGAR, P. P., "The Scope for Development of Magnesium Industry in India"

*Published in the special issue of the *Statesmen* Supplement dated 15th February 1960, entitled "Pilot Plants in Metallurgical Research & Development" issued on the occasion of the inauguration of the Symposium organized by the National Metallurgical Laboratory.

4. GUPTA, P. K. & SRIVASTAVA, K. N., "Progress of Light Metal Industry in India"

Letters to the Editor

1. RAMACHANDRAN, E. G. & DASARATHY, C., "Stability of Retained Austenite on Ageing", *Acta Metallurgica*, 8(4) (1960), 274, 275.
2. RAMACHANDRAN, E. G. & DASARATHY, C., "Athermal Stabilization of Austenite", *Acta Metallurgica*, 8(9) (1960), 666, 667.
3. RAMACHANDRAN, E. G. & DASARATHY, C., "Hydrogen and Austenite Stabilization", *Acta Metallurgica*, 8(10) (1960), 729, 730.

Papers Presented at Symposia and Technical Sessions

The following paper was presented at the International Mineral Processing Congress held at London in April 1960:

Beneficiation of Low-grade Manganese Ores with Particular Reference to Semi-pilot Plant Studies on a Low-temperature Magnetizing Reduction Process for Ferruginous Manganese Ores — G. V. Subramanya & P. I. A. Narayanan

The following papers were presented at the Symposium "Light Metal Industry in India", held under the auspices of this Laboratory from 14th to 17th February 1961:

1. The Role of the National Metallurgical Laboratory in Research and Development of Light Metal Industry in India — B. R. Nijhawan
2. A Study of the Production of Soderberg Paste from Indian Raw Materials — P. Prabhakaram & H. P. S. Murthy
3. Development of Light Metal Industry in India — P. K. Gupta & K. N. Srivastava
4. The Scope for Development of Magnesium Industry in India — R. N. Misra, V. S. Sampath & P. P. Bhatnagar
5. Electrolytic Production of Magnesium and Magnesium Alloys — P. K. Som, P. B. Chakravarti & T. Banerjee
6. The Effect of Misch-metal Additions on the Structure and Workability of Al-Mg (7-10 per cent) Alloys — Y. N. Trehan, P. K. Gupte & B. R. Nijhawan
7. Recent Advances in the Technology of Al-Mg Alloys — Y. N. Trehan & S. P. Bhadra

The following paper was presented at the Annual Meeting of the Indian Institute of Metals held at Bhilai in December 1960:

Investigation into Oxygen Steel-making Process Employing Indian Raw Materials and Refractories — B. L. Sen, B. R. Nijhawan & A. B. Chatterjea

The following papers were presented at the Annual Meeting of the Indian Ceramic Society held at Kumardhubi in March 1961:

1. Indian Dolomites — A Raw Material Study — P. C. Sen, M. R. K. Rao & H. V. Bhaskar Rao
2. Indian Dolomites — Some Aspects of Complete Stabilization — M. R. K. Rao, P. C. Sen & H. V. Bhaskar Rao

The following papers were presented at the Symposium on "Iron and Manganese Ores" held at Calcutta in March 1960 under the auspices of Mining, Geological and Metallurgical Society of India:

1. Beneficiation of Low-grade Manganese Ores of India — G. V. Subramanya & P. I. A. Narayanan
2. Beneficiation and Agglomeration of Iron Ores — S. K. Banerjee, P. V. Raman & P. I. A. Narayanan
3. Flotation of Manganese Ores — B. L. Sengupta, G. V. Subramanya & P. I. A. Narayanan

The following paper was presented at the Metallurgical Conference held at Paris, France, in October 1960:

Phase-transformation in Electrolytic Alloys with Special Reference to Copper-zinc and Silver-copper System — T. Banerjee & P. L. Ahuja

APPENDIX II

INVESTIGATION AND RESEARCH REPORTS

1. Studies on the Beneficiation of Iron Ores from Tisco — G. V. Subramanya, G. P. Mathur, S. K. Banerjee, P. K. Sinha & B. L. Sen Gupta (IR180/60)
2. Beneficiation of Fluorspar from Thurwali, Rajasthan — P. V. Raman & P. I. A. Narayanan (IR181/60)
3. Moulding and Core Characteristics of Batala Sands received from Messrs Batala Engineering Co. Ltd., Punjab — K. K. Balasankar, P. C. Das, G. L. Saha, Jatinder Mohan & P. K. Gupte (IR182/60)
4. Investigation on the Moulding Characteristics of River Sand sent by Messrs Southern Railway, Golden Rock, Madras — T. A. Beck, Jatinder Mohan & P. K. Gupte (IR183/60)
5. Screen Analysis and Chemical Analysis of Iron Ore Samples from Kiriburu — S. K. Banerjee, B. L. Sen Gupta & P. I. A. Narayanan (IR184/60)
6. Studies on the Reducibility of Beneficiated Tisco Iron Ores — M. Subramanian & P. P. Bhatnagar (IR185/60)
7. Reduction of Iron Content in a Manganese Ore Sample from Messrs National Carbon Co., Calcutta — R. Ganesh, G. V. Subramanya & P. I. A. Narayanan (IR186/60)
8. Beneficiation of Low-grade Manganese Ore from Lohandabud group of Mines, Jharsuguda, Orissa — R. Ganesh, G. V. Subramanya & P. I. A. Narayanan (IR187/60)
9. Further Studies on the Beneficiation of Iron Ore Samples from Tisco — S. K. Banerjee, P. K. Sinha, B. L. Sen Gupta & P. I. A. Narayanan (IR188/60)
10. Beneficiation of Low-grade Graphite from Doomara & Lotiyana Villages, Ajmer, Rajasthan — Y. A. Joglekar, G. P. Mathur & P. I. A. Narayanan (IR189/60)
11. Beneficiation of an Amorphous Gypsum Sample from Messrs Bikaner Gypsums Ltd., Rajasthan — Y. A. Joglekar, G. V. Subramanya & P. I. A. Narayanan (IR190/60)
12. Moulding Characteristics of Hyderabad White Sand from Messrs Kirloskar Bros. Ltd., Kirloskarvadi — Jatinder Mohan, B. V. Somayajulu & S. S. Bhatnagar (IR191/60)
13. Moulding Characteristics of Facing and Backing Sands received from Southern Railway, Golden Rock, Madras — K. K. Balasankar, T. A. Beck, B. V. Somayajulu & S. S. Bhatnagar (IR192/60)

14. Moulding and Core Characteristics of Red Core Sand sent by Messrs Kirloskar Bros. Ltd., Kirloskarwadi, Bombay — Jatinder Mohan, B. V. Somayajulu & S. S. Bhatnagar (IR193/60)
15. Sintering of Dolomite in Shaft Kiln of Hindustan Steel Plant, Rourkela — M. R. K. Rao, P. C. Sen & H. V. Bhaskar Rao (IR194/60)
16. Report on Beneficiation Studies on a Complex Low-grade Copper-Lead-Zinc Ore from Bhotang Mines, Rangpo, Sikkim — P. V. Raman, G. P. Mathur, M. V. Ranganathan & P. I. A. Narayanan (IR195/60)
17. Investigation on the Suitability of Asbestos from Singhbhum District for Industrial Purposes — N. V. Shah & H. V. Bhaskar Rao (IR196/60)
18. Reduction of Iron Oxide Content in a Bauxite Sample from Road Side Mine, Jabalpur District, Madhya Pradesh — S. K. Banerjee, G. V. Subramanya & P. I. A. Narayanan (IR197/60)
19. Beneficiation of Low-grade Ferruginous and Siliceous Manganese Ores from Koira, Orissa — S. B. Das Gupta, G. V. Subramanya, P. K. Sinha & P. I. A. Narayanan (IR198/60)
20. Beneficiation of Low-grade Chromite from Kankauli Deposits, Ratnagiri District, Maharashtra — A. Peravadhanulu, G. P. Mathur & P. I. A. Narayanan (IR199/61)
21. Studies on the Development of Zircon Refractories from Travancore Beach Sand — T. V. Prasad, M. C. Kundra & H. V. Bhaskar Rao (RR109/60)
22. Studies on the Extraction of Vanadium Pentoxide from Vanadium Bearing Titaniferous Magnetite Ore of Bihar and Orissa — Mrinal Roy & P. P. Bhatnagar (RR110/60)
23. Spectrographic Determination of Boron in Steel by Rotating Electrode Method — M. K. Ghose, S. Pait & B. C. Kar (RR111/60)
24. Indian Dolomites — A Raw Material Study — P. C. Sen, M. R. K. Rao & H. V. Bhaskar Rao (RR112/60)
25. Indian Dolomites — Some Aspects of Complete Stabilization — M. R. K. Rao, P. C. Sen & H. V. Bhaskar Rao (RR113/60)
26. Constitution and Properties of Stabilized Dolomites — M. R. K. Rao, P. C. Sen & H. V. Bhaskar Rao (RR114/60)
27. Production of Low-carbon Ferro-chrome from High-carbon Ferro-chrome — V. S. Sampath & P. P. Bhatnagar (RR115/61)
28. The Effect of Misch-metal Additions on the Structure and Workability of Al-Mg (7-10 per cent) Alloys — Y. N. Trehan, P. K. Gupte & B. R. Nijhawan (RR116/61)

SURVEY AND SPECIAL REPORTS

1. The Role of the National Metallurgical Laboratory in Research & Development of Light Metal Industry in India — B. R. Nijhawan

2. A Study of the Production of Soderberg Paste from Indian Raw Materials—
P. Prabhakaram & H. P. S. Murthy
3. Development of Light Metal Industry in India — P. K. Gupta & K. N. Srivastava
4. The Scope for the Development of Magnesium Industry in India — R. N. Misra,
V. S. Sampath & P. P. Bhatnagar
5. Electrolytic Production of Magnesium and Magnesium Alloys — P. K. Som, P. B.
Chakravarti & T. Banerjee
6. Recent Advances in the Technology of Aluminium-magnesium Alloys — Y. N. Trehan
& S. P. Bhadra
7. Metals and Alloys in Automobiles — A. B. Chatterjea
8. Recent Trends in Steel Technology — A. B. Chatterjea
9. The Non-destructive Testing of Metal Components Used in Transport — E. G. Rama-
chandran
10. Metals and Alloys in Air Transport — Ved Prakash
11. Service Behaviour of Metals in Transport — B. R. Nijhawan