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JAMSHEDPUR, INDIA

CONTENTS

RESEARCH PROJECTS

PILOT PLANTS

ENGINEERING SECTIONS

OPERATIONAL RESEARCH

APPENDIX I

APPENDIX II

FOREWORD

I HAVE great pleasure in presenting the Report of the Director, National Metallurgical Laboratory, for the year 1961-62.

The projected expansion of the National Metallurgical Laboratory during the Third Five Year Plan, is now taking concrete shape, which includes the establishment of a Pilot Plant for beneficiation of indigenous low-grade ores, studies on sintering, pelletizing etc. at a cost of Rs. 3·5 million. The entire equipment for this Pilot Plant had been procured from the U.S.A., transported to the site and the work of erection has made rapid progress. It is hoped that the plant, one of the biggest of its kind in India, will be in full operation next year. Similarly, the expansion of the main building by the addition of the side and central wings has made considerable headway. It is gratifying to note that these additions with all services costing over Rs. 1·5 million will be fully completed during the coming year. The new wings will house laboratories for radio-active isotope tracer research studies, expansion of corrosion laboratories, X-ray flourescence spectroscopy, vibrophore pulsator, vacuum melting units and equipment for pressure leaching and hydro-metallurgical research activities, etc.

The submerged electric arc ferro-alloy pilot plant, built at a cost of Rs. 0·6 million, went into operation on the 12th February 1962 during the Symposium on " Ferro-alloy Industry in India ", organized by the Laboratory.

The expansion envisaged during the Third Five Year Plan also covers the establishment of a full-fledged Iron & Steel Division and an Alloy Steel Division as also the establishment of a Marine Corrosion Research Station at Digha. The plans and designs for the Marine corrosion Research Station at Digha have already been finalized and contract orders have been placed. It is hoped to man and operate this station fully during 1962-63 which will provide the much needed corrosion data for the industry. The Laboratory is also actively collaborating with the Metals Research Committee in organizing research into diverse phases of corrosion of metals and alloys on an all India basis. Incidentally, it has been said that the National Metallurgical Laboratory is one of the leading research laboratories in the country today where considerable work on different aspects of corrosion of metals and alloys has already been undertaken.

Efforts are being made to obtain through the channel of United Nations Special Fund Assistance Programme pilot plant equipment for iron and steel research investigations including Krupp-Renn kilns, Sturzelberg rotary furnaces, equipment for direct reduction of iron ore, L-D oxygen fully instrumented 3-tons' converters, pilot continuous casting units and a pilot plant for the study of carbon electrodes and Soderberg pastes for electro-metallurgical applications.

The Laboratory has undertaken the design and fabrication of a pilot basic-lined side-blown converter employing non-oxygenated air blast to refine phosphoric pig iron into acceptable grades of steel. The design and fabrication of a vertical smelting furnace with two sets of tuyeres, to undertake investigations on oil injections, low shaft-furnace gas injection, besides injection of powdered fuels and fluxes through the second set of tuyeres, has been completed for the production of foundry grades of pig iron.

The Laboratory has made a beginning in imparting specialized training in metallurgical subjects. Graduate apprentices have been selected and are being given a course of training to meet the requirements of technical posts in metallurgical industries. Likewise at the artisan level, trade apprentices for mechanical, electrical and electronic instrumentation, mechanical workshop and pilot plant installations are being trained. There has also been a substantial increase in the number of junior and senior research fellowships for those working for higher post-graduate degrees. Such measures are steps in the right direction which will pay rich dividends in due course, not only to the Laboratory but also to the metallurgical industries.

During the year under review the following important Project Investigation Reports have been issued:

(1) Pilot plant studies on beneficiation and sintering of Bolani iron ore for Durgapur Steel Plant of Hindustan Steel Ltd.

(2) Iron production with non-coking coals in low-shaft furnace pilot plant at the National Metallurgical Laboratory.

(3) Report on the operation of low-shaft furnace pilot plant at the National Metallurgical Laboratory for the production of foundry grades of pig iron with raw materials from Andhra Pradesh, and

(4) Pilot plant studies on beneficiation and sintering of Barsua iron ores for Rourkela steel plant of Hindustan Steel Ltd. Also during the year, the Laboratory brought out a publication containing proceedings, technical papers, etc. of the Symposium on "Light Metal Industry in India" arranged in 1961. The publication has been well received and reviewed both at home and overseas.

The *NML Technical Journal* has stepped into its fourth year of publication and the technical papers published are abstracted in leading overseas abstracting services. The number of exchange technical publications has steadily multiplied.

A Symposium on "Ferro-Alloys Industry in India" was arranged in February 1962, which was attended by delegates from practically all over the world, besides those from India. In having attracted valuable technical contributions from specialists in their respective fields, the Laboratory could feel justifiably happy on their success in organizing such international Symposia, which offer stimulation of technical and research ideas and practical initiation to important technical themes. During the Symposium, considerable emphasis was laid on research and development work on the family of ferro-alloys, production techniques of different ferro-alloys from indigenous raw materials, utilization of waste products, etc. These aspects certainly represent challenging fields in which the Laboratory is today actively engaged in its broad-based research and development programme to attain self-sufficiency in minerals and metals.

The ferro-alloy pilot plant is of such a size that valuable information can be obtained on important factors such as electric power, electrode consumption, recovery and yield figures, and economics of production which can be scaled up to enable a fairly accurate appraisal on a larger scale plant being made. Customers' raw materials will be accepted on a contract basis and I am sure this unit will serve a useful purpose not only for the Indian ferro-alloy industry but also for the whole of South-East Asia.

Work on the beneficiation and sintering of Indian iron ores, such as from Kiriburu for the National Mineral Development Corporation, from Rajahara for the Bhilai steel plant of the Hindustan Steel Limited, etc., is being continued.

The steel plants in India are having a serious problem in the utilization of the flux grade limestone in steel-making, which due to its high silica content presents considerable operational difficulties. On an assignment given by the Tata Iron and Steel Co. Ltd., the Laboratory has successfully beneficiated in their pilot plant high silica steel-making limestone and reduced its silica content to below 2 per cent. The limestone briquettes made after beneficiation have undergone successful trial tests in a 235 tons open-hearth steel smelting furnace. Based on the work done, a comprehensive report was submitted suggesting a proposal for setting up a beneficiation plant to treat low-grade limestone at the rate of 250 tons/day detailing the equipment, cost of beneficiation and briquetting of the limestone. Similar work has also been carried out on behalf of Hindustan Steel Limited on four different consignments of limestone from Purnapani quarries, which have also been successfully beneficiated for steel-making use.

The low-shaft furnace pilot plant of the Laboratory has been in operation during the year. Extensive smelting trials have been conducted on raw materials from Andhra Pradesh, East Punjab and Maharashtra State to determine the feasibility of the production of foundry grades of pig iron. On the basis of the work done at the Laboratory, it has been recommended that the setting up of iron production units based on low-shaft blast furnace smelting would be both metallurgically and economically feasible in Andhra Pradesh. The Laboratory has been in continuous touch with the Nepal Government for the establishment of a small scale iron production plant in Nepal, and smelting operations with their raw materials are being initiated at the low-shaft furnace pilot plant.

The pilot plant for the aluminizing of steel has been operated for a considerable period to aluminize the hardware supplied by the Post and Telegraph Department and auto-muffler parts and silencer pipes furnished by the army base camps and automobile industry. The aluminized telegraph wire which was under service trial under marine atmospheric conditions in the Visakhapatnam area has given most satisfactory results and the Post and Telegraph Department now wish to introduce the aluminizing of their hardware products instead of galvanizing them.

The beneficiation of fluorspar for the Rajasthan Government was undertaken and a project report is being worked out for the Government of Rajasthan. The fluorspar has been up-graded to the metallurgical grade standard as also acid grade standard; the latter has been used for the production of cryolite for trials in aluminium reduction cells in the aluminium industry in India.

In the context of our successive Five Year Plans, priority is given to the operational and applied research projects, but sufficient emphasis has also been given to fundamental research schemes which have made steady progress.

Continued collaboration has been maintained with the different Ministries of the Government of India for research and development programmes, and I am very happy to say that the National Metallurgical Laboratory is acting as a nucleus for many of the long-range and short-term projects connected with the development of metal and mineral industries such as for the National Mineral Development Corporation for laboratory and pilot plant scale investigations into Khetri copper ores.

Significant progress has been maintained on fundamental research studies such as isothermal transformation studies (T.T.T curves) of Indian plain carbon and alloy steels for which an atlas is now being compiled for use of research and industry, preferred orientation in rolled sheets, solubility of nitrogen in plain carbon and alloy steels, carbon activity in iron and its alloys, study on age-hardening of light metal alloys, base exchange studies on clays, reducibility studies of iron ores and sinter. The work carried out attempts an understanding and formulation of certain basic concepts of mechanism of phase transformations, crystal structure and its orientation etc. In the field of refractories, basic studies on the development of basic refractories for L-D oxygen converter, on welding flux, on base-exchange properties of clays etc. have yielded most interesting results. Several problems relating to operational processes, service failures etc. referred to by the industries were successfully investigated and concrete suggestions were offered for the solution of their problems. The Liaison, Information and Operational Research Division undertook preparation of several project reports specially for the foundry industries for the production of different grades of malleable iron products.

Prof. M. S. Thacker, Director-General, Scientific and Industrial Research, during his visit to the Laboratory early this year, spoke of the high standard of research work in progress and the excellent work done by the Director and his colleagues. The Laboratory has always enjoyed the full support and active goodwill of Prof. M. S. Thacker, who has taken a keen interest in the Laboratory's activities.

I should like to express on my behalf and that of other Members of the Executive Council our sincere appreciation of the sustained and devoted work that is being done by Dr. B. R. Nijhawan and his able colleagues. I should like them to know that the Members of the Executive Council of the National Metallurgical Laboratory appreciate their highly creditable and praiseworthy efforts. Much of the success of the laboratory's ever-widening scope of research and pilot plant activities is undoubtedly due to the hard work, research and organizational ability of its Director, Dr. B. R. Nijhawan. The National Metallurgical Laboratory is undergoing rapid expansion in many fields during the Third Five Year Plan, and I have great pleasure in recording our appreciation of the commendable success achieved which augurs well for the future.

Sd/- J. J. GHANDY

Chairman
Executive Council of the
National Metallurgical Laboratory

August 1, 1962,
Jamshedpur

INTRODUCTION

The National Metallurgical Laboratory has now entered the Third Five Year Plan in the midst of sustained growth of research and pilot plant activities geared to the needs of mineral and metal industries. The expansion projected during the Third Five Year Plan is now taking concrete shape. During the very first year of the Third Five Year Plan, it is to the credit of the National Metallurgical Laboratory that practically the entire equipment for the integrated pilot plant on Beneficiation of Low-Grade Indian Ores, involving an outlay of Rs 50 lakhs (Rs 5 million) has been transported to the site and is now in the process of speedy installation. Similarly, expansion to the National Metallurgical Laboratory and main building's side and central wings projected during the Third Five Year Plan has made considerable headway during the first year of the Plan itself.

The Pilot Submerged Electric Arc Ferro-Alloy Pilot Plant, which was to go into production during the Third Five Year Plan, went into operation on 12th February, 1962, during the National Metallurgical Laboratory's Symposium on ' Ferro-Alloy Industry in India '. These developments during the early part of the Third Five Year Plan speak well of the well organized planning undertaken by the National Metallurgical Laboratory whose systematic expansion and growth are keeping fairly ahead of schedule. The establishment of regional foundry units has also made good progress. Likewise, the operations of the Low-shaft Furnace Pilot Plant have maintained active progress during the year under review.

Due steps are also being taken to implement the decisions of the Executive Council of the National Metallurgical Laboratory and the Governing Body of the Council of Scientific & Industrial Research to establish full-fledged Divisions on " Iron & Steel " and on " Alloy Steel " at the National Metallurgical Laboratory. Amongst the various items of equipment being installed, mention may be made of the X-ray fluorescence spectroscope which is the first of its type in the country.

Equally gratifying is the position that the Council of Scientific & Industrial Research and the National Metallurgical Laboratory have undertaken the construction of housing for their staff on a vast scale including six-storeyed multiple residential flats and bungalows.

During the period under review, the National Metallurgical Laboratory brought out an excellent publication containing proceedings, technical papers, etc. of the Symposium on " Light Metal Industry in India " arranged by the National Metallurgical Laboratory early in 1961 which has been exceedingly well received and reviewed both at home and overseas. In this connection a reference is made to the Monograph on " Foundry Moulding Sands of India " recently published by the National Metallurgical Laboratory, which has received commendable appreciation from eminent foundry technologists both at home and overseas countries. About this monograph Mr. O. P. Einerl, one of the foremost foundry scientists, has stated in his review, published by the Institute of Metals, London, that " The book presents in a most lucid manner the results of what must have been a monumental research programme. If a similar modern work for the sands of the United Kingdom were to be undertaken, it would, with profit, be modelled on the same line as this monograph." The Laboratory has before published research monographs on other subjects based on research and pilot plant trial results obtained during the last few years. The cataloguing of technical literature, documentation and patent service have been established on a scientific basis in the National Metallurgical Laboratory.

Pilot Plant scheme on the production of soft and permanent magnet alloys and sintered magnetic alloys will also be implemented during the Third Five Year Plan and steps are being taken to establish this pilot plant at the very earliest.

Based on the Report issued by the National Metallurgical Laboratory on " Pilot Plant Studies on Beneficiation and Sintering of Bolani Iron Ore for Durgapur Steel Plant ", Messrs. Head Wrightson & Co. Ltd, a leading U.K. firm, who collaborated with the National Metallurgical Laboratory on this investigation, have commented on the results obtained and have stated that: " The results given provide an excellent basis for the process evaluation of a plant to handle the Bolani ore from mines to blast furnaces ". Further work on the beneficiation and sintering of Indian iron ores, such as from Kiriburu for the National Mineral Development Corporation, from Rajahara for the Bhilai Steel Plant of the Hindustan Steel Limited, etc., is now underway at the National Metallurgical Laboratory. Shri J. M. Shrinagesh, Chairman, Hindustan Steel Limited, in this connection acknowledged the appreciation of the technical assistance received from the National Metallurgical Laboratory by the Hindustan Steel Limited in many spheres.

Successful work has also been undertaken on beneficiation on pilot plant scale of Salem magnetite iron ore for iron smelting with lignite coke in a small smelting furnace. Similar trials will be made in the submerged arc electric furnace for electric smelting of Salem magnetite. At the instance of the Government of Madras, the National Metallurgical Laboratory prepared 60 tons of the Salem magnetite concentrate for shipment to Norway for further smelting tests.

In the present stage of the laboratory's development work, considered in the context of our successive Five Year Plans, while priority is given to the operational and applied research projects, equal emphasis has also been given to fundamental metallurgical projects which have made steady progress. The progress on the various research projects in different fields has been reported in the appropriate sections and it can be seen that the projects on nickel and cobalt-free electrical resistance alloys, development of techniques of bimetals, production of ceramic magnets, production of ferro-alloys by aluminothermic reaction, preparation of iron powder from ' blue dust ', production of flux for submerged arc welding etc., are now ready for industrial scale implementation. Preliminary Project Reports on the above technical processes have been issued to the industry. The Laboratory has maintained active liaison with the National Research Development Corporation to whom the following preliminary Project Reports have been issued for industrial scale implementation:

1. Production of Iron Powder for Autogenous cutting.
2. Manufacture of Alnico Permanent Magnet alloys.
3. Manufacture of Ceramic Magnets.
4. Flux for Submerged Arc Welding.
5. Electrical Resistance Alloys for Heating Elements.
6. Production of Thermostatic Bimetals.
7. Production of Carbon Free Ferro-alloys by Alumino-thermic Reactions.

It is hoped that these processes will find extensive applications in the industry like the process on aluminizing of steel wire and hardware items, which has already been leased out to over a dozen industrial units in India. Similarly, the release of the process on the production of electrolytic manganese metal and manganese dioxide is now taking concrete shape with the collaboration of Messrs. Devidayal (Sales) Pvt. Ltd, Bombay, who are projecting a plant for the production of 3 tons of electrolytic manganese per day. The National Metallurgical Laboratory undertook different investigations for private and public sector plants including the Defence Organization. The Laboratory has continued to act as a nucleus for many of the long range and short-term projects connected with the development of metal and mineral industries, such as for the National Mineral Development Corporation for laboratory and pilot plant scale investigations into Khetri copper ore.

During the year, the Laboratory has taken six new patents.

1. Indian Patent No. 76415 — Improvement in or relating to the modification of aluminium base alloys containing silicon.
2. Indian Patent No. 79598 — An improved device for the continuous vapour phase degreasing of metallic wire and strip.
3. Indian Patent No. 79597 — Improvements in a continuous vertical countercurrent solid — gas reactor.
4. Indian Patent No. 81402 — Improvements in or relating to electrodeposition of metals by direct current electrolysis of aqueous solutions containing metal ions.
5. Indian Patent No. 81403 — Improvements in a pneumatic basic-lined side blown vessel for the conversion of phosphorus containing pig irons into high grade steels.
6. Indian Patent No. 76997 — Improvements in or relating to the production of copper powder by electrolytic process.

Practical demonstrations were arranged to acquaint the interested manufacturers with the technical know-how of NML patented processes on electroplating on aluminium, chemical polishing of aluminium and electroplating of non-metallics, etc.

Significant progress has been maintained on fundamental research studies such as isothermal transformation studies (T.T.T. curves) of Indian plain carbon and alloy steels for which an Atlas is now being compiled for use of research and industry, preferred orientation in rolled sheets, solubility of nitrogen in plain carbon and alloy steels, carbon activity in iron and its alloys, study on age-hardening of light metal alloys, base exchange studies on clays, reducibility studies of iron ores and sinter. The work carried out attempts an understanding and formulation of certain basic concept of mechanism of phase transformations, crystal structure and its orientation etc. In the field of refractories, basic studies on the development of basic refractories for L-D oxygen converter, on welding flux, on base-exchange properties of clays, etc. have yielded most interesting results. It is also of interest to state that the National Metallurgical Laboratory has undertaken a comprehensive study on bentonite samples from different parts of the world to formulate their basic characteristics under an international code of procedure.

In the field of operational research, active progress has been made on the utilization of low-shaft furnace slag for the preparation of slag cement as well as light weight aggregates for insulating and structural concrete. The aggregate produced from the low-shaft furnace slag has been found to possess the requisite low unit weight and may be useful as a light weight aggregate for insulation concrete. Several problems relating to operational processes, service failures, etc., referred to by the industries were successfully investigated. The Liaison, Information and Operational Research Division undertook preparation of several Project Reports specially for the foundry industries. A summary of the highlights of important problems under progress is given in the following pages.

RESEARCH PROJECTS

1.0 Pilot Plant Studies on Beneficiation and Sintering of Barsua Iron Ores for Rourkela Steel Plant of Hindustan Steel Ltd

Systematic pilot plant investigations were taken up at the National Metallurgical Laboratory on beneficiation and sintering of iron ore for Rourkela Steel Plant of Hindustan Steel Ltd including the optimum degree of beneficiation needed and economics of iron production therefrom resulting from the use of self-fluxing sinter. A 100 tons representative sample of run-of-mine ore from Barsua mine of Hindustan Steel Ltd, assaying Fe, 52·18; SiO_2, 4·80; Al_2O_3, 11·34; MgO, 0·84; CaO, 0·42; P, 0·07; S, 0·40, and L.O.I., 9·08 per cent, was selected for pilot plant scale beneficiation and sinter investigations at the National Metallurgical Laboratory. Petrological studies of the iron ore were followed by screenability tests at different iron ore's moisture contents carried out to determine the ore's screenability characteristics during rainy monsoon weather. Ore washing tests performed after crushing the sample to -3 in., -2 in. and -1 in. sizes separately indicated that a clean, sized, washed product free from adhered ore fines and $-\frac{3}{8}$ in. classifier sand product free from high-gangue slime could be obtained.

Washing of the ore after crushing it separately to -3 in., -2 in. and -1 in. sizes resulted in (1) improvement in the Fe grade of washed product by 2·54 to 3·36 per cent, (2) a decrease in total insoluble contents by 2·49 to 3·24 per cent. The rejections through the slime of silica and alumina varied from 39·6 to 44·1 per cent and 18·4 to 26·1 per cent of the total respectively, for a corresponding iron loss in the deslimed washed ore product of 6·8 to 9·3 per cent. When washing was followed by heavy media separation, there was considerable improvement in the grade of the sink product and an appreciable decrease in its total alumina and silica content. The results of washing and heavy media separation after crushing the ore to -3 in., -2 in. and -1 in. sizes are given in Figs. 1, 2 and 3 respectively. It would be seen that heavy media separation of $+\frac{3}{8}$ in. washed products at sp. gr. 3·0 could produce sink products assaying 63·06 to 62·52 per cent Fe, 1·17 to 1·6 per cent SiO_2 and 4·06 to 4·90 per cent Al_2O_3 with recovery figures of 45·5 to 47·4 per cent Fe in the upgraded ore apart from the iron content of the $-\frac{3}{8}$ in. fraction. The results obtained also showed that crushing to about 1 in. followed by washing/heavy media separation would reject a float higher in alumina and silica contents in relation to the results obtained by washing/heavy media separation at 3 in. and 2 in. crushed ore size.

The $-\frac{3}{8}$ in. classifier sand obtained by washing at 1 in. ore size constituted 34·8 per cent by weight of the total and assayed Fe, 52·81; SiO_2, 3·73 and Al_2O_3, 11·54 per cent whilst containing 38·1 per cent of the total iron metallic content of the ore; this could be used in the self-fluxing sinter for iron smelting. Alternatively, this product could be further beneficiated, if required, employing heavy media separation and jigging. Heavy media separation of $-\frac{3}{8}$ in. $+$ 6 mesh classifier sand at 3·0 sp. gr. yielded a sink product assaying Fe, 63·1; SiO_2, 1·1 and Al_2O_3, 4·3 per cent yielding thereby additional recovery of 13·7 per cent Fe by weight. Jigging of

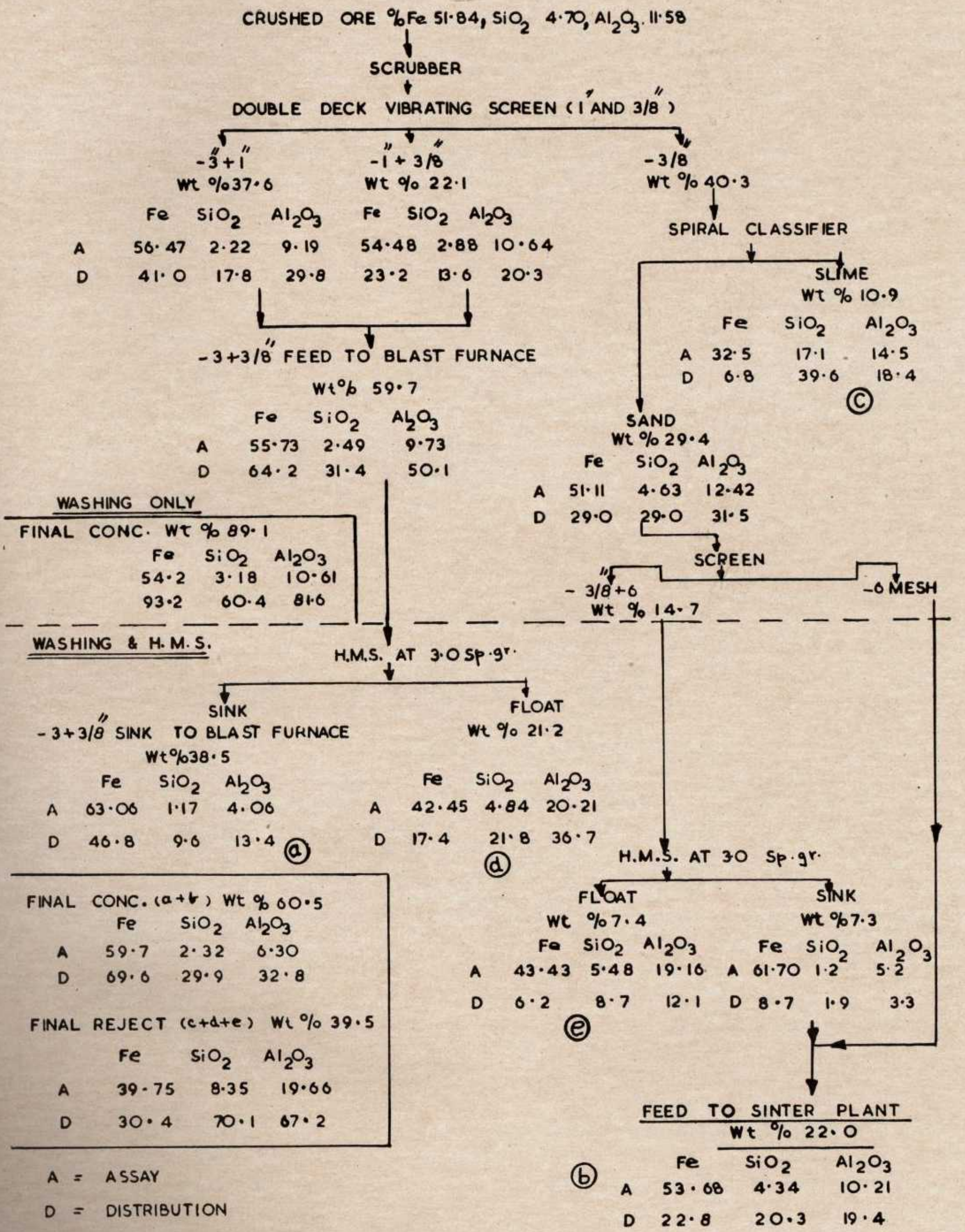


Fig. 1 — Summary of results of beneficiation at 3 in. size of Barsua Iron Ore

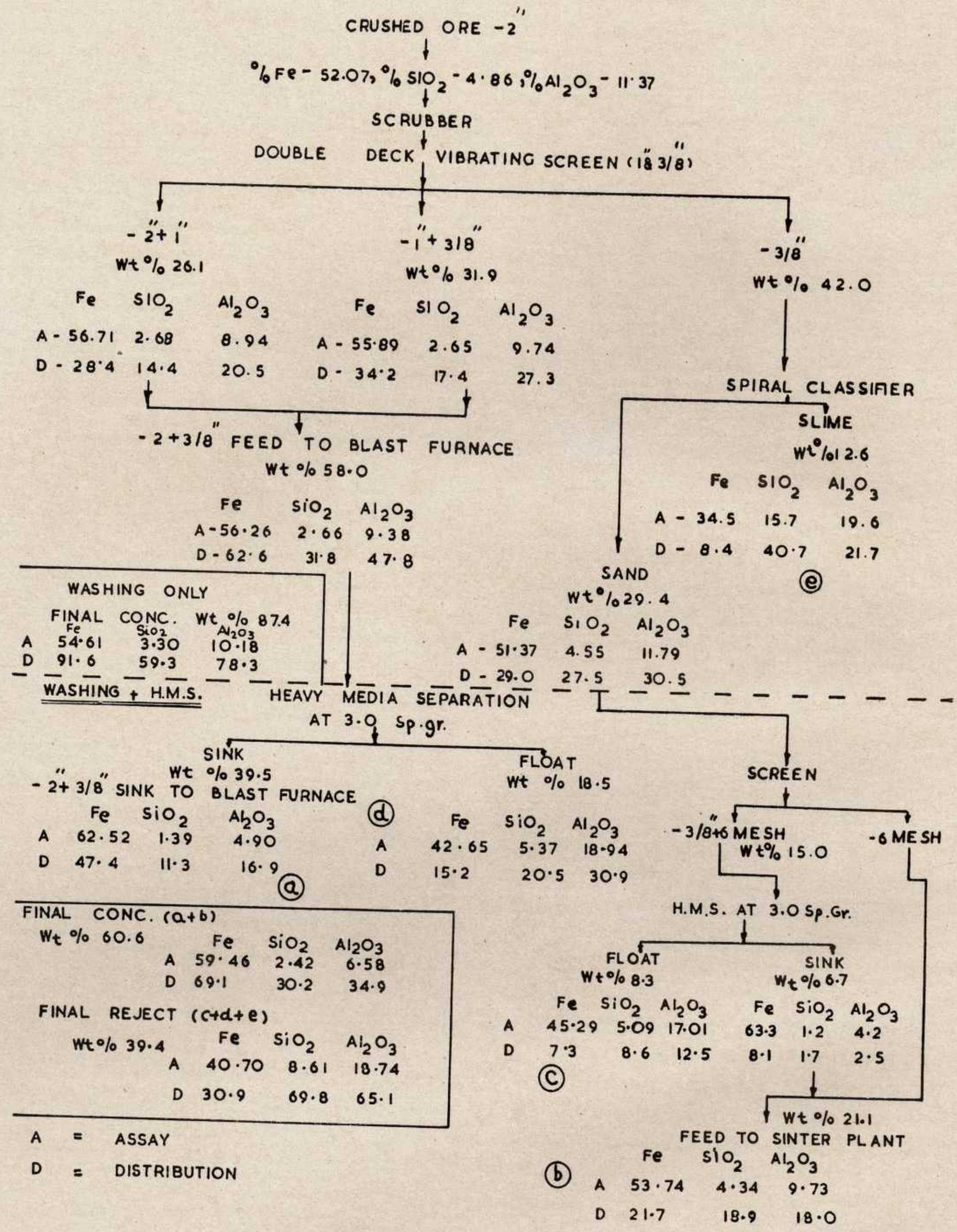


FIG. 2 — SUMMARY OF RESULTS OF BENEFICIATION AT 2 IN. SIZE OF BARSUA IRON ORE

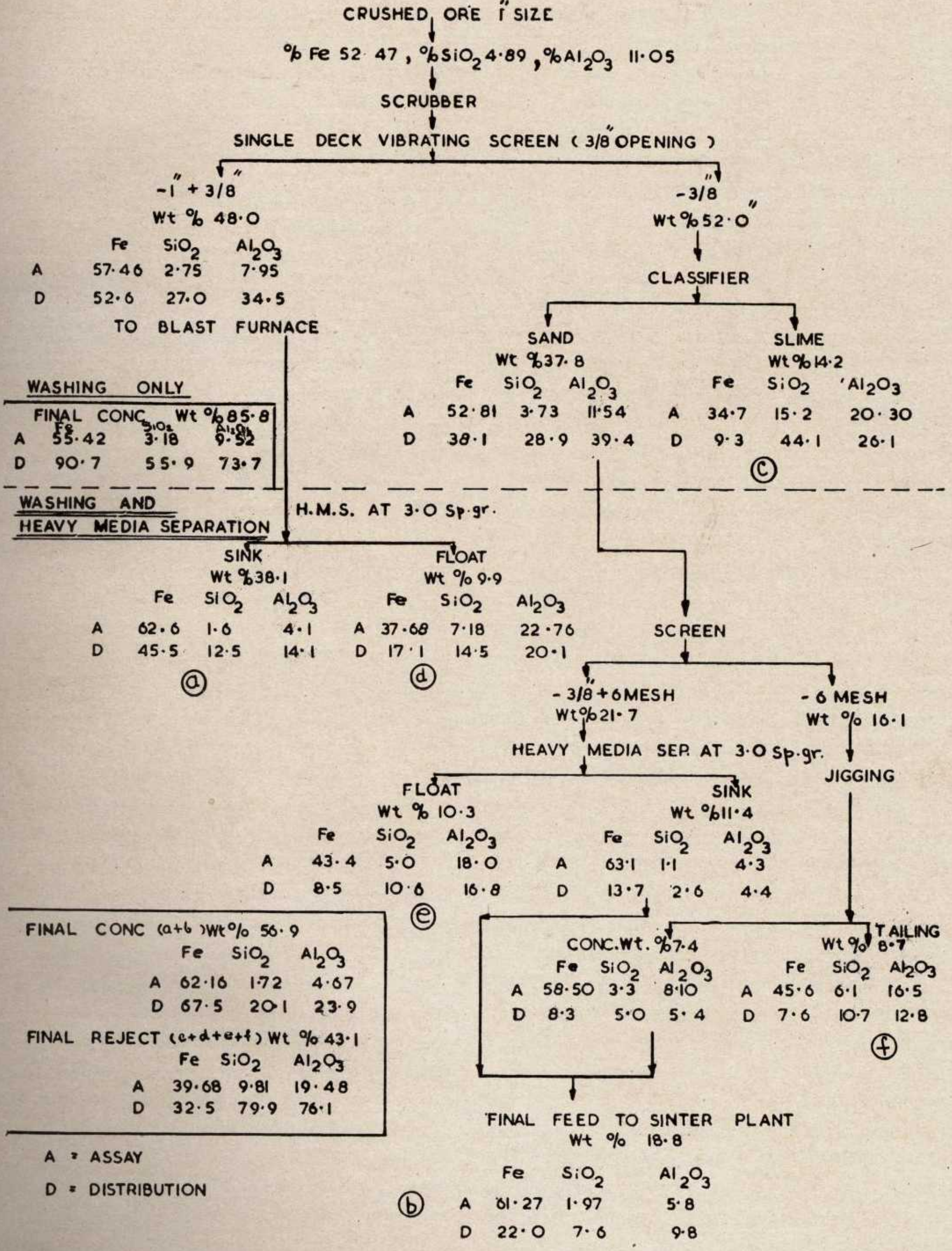


FIG. 3 — SUMMARY OF RESULTS OF BENEFICIATION AT 1 IN. SIZE OF BARSUA IRON ORE

—6 mesh fraction of the classifier sand yielded a concentrate assaying Fe, 58·5; SiO_2, 3·3 and Al_2O_3, 8·17 per cent, with a further recovery of 8·3 per cent Fe therein. The —⅜ in. +6 mesh heavy media concentrate product combined with the —6 mesh jig concentrate, constituting the basic feed for sinter-making would assay Fe, 61·27; SiO_2, 1·87 and Al_2O_3, 5·8 per cent. The entire operations, i.e. washing at 1 in. followed by heavy media separation, would produce reject products constituting 43·1 per cent by weight of the original ore, through heavy media floats at 3·0 sp. gr., jig tailing and the classifier slime and assaying Fe, 39·68; SiO_2, 9·81 and Al_2O_3, 19·48 per cent with a total loss of 32·5 per cent Fe by weight of the original ore sample investigated.

Sintering characteristics of the —⅜ in. classifier sand obtained after washing of —1 in. ore and a combined heavy media and jig concentrate obtained therefrom were comprehensively investigated. It was observed that optimum moisture content necessary was 7 per cent for obtaining good metallurgical grade self-fluxing as well as non-fluxing sinters. The coke content for a good sinter was 4 per cent. Self-fluxing sinters had higher degree of oxidation than those of the non-fluxing variety. It was interesting to note that self-fluxing sinters were invariably stronger than non-fluxing sinters. The strength of the sinter increased with the increase in basicity ratio up to a certain limit. Good metallurgical grade sinters were also produced when dolomite was added along with limestone, for increasing the MgO content in the sinter. The —⅜ in. washed material was of a poor grade and the sinter produced therefrom was high in silica and alumina contents, and also possessed poor strength. The total insoluble contents of the sinters produced from the —⅜ in. washed and beneficiated product were appreciably low.

To achieve full benefits of beneficiation mentioned above, the existing iron ore crushing and screening plant at Barsua will have to be suitably modified for introducing scrubbing, wet screening, dewatering and heavy media separation to the optimum extent needed.

1.1 Pilot Plant Studies on Beneficiation and Sintering of Bolani Iron Ore for Durgapur Steel Plant of Hindustan Steel Ltd

Pilot plant studies were undertaken on the beneficiation and sintering of representative sample of iron ore from Bolani mines, for use in Durgapur Steel Plant of Hindustan Steel Ltd with a view to solve the problem of handling sticky wet iron ore and to produce properly sized lumpy ore and sinter from ore fines low in silica and alumina for iron smelting.

A 100-tons representative sample of run-of-mine ore from Bolani ore mines, assaying Fe, 56·60; SiO_2, 5·00; Al_2O_3, 6·30; MgO, 0·57; CaO, 0·60; P, 0·06; S, 0·20; and loss on ignition, 7·30 per cent, was selected for comprehensive beneficiation and sinter-making studies.

The sample contained predominantly hematite, followed by martite, goethite, lepidocrocite, and ochre. Quartz was present in cavity walls and transverse cracks or displacement zones. Kaoline and feldspars which filled the cavities as well as pores in the iron oxides and laterite yielded the alumina content of the ore.

Screenability tests with different moisture contents of the ore indicated that maximum screening difficulty would be encountered when the moisture content of the ore was 10 per cent.

Washing tests (scrubbing followed by wet screening) indicated that a clean, sized, washed product (+ ⅜ in.) free from adherent ore fines for direct lumpy charge for the blast furnaces and a —⅜ in. classified sand product freed from undesirable slime for making self-fluxing sinter could be obtained.

Washing of the ore after crushing it separately to -3in., -2 in. and $1\frac{1}{2}$ in. resulted in (i) improvement in the Fe grade of washed products by 3·05 to 3·52 per cent and (ii) a decrease in the insoluble contents by 3·4 to 4·18 per cent. The rejections of silica and alumina in the slime were 53 to 59 and 25 to 31 per cent respectively of the total in each case for an iron loss in the washed product of 4·4 to 6·1 per cent through the slime.

When washing was followed by heavy media separation, there was considerable further improvement in the grade of the sink product and an appreciable decrease in its total silica and alumina contents. For ready comparison and evaluation, the results of washing and heavy media separation carried out after crushing the ore to -3 in., -2 in. and $-1\frac{1}{2}$ in. sizes are summarized in Figs 4, 5 and 6 respectively.

It would be seen that heavy media separation of the $+\frac{3}{8}$ in. washed products at medium sp. gr. 3·0 could produce sink products assaying 62·80 to 63·87 per cent Fe, 1·10 to 1·29 per cent SiO_2 and 2·70 to 2·84 per cent Al_2O_3 with recoveries ranging from 47 to 51 per cent Fe in the upgraded ore, excluding the iron content of metallics in $-\frac{3}{8}$ in. ore fine fractions.

The results of ore washing and heavy media separation thereafter also indicated that the grade of the product obtained at -2 in. was better than that at -3 in. There was no appreciable difference in the results obtained at -2 in. and $-1\frac{1}{2}$ in. sizes. However, beneficiation at $-1\frac{1}{2}$ in. size may only be necessary if the lattice showed superior reducibility characteristics in relation to coarser lumpy ore size.

The $-\frac{3}{8}$ in. classifier sand obtained by washing at -2 in. size, constituting 34·9 per cent by weight, assayed Fe, 57·32; SiO_2, 4·03 and Al_2O_3 6·47 per cent and contained 35·4 per cent of the total iron content of the ore; this can be used as such for sinter-making. Alternatively, this product could be further beneficiated, if required, extending heavy media separation up to $+6$ mesh and using the heavy media concentrate and the -6 mesh fraction mixed together for making optimum metallurgical grade sinter. Thus, the $-\frac{3}{8}$ in. classifier sand obtained by washing at -2 in. when subjected to heavy media separation yielded a $-\frac{3}{8}$ in.$+6$ mesh sink product assaying Fe, 63·9; SiO_2, 1·52 and Al_2O_3, 2·70 per cent; this product when mixed with the -6 mesh fraction would form a sinter feed assaying Fe, 58·56; SiO_2, 3·89 and Al_2O_3, 5·61 per cent with a recovery in the -6 mesh fraction of 26·3 per cent Fe.

The -6 mesh fraction could also be further beneficiated by jigging, if required. The $-\frac{3}{8}$ in.$+6$ mesh sink at 3·0 sp. gr. and -6 mesh jig concentrate combined together to form the sinter feed assaying Fe, 62·23; SiO_2, 2·41 and Al_2O_3, 3·75 per cent would then constitute 18·9 per cent by weight of the total ore sample investigated with a recovery of 20·8 per cent Fe in the -6 mesh jigged ore fraction. The entire operations of washing at -2 in. followed by heavy media separation and jigging would produce reject products constituting 38·7 per cent weight of the ore sample comprising floats at 3·0 sp. gr., jig tailing and slime assaying Fe, 45·58; SiO_2, 11·03 and Al_2O_3, 11·66 per cent with a total loss of 31·2 per cent Fe and total rejection of 82·1 per cent SiO_2 and 70·6 per cent Al_2O_3 contents of the ore sample investigated.

Washing of only the $-2\frac{1}{2}$ in. fraction of the run-of-mine ore constituting two-thirds of the ore sample and wet screening only of the $+2\frac{1}{2}$ in. fraction after prior crushing it to -2 in., produced a combined $+\frac{3}{8}$ in. product assaying Fe, 61·28; SiO_2, 1·78 and Al_2O_3, 6·36 per cent with a recovery of 37·5 per cent Fe. The -2 in.$+\frac{3}{8}$ in. wet-screened material obtained after crushing the original $+2\frac{1}{2}$ in. screened fraction to -2 in., was of a good grade and hence its further treatment by heavy media separation was not necessary. Heavy media

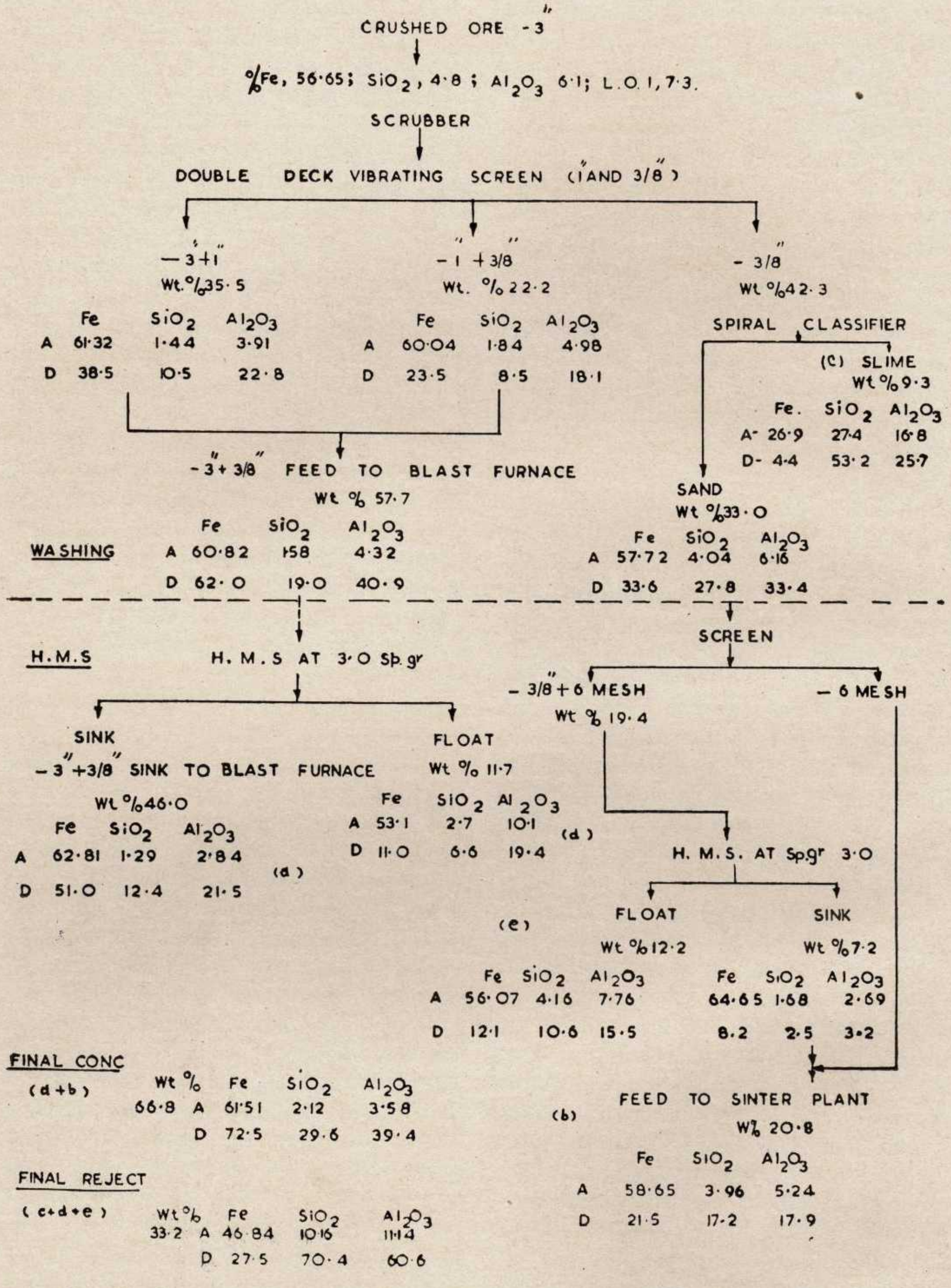


FIG. 4 — SUMMARY OF RESULTS OF BENEFICIATION AT 3 IN. SIZE OF BOLANI IRON ORE

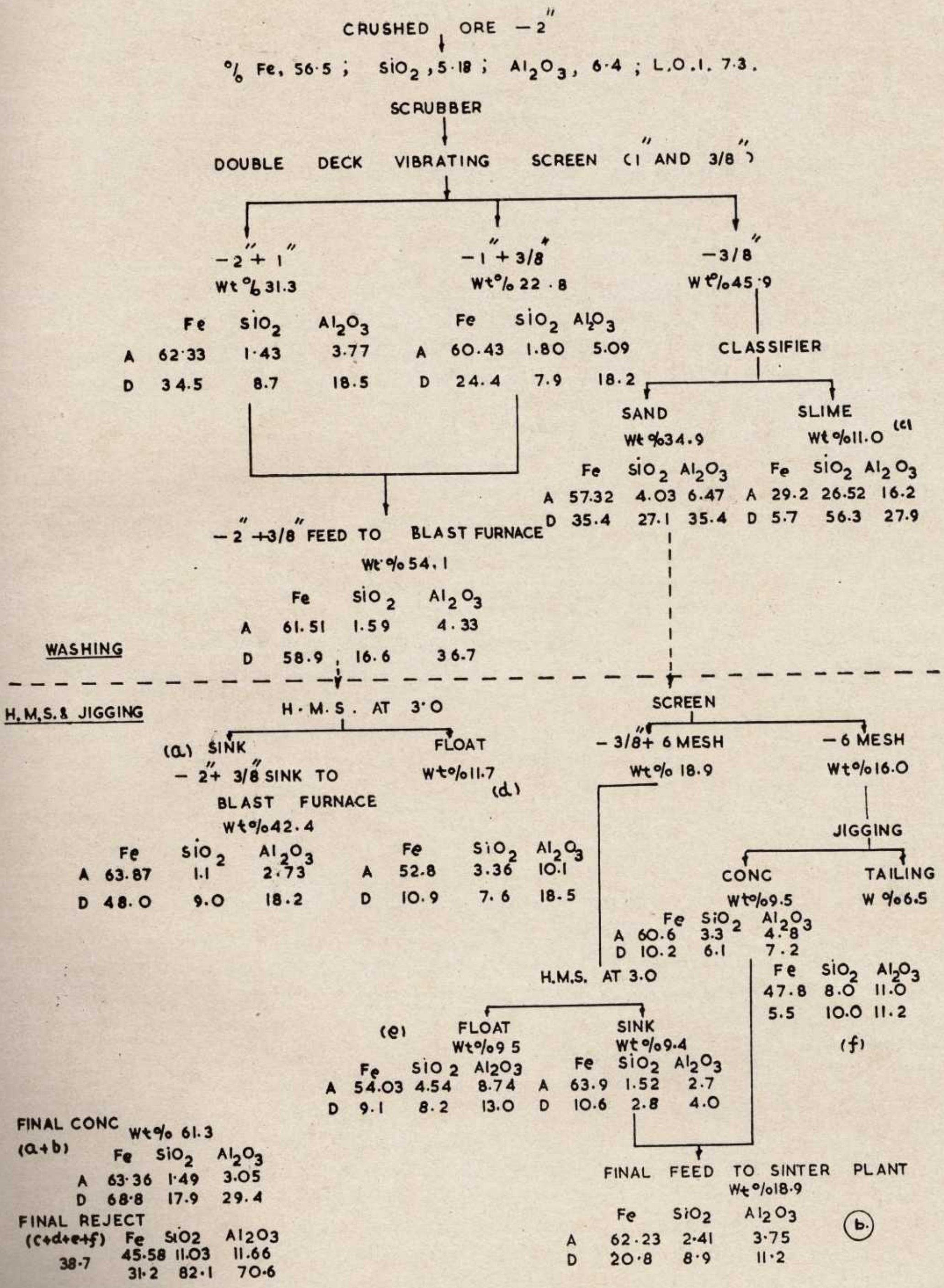


FIG. 5 — SUMMARY OF RESULTS OF BENEFICIATION AT 2 IN. SIZE OF BOLANI IRON ORE

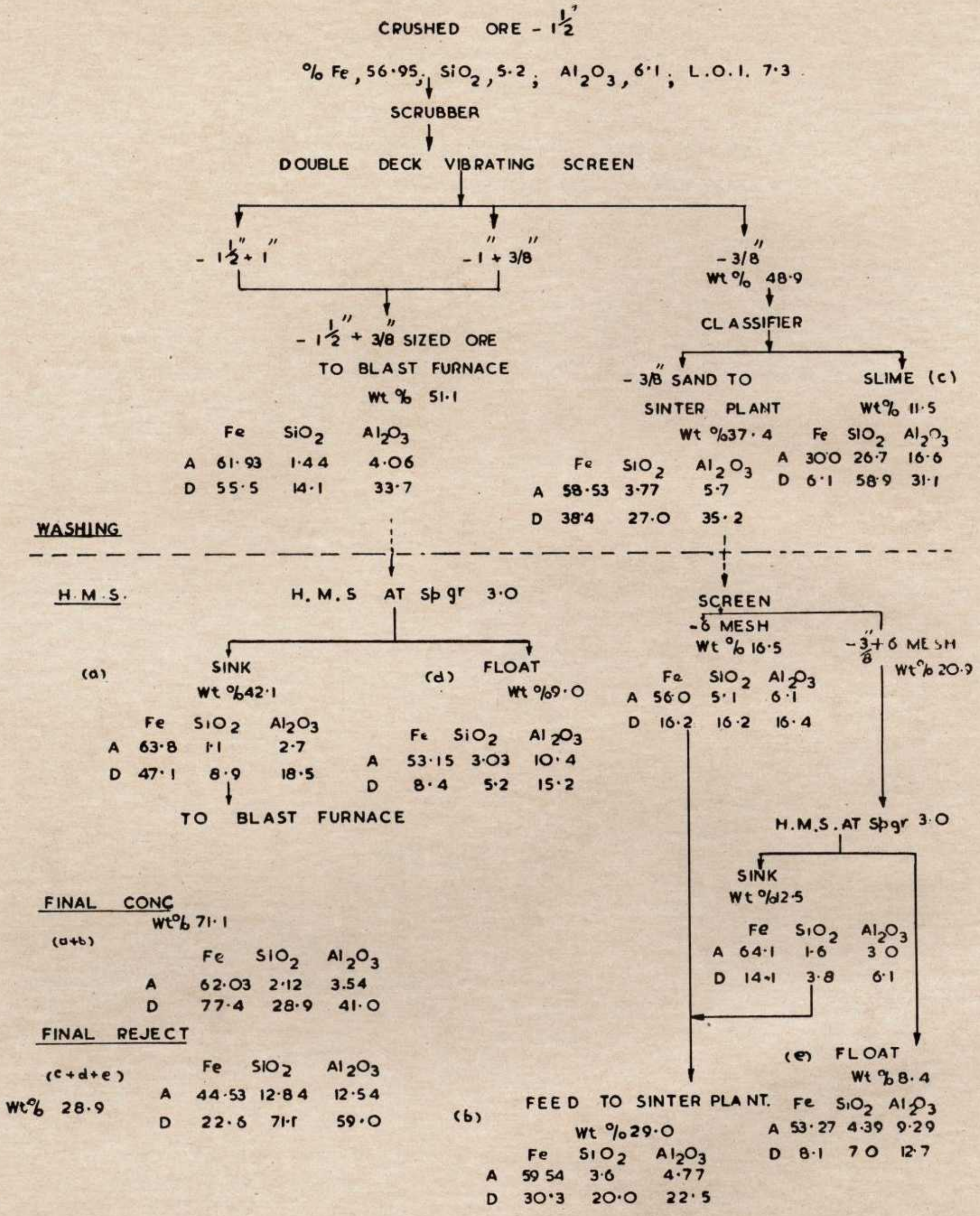


FIG. 6 — SUMMARY OF RESULTS OF BENEFICIATION AT 1½ IN. SIZE OF BOLANI IRON ORE

separation of the original $-2\frac{1}{2}$ in. $+\frac{3}{8}$ in. washed product yielded a sink product at 3·0 sp. gr. containing high iron content. This sink product when combined with the -2 in. $+\frac{3}{8}$ in. wet screened product would produce a material suitable for blast furnace lumpy charge assaying Fe, 62 90; SiO_2, 1·35 and Al_2O_3, 3·74 per cent with a recovery of 49·6 per cent Fe. The $-\frac{3}{8}$ in. classifier sand obtained by wet screening was of a good grade and further treatment of this product to improve the grade might also be not necessary. The grade of the $-\frac{3}{8}$ in. classifier sand obtained by washing the $-2\frac{1}{2}$ in. fraction of the ore could further be improved by heavy media separation and jigging.

The results obtained by (i) washing/heavy media separation after crushing the whole of the ore to -2 in., when compared to results obtained by (ii) washing and heavy media separation of only the $-2\frac{1}{2}$in. fraction of the ore and (iii) wet screening only of the -2 in. fraction obtained from the original $+2\frac{1}{2}$ in. fraction after prior crushing it to -2 in., showed that $+\frac{3}{8}$ in. fraction in the latter two cases (ii + iii) was higher in alumina content by 1·01 per cent, and lower in grade (Fe) by 1·0 per cent whilst yielding an improved iron recovery of only 1·6 per cent in relation to (i) above; the only advantage gained by the latter two cases (ii + iii) was that on an overall basis, 34·3 per cent of the bulk of the run-of-mine ore can " by-pass " the crusher and heavy media plant. But the flowsheet will thereby be more complex and capital expensive since a separate screen, classifier and conveying system will have to be provided for flow cycles (ii + iii) above. Any variations of the ore characteristics would also affect the operations of such a plant. On the other hand, treatment of the entire run-of-mine ore after crushing it to -2 in. (i above) will involve a simpler flow-sheet and also ensure uniform quality of the products.

Sintering characteristics individually of (i) $-\frac{3}{8}$ in. classifier sand obtained after washing of -2 in. size ore, (ii) a combined heavy media and jig concentrate obtained from the $-\frac{3}{8}$ in. classifier sand and (iii) $-\frac{3}{8}$ in. dry screened product produced from -2 in. ore, were comprehensively investigated; it was observed that optimum moisture contents necessary for obtaining high sintering rates were 7, 7 and 8 per cent respectively for the three types of ore fines.

It was also observed that self-fluxing sinters had invariably a higher degree of oxidation in relation to non-fluxing sinters. Good sinters were produced when dolomite was also added alongwith limestone to increase the MgO contents of sinter.

Silica and alumina contents were obviously higher in the sinters produced from $-\frac{3}{8}$ in. ore fines obtained after dry screening -2 in. ore size than the sinters made from the other two types of treated ore fines, apart from the position that the former were not of good metallurgical grade of strength. A general flowsheet recommended for treatment of whole of the ore is given in Fig. 7. The run-of-mine ore is fed on to a roll grizzly with 4 in. opening. The oversize $+4$ in. is crushed in a primary crusher (Jaw or Gyratory crusher) and joins the -4 in. material and conveyed to a single deck punched plate vibrating screen with $2\frac{1}{2}$ in. round hole openings (equivalent to 2 in. square opening). The oversize is crushed in a secondary crusher to -2 in. size and joins the -2 in. from the single deck punched plate vibrating screen before going to the scrubber. Provisions should be made for fast jets of water sprays on the punched plate screen in case screening difficulties were encountered during rainy monsoon weather. The scrubber should, therefore, be suitably located so as to be able to receive the -2 in. screen undersize along with considerable water.

The scrubber discharges the ore on to a double deck vibrating screen having water

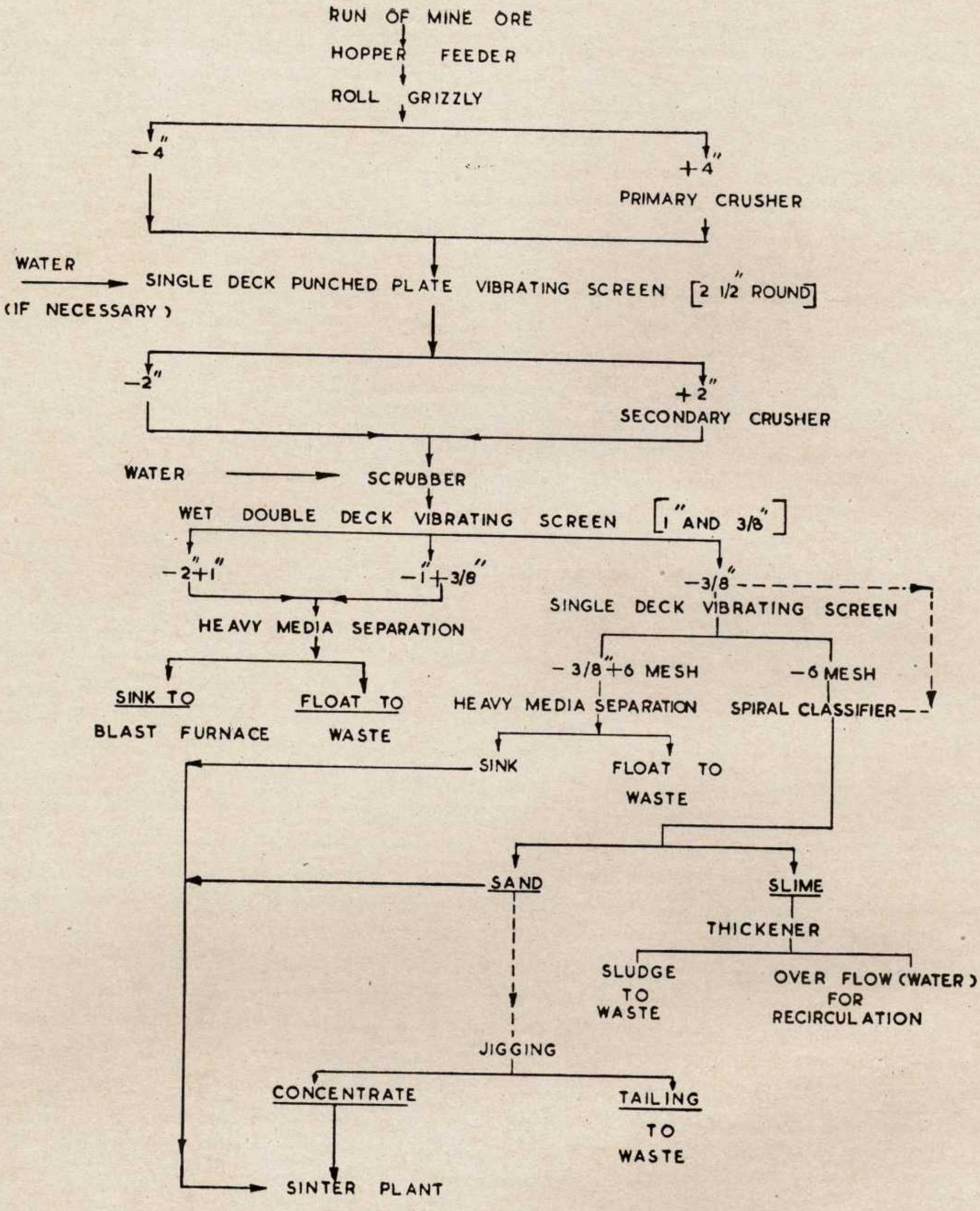


FIG. 7 — GENERAL FLOWSHEET FOR BENEFICIATION OF IRON ORE FROM BOLANI

sprays and separates the $+\frac{3}{8}$ in. ore for further treatment in heavy media separation plant. The $-\frac{3}{8}$ in. washed material is further screened on a single-deck vibrating screen to obtain $-\frac{3}{8}$ in.$+$ 6 mesh fraction for heavy media separation in a separate unit. The -6 mesh fraction is dewatered and the sand portion is jigged to yield a concentrate to be mixed with $-\frac{3}{8}$ in.$+$ 6 mesh heavy media separation concentrate for thickener treatment for reclamation of water. Such as flow sheet can form the basis for the design of a plant, engineering details of which will have to be separately worked out. The plant may be advantageously located at the mine-site since disposal of the slime, reject float obtained by heavy media separation and jig tailing can be readily effected and transport of the rejects constituting 30 to 40 per cent by weight of the ore will be avoided to disposal sites.

1.2 Pilot Plant Studies on the Beneficiation of Iron Ore from Kiriburu

At the instance of the National Mineral Development Corporation, Govt. of India, beneficiation studies were undertaken on a representative sample of iron ores from Kiriburu iron ore mines, so as to yield iron ores of suitable qualities to meet the expanding need of the country's iron and steel industries as well as for export purposes.

A representative 100 tons sample was obtained for investigation. The original sample assayed 60·96 per cent Fe, 2·4 per cent SiO_2, 5·2 per cent Al_2O_3 and 5·3 per cent loss on ignition. The $+$ 4 in. fraction of the sample as received, constituted 35·6 per cent by weight of the ore sample and assayed Fe, 65·3; SiO_2, 0·75 and Al_2O_3, 3·1 per cent. Washing and heavy media separation tests were performed with the sample at -1 in., -2 in. and -3 in. size.

Washing and heavy media separation at -1 *in. size* — Washing and wet screening the ore after crushing it to -3 in. followed by crushing the -3 in.$+$ 1 in. fraction to -1 in. and then wet screening produced a combined -1 in.$+\frac{3}{8}$ in. product assaying Fe, 64·28; SiO_2, 0·91 and Al_2O_3, 3·24 per cent with a recovery of 67·2 per cent Fe. The $-\frac{3}{8}$ in. classifier sand assayed Fe, 59·07; SiO_2, 2·41 and Al_2O_3, 5·64 per cent with a recovery of 26·5 per cent Fe. The slime assayed Fe, 43·21; SiO_2, 14·2 and Al_2O_3, 12·48 per cent with a loss of 6·3 per cent Fe and rejection of 50·6 per cent SiO_2 and 23·6 per cent Al_2O_3 of the ore. Heavy media separation at 2·9 sp. gr. of -1 in.$+$ 6 mesh product assayed Fe, 66·04; SiO_2, 0·61 and Al_2O_3, 2·0 per cent with a recovery of 67·7 per cent Fe.

Washing and heavy media separation at -2 *in. size* — The washed -2 in.$+\frac{3}{8}$ in. ore fraction after crushing the ore to -2 in., assayed Fe, 64·18; SiO_2, 1·81 and Al_2O_3, 3·55 per cent with a recovery of 78·0 per cent Fe. The $-\frac{3}{8}$ in. classifier sand assayed Fe, 57·37; SiO_2, 3·58 and Al_2O_3, 5·96 per cent with a recovery of 16·3 per cent Fe. The slime assayed Fe, 43·58; SiO_2, 13·3 and Al_2O_3, 11·27 per cent. The heavy media separation of the washed -2 in.$+$ 6 mesh product yielded a sink at sp. gr. 2·9 assaying Fe, 65·69; SiO_2, 0·85 and Al_2O_3, 2·72 per cent with a recovery of 72·2 per cent Fe.

Washing and heavy media separation at -3 *in. size* — Washing of the ore after crushing it to -3 in. produced a -3 in. $+\frac{3}{8}$ in. product assaying Fe, 63·83; SiO_2, 0·85 and Al_2O_3, 2·96 per cent with a recovery of 79·4 per cent Fe. The $-\frac{3}{8}$ in. classifier sand assayed Fe, 56·46; SiO_2, 3·23 and Al_2O_3, 6·82 per cent with a recovery of 15·3 per cent Fe. The slime assayed Fe, 41·09; SiO_2, 15·83 and Al_2O_3, 13·33 per cent with a loss of 5·3 per cent Fe and rejection of 51·0 per cent SiO_2 and 23·6 per cent Al_2O_3 of the total. Heavy media separation at 2·9 sp. gr. of -3 in.$+$ 6 mesh product yielded a concentrate assaying Fe, 65·48; SiO_2, 0·57 and Al_2O_3, 1·67 per cent with a recovery of 75·7 per cent Fe. Further work is in progress.

1.3 Pilot Plant Beneficiation of Low-grade Magnetite from Salem

With a view to determine the suitability of smelting magnetite ore from Salem in South India for production of pig iron, pilot plant studies were undertaken to produce 75 tons of concentrate from low-grade Salem magnetite ore. The ore as received assayed Fe, 36·57; and SiO_2, 44·88 per cent. The concentrate obtained by grinding the ore to about 65 mesh followed by wet magnetic separation assayed Fe 65·62 per cent and SiO_2 7·74 per cent with a recovery of 90·8 per cent Fe. Cleaning of a portion of the concentrate improved the grade to 70·4 per cent Fe and 2·74 per cent SiO_2 with a recovery of 88·8 per cent Fe.

Sixty tons of the concentrate obtained were sent to Govt. of Madras for despatch to Norway for undertaking electric smelting trials. Electric Smelting trials are also being conducted at the National Metallurgical Laboratory employing Salem magnetite concentrate.

1.4 Beneficiation of Classifier Sand and Heavy Slime from the Noamundi Washing Plant, Tisco

Representative sample of classifier sand from the Noamundi Washing Plant of Tata Iron & Steel Co. Ltd, assaying Fe, 59·40; SiO_2, 1·95 and Al_2O_3, 6·28 per cent was received for conducting Humphrey Spiral tests so as to obtain a high grade concentrate product.

Jigging of the $-6 + 28$ mesh fraction of the sample produced a concentrate assaying Fe, 61·15; SiO_2, 1·40 and Al_2O_3, 4·83 per cent with a recovery of 35·8 per cent Fe. Spiral treatment of the -28 mesh fraction could produce a concentrate assaying Fe, 63·63; SiO_2, 1·12 and Al_2O_3, 3·70 per cent with a recovery of 30·0 per cent Fe. The jig and spiral concentrate mixed together would assay Fe, 62·26; SiO_2, 1·27 and Al_2O_3, 4·32 per cent with a total recovery of 65·8 per cent Fe.

Straight jigging of the sample produced a concentrate assaying Fe, 61·01; SiO_2, 1·46 and Al_2O_3, 3·84 per cent with a recovery of 82·6 per cent Fe, indicating that it would be much better than a jigging/spiral combined treatment.

A sample of heavy slime from the settling tank at Noamundi washing plant assaying Fe, 59·12; SiO_2, 5·19; Al_2O_3, 6·41 per cent and containing 84·3 per cent by weight of -325 mesh material when subjected to spiral treatment, produced a concentrate assaying 60·54 per cent Fe but with a low recovery of only 29·9 per cent Fe; this heavy slime was too fine to ensure satisfactory separation of alumina by spiral treatment.

1.5 Beneficiation of Top Bench Iron Ore and Kanga Iron Ore from the Western Ridge, Noamundi

In the process of mechanized mining at Noamundi Iron Ore mines, it was observed that laterite and slab were associated with the ore from the top bench, whereas the lower levels were comparatively free from lumpy aluminous gangue. Beneficiation of the ore produced from the top bench was, therefore, considered necessary to bring down its alumina content.

Four samples of the ore from the top bench were received for conducting sink and float, as well as jigging tests for comparative studies.

Sample No. 1 — A sample of iron ore from top bench at Noamundi, assayed Fe, 51·07; SiO_2, 6·48 and Al_2O_3, 8·96 per cent. Jigging of the sample after crushing to -3 mesh produced a concentrate assaying Fe, 57·00; SiO_2, 3·57 and Al_2O_3, 6·40 per cent with a recovery of 55·9 per cent Fe. Heavy media separation of the sample up to + 6 mesh fraction after crushing it to about 2 in. size produced a concentrate assaying Fe, 63·59; SiO_2, 1·33 and Al_2O_3, 3·85 per cent with a recovery of 49·0 per cent Fe. Jigging of the −6 mesh fraction produced a concentrate assaying Fe, 56·24; SiO_2, 3·16 and Al_2O_3, 7·29 per cent with an additional recovery of 3·8 per cent Fe; this jig concentrate when mixed with the heavy media concentrate would yield a combined concentrate assaying Fe, 63·01; SiO_2, 1·48 and Al_2O_3, 4·13 per cent with an overall recovery of 52·8 per cent Fe.

Sample No. 2 — A sample of Kanga iron ore from Noamundi, assayed Fe, 64·80; SiO_2, 1·27 and Al_2O_3, 2·22 per cent. Crushing the sample to about 2 in. size and screening out the −3 mesh fraction produced a −2 in.+ 3 mesh fraction assaying Fe, 65·63; SiO_2, 1·05 and Al_2O_3, 1·83 per cent with a recovery of 91·3 per cent Fe. The −3 mesh fraction assaying Fe, 57·33; SiO_2, 3·24 and Al_2O_3, 5·78 per cent when subjected to jigging produced a concentrate assaying Fe, 62·50; SiO_2, 1·99 and Al_2O_3, 3·58 per cent with an additional recovery of 6·9 per cent Fe; this jig concentrate when mixed with the −2 in.+ 3 mesh fraction would assay Fe, 65·40; SiO_2, 1·12 and Al_2O_3, 1·96 per cent with a total recovery of 98·2 per cent Fe.

The sample of Kanga iron ore was already of a high grade, no beneficiation treatment was, therefore, considered necessary for it.

Sample No. 3 — A sample of the feed to Noamundi heavy media separation pilot plant prepared by washing and screening out the $-\frac{1}{8}$ in. fraction from a truly representative sample of top bench ore from Noamundi after crushing it to 2-3 in. assayed Fe, 60·50; SiO_2, 2·18 and Al_2O_3, 5·51 per cent. This sample was reported to be more representative than sample No. 1.

Jigging of the sample after crushing it to −3 mesh produced a concentrate assaying Fe, 65·83; SiO_2, 0·50 and Al_2O_3, 2·24 per cent with a recovery of 66·8 per cent Fe. Heavy media separation of the sample at the size received and employing a medium of sp. gr. 3·0 produced a concentrate assaying Fe, 65·90; SiO_2, 0·76 and Al_2O_3, 2·00 per cent with a recovery of 74·6 per cent Fe. The grades of concentrates obtained by jigging and by heavy media separation treatment were identical but heavy media separation yielded higher iron recovery. Thus jigging of this sample after prior crushing it to −3 mesh appeared to be unnecessary when heavy media separation could produce the same grade with better iron recovery at a coarse size. The heavy media separation float portion when subjected to jigging after crushing it to −3 mesh, could increase the iron recovery by another 5·7 per cent Fe yielding a concentrate assaying Fe, 57·2; SiO_2, 2·68 and Al_2O_3, 7·91 per cent. The jig concentrate and heavy media separation concentrate when mixed together would assay Fe, 65·21; SiO_2, 0·91 and Al_2O_3, 2·48 per cent with a total recovery of 80·3 per cent Fe.

Sample No. 4 — A sample of sink product obtained at Noamundi Heavy Media Separation Pilot Plant treating the ore from a top bench of Noamundi mines, assayed Fe, 63·46; SiO_2, 1·14 and Al_2O_3, 3·45 per cent. Further heavy media separation of the sample at the size received and employing a medium of sp. gr. 3·0 produced a sink assaying Fe, 65·55; SiO_2, 0·62 and Al_2O_3, 2·25 per cent with a recovery of 77·3 per cent Fe, which indicated that better results can be obtained with adequate density control of the medium and operational efficiency.

1.6 Studies on the Morphology of Phosphorus in Indian Iron Ores

Under the Council of Scientific & Industrial Research Fellowship Scheme, studies were initiated on the morphology of phosphorus in Indian iron ores, starting with iron ore from Noamundi top bench. Various fractions obtained by heavy media separation were sampled and chemically analysed. The result showed that phosphorus concentration was high in float at 3·0 in relation to float at 2·9 and sink at 3·0. Float at 2·9 had the maximum and sink at 3·0 the minimum laterite but the phosphorus content was more or less the same in both these fractions. It was concluded that the phosphorus did not increase with increase of the laterite. Two sets of samples, i.e. (1) Noamundi soft and (2) Noamundi hard, were taken for further study. Study of the Noamundi soft ore further confirmed that the phosphorus content was high in sink at 2·9. The intermediate product (in the float at 3·0 but sink at 2·9) had the maximum phosphorus content and the causes responsible for it were under investigation.

2.0 Pilot Plant Beneficiation Studies on a Limestone Sample from Tisco

Comprehensive laboratory and pilot plant investigations were taken up on the beneficiation of a limestone sample received from Tata Iron & Steel Co. Ltd., with a view to reduce its silica content so that the beneficiated product can be advantageously and effectively utilized in steelmaking.

About 20 tons of the limestone sample was received for beneficiation trials. The sample assayed CaO, 45·28; SiO_2, 7·8; MgO, 3·1; Al_2O_3, 1·9 and Fe_2O_3, 1·1 per cent. Chemical and mineralogical examinations showed that the problem of beneficiation consisted in the elimination of siliceous gangue from the calcite. The nature of association of calcite and the associated gangue suggested that froth flotation was the only practical method for their separation.

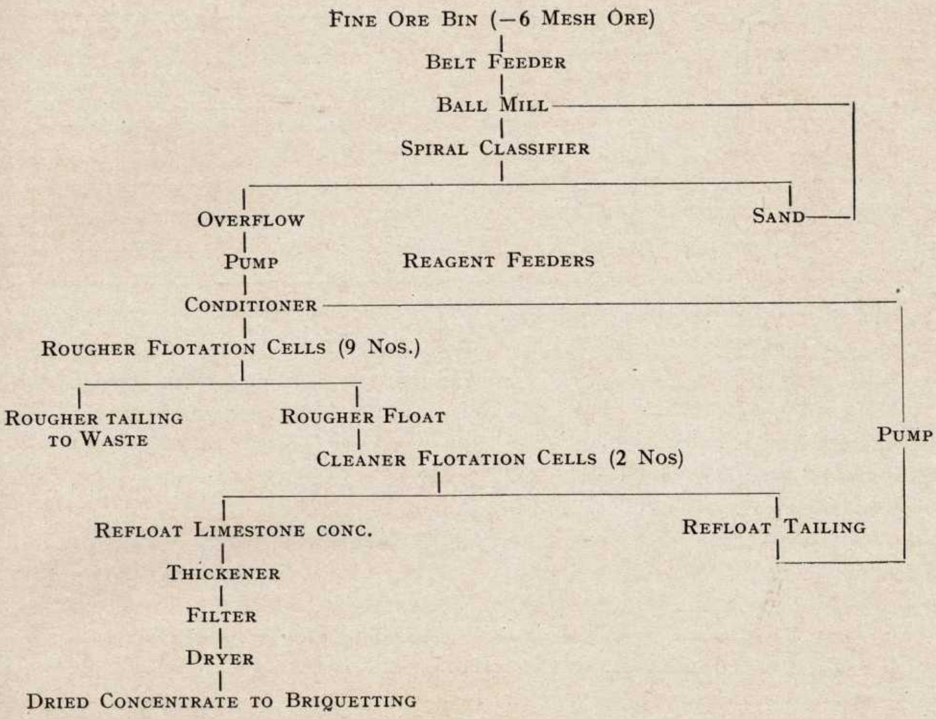
Initial batch scale experiments were carried out to determine the effects of (i) size of flotation feed and (ii) quantity of collector, depressant and frother on the recovery and grade of flotation concentrate. Having formulated optimum conditions for flotation on a laboratory scale, pilot plant studies were undertaken, to determine (i) optimum conditions for flotation in a continuous circuit where provision existed for recirculation of the reflotation tailing and (ii) to produce optimum high grade concentrate and briquette for steelmaking trials in a 250 tons open-hearth furnace.

Crushing of the sample to $\frac{1}{8}$ in. was done in a jaw crusher and a double roll crusher in closed circuit with a vibrating screen. A flowsheet of the grinding and flotation circuit of the pilot plant having a capacity of 250-300 lb. of ore/hr. is given on top of next page.

The ore crushed to about 6 mesh was fed at the rate of about 250 lb./hr. to a ball mill (16 in. × 32 in.) in closed circuit with a spiral classifier (9 in. dia.). The sand from the classifier was fed back to the ball mill for regrinding whereas the overflow was pumped to the conditioner. The pulp density in the ball mill was maintained at about 65 per cent solids whereas the classifier overflow was about 18-19 per cent solids.

Sodium silicate and oleic acid emulsion were added to the conditioner at the rate of 1·5 lb./ton and 1·0 lb./ton of limestone respectively. The conditioned pump was floated in 9 cells of No. 7 Denver Subaeration flotation unit. Additional quantities of oleic acid emulsion (0·25 lb./ton) were added into the 3rd and 6th cells of the unit. Primary tailing from the 9th cell was allowed to go as waste whilst the rougher float was led to another 2 cells of similar type for reflotation. 0·5 lb. per ton of sodium silicate was added at this stage. The reflotation tailings were recirculated in the flotation system by pumping them to the conditioner. The final concentrate was thickened or settled in settling ponds, filtered and dried for briquetting studies.

Water employed for the pilot plant studies was partly soft water and partly



tap water, since the water softener available did not have sufficient capacity to meet the entire requirements of the plant. Due to this, consumption of reagents was somewhat higher in pilot plant trials in relation to laboratory scale experiments. The pilot plant was found to operate satisfactorily when following conditions in the grinding circuit were maintained:

Feed rate — 250 lb. ore per hour.
Weight of balls — 520 lb.
Mill speed — 40 R.P.M.
Percentage solids in Mill discharge — 65 per cent.
Percentage solid in. in classifier overflow — 18-19 per cent.

Sieve analyses of the ball mill feed, discharge and the classifier overflow (flotation feed) when the ball mill was operating under optimum conditions are shown in Table 1.

Samples of tailing as well as concentrate were collected regularly for check analysis. The concentrate which was allowed to settle in settling pits was filtered, dried in a rotary drier and weighed before briquetting. The overhaul results of the campaign are given in Table 2.

TABLE 1 — SIEVE ANALYSES OF BALL MILL FEED, DISCHARGE AND CLASSIFIER OVERFLOW

SIZE IN MESH (T.S.S.)	ORIGINAL FEED %	BALL MILL DISCHARGE %	CLASSIFIER OVERFLOW %
+6 mesh	1·0	—	—
−6+8	8·0	—	—
−10+14	12·3	—	—
−8+10	13·0	—	—
−14+20	10·5	—	—
−20+28	8·7	0·5	—
−28+35	6·1	1·2	—
−35+48	5·7	2·1	—
−48+65	4·5	5·0	0·1
−65+100	4·1	9·0	0·3
−100+150	4·6	15·0	3·0
−150+200	4·5	20·4	8·9
−200	17·0	46·8	87·7
	100·0	100·0	100·0

TABLE 2 — OVERHAUL RESULTS OF FLOTATION ON A PILOT PLANT SCALE

PRODUCT	WT. %	ASSAY %		DISTRIBUTION %	
		CaO	Insolubles	CaO	Insolubles
Flotation conc.	78·5	49·84	2·9	86·5	21·7
Flotation tailing	21·5	28·45	38·3	13·5	78·3
Head (calc.)	100·0	45·25	10·5	100·0	100·0

The concentrate (moisture free) weighing 16·5 tons, assayed CaO, 49·84; MgO, 3·37; SiO_2, 2·3; Al_2O_3, 0·7 and CO_2, 39·1 per cent and represented a recovery of 86·5 per cent CaO; this product was later employed for briquetting studies. Extensive studies undertaken on the various aspects of briquetting indicated that fairly strong briquettes can be obtained using binders like molasses alone or a combination of molasses and cement. Green briquettes made with 6 per cent molasses and 8 per cent water are generally weak but after heating for drying in a pan, the briquettes were hard and strong to withstand handling. On the other hand, green briquettes made with 4 per cent molasses, 1 per cent cement and 8 per cent water after curing for a day or so were fully strong and could withstand handling — when 1 per cent cement was used, no heating of the briquettes for drying them will be required.

Studies on the utilization of the limestone concentrate fines in a pelletizing drum indicated that pelletizing also can be successfully employed for producing strong pellets for use in steel making. For pelletizing also binders employed for briquetting could effectively be used. However, it may be more economical to produce pellets rather than briquettes for use in steelmaking. Further studies are underway on pelletizing of fine limestone concentrates. Based on the above trials, a Report was prepared and proposals put forward for setting up a beneficiation plant to treat low-grade limestone at the rate of 250 tons/day to ultimately produce limestone concentrate briquettes for use in steelmaking. Economics of beneficiation of limestone and agglomeration of the concentrate were evaluated.

2.1 Beneficiation Studies on Limestone Samples from Purnapani Quarries, Sundergarh Dist., Orissa

Four limestone samples (Nos. 1, 2, 3 and 4) were received from the Hindustan Steel (P) Ltd, Rourkela, 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; acid insolubles, 9·65; Al_2O_3, 1·7; Fe_2O_3, 0·65 and SO_3, 0·3 per cent. Quartz was the chief gangue mineral. Calcite and dolomite constituted the carbonate minerals and were fairly liberated from the gangue at about 150 mesh size. Optimum conditions for flotation of limestone were determined employing an emulsion of oleic acid as collector and 1·0 lb./ton of sodium silicate. The concentrate assayed CaO, 49·73; MgO, 5·15; SiO_2, 1·92; Al_2O_3, 1·18; Fe_2O_3, 0·52 and S, 0·2 per cent with a recovery of 99·3 per cent CaO; this product was chemically suitable for use as a flux in iron and steel industry.

(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 total S, 0·25 per cent. Quartz which was the chief gangue was fairly liberated at about 150 mesh. Flotation tests employing 1·0 lb./ton of oleic acid emulsion and 1·0 lb./ton of sodium silicate produced a concentrate assaying 49·67 per cent CaO with a recovery of 97·3 per cent CaO. Reflotation yielded a concentrate assaying CaO, 50·1; MgO, 4·5; SiO_2, 1·44; Al_2O_3, 0·8; Fe_2O_3, 0·9 and S, 0·18 per cent with a recovery of 94·4 per cent CaO; this product was chemically suitable for use as a metallurgical flux.

(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. The gangue minerals in the sample consisted of quartz, phlogopite-biotite-chlorite, muscovite, feldspar and minor amounts of pyrrhotite, chalcopyrite and pyrite. The gangue was fairly well liberated at about 150 mesh size. Flotation using 1·0 lb./ton of sodium silicate and 0·75 lb./ton of oleic acid emulsion produced a concentrate assaying CaO, 49·83; MgO, 4·73; CO_2, 42·14; SiO_2, 2·2; Al_2O_3, 1·0; Fe_2O_3, 1·0; SO_3, 0·29; with a recovery of 95·0 per cent CaO; this product was suitable chemically for use as a metallurgical flux.

(iv) Sample No. 4 — The sample as received assayed CaO, 40·95; CO_2, 35·0; MgO, 5·4; SiO_2, 14·04; Al_3O_3, 3·08; Fe_2O_3, 1·1 and S, 0·2 per cent. Calcite and dolomite constituted the carbonate minerals and the gangue consisted primarily of quartz followed by chlorite, phlogopite, biotite, muscovite, feldspars and small amounts of pyrrhotite, chalcopyrite and pyrite. The gangue was fairly well liberated at about 150 mesh. Flotation with an optimum feed containing 73·1 per cent —200 mesh and using 0·75 lb./ton of oleic acid emulsion as collector and 1·0 lb./ton of sodium silicate as depressant for siliceous gangue, yielded a concentrate assaying CaO, 48·9; MgO, 4·76; CO_2, 40·9; SiO_2, 2·53; Al_2O_3, 1·28; Fe_2O_3, 1·3 and S, 0·3 per cent with a CaO recovery of 95·9 per cent. This product was suitable for use as a flux in steel-making.

3.0 Beneficiation of Calcitic Fluorspar from Matamagri Deposit in Rajasthan

The investigation was undertaken on behalf of Govt of Rajasthan to produce metallurgical grade of fluorspar through flotation.

A sample of low grade calcitic fluorspar from Matamagri deposit in Dungarpur District, Rajasthan, received for beneficiation assayed CaF_2, 26·9; $CaCO_3$, 27·0; SiO_2, 45·0; Al_2O_3, 1·13; Fe_2O_3, 0·70; Pb, 0·03 and S, 0·2 per cent. Quartz and calcite constituted the main gangue in the sample. Straight flotation of fluorspar followed by three cleanings yielded a metallurgical grade of concentrate assaying CaF_2, 90·93; $CaCO_3$, 7·05 and SiO_2, 1·14 per cent with 74·8 per cent CaF_2 recovery, indicating thereby that a flotation plant installed in Rajasthan for a siliceous type of fluorspar could also efficiently treat a calcitic type as well, for production of metallurgical grade of concentrate, but under slightly different flotation conditions.

Heavy media separation of the $-\frac{3}{4}$ in. + 10 mesh sample at 2·65 per cent specific gravity rejected 50·1 per cent by weight of the original ore in the float in which the elimination of $CaCO_3$ was 70·4 per cent; the loss of fluorspar was 9·7 per cent only. Flotation performed with the sink after mixing with the —10 mesh untreated fraction, employing the same reagent combinations, yielded a refloat concentrate assaying CaF_2, 92·35; $CaCO_3$, 4·8; SiO_2, 1·14; with CaF_2, 73·2 per cent recovery. The concentrate obtained either by heavy media separation or by flotation were considered suitable for metallurgical use after agglomeration.

3.1 Studies on the Suitability of Water from Lakshman Sagar in Dungarpur Dist., Rajasthan, for Flotation of Fluorspar from nearby Deposits

Earlier investigations had indicated that fluorspar samples from (1) Bhagatwali mine, (2) Ramorwali mine, (3) Thurwali mine and (4) Matamagri mine from Dungarpur District of Rajasthan could be concentrated for metallurgical purposes and acid manufacture by straight fatty acid flotation employing water demineralized in a Permutit water softener. The nearest source of water supply to the above fluorspar deposits is at Lakshman Sagar, situated about 2 miles away. Flotation studies were undertaken with the fluorspar sample employing water from Lakshman Sagar instead of soft water received for this purpose. Average values for the temporary and permanent hardness as $CaCO_3$ were reported to be 27·52 per cent and 3·47 respectively. The water had a *p*H of about 7·0. Results of experiments carried out showed that water from Lakshman Sagar can be successfully employed for fluorite flotation but the collector requirements may be higher by about 0·25 lb./ton for rougher flotation to get optimum results.

3.2 Briquetting Studies with Fluorspar Concentrate from Low-grade Ore from Rajasthan

Briquetting tests were carried out with flotation fluorspar concentrates. Briquettes made with 2 and 1 per cent cement keeping lime and sulphite lye constant at 6 per cent and 4 per cent respectively gave green compression strengths of 400 lb. and 375 lb./sq. inch respectively. The shatter values of the briquettes were also satisfactory. Briquettes made with varying sulphite lye and lime indicated that 3 per cent sulphite lye and 6 per cent lime were optimum for making hard and strong briquettes. Briquettes using cement and sulphite lye as binder were also prepared.

3.3 Pre-concentration of Rajasthan Fluorspar by Heavy Media Separation

At the instance of Director of Mines & Geology, Govt of Rajasthan, heavy media separation studies on the 50 tons sample of low-grade fluorspar sample were taken up for pre-concentrating the ore before flotation aiming at rejection of the barren gangue at a coarse size.

The sample as received was crushed to -3 in. size. It assayed CaF_2, 21·2; $CaCO_3$, 6·6; SiO_2, 58·3; Al_2O_3, 1·6 and Fe_2O_3, 1·8 per cent. The ore was crushed to -1 in. size and screened into the fractions: -1 in. $+\frac{3}{8}$ in., $-\frac{3}{8}$ in. $+$ 6 mesh and -6 mesh. Heavy media separation tests were carried out with the first two fractions only, at sp. gr. 2·6 and 2·65. Another test was done after crushing the ore to $-\frac{3}{4}$ in. size and sizing into the fractions: $-\frac{3}{4}$ in. $+\frac{5}{8}$ in., $-\frac{3}{8}$ in. $+$ 6 mesh and -6 mesh. The first two fractions were again subjected to heavy media separation at sp. gr. 2·60 and 2·65.

Results of heavy media separation studies indicated that the floats obtained at 2·6 sp. gr. assayed CaF_2 8·18 per cent and 7·66 per cent with losses of 13·9 and 15·0 per cent of original CaF_2 when heavy media feed was -1 in. and $-\frac{3}{4}$ in. respectively.

As fluctuations in the density of the medium would adversely affect the separation, it has to be closely controlled in the plant even to obtain laboratory scale results. It was, therefore, considered worth while to adopt heavy media separation for pre-concentrating the ore.

4.0 Beneficiation of Lateritoid Manganese Ore from Malaya

About 1 ton of ferruginous manganese ore sample was received from the Director, Eastern Mining and Metal Co. Ltd, Malaya, for beneficiation studies. The ore consisted of psilomelane/cryptomelane in predominance followed by wad with minor quantities of manganite. Lateritoid material formed the major gangue material with minor amounts of goethite, quartz and clayey minerals.

The sample as received assayed Mn, 18·9; MnO_2, 25·2; Fe, 19·85; SiO_2, 14·6; Al_2O_3, 15·5; BaO, 3·7; CaO, 1·6 and P, 0·05 per cent. The sample was passed over 2 in. screen. The oversize was crushed to −2 in. size. The two fractions were washed. Heavy media separation tests were conducted with the washed products at sp. gr. 3·0, 2·9 and 2·8 using galena. The combined concentrates after heavy media separation at 2·9 yielded a sink product assaying Mn, 33·1; Fe, 17·5; and SiO_2, 4·4 per cent with a recovery of 62·7 per cent Mn. As the grade of manganese was not good, an attempt was made to get a cleaner product by heavy media separation at sp. gr. 3·3, but the result was not satisfactory and the concentrate was still high in iron. A sample of the washed ore was then crushed to −3 mesh size and a jigging test was performed without success.

The original washed ore, crushed to −3 mesh size, was then subjected to magnetizing reduction roast treatment. Wet magnetic separation tests with the reduced ore at different sizes, viz., −3, −10, −28 and −65 mesh yielded non-magnetic manganese concentrates assaying Mn 48·1 and Fe, 3·7 per cent; Mn, 48·5 and Fe, 4·2 per cent; Mn, 48·3 and Fe, 4·1 per cent; Mn, 46·3 and Fe, 5·4 per cent with respective manganese recoveries of 54·7, 62·7, 67·2 and 68·2 per cent.

Dry magnetic separation after desliming the original sample crushed to different sizes did not yield satisfactory results. Heat-treatment at about 400°C. for an hour followed by dry magnetic separation at −10 and −28 mesh of the washed ore yielded concentrates assaying Mn, 41·8; Fe, 8·9; SiO_2, 3·5 and Mn, 42·3; Fe, 8·6; SiO_2, 3·9 with manganese recoveries of 63·7 per cent and 66·0 per cent respectively. Magnetic separation of the −28 mesh washed ore after heat treatment and sizing yielded a concentrate assaying Mn, 42·2; Fe, 8·8; SiO_2, 3·0 and Al_2O_3, 6·8 per cent with a manganese recovery of 64·0 per cent.

4.1 Briquetting and Sintering Studies on Manganese Concentrate from Low-grade Ore from Siljora-Kalimati Mines, Orissa

The investigation was taken up on behalf of Messrs Rungta & Sons, for preparing manganese concentrates from low-grade manganese deposits of their mines and examine their briquetting and sintering characteristics for charging directly in the furnace for ferro-manganese production.

Laboratory studies were made on the sintering characteristics of manganese concentrate from low-grade manganese ore from Siljora-Kalimati mines, Orissa. The effects of variables such as, coke, moisture and sinter fines in the sinter mix on the rate of sintering and their effects on the quality of sinter produced were studied with a sintering unit, designed and fabricated at the National Metallurgical Laboratory. Research results showed that the optimum amounts of coke and water contents were 6·0 per cent and 10·0 per cent respectively for producing a sinter with good-size stability. The sinter assayed Mn, 53·5; Fe, 8·1; SiO_2, 3·4; Al_2O_3, 10·2 and P, 0·11 per cent and was suitable for production of standard grade ferro-manganese. Briquetting tests with −20 mesh manganese concentrate using different percentages of lime, cement and sulphite lye as well as of pitch and sulphite lye in different proportions as binder were completed. Sintering studies were also undertaken on the manganese concentrate obtained from the low-grade ore of the locality. Effects of variables such as coke, moisture and sinter fines in the sinter mix on the rate of sintering and their effects on the quality of the sinter produced were determined. Results showed that optimum coke and water contents were 6·0 per cent and 10·0 per cent respectively for producing a sinter with good size-stability. The sinter assayed Mn, 53·5; Fe, 8·1; SiO_2, 3·4; Al_2O_3, 10·2; and phosphorus 0·11 per cent and was suitable for production of standard grade ferro-manganese.

4.2 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.

Scanty data are 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 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.

Samples high in phosphorus were collected from Banswara District, Rajasthan; Nagpur and Bhandara District, Maharashtra, and Balaghat District of Madhya Pradesh. Phosphorus content of the samples varied from 0·2 to 0·4 per cent and in one sample from Rajasthan, it was as high as 0·57 per cent.

Rajasthan samples showed that the phosphorus content increased with the increase in the iron content. Samples from Maharashtra and Madhya Pradesh showed that in most samples, phosphorus was present as apatite. It was observed that apatite could be separated as a non-magnetic fraction by magnetic separation and a comparatively low phosphorus manganese concentrate could be obtained as a magnetic product. Microscopic studies revealed that apatite also possessed some affinity for garnets. A garnet sample picked up from the manganese ore showed phosphorus content as high as 0·62 per cent equivalent to 3·36 per cent of apatite and removal of garnet could, therefore, reduce the phosphorus content in the ore to some extent. Chemical analyses were undertaken to study variations of phosphorus with Mn, Fe and SiO_2 contents in different manganese ores. Products of sieve analyses products and isodynamic separation were analysed for their Mn, Fe, SiO_2 and P content which yielded useful data. Studies of the polished sections under the ore-microscope threw some light on the mineralogical assemblage, textural features and paragenesis of the ore-bodies. X-ray powder diffraction studies were also undertaken for confirming the structure of manganese minerals petrologically identified.

4.3 Flotation of Ferruginous Manganese Ores

Under the Council of Scientific & Industrial Research Fellowship Scheme, a project was initiated on the separation of manganese minerals from iron minerals by flotation techniques. The scheme consisted of three stages, viz. (i) collection of literature pertaining to the subject, (ii) collection and purification of the various manganese minerals and the associated gangue minerals and (iii) flotation experiments with purified minerals under varying conditions.

The first two steps of the scheme were successfully completed. Various manganese minerals and the commonly associated gangue minerals — pyrolusite, psilomelane, braunite, hollandite, sitaparite, jacobsite, rhodonite, hematite, goethite, and quartz were collected and purified.

Hallimond tube apparatus was fabricated with all the accessories. The air inlet tube was slightly modified for handling convenience. The equipment was standardized with a limestone sample employing sodium silicate as depressant for siliceous gangue and oleic acid as collector for the carbonates. Some tests with synthetic mixture of Sambalpur manganese ore and quartz were conducted employing sodium silicate as depressant for quartz and commercial oleic acid as collector for manganese minerals in varying amounts but the results were not encouraging. The oleic acid was purified by steam distillation and the purified acid had also been employed in subsequent studies.

5.0 Recovery of Diamonds from Diamondiferous Ores from Panna, Madhya Pradesh

The National Mineral Development Corporation had projected the establishment of a modern milling plant for the recovery of diamond from two types of ores near Panna. In order to procure data for selecting the correct type of equipment for the plant, laboratory scale studies were initiated on samples of diamondiferous ores from Panna.

(i) *Diamondiferous Gravel from Ramkheria Mines, Panna* — Washing and screening of the ore straightway rejected 12·93 per cent by weight as + 3 in. and 55·55 per cent by weight as slime, total rejection being 68·48 per cent by weight. The −3 in. + 1 in. material was subjected to handpicking. Heavy media separation of −1 in. + 7 mesh and jigging of −7 + 20 mesh washed gravel respectively yielded concentrates containing 1·09 per cent and 0·29 per cent by weight of heavy minerals. Differential grinding and grease belt separation tests were next carried out with the total heavy mineral concentrates obtained from heavy media separation and jigging. Diamonds were employed in the feed. It was found that diamonds could be successfully recovered by this process. The −20 mesh fraction, 13·94 per cent by weight, was sampled into two fractions. One sample was hydraulically classified into three fractions. Tabling and electrostatic separation of the table concentrates obtained from the three classified products yielded three non-conducting concentrates, total being 0·22 per cent by weight. A few diamond grains in the concentrate were identified. The other sample from −20 mesh fraction was ground to −65 mesh size and subjected to froth flotation. It was found that 0·5 lb./ton of light diesel oil and 0·18 lb./ton of pine oil could efficiently float diamonds.

Based on these studies, a comprehensive beneficiation flowsheet was formulated for the treatment of diamondiferous gravels from Ramkheria Mine, Panna.

(ii) *Diamondiferous Tuff from Majhgawan Mines, Panna* — Primary crushing of the tuff followed by scrubbing and classification showed that the total loss of slime was of about 12 per cent of the original. The results of heavy media separation of the $-1\frac{1}{2}$ in. + 7 mesh fraction had shown that the total percentage of heavy minerals including diamonds (if present) was only 1·4 per cent. Examination of the sink product revealed the presence of a coarse piece of diamond of $-\frac{1}{4}$ in. + 7 mesh size, weighing about one carat. Jigging followed by subsequent treatment over the grease belt gave satisfactory results. A number of tests performed introducing a few diamonds in the feed confirmed the observation that diamonds (if present in the sample) could be recovered efficiently by grease belts. Lastly, flotation of the −20 mesh fraction, ground to −65 mesh, using light diesel oil, pine oil and oleic acid, gave a concentrate, which on petrological examination revealed the presence of a large number of deep blue grains (resembling those of diamonds) of very high refractive index (higher than 1·88) and high specific gravity (greater than 2·96). Those grains may be diamonds of the industrial type, but this observation needed confirmation. A flow sheet for the treatment of 200 tons of tuff/hr was formulated for the National Mineral Development Corporation.

6.0 Beneficiation of Low-grade Chromite from Vagda Deposit, Maharashtra

A low-grade sample of chromite from Vagda deposit, Ratnagiri district, Maharashtra, received from Geological Survey of India assayed Cr_2O_3, 33·14; FeO, 21·57; Soluble Fe, 1·3; MgO, 14·50; SiO_2, 14·74; Al_2O_3, 11·0; and CaO, 2·24 per cent. The chief gangue minerals in the sample were olivine followed by chlorite, serpentine, talc, muscovite and traces of hypersthene, magnetite and ilmenite. Liberation of chromite was found to be fairly good at −65 mesh and tabling of the sample at this size yielded a concentrate assaying Cr_2O_3, 47·4 and FeO, 26·2 per cent, with a recovery of 75·2 per cent Cr_2O_3. The table concentrates obtained at coarser sizes were of poorer grade. Neither flotation under the conditions investigated nor magnetic separation yielded satisfactory results.

7.0 Beneficiation of Low-grade Garnet from Guda, Ajmer

Five samples of garnet from Guda area, Ajmer, were received from Director of Mines and Geology, Government of Rajasthan, for beneficiation studies for use in abrasive manufacture. The samples were mixed and the mixed sample contained 57·0 per cent garnet. Almandite and spessartite constituted the garnet minerals in the sample, whereas the gangue minerals were biotite, chlorite, muscovite, quartz, feldspar, ilmenite and epidote. Tabling of sampling at −65 and −100 mesh sizes followed by magnetic or electrostatic separation of table concentrates did not yield concentrate suitable for abrasive purposes due to intimate association of gangue minerals with garnet.

8.0 Production of Ferro-alloys in Pilot Submerged Arc Smelting Furnace

With the broad-based objectives of developing technical ‘know-how’ for the production of diverse ranges of ferro-alloys from indigenous raw materials and formulation of electric smelting techniques, a Pilot Submerged Arc Furnace has been in operation at the National Metallurgical Laboratory with a production capacity of 1-3 tons of ferro-alloys per day depending on the types of ferro-alloys required. Raw materials will be accepted on a research-contract basis for testing their amenability to electro-thermal reduction and for determining the most suitable electro-thermal reduction techniques. A full report giving recommendations on smelting practice, power consumption, size of installation, economics of production required for a given output will be furnished to interested industrial organizations based on these smelting trials.

The Submerged Arc Electric Smelting Furnace is housed in a 32 ft. high specially built bay of 33 ft × 51 ft, served by a 3-ton crane-hoist. The substation containing the circuit breaker, furnace transformer and amplidyne controller and low-tension distribution board has been built as an integral unit adjacent to the smelting bay. The furnace, being of the open type, is provided with a fume extractor which is housed in an adjoining building. The ferro-alloy smelting plant is equipped with its own ore handling and preparation plant including crushing, sizing, briquetting or pelletizing etc.

The furnace with a shell of 8 ft. internal diameter is surrounded by a charging

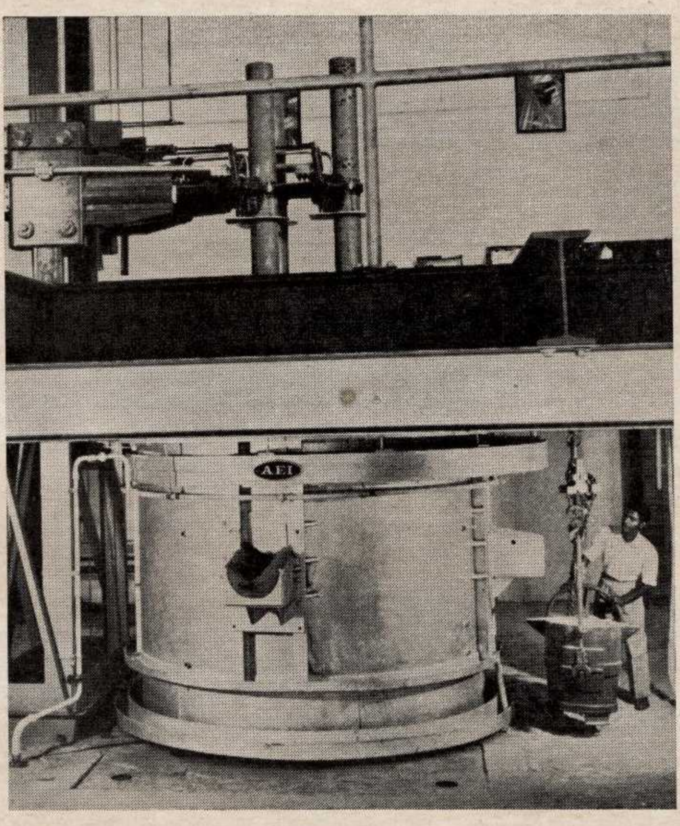


FIG. 8 — THE PILOT SUBMERGED ARC SMELTING FURNACE (1-3 TONS/DAY CAPACITY) FOR PRODUCTION OF FERRO-ALLOYS, INSTALLED AT THE NATIONAL METALLURGICAL LABORATORY

platform at a height of 9 ft from the main floor level. The furnace controls are suitably housed on a panel adjoining the operating platform. The furnace has a normal rating of 500 kVA. and can be operated at any desired voltage to permit requisite smelting operations including the preferential reduction of iron from ilmenite, direct smelting of iron ores, etc. The furnace shell can be rotated or oscillated on either side at various speeds through a reduction gear.

A mono-rail loop running round the shell beneath the platform level permits handling of metal and slag in ladles mechanically. Several thermo-couples are provided in the shell built in through refractory lining to facilitate temperature measurements of the hearth and side walls at different points.

The furnace is equipped with electrode holders suitable for 8 in. dia. electrodes and the electrode clamps are arranged for remote control by pneumatically operated cylinders. A water cascade is also provided for circulation of cooling water. Ample space has also been provided for the storage of refractories, electrodes and other materials.

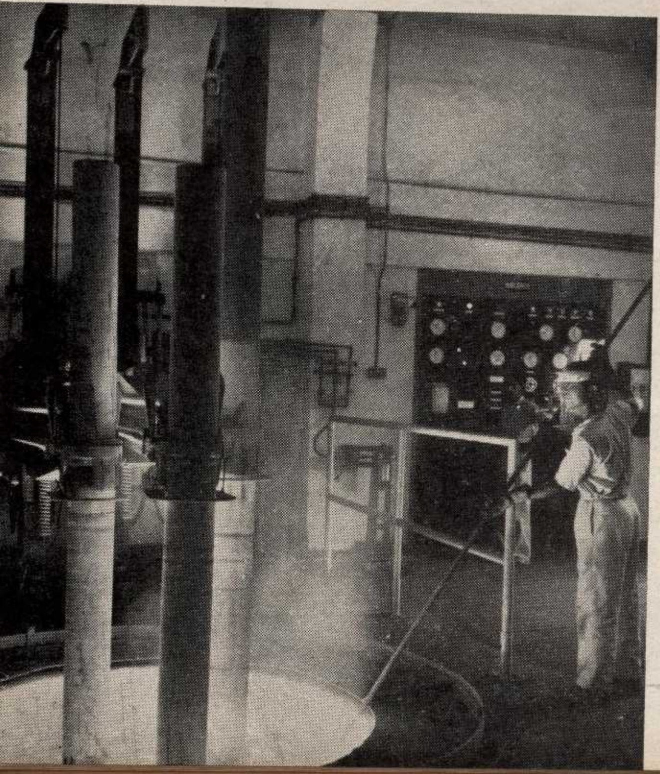
Following are some of the electro-thermal reduction studies, planned for execution:

(i) *Production of High Carbon Ferro-chrome and Silico-chrome* — The production of ferro-chrome has not been established in the country, though a number of industrial licences have been granted for a total production of 45,000 tons of ferro-chrome. The specifications for ferro-chrome limit the phosphorus content to 0·05 per cent and the investigations envisaged will involve the selection of suitable reductants and formulation of electrothermal smelting techniques.

(ii) *Production of Low-phosphorus Ferro-manganese* — Specifications for the export grade of ferro-manganese limit the phosphorus to 0·25 per cent and with the raw materials available in India, it is difficult to produce ferro-manganese with less than 0·35 per cent phosphorus. The possibilities of preferential removal of iron and phosphorus through thermal beneficiation will also be examined.

(iii) *Electric Smelting of Iron Ores* — In areas where deposits of coal are not available in close proximity to iron ore deposits, and where cheap hydroelectric power is available, electro-thermal reduction of iron ore deserves special consideration.

FIG. 9 — A VIEW OF THE PILOT SUBMERGED ARC SMELTING FURNACE IN OPERATION



(iv) *Smelting of Ilmenite* — Considerable difficulties are being encountered in the use of indigenous ilmenites for the production of pigment grade of TiO_2 as Indian ilmenites contain small quantities of vanadium and chromium. The possibilities of preferential reduction of iron along with vanadium and chromium are being examined on a laboratory scale which will subsequently be tried in the pilot ferro-alloy furnace. The establishment of electro-thermal smelting of ilmenite will not only help in promoting the export of titania-rich slag but substantial quantity of pig iron will also be thereby available.

9.0 Extraction of Magnesium Metal Electrolytically from Magnesium Chloride

The work was taken up initially at the instance of M/s. Tata Chemicals Ltd, for utilization of by-product bittern from their salt works by the extraction of magnesium metal. Nearly 1·1 million tons of magnesium salt are being produced annually from Indian salt industry as by-product, with a very limited market. This bittern can, however, be utilized for production of magnesium metal by electrolytic process.

Systematic laboratory scale investigations into drying characteristics of the hydrated magnesium chloride with and without any mixture of alkali chlorides in various proportions were completed in a small oven, fitted with hot air circulation and mechanical stirrer arrangement. On the basis of data collected, an oven having a drying capacity of 24 kg. per day on an average was designed and fabricated. The total dried salt produced in the small oven was nearly 500 kg. during the year, this salt was used for subsequent batch trial in electrolysis.

Electrolytic test trials were carried out with different diaphragm compositions and different cell assembly. The electrolysis was conducted for 18 hours, including premelting of the charge for 16½ hours and actual electrolysis for 1½ hours. Current efficiency was 58 per cent and the metal purity obtained was 99·8 per cent in a vat of 110 kg. capacity of fused salt. Difficulties with ceramic diaphragm were partially overcome. The diaphragm withstood 18 hours of working from the start of feeding to the completion of electrolysis.

The cell dimensions and its assembly including anode drift mechanism were modified in the light of the difficulties encountered. The fabrication of the cell, anode lifting mechanism, furnace and anode draft were completed. In the present vat nearly 300 kg. of total dried salt were required as the first charge, of which nearly 280 kg. of dried salt were collected. The D.C. load in the present cell was found to be in the region of 380-410 amps. at 7-8 volts. For this purpose, two 250 amps. 0-15 volts rectifier units were connected in parallel with suitable bus bar supply lines.

It was possible to dehydrate the hydrated magnesium chloride from waste bittern suitable for use as cell feed for extraction of magnesium metal. A scheme for 1000 amps. cell was prepared for subsequent work and necessary materials are under collection.

10.0 Electrolytic Production of Chromium

Good deposits of pure chromium can be obtained by the electrolysis of chromic acid solutions containing small percentages of sulphate, but high cost of this operation, owing principally to the fact that the current efficiency was 15 per cent, rendered the metal very expensive.

Because of its difficult and costly preparation, potential use of electrolytic chromium was limited and as such this work was taken up with a view to improving the current efficiency in chromium deposition which ultimately would determine the economics of the process.

Experiments were carried out in 1500 ml. beaker with 1000 ml. solution. In the preliminery experiments, lead was used as anode, but this was found to become passive within a short period. It was, therefore, replaced by a 7 per cent tin-lead anode. The cathode used was brass. Results are shown in Tables 3, 4 and 5.

From the results obtained in Tables 3 and 4, it could be observed that the addition of hydrofluoric acid to the chromic acid bath increased the current efficiency up to a certain extent and beyond that further addition of hydrofluoric acid decreased the current efficiency. There was a limiting concentration of hydrofluoric acid to get the maximum current efficiency. Moreover, it was also clear that the hydrofluoric acid increased the current efficiency up to 23 per cent which was 60 per cent more than the usual chromium plating solution. However, due to the presence of two catalysts, the control of the plating solution was more difficult and complicated. In ordinary chromium plating bath, there were only two variables, namely chromium acid and sulphuric acid or sulphate. In

TABLE 3 — EFFECT OF HYDROFLUORIC ACID ON THE CURRENT EFFICIENCY OF THE BATH IN PRESENCE OF SULPHATE

Composition of the bath:
Chromic acid = 300 *gm./l.*
Sulphuric acid = 1 *gm./l.*
Temperature = 55°±3°*C.*
Current density = 360 *amp./sq. ft*

Test No.	Hydrofluoric Acid in gm. per Litre	Current Efficiency in Percentage
1	0·45	15·5
2	0·60	19·2
3	0·80	23·1
4	1·20	22·8
5	1·60	22·3
6	2·00	21·2
7	2·40	19·3

TABLE 4 — EFFECT OF SULPHURIC ACID ON THE CURRENT EFFICIENCY OF THE BATH IN PRESENCE OF HYDROFLUORIC ACID

Composition of the bath:
Chromic acid = 300 *gm./l.*
Hydrofluoric acid = 0·8 *gm./l.*
Temperature = 55°±3°*C*.

TEST No.	SULPHURIC ACID IN GM. PER LITRE	CURRENT DENSITY IN AMP. PER SQ. FT	CURRENT EFFICIENCY IN PERCENTAGE
1	0·489	360	20·3
2	0·979	360	23·0
3	1·959	360	20·8
4	2·447	360	19·4
5	3·427	360	16·8
6	0·489	504	21·7
7	0·979	504	23·2
8	1·959	504	22·6
9	2·447	504	21·1
10	3·427	504	17·9

TABLE 5 — EFFECT OF CHROMIC ACID CONCENTRATION ON THE CURRENT EFFICIENCY OF THE BATH IN PRESENCE OF HYDROFLUORIC ACID, RATIO OF CHROMIC ACID TO HYDROFLUORIC ACID BEING MAINTAINED AT 100:1·6 THROUGHOUT

Temperature of the bath = 55°±3°*C*.

TEST No.	CHROMIC ACID IN GM. PER LITRE	CURRENT DENSITY IN AMP. PER SQ. FT	CURRENT EFFICIENCY IN PERCENTAGE
1	200	360	22·8
2	250	360	24·3
3	325	360	22·3
4	400	360	19·8
5	500	360	14·7
6	200	504	25·3
7	250	504	26·5
8	325	504	24·8
9	400	504	21·6
10	500	504	18·3

the case of two catalyst solutions, there were three variables and it made the solution more difficult to control. The effect of chromic acid concentration on the current efficiency of the bath is recorded in Table 5. Highest current efficiency was obtained when the concentration of chromic acid was 250-300 gm. per litre. Further work with other addition agents to increase the current efficiency was in progress.

11.0 Electro-refining of Lead

The work was initiated at the instance of the Metal and Steel factory, Ichapore, Ministry of Defence, with a view to obtaining 99·9 per cent pure lead from secondary lead ingots obtained from spent bullet cores containing appreciable amounts of antimony, tin and bismuth. This recovery of pure lead will reduce the import of the pure metal used by the Defence Department for making bullet cores.

The process of electro-refining of lead was restricted to bullion containing not more than 2 per cent total impurities, in which copper should not be more than 0·04 per cent, tin 0·05 per cent and antimony 1·5 per cent. Lead bullion weighing 160 kg. from Defence Department was on chemical analyses found to vary from ingot to ingot in composition. To make the impurities almost uniform, lead bullions were melted in a lot in a melting furnace and cast into pig. Average chemical analyses of the original blocks were as follows: antimony, 1·42 to 1·63; tin, 0·12 to 0·21; copper, 0·05 to 0·081; bismuth, 0·05 to 0·09; iron trace, 0·10; arsenic and zinc trace. Balance was lead. The composition after mixing was found as follows:

	LOT	
	No. I	No. II
%		
Sb	1·56	1·56
Sn	0·12	0·15
Cu	0·084	0·075
Bi	0·08	0·082
Fe	0·09	0·075
As	Trace	Trace
Cd	do	do
Zn	do	do
Ag	do	do
Total impurities	1·934	1·942

The blocks were subjected to dressing and softening operations one after another in the same unit with 10 kg. of metal in each batch, to reduce mainly copper and tin. Reproducible and effective reductions were obtained. The bullion was melted with sulphur to convert the copper to copper sulphide (Cu_2S) with a little excess of sulphur. The melting temperature was raised to dull red and the mass was agitated. The temperature was brought down after some period whilst constantly removing the accumulated scum. The metal was sampled. Without cooling, the softening treatment was carried out by raising the temperature. A continuous air jet agitation was maintained. The metal was then cooled and the scum mostly oxides and a good quantity of litharge separated and skimmed out. The metal was sampled and cast into blocks. The average results of chemical analyses of the blocks are given below:

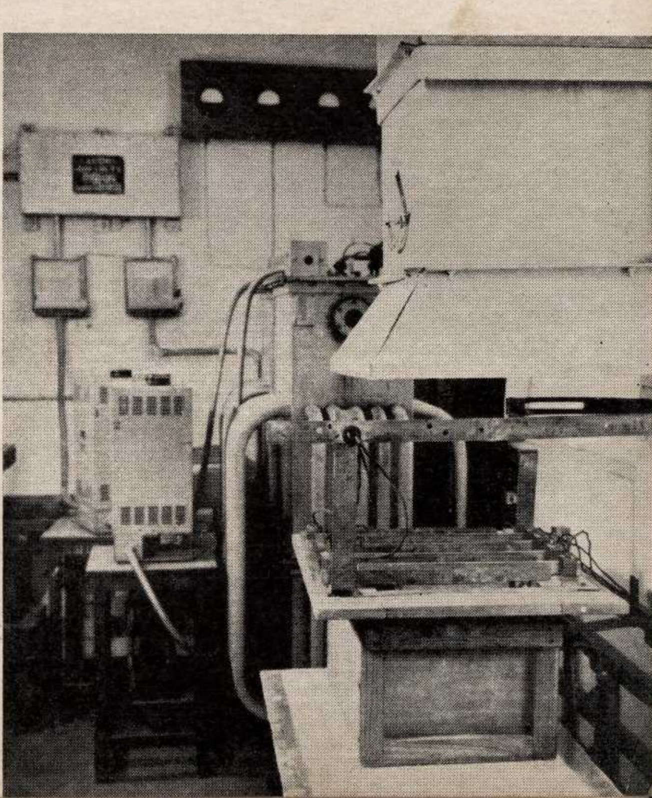
	AFTER DROSSING		AFTER DROSSING	
%	I	II	I	II
Sb	1·20	1·25	0·930	0·920
Sn	1·138	0·158	0·050	0·056
Cu	0·035	0·032	0·043	0·042
Bi	0·10	0·11	0·142	0·138
Fe	0·09	0·085	0·065	·060
As	—	—	—	—
Cd	Trace	Trace	Trace	Trace
Ag	—	—	—	—
Zn	—	—	—	—

The blocks were melted and cast into slabs for use as soluble anode.

Electrolyte was prepared batchwise in two stages in perspex vat. Finely crushed quartz was digested with A.R. grade hydrofluoric acid. To ensure complete commission of hydrofluoric acid to the corresponding fluosilicic acid, the amount of quartz was maintained slightly above the theoretical requirements. To the supernatant liquid (decanted), a calculated amount of basic lead carbonate was added. The composition of the electrolyte was then adjusted to the requisite value. Various factors affecting the overall performance, particularly the current efficiency and purity of the metal, were studied on laboratory scale experiments. From study of electrolytic conductivity of the electrolyte containing lead fluosilicate and a little excess silicofluoride, it was found that 40-50 gm./litre lead as lead fluosilicate, 90-100 gm./litre free fluosilicate and 130-150 gm./litre total acid had a very high conductivity.

During bench scale experiments, a perspex vat of 8 in. × 5 in. × 6 in. was used as electrolytic cell with stainless steel (18/8) sheet as cathode. Electrolysis was continued initially for 2 hours and then increased to 4 hours and 24 hours duration; metal purity of 99·96 per cent with current efficiency 96-97 per cent was obtained in 2 hours period which dropped to 95/96 per cent in 4 hours and to 92/93 per cent in 24 hours period. Nearly 13·6 kg. of metal was collected by these trials. Collected deposits were stripped out from cathode starting sheets and melted into one block.

FIG. 10 — SET-UP FOR THE ELECTROLYTIC REFINING OF LEAD



Results were encouraging enough to carry out bigger scale trials. For this purpose 12 in. × 12 in. × 18 in. alkathene welded vats were fabricated. Electrolysis was conducted for 4 hours and 24 hours period. Current efficiency was 91 to 93 per cent and purity of metal was 99·95 per cent. Continuous electrolysis for 3 days was attempted but due to difficulty of stainless (18/8) steel cathode corrosion, the metal deposits were spongy during the latter part of the electrolysis though a purity of 99·94 per cent lead was obtained with only 83 per cent current efficiency.

It was decided to use the refined lead already collected as starting cathode. For the purpose, the refined lead blocks were rolled to sheets and after giving necessary shapes, copper connections were made. Fresh electrolyte was prepared, and electrolysis for 5 days' duration was done with current efficiency of 90·2 per cent. Nearly 12·6 kg. of metal was collected by this operation. All electrolytic trials were carried out at room temperature with stationary electrolyte, occasional hand stirring was made. Further work is in progress.

12.0 Smelting of Ilmenite for Removal of Iron

Beach sands of Tranvancore, Madras and Maharashtra contain large deposits of ilmenite associated with rutile, zircon, monazite, etc. Indian ilmenites though high in titania are associated with small quantities of chromium and vanadium. The treatment of ilmenite with concentrated sulphuric acid for the production of pigment grade TiO_2 was not only wasteful since considerable acid was consumed by iron, but the presence of vanadium and chromium also affected the quality of the pigment.

The study was taken up with the idea of producing a pig iron and a titania-rich slag by smelting ilmenite with carbon and suitable fluxes. The pig iron so produced would contain most of the chromium and vanadium present in the ore and the slag would be practically free from iron and other harmful impurities.

The smelting trials were conducted in a 50 lb. direct arc furnace, lined with either magnesite or sillimanite. The bath temperature was controlled to prevent the formation of titanium carbides and nitrides, which resulted in very viscous slag. The effect of various fluxing agents, quantity and rate of additions of coke was investigated. It was observed that sillimanite lining was rather badly affected and the slag picked up considerable quantity of alumina and silica. In preliminary experiments carried out, though most of the ore was reduced to the metallic state and a considerable proportion of vanadium and chromium was also reduced, the slag was invariably contaminated with the lining material. Magnesite was found to be the best lining for ilmenite smelting and further studies were conducted on the behaviour of lining on repeated smelting. The effect of fluxes in yielding a fluid slag would also be investigated. Pre-reduction of the ilmenite with carbon in the solid state (at a temperature of 1100-1200°C.) and melting of the product with magnesite flux would also be examined.

13.0 Production of Ferro-alloys by Alumino-thermic Reaction

The alumino-thermic reactions for the production of ferro-alloys are known for their versatility, ease of operation and adaptability on cottage industry scales. The process consists in the reduction of oxides by aluminium. On ignitions of the reaction mass, powdered aluminium combines with the oxygen content of the metallic oxide until it is consumed. Slag consisting mostly of aluminium oxide and a metal regulus are obtained. The reactions are highly exothermal and temperature as high as 2500°C. is obtained. The development of alumino-thermal reactions is of great importance under Indian conditions as the present demands of special ferro-alloys — such as those of vanadium, titanium, tungsten, carbon-free ferro-chrome, chromium metal, etc., may not warrant setting up individual electro-thermal capital intensive plants. Systematic studies were undertaken to produce the different grades of ferro-alloys.

(i) *Carbon Free Ferro-chrome* — The studies on the production of carbon-free ferro-chrome were completed and 230 kg. of alloy was produced.

(ii) *Production of Ferro-titanium* — Systematic studies for the production of different grades of ferro-titanium were undertaken. The factors being studied included the effects of particle size, rate of heating, preheating temperature, quantities of reducing agent, energizers, fluxes, size and shape of vessel. It was observed that for the production of 25 per cent grade ferro-titanium using ilmenite and iron ore energizers, recoveries of titanium were poor while recoveries of iron were almost 100 per cent. Further studies were continued to get improved titanium recoveries. For the production of ferro-titanium containing 40 per cent titanium, it was observed that rutile was to be used as raw material instead of ilmenite and mill scale to be used as an energizer.

14.0 Thermal Beneficiation of Low-grade Chrome Ore

Large reserves of low-grade Indian chrome ores having Cr: Fe ratio less than 3 cannot be directly used for the production of high grade ferro-chrome. The conventional physical methods fail to improve this ratio as FeO and Cr_2O_3 are in a chemically combined state in the chrome spinel. Studies were undertaken to upgrade the ore by preferential reduction of FeO and its subsequent removal by acid leaching.

An extensive investigation was carried out to beneficiate a low-grade chrome ore from Mysore by preferential reduction with gaseous as well as solid carbonaceous reducing agents. It was possible to beneficiate the ore with an initial Cr: Fe ratio of 1·01 to an upgraded ratio of 3 or higher by preferential reduction of the ore with coke and subsequent acid leaching. A standard procedure for testing different low-grade chrome ores was then evaluated. Three different samples were received from the Director, Geological Survey ot India, for beneficiation studies. Normal ore-dressing methods could not improve the Cr: Fe ratio of such chrome ores although the gangue materials could be removed to yield high chromium concentrates by gravity treatments. These table concentrates were used for thermal beneficiation studies.

(i) *Low-grade Ore from Kankauli Deposits of Maharashtra* — The table concentrate of Kankauli chromite contained 50·83 per cent Cr_2O_3 and had an initial Cr: Fe ratio of 1·6: 1. The concentrate was reduced with 10 per cent by weight of coke at different temperatures from 1100° to 1300°C. and optimum temperature for reduction was then found to be 1250°C. Studies on the effect of particle size on reduction suggested an optimum ore size of −52 mesh (B.S.S.) and coke of −200 mesh (B.S.S.). From observations on kinetics of reduction at 1250°C., it was observed that the reduction was almost complete in 2 hours. Under such optimum conditions, an upgraded Cr: Fe ratio of 10 with a chromium recovery of 85 per cent was obtained. Under similar conditions the original ore yielded a Cr: Fe ratio of 4 with a chromium recovery of about 90 per cent. Leaching of reduced ore samples with hot dilute sulphuric acid was completed in 40 minutes. The dissolution of chromium could be suppressed

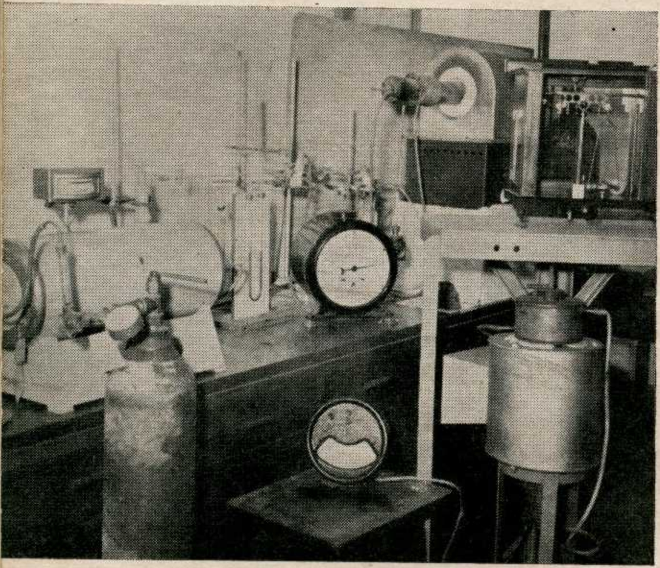


FIG. 11 — SET-UP OF THE APPARATUS FOR STUDYING THE REDUCIBILITY OF ORES

by controlled adjustment of the amount of acid in the leaching medium.

(ii) *Low-grade Ore from Vagda deposits of Maharashtra* — The original ore contained 31·95 per cent Cr_2O_3 with a Cr: Fe ratio of 1·4. Table concentrate of the ore containing 44·93 per cent Cr_2O_3 with a Cr: Fe ratio of 1·5 was used for thermal beneficiation studies. Optimum temperature for preferential reduction of the ore was found to be 1250°C. Optimum sizes of ore and coke for reduction at 1250°C. were found to correspond to -100 mesh (B.S.S.) and -170 mesh (B.S.S.) respectively. A minimum period of $2\frac{1}{2}$ hours was necessary for maximum preferential reduction. Under such optimum conditions, the concentrate could be beneficiated up to a Cr: Fe ratio of 8 with 90 per cent Cr recovery. The leaching was completed in 30 minutes with hot dilute sulphuric acid. The chromium dissolution could be suppressed subsequently by fixing the amount of acid at 10 per cent less than the theoretical requirement for removing the whole of reduced iron.

(iii) *Low-grade Ore from Enuconda Mines of Andhra Pradesh* — A table concentrate of the ore containing 40·83 per cent Cr_2O_3 and an initial Cr: Fe ratio of 1·23 was used for thermal beneficiation studies. The sample could be beneficiated by reduction at 1250°C. for a period of $2\frac{1}{2}$ hours maintaining the particle sizes of ore and coke at -100 mesh and -200 mesh (B.S.S.) respectively. Under these conditions, a beneficiated ratio of 12·6 could be obtained with 91 per cent chromium recovery. Almost all soluble iron could be leached out in 30 minutes by using hot dilute acid for leaching. Chromium dissolution could also be suppressed by controlling the amount of leach acid to 10 per cent less than the theoretical amount.

15.0 Reducibility of Bolani Iron Ores

At the instance of Hindustan Steel Ltd, systematic studies were taken up on reducibility of iron ores from Bolani mines after its beneficiation, so as to evaluate the optimum size for iron smelting. Reducibility tests were carried out on the crushed and washed as well as heavy media separated products of $-3+\frac{3}{8}$ in., $-2+\frac{3}{8}$ in. and $-1\frac{1}{2}+\frac{3}{8}$ in. sizes and on sinters prepared from $-\frac{3}{8}$ in. Bolani iron ores. Preliminary experiments indicated that a temperature of 800°C. and 5·3 litres/minute hydrogen flow considerably decreased the time for complete reduction of the sample to about $3\frac{1}{2}$ hours. In order to study the reducibility of the different products under similar conditions, the three fractions of the ore sample weighing about 150 gm. were reduced with hydrogen at 800°C. and 5·3 litres/minute flow rate. It was found that there was not much difference in the inherent reducibility of the original ore as a result of prior crushing to different sizes and washing the separately crushed products comprising $-3+\frac{3}{8}$ in., $-2+\frac{3}{8}$ in. and $-1\frac{1}{2}+\frac{3}{8}$ in. fractions. The effect of ore size on the rate of reduction was studied by sub-dividing the washed and heavy media products to $-3 + 2\frac{1}{2}$ in., $-2\frac{1}{2} + 2$ in., $-2 + 1\frac{1}{2}$ in., $-1\frac{1}{2} + 1$ in,$-1 + \frac{3}{4}$in.—and $\frac{3}{4} + \frac{3}{8}$ in. fractions and testing the reducibility of each fraction at 800°C. with hydrogen at a flow rate of 5·3 litres/minute. The time for 90 per cent reduction of different fractions is given in the following table:

TABLE 6 — EFFECT OF SIZE OF WASHED AND HEAVY MEDIA SEPARATED ORE FRACTION ON THE RATE OF REDUCTION

Weight of ore = 500 *gm.*
Rate of flow = 5·3 *litres/minute*
Temperature = 800°*C.*

SIZE OF ORE	TIME IN MINUTES FOR 90% REDUCTION	
	Washed ore fractions	Heavy media separated fractions
$-3 + 2\frac{1}{2}''$	255	260
$-2\frac{1}{2} + 2''$	200	200
$-2 + 1\frac{1}{2}''$	175	160
$-1\frac{1}{2} + 1''$	205	185
$-1 + \frac{3}{4}''$	185	150
$-\frac{3}{4} + \frac{3}{4}''$	195	160

It was observed that the rate of reduction decreased with reduced size of ore sample though no marked difference was observed in the different fractions below 2 in. The products obtained after heavy media separation were more reducible than those obtained on washing alone which was attributed to the removal of gangue products. It may be concluded that ore crushed to $-2 + \frac{3}{8}$ in. washed and beneficiated by heavy media separation would be optimum for iron smelting in a blast furnace.

Reducibility tests were done on sinters produced from $-\frac{3}{8}$ in. size fraction of Bolani iron ores. Self-fluxing and non-fluxing sinters were prepared under different conditions. 150 gm. of each sinter crushed to $-0{\cdot}263 + 0{\cdot}185$ in. size were reduced in a vertical tube furnace, the sample being suspended in a stainless steel wire mesh basket. The course of reduction was followed by readings in a thermal balance. The sample was reduced at 800°C. with hydrogen at a flow rate of 5·3 litres/minute. It was observed that non-fluxing sinters took 130-150 minutes for 9 per cent reduction while the washed ore took only 65 minutes. The self-fluxing sinter produced with the basicity of 1·3 with higher lime content was assessed to be the best so far as reducibility was concerned and took 49 minutes for 90 per cent reduction. The sinter with 10 to 17 per cent FeO content reduced in a minimum time of 49-56 minutes for 90 per cent reduction.

16.0 Utilization of Amjhore Pyrites

Native sulphur does not occur in India in any workable quantities and the only sulphur bearing deposits wherefrom sulphur can be recovered are the pyrites at Amjhore in Bihar and Tugaldal near Chitaldurg, Mysore. The reserves of Amjhore deposits have been estimated at about 100 million tons with sulphur content up to 48 per cent. The Pyrites Chemical and Development Company set up by the Government of India has been examining the possibilities of the production of elemental sulphur from the Amjhore pyrites by the Orkla process. The pyrites for charging in the shaft furnace should be in massive form and should retain due strength without crumbling under load up to softening temperatures in the furnace. The decomposition studies of Amjhore pyrites were undertaken to investigate the possibilities of charging the pyrites in the Orkla furnace.

It was observed that the Amjhore specimens lost labile sulphur much faster, 20 per cent being lost within 200 minutes while the Orkla pyrites lost about 14 per cent sulphur during 200 minutes of heating. Eleven specimens examined from different areas of Amjhore deposits were found to crumble into small pieces and none of them retained any strength. The sample from Orkla in Norway, however, retained its strength after firing at 700°C.

The possibilities of using briquettes in the shaft furnace were examined and the properties of briquettes made with binders like sodium silicate, and cement and lime before and after heating to remove labile sulphur were under study. The possibilities of alternative methods for the production of elemental sulphur were also under active scrutiny.

17.0 Utilization of Red Mud for the Recovery of Metallic Values

Red mud containing Fe_2O_3, TiO_2 and unrecovered alumina is a waste product of the aluminium industry and considerable quantities are dumped as waste. Investigations were taken up on the utilization of red mud for recovering alumina and other metallic values.

For recovery of alumina, the red mud was roasted with soda ash and limestone at 1050-1100°C. The roasted product was leached with water to extract the soluble sodium-aluminate and alumina subsequently precipitated from solution. Various factors studied were the effects of temperature, quantity of soda ash, limestone, particle size and time on the above treatment. The study of the effect of increasing soda ash additions on alumina recovery at 1050°C. showed that as the soda ash in the charge increased, the alumina recovery also increased but resulted in SiO_2 pick up in solution to the extent of 2-4 per cent of the total alumina in solution. It was observed that 160 gm. of commercial soda ash was required for 100 gm. of red mud assaying 21·5 per cent Al_2O_3 for a maximum recovery of 92 per cent alumina. It was also observed that 80 per cent of total Al_2O_3 in red mud could be easily recovered by roasting 100 gm. of red mud with only 35 gm. of soda ash at 1050°C. for four hours. Further studies on the effect of temperature on Al_2O_3 recovery with 35 per cent soda ash in the charge indicated that Al_2O_3 recovery increased with temperature from 850°C. to 1150°C. Optimum roasting time was observed to be four hours. Studies on leaching temperature and time showed that 90°C. and 60 minutes were optimum. It was observed that borax additions helped in the alumina recovery.

The leach liquor obtained contained nearly 2 to 3 per cent SiO_2 of the weight of alumina in solution. This solution as such was unsuitable for precipitation since SiO_2 would also be precipitated. Since the precipitated alumina should contain less than 0·05 per cent SiO_2, the solution was to be desilicated. The study of desilicating the solution was taken up. Synthetic sodalite type of compounds were prepared to be used as desilication seed.

18.0 Pilot Plant Study on the Recovery of Vanadium Pentoxide from Vanadiferous Magnetite Ore

Vanadium bearing titaniferous magnetite occur in Bihar and Orissa. These ores analyse on the average 55 to 65 per cent Fe, 8 to 12 per cent TiO_2 and 0·8 to 1·8 per cent vanadium. Based on the laboratory scale studies, a pilot plant treating one ton of ore per day was installed and systematic pilot plant trials for roasting and leaching and precipitation of V_2O_5 were taken up.

Vanadiferous magnetite containing 0·75 per cent vanadium was collected from Rairangpur (Orissa) and crushed to —30 mesh B.S.S. Systematic studies on optimum roasting conditions were completed.

The studies were conducted in a 12 ft. long and 9 in. inside diameter rotary kiln and rotating at a speed of about 0·8 r.p.m. The kiln is lined with sillimanite bricks in the high temperature zone and high duty fire clay rings elsewhere. The following variables for the maximum recovery of vanadium in the leach liquor were studied:

i) Effect of particle size of ore,
ii) Effect of sodium salts,
iii) Optimum percentage of sodium compounds,
iv) Temperature of roasting,
v) Period of roasting.

i) *Effect of Particle Size* — Vanadium was found to be very finely distributed in the vanadium ores and the different size fractions were found to contain the same vanadium content. The batch studies indicated that the recovery of vanadium rose with decrease in particle size of charge, but due to excessive dust losses in the rotary kiln, finer fractions could not be effectively studied. The optimum size of —30 mesh grind was used for further roasting studies.

ii) *Effect of Various sodium Compounds* — Sodium chloride and sodium carbonate were individually tried after mixing in various proportions. Sodium chloride did not prove to be suitable for that particular ore and sodium carbonate was finally selected for further studies.

iii) *Optimum Percentage of Sodium Carbonate* — The ore was roasted with varying quantities of soda ash under similar conditions. The recovery of vanadium rose steadily from 50 per cent to 80 per cent with increase in soda ash up to a maximum of 20 per cent. It was observed that additions of soda ash above 12 per cent did not improve the recovery to a sufficient extent to justify the extra cost involved in the process, i.e. cost of extra soda ash and cost of extra sulphuric acid for precipitation. Further studies on the temperatures of roasting and retention period in the kiln were carried out, using 12 per cent of soda ash.

iv) *Effect of Temperature* — The crushed ore was roasted with 12 per cent soda ash at different temperatures varying from 800° to 1000°C. On raising the temperature above 1000°C., the ore exhibited a tendency to sinter and the recovery dropped considerably. Further roasting studies were carried out at 900-950°C.

v) *Effect of Period of Roasting* — Laboratory scale studies called for roasting for 3 to 4 hours for the maximum recovery and the rotary kiln used for pilot scale studies could retain the charge at the high temperature zone for a maximum period of 30 minutes. To study the effects of increased retention period, the charge was recirculated through the kiln a number of times. The recovery improved only on first recycling and decreased thereafter as the charge had a tendency to sinter on recycling more than once.

Roasting studies on the ore collected from Rairangpur were completed. It was observed that 85 per cent of the vanadium was recovered by roasting at 900-950°C. with 12 per cent soda ash and soaking the mixture for 1 hour at the high temperature. The roasted ore was leached at elevated temperature and vanadium pentoxide precipitated from the solution containing 10 to 15 gm./litre of vanadium at *p*H 2. Further studies were aimed at establishing the number of stages required for counter-current leaching and solid to liquid ratio, duration of leaching. Systematic studies on the recovery and purification of sodium sulphate as a by-product would also be taken up.

19.0 Production of Magnesium Powder

Magnesium powder is prone to oxidation and during storage for longer periods an oxide coating is formed, thus rendering the powder unsuitable for pyro-technics. The investigation was taken up at the instance of the Defence Establishment with a view to upgrade the deteriorated magnesium powder analyzing 90-93 per cent metallic magnesium to 99·0 per cent. The conditioned powder analyzing metallic magnesium greater than 99 per cent can be used for pyro-technics.

The sample as received had a dark appearance and contained 93·33 per cent of metallic magnesium. The degree of oxidation increased with the particle size and the + 200 fraction contained 93·91 per cent magnesium while the −200 fraction contained only 85·74 per cent magnesium.

Preliminary studies were undertaken to preferentially remove the oxide layers by subjecting the powder to surface grinding tests but no marked improvement was observed. A number of chemical methods with organic and inorganic agents were investigated to remove the oxide layer and it was finally observed that with inorganic agents, the oxide layer could be preferentially removed. Systematic studies were taken up to arrive at optimum conditions for obtaining products of high magnesium contents. Specimens were taken from a magnesium ingot in the form of cubes and treated for 24 hours with solutions having varied concentrations of the inorganic compound. The results are tabulated below:

Expt No.	Percentage by Weight of the Inorganic Compound in 100 c.c. of Solution	Percentage Loss in Weight of the Specimens
1	5	0·6
2	10	0·5
3	15	0·3
4	20	0·1
5	25	0·09
6	30	0·08

Similar studies were made at 50°C. for periods of 6 hours and the results are tabulated below:

Expt No.	Percentage by Weight of the Inorganic compound in 100 c.c. of Solution	Percentage Loss in Weight of the Specimens
1	5	0·6
2	10	0·5
3	20	0·1

It was thus possible to preferentially remove oxide coating and the reconditioned powder analysed 99·7 per cent metallic magnesium. It was further observed that treated samples had greater stability to ordinary atmospheric conditions than the original oxidized sample received for processing. The two samples were left overnight in contact with water and the treated sample analysed 99·06 per cent magnesium while the original oxidized sample deteriorated from 93 per cent to 84·03 per cent metallic magnesium. The details of a plant processing 50 kg. of powder per batch were worked out.

20.0 Catalytic Distillation of Titanium from Titanium Bearing Alloys

Aluminium-titanium alloys containing 50 to 60 per cent titanium can be made by alumino-thermic reactions in presence of energizers. It was proposed to study the recovery of titanium from the aluminium-titanium alloys by treatment with $TiCl_4$ or $AlCl_3$. The reaction of $TiCl_4$ with titanium and the disproportionation of subhalides produced may be represented as follows:

$$\begin{aligned} Ti &+ TiCl_4 \rightleftharpoons 2TiCl_2 \\ TiCl_2 &+ TiCl_4 \rightleftharpoons 2TiCl_3 \\ 2TiCl_3 &+ Ti \rightleftharpoons 3TiCl_2 \\ 3TiCl_4 &+ Ti \rightleftharpoons 4TiCl_3 \end{aligned}$$

It was necessary to study initially the reaction between titanium and $TiCl_4$. The

TABLE 7—AMOUNT OF TURNINGS: 2 GM. TEMPERATURE: 600°C. DURATION: 1 HOUR

PARTIAL PRESSURE OF $TiCl_4$ IN THE ARGON STREAM IN MM. MERCURY	AMOUNT OF TITANIUM REMOVED	AMOUNT OF Ti WITH $TiCl_2$	AMOUNT OF Ti WITH $TiCl_3$ FORMED	OBSERVATION
16	0·0775	0·862	0·01245	Near the reaction zone a shiny metallic deposit was observed
24	0·089	0·08956	0·015	No shiny deposit
32	0·080	0·0958	0·0431	No shiny deposit
42	0·080	0·1006	0·0594	No shiny deposit

partial pressure of $TiCl_4$ at various temperatures required to suppress the formation of $TiCl_3$ was calculated from thermodynamic data. An assembly for the reaction of a gas mixture containing $TiCl_4$ at various vapour pressures was set up. The crude titanium tetrachloride was reflexed and distilled with Cu turnings, obtaining a clear and colourless titanium tetrachloride liquid. Equilibrium vapour pressures of $TiCl_4$ at various temperatures were determined by bubbling argon gas through $TiCl_4$ liquid for definite small periods of time and determining the amount of $TiCl_4$ in the gas stream by absorbing $TiCl_4$ in hydrochloric acid solutions. Table 7 gives some typical results obtained by reacting titanium turnings with titanium tetrachloride at various partial pressures of the latter using argon as a carrier gas.

Further work will be carried out at higher temperatures and longer durations.

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

Systematic studies were taken up on the Indian bauxites for their suitability for recovery of alumina by Bayer's process. A small autoclave was fabricated for operation at high temperatures and pressures.

The properties studied included relative grinding characteristics, mineralogical and chemical composition and various factors which affect the alumina extraction, viz., particle size, concentration of alkali, molar ratio of alkali to Al_2O_3, pressure and settling rate of red mud. Samples were collected from twelve different deposits in India. An electrically heated autoclave was fabricated which gave satisfactory service. A sample of bauxite from Kutch was taken up for investigation. As the Masletinskii method of determination of Al_2O_3 mineral was found unsatisfactory, the usual plant method for estimation of gibbsite in bauxite was followed and the sample was found to contain 85 per cent gibbsite. Preliminary extraction tests were carried out both at atmospheric pressure and at 105 lb./sq. in. In the former case, it gave 85 to 90 per cent, in the latter about 95 per cent Al_2O_3 recovery. Further experiments were in progress to determine the optimum conditions for the maximum extraction of Al_2O_3 from this bauxite sample.

22.0 Chlorination of Vanadium Bearing Titaniferous Magnetite Ores

The removal of vanadium bearing titaniferous magnetites by chlorination offered the advantage that most of the vanadium could be removed at comparatively low operating temperatures of 150-300°C. HCl gas was reported to selectively remove vanadium. Systematic studies on the volatilization of vanadium from the ores were carried out; it was observed that more than 80 per cent of the vanadium could be volatilized at as low a temperature as 190°C. HCl gas generated by treating the HCl with strong H_2SO_4 was passed over the vanadium ore contained in a horizontal tubular furnace. It was observed that the recoveries improved with decrease in particle size up to 72 mesh B.S.S. sieve, and thereafter no improvement was observed. The effect of temperature on the chlorination rate is given in the following table. It would be observed that maximum recoveries were obtained at 200°C.

TABLE 8—EFFECT OF TEMPERATURE ON THE RATE OF REACTION. RATE OF HCl GAS: 280 C.C. PER MINUTE. DURATION: 2 HOURS

TEMPERATURE	% VANADIUM REMOVED
150	44
170	46
190	54
200	64
210	56
250	44
300	40
350	37
400	33

No marked improvement was observed on chlorinating for longer period at 200°C., though at 190°C. the percentage of vanadium chlorinated increased to 81·8 after chlorinating for 4 hours. Further experiments were carried out to study the effect of added salts on the rate of volatilization of vanadium and iron in the ore. Chlorination experiments with changes in the partial pressure of HCl gas by introducing an inert gas and by introducing hydrogen alone with HCl gas were planned including trials with chlorine and carbon or hydrogen in a vertical furnace to increase the efficiency of the chlorination agent.

23.0 Preparation of 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 are met entirely by import chiefly from Denmark and England. In view of the expanding demand of this material in India, need for its synthetic preparation is 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 at the first instance. Extensive work on laboratory and pilot plant scales on beneficiation of fluorspar was conducted in the laboratory.

With a view to prepare synthetic cryolite, beneficiated metallurgical grade fluorspar was used as the starting material.

The method consisted in leaching fluorspar with sulphuric acid and boric acid in the presence of sodium sulphate. The reactions in the leaching and precipitation are represented by the following equations:

$$2CaF_2+H_2BO_3+2H_2SO_4+H_2O = HBF_4+2CaSO_4\cdot 2H_2O \quad (1)$$

$$3HBF_4+Al_2O_3\cdot 3H_2O+3Na_2CO_3 = 2AlF_3\cdot 3NaF+3H_3BO_3+3CO_2 \quad (2)$$

Silica was not attacked by fluoboric acid and thus remains in the insoluble residue $CaSO_4\cdot 2H_2O$. Some calcium fluoborate which was soluble was also formed according to the following reactions:

$$4CaF_2+2H_3BO_3+3H_2SO_4 = Ca(BF_4)_2+3CaSO_4\cdot 2H_2O \quad (3)$$

It was, however, necessary to prepare pure fluoboric acid solution free from calcium ions in order that a relatively pure cryolite could be made. Use of an excess sulphuric acid could furnish sulphate ions to suppress the solubility of calcium ions; but free sulphuric acid in the fluoboric acid solution was undesirable because a cyclic process for the production of cryolite would not function properly. Consequently, it was necessary to employ sodium sulphate as a constant component to supply sulphate ions, the negative radical of sodium sulphate. Sodium sulphate was selected because sodium ions were common in the process liquors.

The digest liquor free from sulphuric acid and calcium ions was taken and precipitation was carried out with sodium carbonate and hydrated alumina according to equation (2). Hydrated alumina was first added and digested until dissolved. Sodium carbonate was then slowly added and the slurry digested for some time so as to complete the reaction. Cryolite was filtered, washed and dried. The spent liquor was used again to utilize its boric acid and sodium sulphate contents.

Preliminary leaching experiments with synthetic leach liquors with varying amounts of sulphuric acid, boric acid and sodium sulphate at different temperatures and for different intervals of time were carried out with 100 gm. of fluorspar. Excess sulphuric acid was always found in the digest liquors even if the sulphuric acid used was less than theoretical. Theoretical amounts of sulphuric acid were used in all the experiments. Boric acid was always used in excess to the quantity theoretically required. 100 to 120 gm./litre of boric acid gave good leaching efficiency. 50 gm./litre of sodium sulphate was required to keep the calcium content of the digest liquors at a minimum and was consequently used for all experiments. Time and temperature were complementary to each other. At 95°C. leaching for two hours gave leaching efficiencies from 95 to 98 per cent. After standardizing the leaching conditions, trials for precipitation of cryolite were carried out with varying amounts of hydrated alumina and sodium carbonate for different times and temperatures. Following conditions were found suitable in the precipitation.

When the ratio of hydrated alumina to sodium carbonate during precipitation was kept 1: 1, the spent liquor was found to give better leaching. Material data of a cyclic experiment up to 7 cycles is given in Table 9 where the ratio is kept 1: 1.

TABLE 9 — DATA OF CYCLIC EXPERIMENTS

Volume of the solution = 500 *c.c.*
Boric acid content = 55 *gm.*
Sodium sulphate = 25 *gm.*

No. of Cyclic Expts	Amount of Fluorspar gm.	H_2SO_4 Added	Boric Acid Added gm.	Residual H_2SO_4 gm.	Cryolite Obtained gm.
1	100	Theoretical	55	3·42	60
2	100	,,	nil	14·37	66
3	100	,,	nil	28·4	48
4	100	,,	nil	46·7	45
Soln purified					
5	100	,,	8	26·45	45
6	100	,,	nil	30·20	48
7	100	,,	nil	40·29	46

Boric acid in spent liquor = 46 gm.
Loss of boric acid = 15 gm.
Total fluorine in 700 gm. fluorspar = 287·4 gm.
Fluorine leached = 213·7 gm.
Fluorine recovered in cryolite = 178·80 gm.
Overall recovery = 62·2%

Further experiments were under progress to decrease the loss of H_3BO_3 to improve the leaching efficiency, to study the factors which affect efficiency and to thereby improve the overall fluorine recovery. One kilogram of cryolite sample was prepared and its suitability as an electrolyte in the aluminium reduction cell was determined.

24.0 Preparation of Anhydrous Magnesium Chloride

Hindustan Aircraft Limited require approximately 50-100 lb. anhydrous magnesium chloride monthly to be used as flux material for melting, casting and refining of magnesium base alloys. This work was taken up with a view to examine the possibilities of preparing anhydrous magnesium chloride to be used as fluxing material.

Preparation of dihydrate magnesium chloride ($MgCl_2$, $2H_2O$) can be made without much hydrolysis and sintering. In the preparation of anhydrous magnesium chloride from dihydrate by dehydration, the decomposition of the salt into hydrochloric acid, magnesium oxide and oxychloride could not be checked unless special precautions were taken. Apart from heating in vacuo, which had not yet been carried out on an industrial scale on account of difficulties connected with apparatus and probable high cost, the methods mainly adopted for the final drying were as follows:

a) Dehydration in presence of circulating dry hydrochloric acid atmosphere.
b) Dehydration in presence of ammonium chloride.
c) Chlorinating magnesium oxide mixed with carbon into anhydrous magnesium chloride.

Dehydration in presence of ammonium chloride was attempted in the course of the work on drying of hydrated magnesium chloride for the production of magnesium metal. Further attempts were made for dehydrating magnesium chloride hydrate by using different molar ratios of ammonium chloride to magnesium chloride hydrate varying from 1: 1 to 1: 16. The results obtained with different ratios of ammonium chloride and magnesium chloride hydrate are given below:

TABLE 10 — RESULTS WITH DIFFERENT RATIOS OF NH_4Cl AND $MgCl_2$

MOLAR RATIO OF NH_4Cl TO $MgCl_2$		MgO %	NH_4Cl %	$MgCl_2$ %	H_2O %
Eq. parts by weight	(a) (b)	0·36	65·00	29·00	5·64
Eq. mole by weight	(a) (b)	0·38	26·85	55·42	17·35
1: 1·5	(a)	0·42	17·63	63·50	18·45
	(b)	0·93	14·20	66·82	18·05
1: 2	(a)	0·67	13·45	66·06	19·82
	(b)	1·06	12·92	68·12	17·90
1: 4	(a)	0·88	10·16	66·96	22·00
	(b)	1·86	8·11	69·56	20·47
1: 6	(a)	1·68	7·24	70·24	20·84
	(b)	2·31	4·82	73·32	19·55
1: 8	(a)	2·12	5·53	74·15	18·20
	(b)	2·67	3·88	75·35	18·10
1: 10	(a)	2·34	4·22	75·59	17·87
	(b)	3·35	2·94	76·47	17·24
1: 12	(a)	2·62	4·00	75·72	17·66
	(b)	4·16	2·37	75·62	17·85
1: 14	(a)	6·12	3·50	73·25	17·13
	(b)	7·15	1·76	74·52	14·57
1: 16	(a)	7·63	3·10	72·39	16·98
	(b)	11·96	1·45	74·96	11·63

The mixture was prepared and boiled with sufficient water for a period of 4 to 6 hours and cooled, the crystallized lumps were kept at a temperature of 76°C. for 24 hours and then at 110°C. for another 24 hours in an ordinary air oven, to remove absorbed moisture. The dry salts were then crushed to —16 mesh and dried at various ranges of temperature for definite periods in a drier fitted with hot air circulation and mechanical stirrer during the drying operation. The maximum hot air temperature of 350°C. and salt temperature of 178-182°C. could be attained in this oven. The results of analysis after 2 days of continuous drying are given under (a). Under (b), results are reported for similar experiments whilst holding the salt at the final drying temperature for a longer period. Possibilities of preparing anhydrous magnesium salt by drying under reduced pressure was also considered. As vacuum drying unit with all accessories was not readily available indigenously, the necessary equipments for bench scale operation were assembled.

25.0 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 also done 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 conducted to determine the low temperature impact property, high temperature short time tensile strength and ageing characteristics of the steel.

During the period under review, investigations were carried out with low-carbon chromium - manganese - nitrogen stainless steels at 17 per cent chromium level to throw light on the phase characteristics, structural stability, corrosion resistance, mechanical and welding properties of compositions having manganese in the range of 2-12 per cent and nitrogen 0·2-0·5 per cent. It was observed that whilst nitrogen and carbon contents in these steels determined the amount of delta ferrite, the manganese content affected the stability of the austenite and controlled its transformation to martensite. As the manganese content was reduced to a level of 8 per cent, these steels showed a martensitic structure although no noticeable increase in hardness was obtained. When the manganese content was further reduced to the order of 4 per cent or less, large quanti-

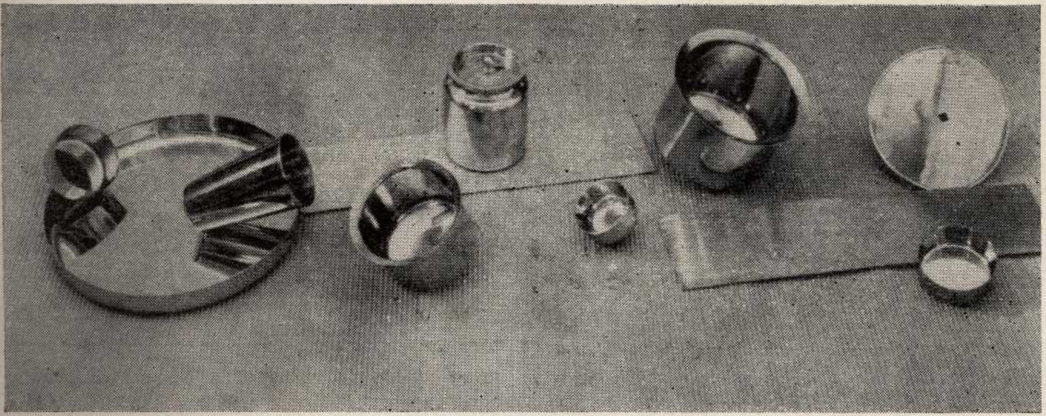


FIG. 12 — MATERIALS FABRICATED OUT OF THE NICKEL-FREE AUSTENITIC STAINLESS STEEL DEVELOPED AT THE NATIONAL METALLURGICAL LABORATORY

ties of martensite were formed with accompanying increase in hardness. Cold working trials showed that a minimum of 17 per cent manganese was required in these steels to render their austenitic structure completely stable and a lower manganese content would lead to structural instability resulting in the transformation of austenite to martensite on cold working. The ferrite content of these steels was determined. It was possible to derive an equation to correlate the δ ferrite content of the steel with its chemical composition. The relationship is expressed as follows:

$$\% \delta \text{ ferrite} = 95 \{0{\cdot}12 (\% \text{Cr} - 13{\cdot}8) - (\% \text{C} + \% \text{N})\}$$

Corrosion tests showed that the manganese as well as δ ferrite contents did not materially affect the corrosion resistance of the steels in boiling 65 per cent nitric acid. Results of salt spray test, however, showed that manganese contents above 8 per cent improved resistance to corrosion. Additions of small amounts of copper and nickel also improved corrosion resistance to salt spray.

Results of deep drawing tests carried out in Swift deep drawing machine indicated a good drawability for the nitrogen containing austenitic 17: 17 Cr: Mn steels, although the drawing pressure required was considerably greater than that required for 18: 8 Cr: Ni stainless steels.

Attainment of high hardness in steels containing about 4 per cent manganese led to the work on 17: 8 Cr: Mn and 17: 6 Cr: Mn steels for the development of high tensile substitute stainless steels. Experiments were carried out to determine physical properties after different cold work and heat treatment cycles. The steels were tested under the following conditions: (1) Liquid air quenching, (ii) 30 per cent cold reduction, (iii) 30 per cent cold reduction followed by liquid air quenching (iv) tempering at high temperatures. Results of these trials showed that an increase of about 50 per cent in the tensile strength could be achieved by 30 per cent cold reduction. Sub-zero treatment, however, did not prove effective in improving the strength characteristics. Tempering treatment at 750°C. gave excellent mechanical properties for some of the steels and a tensile strength as high as 92 tons/sq. in. with an elongation of 12 per cent could be obtained. Isothermal treatment carried out on one of the 17: 17 Cr: Mn steels at 400°C. showed no change in strength and elongation even after 312 hours of treatment. Treatments at higher temperatures did not affect the strength but seriously impaired the ductility. Welding tests with these steels by the argon arc process showed good results. Acidified copper sulphate tests showed these steels to be prone to " weld decay " when the carbon content was above 0·03 per cent. Steels having carbon contents lower than this value did not show propensity to " weld decay ". Presence of δ ferrite in these steels considerably decreased weld decay but did not completely eliminate it when the carbon content was over 0·03 per cent.

25.1 Ageing Characteristics at Elevated Temperature of Ni-free Cr-Mn-N Austenitic Stainless Steel

(i) *Hardness and Corrosion Studies* — In continuation of the earlier work done, further studies were carried out on the ageing characteristics at high temperature of nickel-free chromium-manganese-nitrogen austenitic stainless steels. After ageing at the holding temperatures for a certain period, the specimens were quenched in water at room temperature and then subjected to metallographic examinations, hardness tests, and corrosion tests in different media such as boiling nitric acid, citric acid, vinegar, lime juice and salt spray. The test specimens for experiments were taken from the stainless steel sheets of heat Nos. B_2, B_4, B_6, A160, A171, and A180, as given in Table 11. The test specimens were given a solution-treatment (W/Q, 1050°C.) before ageing. The

TABLE 11 — SHOWING CHEMICAL COMPOSITION AND AGEING TREATMENT

HEAT NO.	COMPOSITION, %					AGEING TEMP., °C.	AGEING PERIOD hr
	Cr	Mn	N	C	Si		
B_4	21·56	18·47	0·80	0·06	0·37	300	1000
B_4	—	—	—	—	—	400	1000
B_4	—	—	—	—	—	500	1000
B_6	18·25	17·70	0·53	0·08	0·27	300	1000
B_6	—	—	—	—	—	400	1000
B_6	—	—	—	—	—	500	1000
A160	17·17	9·8	0·40	0·02	0·53	400	1000
A160	—	—	—	—	—	500	1000
A160	—	—	—	—	—	600	1000
A171	16·65	10·7	0·20	0·03	0·38	400	1000
A171	—	—	—	—	—	500	1000
A171	—	—	—	—	—	600	1000
A175	17·91	5·93	0·32	0·05	0·43	400	1000
A175	—	—	—	—	—	500	1000
A175	—	—	—	—	—	600	1000
A180	17·2	8·05	0·26	0·04	0·48	400	1000
A180	—	—	—	—	—	500	1000
A180	—	—	—	—	—	600	1000

trials completed during the period under review are given in Table 11.

Corrosion tests in boiling 65 per cent nitric acid showed that aged specimens were susceptible to maximum corrosion attack after ageing at 600-700°C. Above these ageing temperatures, rate of corrosion decreased, despite increase in the precipitated phase at higher ageing temperatures which may be attributed to diffusion of chromium at higher ageing temperatures into chromium-depleted areas. Figure 14 shows the data on corrosion at different temperatures for varying periods of ageing. Corrosion attack in vinegar, lime juice, citric acid and salt-spray was less and the results were satisfactory. Hardness measurements of the specimens showed slight change in their values. In general, there was a tendency towards increased values with ageing as shown in Figure 15.

Micro-examination of aged specimens showed that ageing at 300°C. or 400°C. did not bring about any marked precipitation even after prolonged periods. With increase in ageing time and temperature, the precipitated phase increased which appeared to be followed by nucleation and growth process. The precipitated phase formed mostly along the grain boundaries as dark etching nodules having lamellae. The nodules increased in size with time and temperatures. It was observed that the inter-lamellar spacing of the lamellae in the nodules was smaller at lower temperatures but increased at higher

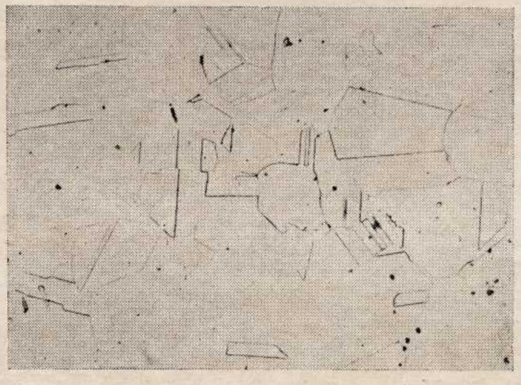


FIG. 13 — PHOTOMICROGRAPH OF NICKEL-FREE AUSTENITIC STAINLESS STEEL AGED AT 300°C. FOR 1000 HRS SHOWING NO PRECIPITATION. × 450

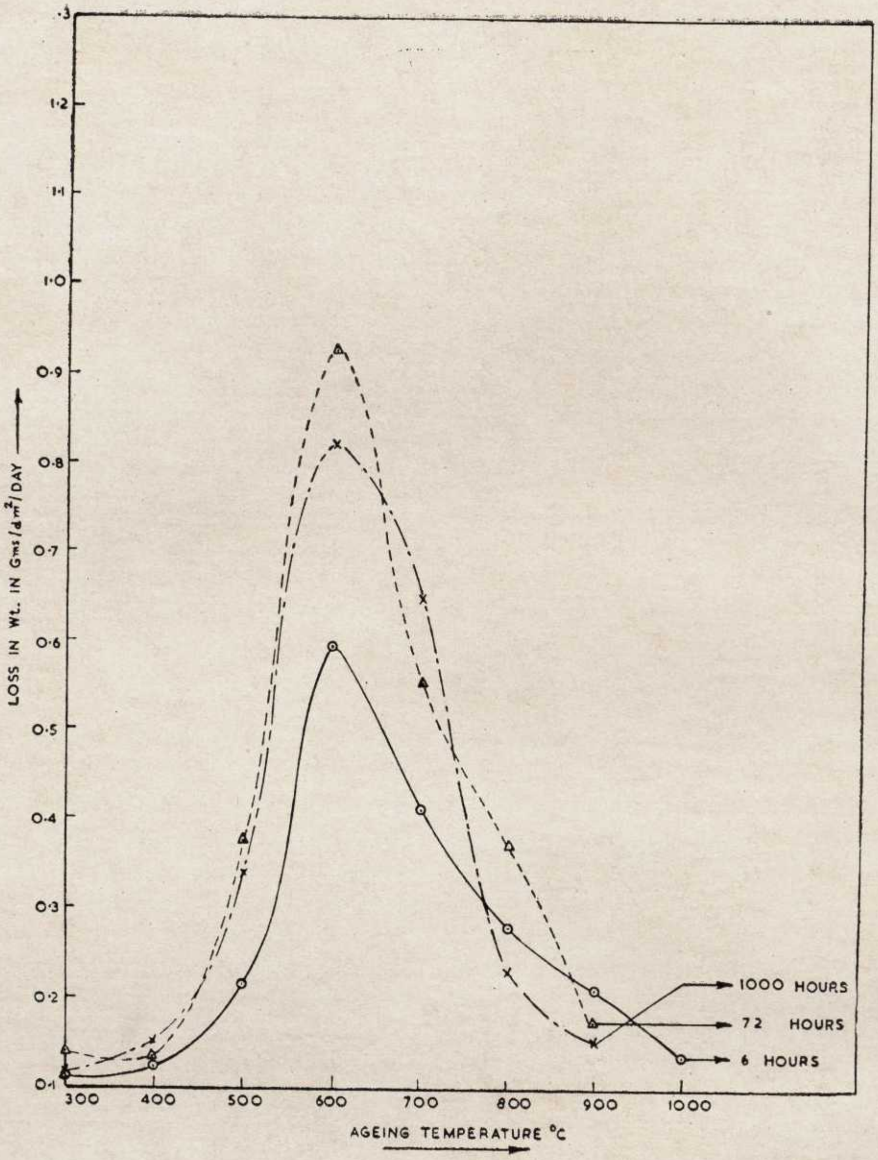


FIG. 14 — CORROSION TEST IN 65 PER CENT BOILING NITRIC ACID OF Cr-Mn-N STAINLESS STEEL (Cr, 21·5; Mn, 17·8; N, 0·75 PER CENT)

ageing temperatures. It was further observed that high nitrogen contents promoted more precipitation. Further work on these lines is in progress.

(ii) *X-ray Diffraction Studies* — During the period under review, investigations related to the study of cold working in relation to the stability of the γ-phase were conducted. Steel No: B_6 only showed duplex structure (i.e. both α and γ phases) after 30 per cent cold reduction. Steels Nos.: B_2, B_3, B_4 and B_5 showed single phase structure (only γ-phase) even after 30 per cent cold reduction. Further studies related to the effects of still heavier reductions and on the effect of subsequent ageing at high temperatures were carried out. In the powder X-ray diffraction photographs (both solution treated and aged samples) of some of these steels (B_5, B_4 and B_9), presence of some extra lines, apart from ferrite, austenite or Cr_2N type phase, was detected. In some cases, the lines were very faint and in most cases complete spectrograph for a particular phase could not be accounted for.

Studies were also continued on the estimation of the relative concentrations of iron, chromium and manganese in the lattice of the precipitated phase, i.e., Cr_2N type, for different temperatures and periods of ageing. Chemical analyses of the extracted residues were also undertaken. As very small quantities of the extracted residues are available, it was not possible to employ wet conventional methods of analyses. Attempts were made to estimate the relative concentration of these elements (i.e., Fe, Cr and Mn) by X-ray fluorescence technique.

TABLE 12 — X-RAY DIFFRACTION DATA OF THE PREPARED ALLOY FOR COMPARISON (SIGMA PHASE)

d-Å.	Intensity (visual)	(A.S.T.M.) Values for FeCr Phase	
		d-Å.	I/Io
2·580	Faint		
2·374	V.F.		
2·267	F.	2·261	100
2·125	V.S.	2·128	100
2·071	F.	2·063	80
2·023	S.	2·015	60
1·975	M.	1·964	80
1·932	V.S.	1·928	100
1·885	M.	1·877	80
1·771	F.	1·755	10
1·500	V.F.		
1·435	V.F.		
1·391	V.F.	1·390	(1)
		1·327	(1)
1·334	Faint		
1·271	F.	1·258	(10)
1·245	M.	1·244	(10)
1·210	M.	1·209	(40)
1·170	V.S.	1·169	(40)
1·145	V.F.		
1·069	M.	1·138	(40)
1·040	M.	1·004	(20)
1·013	M.		
0·990	M.	0.991	(5)

F.: Faint; S.: Strong; M.: Medium.

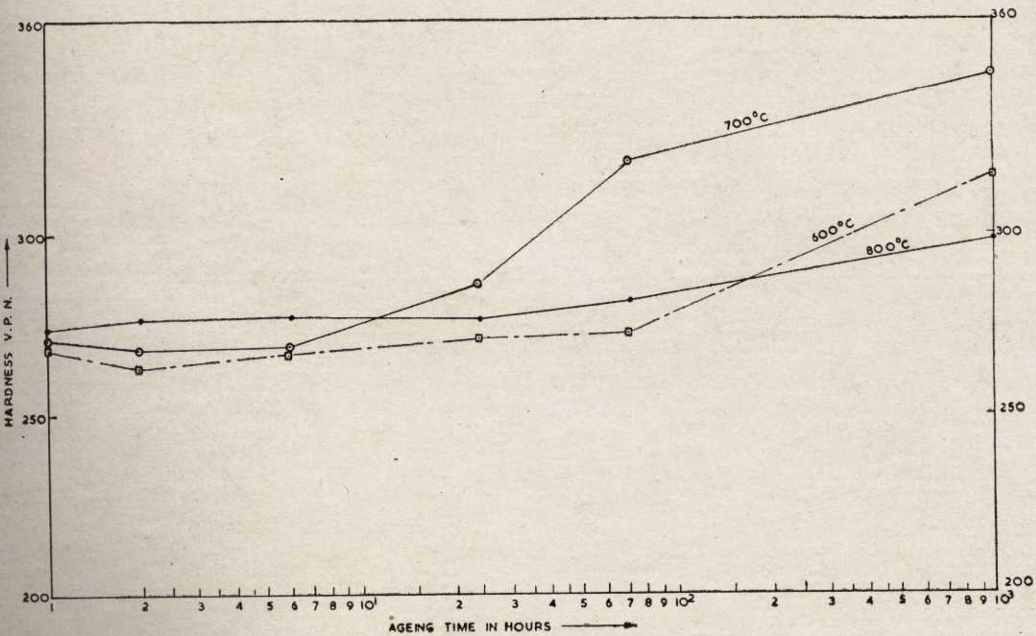


Fig. 15 — Precipitation hardening characteristics of Cr-Mn-N stainless steel (Cr, 21·5; Mn, 17·8; N, 0·75 per cent)

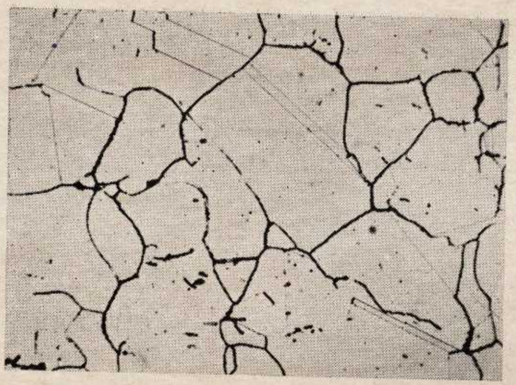


FIG. 16 — PHOTOMICROGRAPH OF NICKEL-FREE AUSTENITIC STAINLESS STEEL AGED AT 500°C. FOR 1000 HRS SHOWING GRAIN BOUNDARY PRECIPITATION. × 450

26.0 Development of Indigenous Alloy Steels

The objective of this project is based on the development of indigenous substitute alloy steels eliminating as far as possible those alloying elements, such as nickel, molybdenum, tungsten, cobalt, etc., resources of which do not exist in India; such substitute alloy steels through judicious combinations of indigenous alloying elements and optimum heat-treatment should conform to the requirements of physical properties and specific service performance characteristics of standard alloy steels. The project covers the important fields of formulating T.T.T. C.C.T. curves, hardenability studies *vis-à-vis* ruling sections and optimum heat-treatment cycles and fabrication techniques.

Alloy steels of compositions given in Table 13 were made in the 0·8-ton electric arc furnace. These steels contained Si, Mn, Cr and V as the chief alloying elements, resources of which are abundant in India. The ingots obtained were forged for studying their mechanical and physical properties, inclusion studies and specific metallurgical characteristics.

26.1 Tool Steels

Work was continued on the development of tool steels based on indigenous alloying elements as far as practicable. The possibility of using nitrogen as an alloying element was also under investigation.

25 heats of 20 lb. each, of tool steels of the following nominal composition were made in 25 KW. high frequency furnace.

C	...	0·7-0·1	per cent
Cr	...	4-5	,,
W	...	6-7	,,
V	...	1-1·5	,,
N	...	0·2-0·25	,,

'N' was introduced by using nitrided ferrochrome. The maximum 'N' obtained in the steels was 0·1 per cent. To some of these steels additions of aluminium, titanium and columbium were made. These tool steels after forging and annealing were hardened and double tempered at various temperatures and their hardness values were evaluated. Out of these 25 heats, four compositions appeared promising, which showed a hardness of about 800 V.P.N. after double tempering. Systematic development work was underway on the more promising compositions to define appropriate hardening and tempering cycles. The steels were hardened by heating in salt bath to 1150°, 1200° and 1250°C., followed by single and double tempering treatments at three different temperatures. Table 14 shows the hardness of

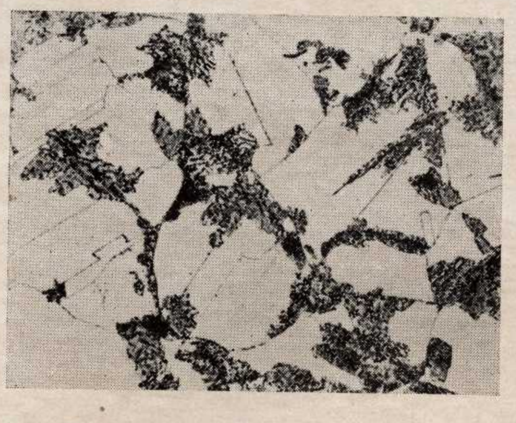


FIG. 17 — PHOTOMICROGRAPH OF NICKEL-FREE AUSTENITIC STAINLESS STEEL AGED AT 600°C. FOR 500 HRS SHOWING SOME PRECIPITATION. × 450

TABLE 13 — COMPOSITION OF ALLOY STEELS MADE IN 0·8-TON ELECTRIC ARC FURNACE

		C %	Mn %	Cr %	V %	Si %
1. Mn-Cr-V Steel	(a)	0·25-0·30	1·0-1·5	0·6-0·9	0·1-0·15	—
	(b)	0·35-0·45	0·5-1·0	1·5-1·8	0·1-0·15	—
2. Mn-Mo steel	(a)	0·30-0·40	1·3-1·7	0·2-0·3	—	—
3. Si-Mn steel	(a)	0·32-0·40	1·1-1·4	—	—	1·1-1·4
4. Si-Cr-V steel	(a)	0·48-0·58	0·6-0·9	0·7-1·0	0·1-0·2	1·2-1·5
5. Mn-V steel	(a)	0·38-0·48	1·6-1·9	—	0·1-0·15	—
6. Si steels	(a)	0·40-0·50	0·4-0·8	—	—	1·6-2·1
	(b)	0·50-0·60	0·4-0·8	—	—	1·6-2·1

the four steels in as cast, forged and annealed conditions. Tables 15, 16 and 17 show the hardness after hardening and tempering at three different temperatures, viz. 550°, 600° and 650°C.

TABLE 14 — HARDNESS OF TOOL STEELS IN AS-CAST, FORGED AND ANNEALED CONDITION

T.S. No.	Hardness in the Ingot Condition V.P.H.N.	Hardness in the Forged Condition V.P.H.N.	Hardness in the Annealed Condition V.P.H.N.
11	543	330	365
12	448	330	367
18	680	270	315
19	644	291	368

26.2 Iron-Aluminium Alloys

The project on iron-aluminium alloys is based on the development of heat-resistant alloys for high temperature applications for appropriate service conditions and requirements.

Study of the 'Binary Iron-Aluminium Alloy System' reported earlier had shown an inherent tendency for grain coarseness which resulted in inadequate mechanical properties although highly favourable oxidation resistance at elevated temperatures was observed.

Grain-size Refinement — For preliminary assessment of the efficacy of Ti, Zr, V, B and Cr for grain refinement, heats containing an arbitrary small amount of each of these elements were made. Results obtained thereon are given in Table 18. It was noticed that additions of grain refining agents had resulted in increase of the as-cast hardness to varying degrees. Tensile tests on the as-rolled sheet samples GR_1 and GR_3 — the only heats which could be effectively rolled — resulted in fracture outside the gauge length, subsequent examination of which showed unsound material.

It may be pointed out that introduction of titanium, zirconium, vanadium and boron was made through their respective ferro-alloys. Purity of the ferro-alloys used was somewhat substandard and impurities, such

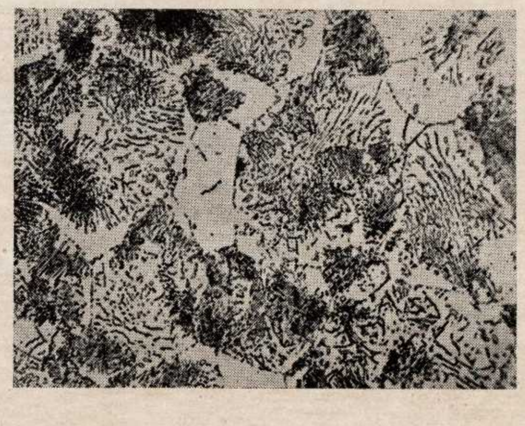


FIG. 18 — PHOTOMICROGRAPH OF NICKEL-FREE AUSTENITIC STAINLESS STEEL AGED AT 800°C. FOR 72 HRS SHOWING PROFUSE PRECIPITATION. × 450

TABLE 15 — HARDNESS OF TOOL STEELS AFTER HARDENING FROM 1150°C. AND TEMPERING AT 550°, 600° and 650°C.

T.S. No.	Hardening Temp. °C.	Hardness after Hardening V.P.H.N.	1st tempering Temp. (1 hr.) °C.	Hardness Values V.P.H.N.	2nd Tempering Temp. (1 hr.) °C.	Hardness Values after 2nd Tempering V.P.H.N.
11	1150	876	550	769	550	804
12	1150	842	550	829	550	804
13	1150	769	550	730	550	842
14	1150	720	550	648	550	829
15	1150	876	600	571	600	680
12	1150	842	600	710	600	746
18	1150	769	600	752	600	780
19	1150	720	600	780	600	850
11	1150	876	650	661	650	635
12	1150	842	650	626	650	680
18	1150	769	650	533	650	557
19	1150	720	650	639	650	786

TABLE 16 — HARDNESS OF TOOL STEELS AFTER HARDENING FROM 1200°C. AND TEMPERING AT 550°, 600° AND 650°C.

T.S. No.	Hardening Temp. °C.	Hardness after Hardening V.P.H.N.	1st Tempering Temp. (1 hr.) °C.	Hardness Values after 1st Tempering V.P.H.N.	2nd Tempering Temp. (1 hr.) °C.	Hardness Values after 2nd Tempering V.P.H.N.
11	1200	934	550	890	550	856
12	1200	804	550	883	550	856
18	1200	912	550	890	550	876
19	1200	836	550	626	550	798
11	1200	934	600	648	600	746
12	1200	804	600	720	600	823
18	1200	912	600	648	600	752
19	1200	836	600	741	600	890
11	1200	934	650	590	650	720
12	1200	804	650	720	650	746
18	1200	912	650	618	650	671
19	1200	836	650	730	650	786

TABLE 17 — HARDNESS OF TOOL STEELS AFTER HARDENING FROM 1250°C. AND TEMPERING AT 550°, 600° AND 650°C.

T.S. No.	Hardening Temp. °C.	Hardness after Hardening V.P.H.N.	1st Tempering Temp. (1 hr.) °C.	Hardness Values after 1st Tempering V.P.H.N.	2nd tempering temp. (1 hr.) °C.	Hardness Values after 2nd Tempering V.P.H.N.
11	1250	842	550	798	550	680
12	1250	836	550	862	550	763
18	1250	862	550	856	550	568
19	1250	720	550	804	550	775
11	1250	842	600	810	600	752
12	1250	836	600	786	600	804
18	1250	862	600	700	600	736
19	1250	720	600	810	600	823
11	1250	842	650	675	650	752
12	1250	836	650	757	650	792
18	1250	862	650	646	650	725
19	1250	720	650	763	650	883

TABLE 18 — RESULTS OF TESTS CONDUCTED ON VARIOUS HEATS

HEAT No.	Al %	GRAIN REFINING ELEMENT %	AS-CAST HARDNESS V.P.H.N.	THE INGOT 1·5″ SECTION	FORGING 1150-900°C. TO BILLET ½″	HOT ROLLING TO 0·1″ SHEET	TENSILE TEST	
							Maximum stress tons/in².	Elongation %
GR 1	9·61	0·51 Ti	235	Sound	Forgeable	Satisfactory	38·19	Broke outside G.L.
GR 2	9·10	0·39 Zr	263	do	do	Cracked in final stages of rolling	Too hard and brittle for making tensile test piece	
GR 3	9·86	0·30 V	241	do	do	Satisfactory	32·82	Broke outside G.L.
GR 4	11·34	0·08 B	244	do	Cracked due to pipe	Could not be worked further		
GR 5	9·69	0·06 Ce	239	Had a cold shut. L.S. showed large shrinkage pipe	Could not be worked further			

as phosphorus, sulphur, hydrogen and nitrogen etc. were responsible for observed embrittlement of these iron-aluminium alloys which however, were found to possess requisite ductility in their binary compositions. Further work employing purer carbon free master alloys obtained by alumino-thermic technique, for grain-refinement was in progress.

27.0 Development of Low-alloy High Tensile Structural Steel

This broad-based investigation was taken up with the objective of developing suitable low alloy structural steels possessing adequate mechanical properties, hot and cold working characteristics, optimum corrosion resistance and favourable welding characteristics. The main aim of the project was to evolve suitable compositions for low alloy high tensile steel based essentially on indigenous alloying elements.

In the first phase of the work, experimental heats were made for standardizing the conditions for their laboratory scale production, with slight modifications in the composition in relation to those currently employed. Main alloying elements of the series investigated were manganese, chromium, silicon and copper. In the next series, a few basic composition were selected to study the effects of vanadium and titanium which harden the ferrite as well as improve the grain structure. Additions of aluminium and zirconium were also made with the objective of getting finer grain size. In order to enable extensive series of tests as well as to study the behaviour under different rates of cooling, bigger heats were made. In another series, the effects of additions of varying amounts of silicon were studied. In this series, basic composition has been kept

TABLE 19 — COMPOSITION OF HEATS

Heat No.	Analysed Chemical Composition, %									Total Charge kg.
	C	Mn	Cr	Si	P	Cu	V	Ti	Al	
35/1	0·18	2·21	0·99	0·46	—	0·34	—	0·06	—	20
35/2	0·07	1·32	0·78	0·15	—	0·31	—	Trace	—	20
37	0·06	1·14	—	0·20	0·11	0·53	0·14	Trace	—	20
38	0·03	0·90	—	1·85	0·09	0·51	0·05	Trace	—	20
37/1	0·05	0·69	—	1·00	0·07	0·44	—	0·02	—	10
37/2	0·09	1·36	—	1·02	0·07	0·42	—	0·08	—	10
37/3	0·07	1·34	—	0·62	0·07	0·44	trace	0·06	—	10
38/1	0·05	1·26	—	1·57	0·02	0·49	0·08	Trace	—	10
38/2	0·07	1·40	—	0·99	0·04	0·44	0·118	—	—	10
38/3	0·04	1·36	—	0·50	0·06	0·46	0·115	—	—	10
39	0·05	1·39	—	1·68	0·07	0·44	—	—	0·02	10
39/1	0·05	1·55	—	0·92	0·07	0·43	—	—	0·02	10
39/2	0·07	1·49	—	0·60	0·07	0·43	—	—	0·05	10
32/1	0·14	1·52	0·97	0·065	—	0·25	—	0·03	—	10
32/2	0·13	2·21	0·13	0·27	—	0·30	0·16	—	—	10
34	0·15	1·84	1·04	0·21	—	0·33	—	—	—	10

within the following limits: C, 0·08-0·1; Mn, 1-1·4; Cu, 0·4-0·45; B, 0·06-0·08; and V or Ti or Al, 0·05 to 0·1 per cent. Chromium was eliminated from this series as it introduced difficulties in welding. In order to improve corrosion resistance, following the elimination of chromium, phosphorus of the order of 0·1 per cent along with copper was used in these steels.

All the heats given in Table 19 were made in the high frequency induction furnace. Heat Nos. 35-38 were made with a total charge of 20 kg. and a single ingot was made out of the whole charge. The rest were 10 kg. heats and two ingots, each of 5 kg., were made out of the charge. The ingots were hot worked from 1150°C. in the usual way. Normalizing treatments were given to all the sections before preparation of the test pieces. The tensile test results of the heats along with their hardness values obtained after different heat treatments and bend test results are given in Table 20. Weldability test results and Charpy impact values at room temperature and subzero temperatures of some of the steels are given in the same table.

Heat Nos. 32/1 to 35/2 were made to repeat melts which gave high tensile values ranging from 50 to 60 tons/sq. in. with good elongation percentage. Bend test results were also satisfactory. Slight air hardening tendency was noticed in them as specimens made from thinner sections gave higher V.P.H.N. values. None of the steels showed any noticeable yield point. 0·1 per cent proof stress values were determined to be in the range of 26-30 tons/sq. in. V-notch Charpy impact test results showed that the transition temperature ranges of these steels were lower than 50°C. and the impact strength at subzero temperatures and room temperature were fairly high. Heat Nos. 32/1 and 35/1 showed fairly good welding characteristics. Weldability test was carried out according to Reeves fillet test method (miniature).

Heat Nos. 37 to 39/2 were made to study the effects of silicon. Nos. 37, 38 and 39 represented vanadium, titanium and aluminium groups respectively. It would be seen from Table 20 that all the steels gave maximum strength of the order of 40 tons per square inch with remarkably high elongation values. The hardness values also showed

TABLE 20 — TEST RESULTS OF HEATS

HEAT No.	TENSILE TEST RESULTS				HARDNESS VALUES (V.P.H.N.) AFTER				BEND TEST RESULTS			WELDABILITY TEST
	Max. S. T.S.I.	0·1% P. stress T.S.I.	R.A. %	Elong. %	Annealing	Normalizing	Oil quenching	Water quenching	Thickness	No. of 180 reversed bend	Remarks on 180 flat bend	
35/1	60·45	35·88	59·00	20·0	—	258	325	343	—	at 2·5	—	May be considered weldable
35/2	58·51	32·98	41·59	21·8	147	253	359	386	—	—	—	Not weldable
37	37·26	24·68	62·5	34·0	170	173	177	182	—	—	—	Weldable
38	41·76	23·4	60·0	31·0	177	180	186	191	—	—	—	
37/1	43·43	22·48	65·9	32·8	130	137	139	153	0·075	3·0	Withstood	
37/2	41·4	24·31	—	25·0	162	185	240	269	0·090	2·0	Failed	
37/3	36·4	25·75	69·3	36·0	—	—	—	—	0·078	3·0	Withstood	
38/1	42·12	20·50	66·2	36·0	160	182	210	215	0·081	3·0	—	
38/2	34·66	22·01	75·0	40·6	135	152	192	234	0·072	—	Withstood	
38/3	39·6	32·25	71·0	37·5	154	172	206	252	0·082	2·0	Failed	
39	45·35	24·65	57·0	31·2	170	194	206	202	0·084	3·0	Withstood	
39/1	42·48	17·22	63·0	32·8	153	174	201	230	0·083	3·0	do	
39/2	42·48	16·20	64·0	34·4	147	163	204	258	0·083	3·0	do	
32/1	51·4	22·5	51·2	21·9	140	282	286	329	1·000	3·0	May be considered weldable	
32/2	57·84	26·23	51·9	21·9	159	271	314	337	1·000	3·0	Not weldable	
34	63·33	29·00	40·2	18·7	200	247	362	375	1·000	Failed	do	

TABLE 21 — RESULTS OF ATMOSPHERIC CORROSION TEST

HEAT No.	CHEMICAL COMPOSITION, %										CORROSION RATE IN GM./DM.^{2} EXPOSURE TIME ONE YEAR
	C	Mn	Cr	Si	S	P	Cu	Al	Ti	V	
2	0·1	0·92	0·67	1·9	0·014	0·034	0·56	—	—	—	1·31
3	0·083	1·88	1·12	0·25	0·037	0·024	0·21	—	—	—	1·18
3R1	0·088	1·46	1·4	0·26	0·037	0·027	0·29	—	—	—	1·09
3R4	0·04	1·61	1·23	0·06	0·01	0·02	0·31	—	—	—	1·6
5	0·065	1·86	0·11	0·55	0·03	0·03	0·49	—	—	—	1·37
6	0·15	0·61	1·03	0·89	0·034	0·017	0·26	—	—	—	1·23
10	0·145	0·1	—	0·35	0·019	0·014	—	0·038	—	—	1·1
11	0·15	0·59	0·53	0·24	0·037	0·067	0·26	—	—	—	0·76
11R	0·14	0·94	0·66	1·4	0·016	0·146	0·25	—	—	—	1·4
12	0·084	0·30	0·69	0·22	0·025	0·082	0·40	0·005	—	—	0·95
13	0·135	0·90	0·884	0·583	0·019	0·125	0·41	—	—	—	1·41
18/3	0·19	1·65	0·15	0·32	0·01	0·02	0·33	—	0·02	—	1·97
21/3	0·11	1·4	0·23	0·14	0·01	0·02	0·38	—	—	0·16	1·8

that none of them were air-hardening type. Weldability test and corrosion test of these steels were undertaken. One year's atmospheric corrosion test of some of the steels were completed. The results along with their chemical compositions are given in Table 21 which shows that steels having copper and phosphorous give good resistance to corrosion.

Further work under way in this project included completion of all the tests including weldability test and corrosion tests. After a critical study of the test results, the heats possessing good mechanical properties as well as adequate weldability and corrosion resistance would be further improved in terms of economy of alloying element, economics of production and yield of finished steel. In the next phase of work, production of tonnage scale heats on industrial scale will be taken up.

28.0 Development of Electrical 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 do not exist in India. There is heavy consumption of nickel and cobalt containing heat-resistant elements in India for domestic and industrial heating application. With increase in availability of electric power and industrial growth, demands 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 leading to the development of indigenous based electrical heating elements.

A number of experimental heats were made using Mn, Si, Ti, Zr, V, misch metal etc. in the high frequency furnace. The hot and cold working behaviour of these were determined along with their physical and mechanical properties. After extensive work it was found desirable to work out in the range of the basic analysis of heat No. E 31 containing relatively high aluminium and low chromium.

During the period under review, this basic composition of heat No. E31, which had shown considerably longer life in relation to other heats, was studied in detail for adjustment of the alloy composition and standardizing the smelting technique and hot and cold-working cycles during various stages of forging, rolling and wire drawing. Experimental heats were made using small amounts of misch metal with or without Zr or Ti. For heats with magnesium addition, Al-Mg alloy of 30 per cent and subsequently 10 per cent Mg concentration were used but it resulted in fire hazards. The misch-metal additions also required proper care to hold the melt to allow inclusions to float up, otherwise streaks of inclusions were observed on the surface of slabs and rods along their rolled lengths. Some heats were also made to study the effects of using Cr-Al master alloy in place of low carbon ferro-chrome to minimize undesirable inclusions.

Most of the heats were generally made in 20 lb. high frequency furnace. The ingot surface was dressed before hot working. Forging of the ingots was generally done at 1200°C. whilst taking care to maintain optimum finishing temperature and mechanical reduction. The forged billets were dressed to remove any surface defects. The machined bars after soaking at about 1050°C. were hot-rolled from 1 in. square size to 3/8 in. dia. rod. It was very important to control the finishing rolling temperature to obtain fine grain-size of the final product. The rods were pickled in 8 per cent HNO_3 1 per cent HF pickling solution to reveal any defect which could be dressed. The rods were annealed at 750°C. and lime coated before cold drawing. A special type of grease mixed with graphite was used for lubrication during cold drawing. It was found necessary to anneal after 2 to 3 passes through tungsten carbide dies. After 20 swg. size, it was also possible to give double die reduction in one pass. During wire drawing, serious trouble of longitudinal cracking was noticed besides occasional lubrication failure; the latter was eliminated after some trials and adequate prior surface preparation. Metallographic examination of the longitudinal cracks showed them to be associated with high inclusions count. Proper care during melting and subsequent mechanical working and sufficient interstage annealing were found helpful in eliminating longitudinal cracking.

Electrical resistivity measurements were carried out at elevated temperature. It was seen (Fig. 19) that after a high rate of rise in the range of 400°-600°C. the curve flattened out.

Accelerated life test of these heating elements was made as per the A.S.T.M. Standard under 2 min. current on and 2 min. off conditions. Results are given below in Table 22.

TABLE 22 — ACCELERATED LIFE TESTS OF HEATS

MATERIAL	TEMPERATURE °C.	ACCELERATED LIFE hr
E31	1200	91
	1150	166
4E31	1200	98
	1150	260
5E31	1200	10
2E28	1200	58
	1150	97
3E20	1200	62
	1150	134
E37	1200	54

It was thus seen that the life of 5E31 was extremely low, which was due to extensive non-metallic inclusions in the heat. Heats 2E28 and 3E20 which were of lower Al and higher Cr content gave less life; manganese in appreciable amounts also had very deleterious effect in this respect. Mechanical tests were carried out at room temperature as given in Table 23. Arrangements for high temperature tensile tests were made.

A preliminary Project Report was prepared for the production of this alloy at a modest rate of 20 tons per annum and the cost figures were worked and found to be much less than

TABLE 23 — RESULTS OF MECHANICAL TESTS

Heat No.	Ultimate Tensile Strength tons/ sq. in.	Elongation % 2 in. G.L.	Reduction Area %	B.H.N. As-cast
4E31	50·0	20·0	62·0	219
2E41B	52·5	20·0	68·0	211
3E32	53·0	18·0	53·0	229
E40B	54·5	16·0	55·0	204

the present market price of imported heating element.

Further work was underway to make larger size heats of 75 lb. in high frequency furnace and study their working characteristics. Studies were also undertaken on the utilization of over 30 per cent scrap for making the heats.

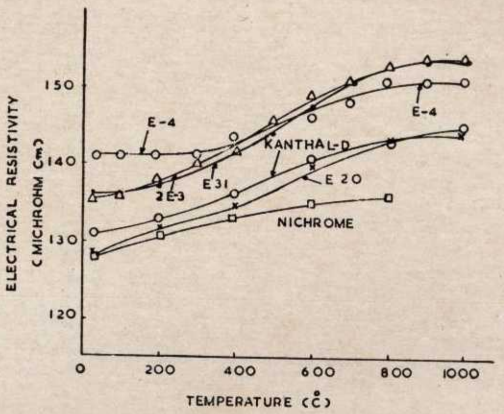


FIG. 19 — ELECTRICAL RESISTIVITY OF HEATING ELEMENTS AT ELEVATED TEMPERATURES

29.0 Development of Nickel-free Coinage Alloys

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. The actual production trials carried out at the India Government Mint, Alipore, starting from melting of the alloying elements to the stamping operations on a few cupro-manganese compositions showed that the ingots obtained by melting scrap in oil-fired and arc-furnace were internally not very sound. Few trials were made to study the effects of melting and pouring temperatures of the alloy, and slag covering, etc. on soundness of ingots cast. No definite improvement could, however, be made thereby. It was decided to try the smelting of scrap in high frequency furnace at the India Government Mint, Bombay. In order to increase the tarnish resistance properties of binary copper-manganese alloys, small additions of other elements were made to compositions to be employed for higher denomination coins.

Accordingly, smelting of these alloy compositions was carried out with varying scrap contents (maximum being 100 per cent scrap) at the India Government Mint, Bombay. The 4½ in. dia. ingots were found to be free from any internal defects even with 100 per cent scrap charge. The ½ in. thick slabs also cast from the same melts were found to be fully satisfactory. Blanks cut from hot and cold rolled sheets on annealing and polishing were found to have red streaks on the surface apart from the general coppery colour which developed within a very short interval after polishing. Studies were in progress to define remedial measures for the above difficulties. Some experiments were carried out at the Bombay Mint by varying the temperature of soaking of ingots, mode of rolling in the cogging mill, variation of gas to air mixture in the annealing furnace and employing different pickling solutions with or without inhibitors during the barrel polishing of blanks.

30.0 Development 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 reducing the copper and zinc contents in brass by their partial substitution by manganese.

Studies were continued on the physical properties of these alloy systems which included the thermal and electrical conductivity of copper-manganese alloys and their variations with change in temperature. Studies on thermal conductivity covered alloys from 0 to 40 per cent Mn with specimens prepared from hot rolled sheets. It was found that though the manganese bearing alloys had marked decrease in thermal conductivity yet they could well compare with stainless steel widely used for domestic utensils. Studies on electrical conductivity were carried out on few wire specimens placed in a specially prepared specimen holder and then maintained at constant temperature between 0 and 150°C. The changes in electrical resistance were found to be interesting, which made the alloys ideal for use in making standard resistances. Arrangements were made to extend these studies to both higher and lower temperatures and also to any other similar alloy system for comparison.

31.0 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 developing detailed technical know-how for their production and to develop, if possible, permanent magnets and other magnetic materials from indigenous sources. The work on development of magnetic materials was pursued actively under the following heads:

(a) Sintering of iron-nickel-aluminium magnetic alloys.
(b) Tempering characteristics of some Alnico type permanent magnets.
(c) Study of magnetic and electrical properties of ceramic permanent magnets of hexagonal structure.
(d) Study of electrical and magnetic properties of some iron-manganese-aluminium alloys.

(a) *Sintering of Iron-Nickel-Aluminium Magnetic Alloys* — Iron, nickel and iron-aluminium metal powders of −200 mesh were mixed in a suitable proportion to get final composition of the mixture: Ni 27 per cent, Al 13 per cent, Fe balance. The mixture was pressed into green compacts in steel dies under a pressure of 30 tons/sq. in. The compacts were 2·0 in. long, 0·5 in. broad and 0·1 in. thick and were sintered in a dry pure hydrogen atmosphere at 1200°, 1250°, 1300° and 1350°C. for durations of 0, $\frac{1}{4}$, $\frac{1}{2}$, 1, 2 and 4 hours. The sintered alloys were slightly ground and their physical properties such as density, hardness, specific electrical resistance, coercive force and remanence were determined. The magnetic measurements were made ballistically using an electromagnet, while electrical resistance was determined using a Kelvin's double bridge. A report on the subject was prepared. Some results of the investigation are as follows:

(i) *Density Changes:* Density-time curves are shown in Fig. 20 for sintering temperatures of 1200°, 1250°, 1300° and 1350°C. The figure showed that density increased almost linearly with time for temperatures of 1200° and 1250°C. while there was a rapid increase in density in the first 1$\frac{1}{2}$ hours of time at 1350°C., after which it became almost constant. The linear increase in density was also associated with progressive homogenization of the alloy as substantiated by the photomicrographs of the alloy sintered at 1200°C. for various durations (Fig. 21).

From the slow rate of densification and homogenization at those temperatures, it may be assumed that reactions governing these processes took place in the solid state, and iron-aluminium alloy powder which could have given rise to a liquid phase was perhaps consumed by solid state reactions much before its melting point was reached; this view was in conformity with the findings of Altman. At 1350°C., the densification was extremely rapid in the first one hour. After two hours of sintering there was practically no increase in density, and sintering on a macro scale had almost come to a standstill. It may be possible that the fusion of the alloy occurred in the first one or two hours and therefore there was no further increase in density afterwards. Micro examination of alloys sintered from 0 to 2 hours of sintering showed that there were very small pores present after sintering for 2 hours and the alloy was a single-phase solid solution.

(ii) *Electrical Resistivity Changes* — The resistivity changes with time for temperatures 1200°, 1250°, 1300° and 1350°C. are shown in Fig. 22. It was seen from the figure that electrical resistivity decreased with time at all these temperatures and there was an abrupt decrease in electrical resistivity in about first half an hour of sintering at 1200° and 1250°C. Thus the sintering of iron-nickel-aluminium powders took place in two steps. The First step was indicated at the early stages of sintering at 1200°C. and 1250°C., where an abrupt decrease in resistivity took place and the second step at the later stages of these temperatures as well as at 1300° and 1350°C. throughout where the decrease was gradual.

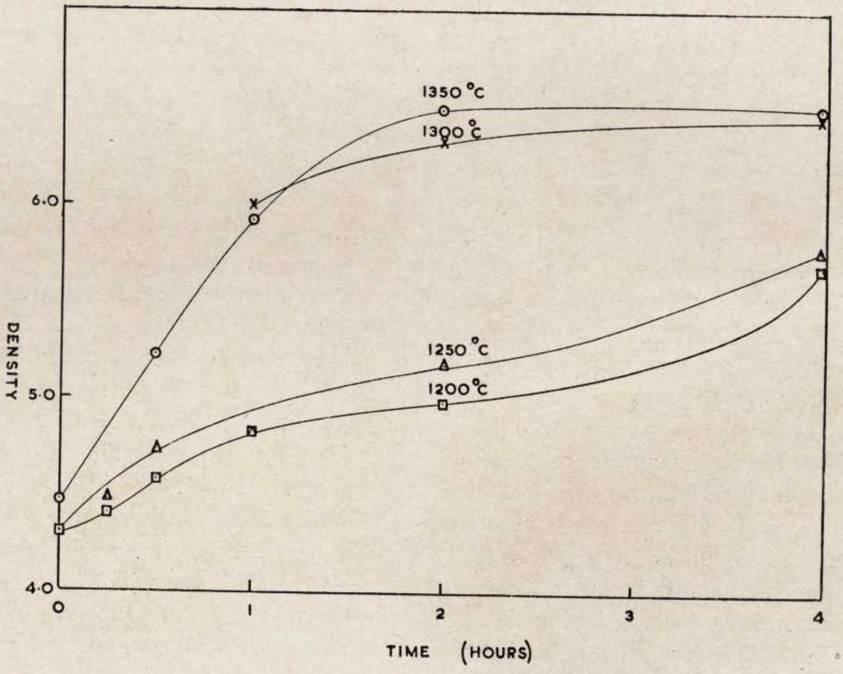


Fig. 20 — Curves showing variation of density with time at different sintering temperatures

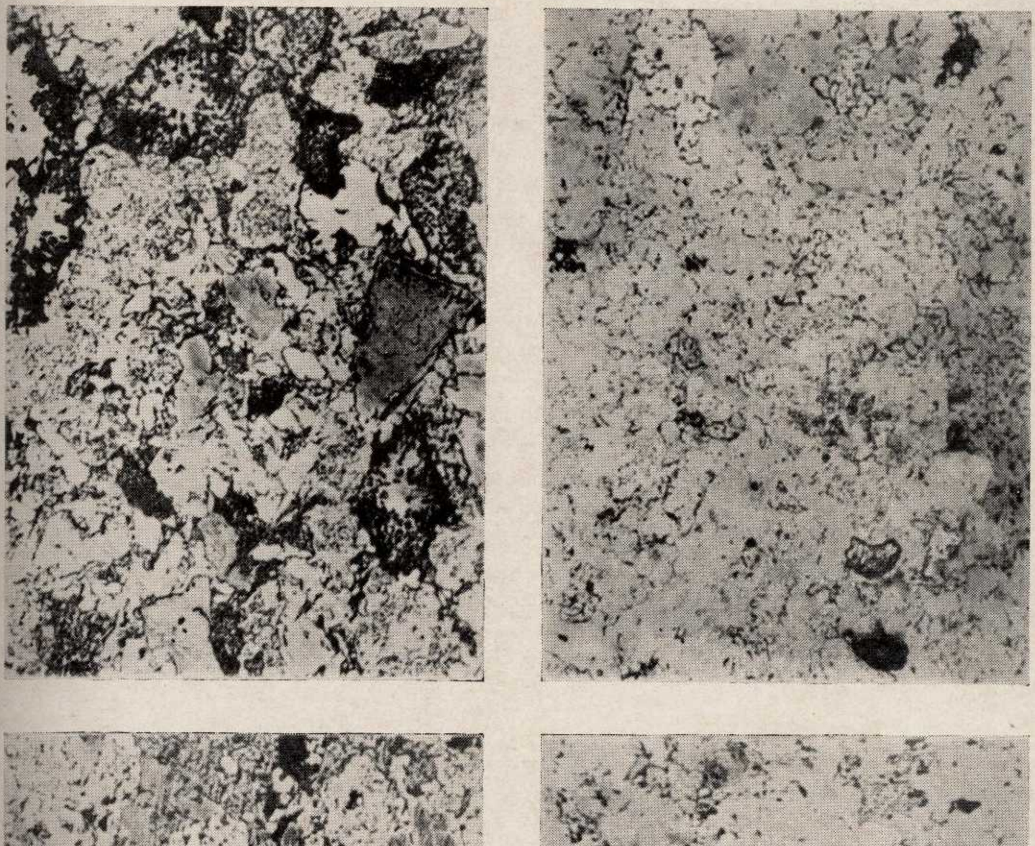


FIG. 21 — PHOTOMICROGRAPHS OF ALLOYS SINTERED AT 1200°C. ×250

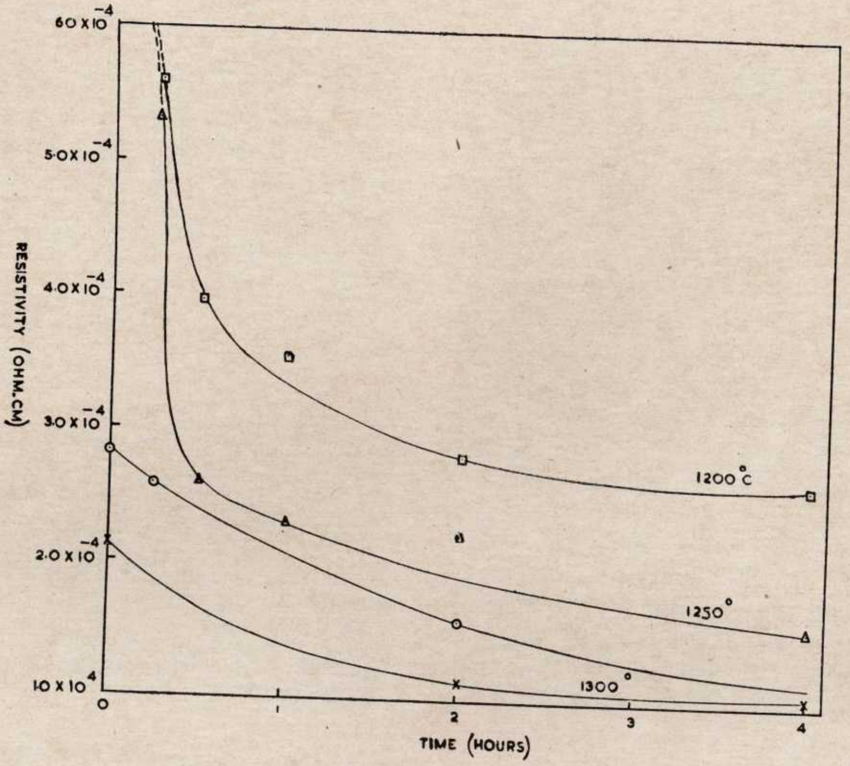


FIG. 22 — CURVES SHOWING VARIATION OF ELECTRICAL RESISTIVITY WITH TIME AT DIFFERENT SINTERING TEMPERATURES

(iii) *Coercive Force and Remanence Changes* — Coercive force-time curves are plotted in Fig. 23 for sintered magnets obtained after sintering at different temperatures. It would be seen that coercive force values were comparatively smaller in early stages of sintering. The values, however, rose steeply after one hour of sintering at 1350° and 1300°C. The curves showed that practical sintering temperature for the production of alloys as magnets would be above 1300°C. and duration of sintering not less than two hours. The fact that precipitation hardening of the alloy could take place even at 0 minute of sintering at 1200°C. again showed that alloy formation of Fe_2NiAl composition, which on decomposition gave permanent magnet properties, was in advanced stage of formation. The maximum value of coercive force was about 400 oersteds after sintering for 4 hours at 1350°C. without any further treatment which compared well with that of the cast magnets. The maximum value of remanence observed in these tests was about 4200 gausses at 1350°C. for a sintering time of 2-4 hours. Comparativrly low values of remanence as compared to cast magnets could be attributed to porosity which was of the order of 6 per cent in the best sintered magnets in this investigation.

(b) *Tempering Characteristics of Some Alnico Type Permanent Magnets* — Effects of cooling rates on the magnetic properties of Alnico type alloys was earlier investigated. The object of present investigation was to study the effects of tempering at various temperatures on the magnetic properties of the alloys cooled at rates to give optimum magnetic properties. The alloys selected were (i) pure Alni composition (27 per cent Ni, 12 per cent Al, balance Fe), (ii) Alni with copper (24·4 per cent Ni, 10·9 per cent Al, 4·5 per cent Cu, bal. Fe), (iii) Alni with cobalt (19·4 per cent Ni, 11·2 per cent Al, 5·4 per cent Co, bal. Fe), (iv) Alnico (19·1 per cent Ni, 11·3 per cent Al, 5·75 Cu, 11·1 per cent Co, bal. Fe). These were heat-treated to give best magnetic properties. The heat-treatment depended upon the alloy composition and consisted in cooling the alloy at a critical rate from the solution temperature to 600°C. in air. The test pieces were then tempered at 600°, 700°, 800° and 900°C. for various durations up to 400 hours depending upon the temperature. Tempering was carried out in purified argon atmosphere. After each tempering time, the samples were tested ballistically for their magnetic properties like (1) saturation induction, (2) remanence, and (3) coercive force. The samples were also tested for their hardness. The results of the investigation are given as below:

Tempering at 600°*C*.— The Alni type magnets showed first increase in coercive force with time up to 30 hours of tempering and then a gradual decrease. The remanence increased for the first few hours, only after which it became nearly constant. Hardness values showed somewhat abnormal behaviour. Though there was marked decrease in coercive force with longer tempering time, the hardness values remained somewhat unaltered which indicated that magnetic hardness was not a factor of mechanical

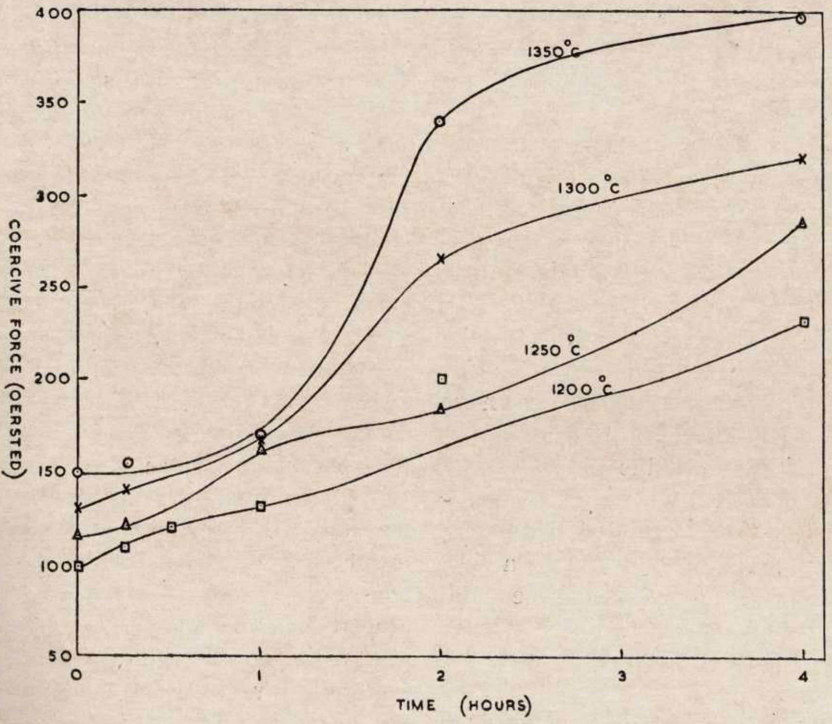


FIG. 23 — CURVES SHOWING VARIATION OF COERCIVE FORCE WITH TIME AT DIFFERENT SINTERING TEMPERATURES

hardness which hitherto had been considered as dependent upon strain in the matrix. Alni with copper also showed somewhat the same behaviour but increase in coercive force was spread over about two hours of tempering only after which gradual deterioration in magnetic properties took place. In case of Alni with cobalt, no increase in coercive force on tempering was observed. From the very beginning, the coercive force started falling. In the case of Alnico type magnet samples, the tempering behaviour could be divided into three stages: (i) In the first stage, the coercive force increased which covered about two or three hours. (ii) After this the magnetic properties deteriorated slightly but remained more or less constant for a duration of about 35 hours. (iii) In the third stage which followed tempering beyond 35 hours, the magnetic properties deteriorated gradually. From these tempering results, it was clearly seen that presence of 4-5 per cent copper or cobalt somewhat accelerated precipitation. However, when cobalt content was raised to 10 per cent in the presence of 4-5 per cent copper, the precipitation was markedly slowed down.

Tempering at 700° *and* 800°C.— At tempering temperatures of 700°C. and 800°C., all the samples showed deterioration in the magnetic properties, which showed that precipitation process was very rapid at these temperatures.

Tempering at 900°C.— Tempering results of Alni, Alni with copper and Alni with cobalt showed marked deterioration of magnetic properties even after short duration of annealing at 900°C. In the case of Alnico magnets, the behaviour was somewhat different. In the first 10 minutes of tempering the coercive force decreased, but in a subsequent tempering for longer times the coercive force not only showed an increase but also a definite tendency to reach its original values.

The results of tempering were under final scrutiny. Micrographic and X-ray analysis were also conducted.

(c) *Study of Magnetic and Electrical Properties of Ceramic Permanent Magnets of Hexagonal Structure* — Hexagonal mixed oxides of the general formula $MO.6Fe_2O_3$, when M may stand for barium, strontium, lead or calcium, are associated with high coercive force, i.e., 2,000-3,000 oersteds. Such magnets are finding applications in light electrical industry due to their low cost and high resistance to demagnetization. The magnets are made in the same fashion as ceramic materials and require to be sintered at temperatures in the range of 1200°-1300°C. to develop best magnetic properties. It is, however, very difficult to produce repeatable magnetic properties due to high rate of grain growth at these temperatures. The problem is, therefore, to reduce the sintering temperature which was recently achieved by small additions of low melting oxides like bismuth oxide which not only lowered the final sintering temperature to about 1100°C. but also improved the coercive force. It was, therefore, necessary to study the effects of additions of other low melting oxides on the magnetic properties, sintering temperature and grain growth of barium-ferrite magnets.

As a first step, sintering characteristics of pure barium-ferrite as well as barium-ferrite containing small quantities of lead oxide were studied. Barium-ferrite was formed by calcination of pure $BaCO_3$ and iron oxide weighed in molar qualities. The calcination was carried for the temperature range 900°-1050°C. for 1½-2 hours. The barium-ferrite compound so formed was pulverized to −200 to −400 mesh compacted at 5 tons/sq. in. and sintered from 1100° to 1300°C. for various duration. It was found that pure barium ferrite gave best properties in the sintering temperature range of 1200°-1250°C. The properties were, however, not reproducible as stated before. The properties could, however, be controlled better in the presence of low melting point oxides like lead oxide. The coercive force in such cases, was as high as 2500 oersteds and remanence 2000 gauss.

The experiments were also tried on commercial type of raw materials such as fine blue dust with iron content in the range of 67-68 per cent. The blue dust contained about 2 per cent of $Al_2O_3 + SiO_2$. The magnets made from such materials, however, showed comparatively lower values of coercive force. The experiments were also carried out by additions of small amounts of five or six other oxides. In some cases, it did not bring about any improvement. The structure of these ceramic magnets was evaluated by X-ray diffraction studies to arrive at precise conditions for highest coercive force.

(d) *Study of Electrical and Magnetic Properties of Some Low-manganese Low-aluminium Steels* — The object of this investigation is to develop low-manganese low-aluminium steels suitable for use as core material in transformers and electrical machines, with a view to improve upon, if possible, the conventional 4 per cent silicon steel sheet now generally employed.

It had previously been established that steels containing 3-4 per cent aluminium and 2 per cent manganese had very low values of coercive force and hysteresis loss as compared to 4 per cent silicon steels. It was, therefore, necessary to make these alloys on a big scale with a view to examine if conditions of deoxidation could be controlled to get magnetic properties obtained in smaller five pound heats. Accordingly, some trial heats were made with starting matreials either as mild steel or armco iron to make a melt of 70 lb. in high frequency induction furnace. It was found that carbon content of the heats made from mild steel dropped during melting to 0·06 per cent. The melts were then made using armco iron, which could be made in about 3 hours with a good recovery. The alloying additions were made to the iron in the furnace, first killing it with ferrosilicon, followed by manganese and aluminium additions. Study of cold rolling behaviour of the steels with good magnetic properties was also undertaken in case of five compositions containing 2-5 per cent aluminium and 1-5 per cent manganese. It was observed that the sheets could be cold rolled from 0·1 in. to 0·005 in. thickness without developing any edge cracks. The work hardening capacity of these steels was also studied. The hardness of these steels in the annealed state varied from 140 to 180 V.P.H.N. and after cold rolling involving 80 per cent reduction, the hardness varied from 200 to 250 V.P.H.N.

A ten pound heat of iron-aluminium alloy containing 10 per cent aluminium was also made. The ingot was hot rolled at 1100°C. to 0·1 in. thickness. Preliminary trial to cold roll this steel was carried out successfully by rolling the steel below its recrystallization temperature. Another heat using super pure aluminium and pure iron was made, in a resistance vacuum furnace. The composition aimed at was 16 per cent aluminium. The chill cast ingot developed a long pipe which made it difficult to work. Further trials were in progress.

32.0 Tinless Bearing Alloys

Tin is extensively used in the manufacture of bearing metals. Due to non-availability of tin in India, the project was taken up with a view to develop aluminium base bearing alloy to replace tin. The alloy was of composition: 4 per cent silicon, bismuth and cadmium 1 per cent each, with a distribution of 3 per cent lead in the alloy. The distribution of lead in aluminium was difficult and several methods were tried to get the lead uniformly distributed in the aluminium matrix without segregation.

One important method tried was to inject lead dioxide to molten aluminium in an atmosphere of nitrogen gas and about 1-1·2 per cent of Pb content was thereby reported. The process was very cumbersome and a lot of dross with oxidation of Al was formed. In another method, lead powder was added to molten aluminium. The lead powder was made by the atomization technique and was directly added to molten

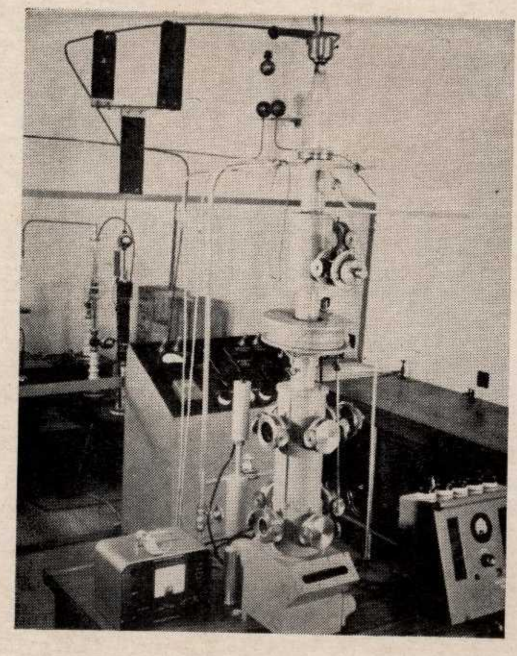


FIG. 24 — A VIEW OF THE FINCH TYPE ELECTRON DIFFRACTION CAMERA UNIT INSTALLED AT THE NATIONAL METALLURGICAL LABORATORY

aluminium by wrapping in an aluminium foil and then stirring the alloy. Nitrogen was used for degassing the metal. Nitrogen was also observed to help uniform distribution of Pb when the microstructures were examined with and without the use of N_2. The Pb per cent was reported on analysis as 1·5-1·9 per cent. There was no segregation of Pb in the ingots. These trials were repeated under varying conditions. Results obtained were under scrutiny.

33.0 Light Metals and Their Alloys

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

In continuation of the work conducted to improve the hot working characteristics of Al-7/10 per cent Mg alloys, different addition agents, e.g. misch metal, manganese, etc., were tried. 0·2-1·2 per cent (by 0·2 per cent increments) of manganese was added to the Al-7/10 per cent Mg alloys, a 75-25 Al-Mn hardner alloy being used for ensuring efficient additions of Mn to the alloy melt. Indications that improvements in hot-forgeability of these alloys could be made by Mn addition were observed. Considerable reduction could be obtained in ingots with medium Mn (0·6 per cent) during hot forging. Undesirable grain coarsening was, however, observed to occur with higher Mn contents. The tensile strength obtained for the as-cast specimens varied from 4 to 11 tons per square inch, higher values being again obtained when 0·4-0·8 per cent Mn had been used.

TABLE 24 — TENSILE TESTS OF AS-CAST SPECIMENS

HEAT No.	CHEMICAL COMPOSITION		ULTIMATE TENSILE STRESS tons/sq. in.
	Mg %	Mn %	
MT_1	9·02	1·66	4·84
MT_2	8·64	1·54	4·65
MT_3	8·68	1·20	4·77
MT_4	8·47	0·98	6·63
MT_5	9·06	0·49	11·0
MT_6	9·27	0·37	9·0
MT_7	8·41	1·15	5·31
MT_8	7·97	1·41	6·64
MT_9	8·19	1·04	7·65
MT_{10}	8·45	0·81	9·24
MT_{11}	8·47	0·57	8·41
MT_{12}	7·93	0·35	6·42

The macro and micro-examination of the alloys in the cast and heat-treated condition were undertaken. Precipitation hardening characteristics of the alloys Al-8 per cent Mg with 3 per cent misch metal and Al-9 per cent Mg with 3 per cent misch metal were studied at 100°, 200° and 300°C., up to a period of 500 hours. The hardness values were found to increase with ageing time and over-ageing was found to occur after a certain

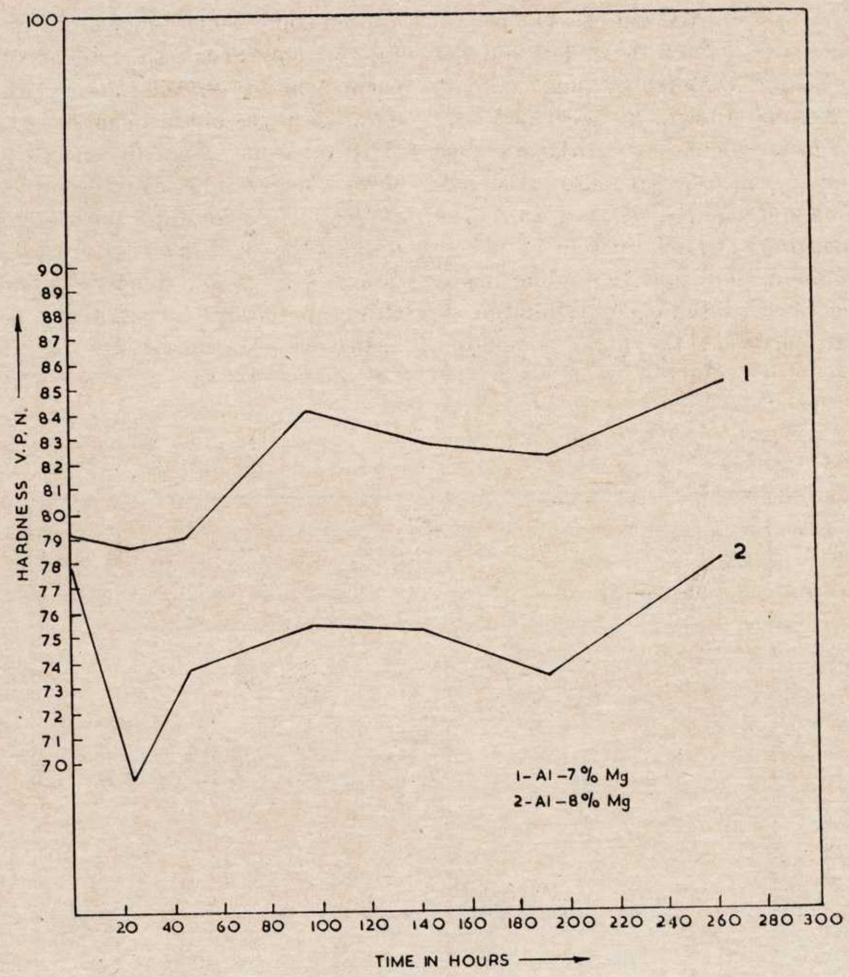


Fig. 25 — Precipitation hardening characteristics of Al-Mg (7-8 per cent) alloys at 1000°C.

ageing time but the increase in hardness value was not very appreciable, and the results in most cases were erratic.

The precipitation hardening characteristics of binary Al-9 per cent Mg alloy and Al-10 per cent Mg alloy without any intentional addition agents were also studied. The results obtained are shown graphically in Figs. 25 and 26. Solution treatments of Al-8 per cent Mg alloy, Al-8 per cent Mg-2 per cent misch metal and Al-8 per cent Mg-3 per cent misch metal alloy were carried out at 430°, 450° and 500°C. in order to find out the effectiveness of these treatments for taking different micro-constituents into solution and to define the temperature ranges for their heat treatment. During hot rolling of Al-8/10 per cent Mg alloy (untreated and misch metal treated) ingots failed in the rolls with crocodile tearing. A detailed investigation was undertaken to determine the cause of such failure of these billets, which had been attributed elsewhere to the presence of traces of sodium. However, chemical analyses of the samples taken from the cracked portions of the billets did not show any sodium. Metallographic examination showed that the cracks developed were mostly of intergranular type although some transgranular cracks were also observed. (Photomicrographs Figs. 27 and 28.) The grain size appeared to be fairly coarse. These observations indicate that the failure might have been caused due to (i) precipitation of some hard phase at the grain boundaries which could not flow during rolling, or (ii) grain coarseness. Further work was carried out on (i) precipitation characteristics of misch-metal and manganese treated Al-Mg (7-10 per cent) alloys, (ii) effect of additions such as Mn, Cr, and Zn on the hot workability of the Al-Mg alloys and (iii) electron-diffraction study of changes in crystal structure brought about by different additions and effects of various homogenizing heat treatments on Al-Mg alloys.

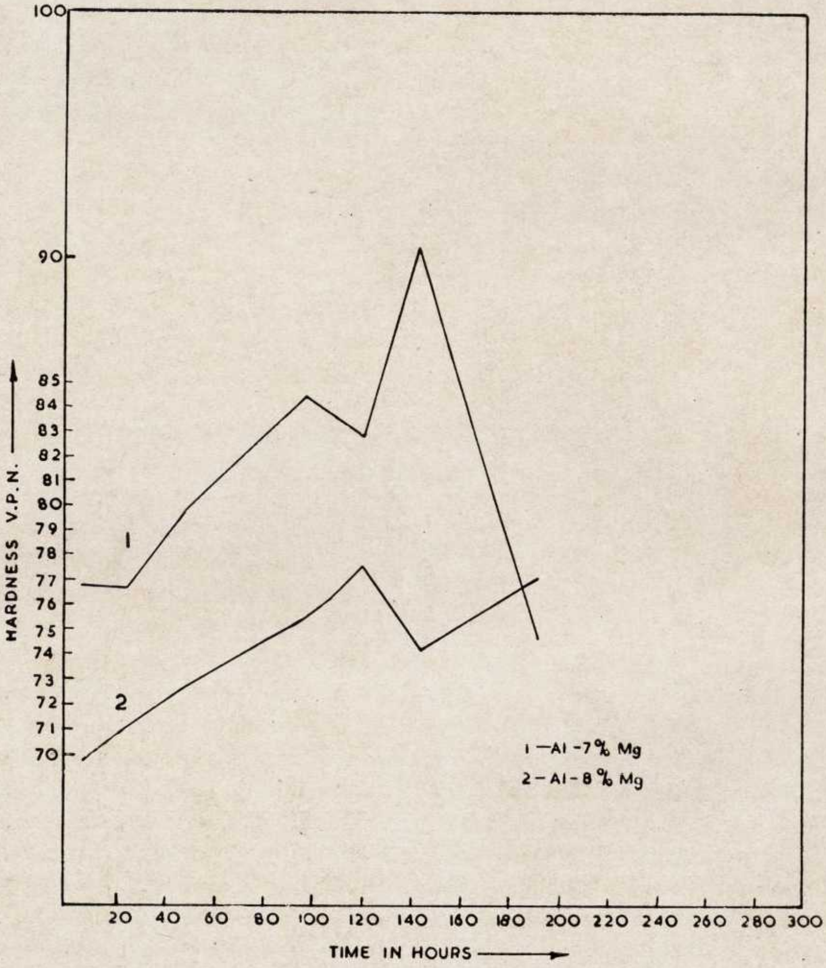


FIG. 26 — PRECIPITATION HARDENING CHARACTERISTICS OF Al-Mg (7-8 PER CENT) ALLOYS AT 200°C.

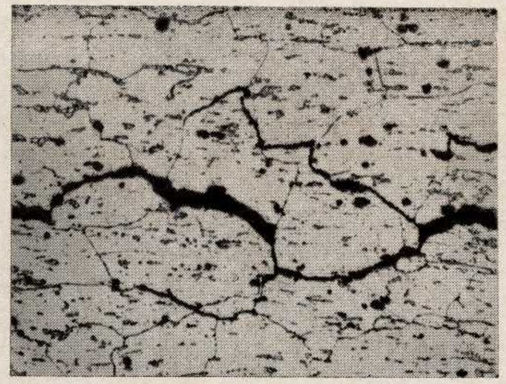


Fig. 27 — Photomicrograph showing intergranular crack in Al-Mg (9 per cent) alloys. × 250. Rolled surface

TABLE 25 — CHANGE IN HARDNESS OF Al-Mg MISCH METAL ALLOYS DURING AGEING AT 100°C.

Al-8% Mg-3% Misch Metal		Al-9% Mg-3% Misch Metal	
Time of ageing hr	Hardness V.P.H.N.	Time of ageing hr	Hardness V.P.H.N.
0	88·3	0	101·29
48	95·89	0·5	102·58
72	94·24	1	96·14
120	92·53	2	94·37
144	96·34	3	97·84
168	91·58	20	98·18
192	96·93	44	103·2
216	95·83	68	101·24
360	91·93	164	87·78
384	95·4	188	97·48
456	93·87	212	99·85
480	93·49	284	96·81
504	91·85	308	99·9
		332	94·6
		356	96·9
		380	103·00
		500	91·4

34.0 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. During the period under review, twelve sand and bonding clay samples were investigated. A summary of the work done is given below:

(i) *Silica Sand sent by M/s. Britannia Engg Co. Ltd, Titaghur, West Bengal* — The sand samples represented a crude silica sand with a grain fineness number of 47, containing 70-80 per cent of SiO_2. The sand was medium coarse. It had a sintering range of 1200-1250°C. and may prove useful for grey iron foundry castings.

(ii) *Moulding Sand sent by M/s. Britannia Engg Co. Ltd, Titaghur, West Bengal* — This sand contained above 50 per cent of A.F.S. clay grade matter. The sand grade assayed nearly SiO_2 73 per cent. The sand was widely distributed on all sieves. With a grain fineness number of 108, it may prove useful for grey iron foundry castings after blending with a sharp silica sand of a coarse grain size in suitable proportions.

(iii) *Londha Yellow Fine Core Sand sent by M/s. Kirloskar Bros., Kirloskarvadi, Bombay* — The sand had a grain fineness number of 65 with a subangular shape and contained 4·3 per cent of A.F.S. clay grade matters. It had 75-80 per cent quartz

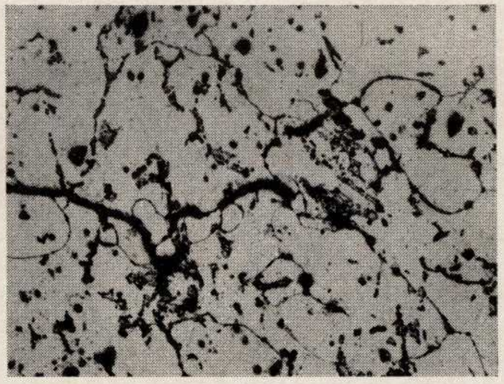


Fig. 28 — Photomicrograph showing intergranular crack in Al-Mg (9 per cent) alloy. × 250. Section perpendicular to rolled surface

and 15-20 per cent feldspar. Its SiO_2 content was 87 per cent approximately and had a sintering range of 1300-1350°C. With linseed oil as binder, it gave good core properties for both cast iron and steel castings. For obtaining green strength, dextrine or bentonite or both might be used.

(iv) *Sabarmaty River Sand* (*Fine*) *sent by M/s. Machinery & Equipment Manufacturers* (*Pvt.*) *Ltd, Ahmedabad* — This was a subangular sand of grain fineness number 129. The sand sintered between 1100° and 1150°C. Its SiO_2 content was 80 per cent approximately and had 52 per cent quartz and 30 per cent feldspar. It contained about 6 per cent of A.F.S. clay grade matter of poor bonding property. Addition of 1 per cent Bihar bentonite was necessary to develop sufficient bonding in this sand which was found useful for non-ferrous and cast iron founding.

(v) *Sabarmaty River Sand* (*Medium*) *sent by M/s. Machinery & Equipment Manufacturers* (*Pvt.*) *Ltd, Ahmedabad* — This sand was also a subangular sand of grain fineness number of 68. It contained 1·5 per cent of A.F.S. clay grade matter with 52 per cent quartz, and 30 per cent feldspar, while SiO_2 content was 84 per cent. The sand showed a sintering range of 1150-1200°C. It can be used for cast iron and non-ferrous founding. For light casting 5 per cent bentonite and for heavy castings 5 per cent bentonite and 2 per cent dextrine additions were found suitable for bonding.

Bonding clays — Investigations were completed on the following samples of clay for bonding moulding sands:

(i) *Bonding Clay sent by M/s. Britannia Engg Co. Pvt. Ltd, Titaghur, West Bengal* — Approximately 95 per cent of the sample passed through 200 mesh sieve (A.F.S.). Sintering range was found to be between 1050° and 1150°C.

(ii) *Bhavnagar Bentonite sent by M/s. Machinery & Equipment Manufacturers Pvt. Ltd* — The sample had a very fine particle size. It had poor gel strength and a steady *p*H value of 8·4. It contained about 52 per cent SiO_2 and 21 per cent Al_2O_3, and showed a poor base-exchange capacity. D.T.A. showed the sand to be mostly halloysite. Five per cent addition of the bentonite to a standard A.F.S. sand developed poor strength characteristics unfit for use in founding.

(iii) *Bentonite samples* (*i.e., select Grill, RNA, K-1, K-2-N, Bentoclay and CH*) *sent by M/s. Industrial Minerals & Chemicals* (*Pvt.*) *Ltd, Bombay, at the instance of Indian Standards Institution* — The samples were studied collectively. Excepting K-1, all bentonites had very fine particle size. Chemical analyses showed 45-50 per cent SiO_2 and 14-19 per cent Al_2O_3. CH, K-1 and Bentoclay contained 6, 3 and 1·5 per cent lime respectively. Alkali content was low whilst magnesia contents varied between 2·5 and 3·5 per cent in all cases. Gel strength was fairly good in K-2-N and Bentoclay, satisfactory in RNA and CH and poor in select Grill and K-1. Swelling indices were in accordance with the gel-strength values. However, base-exchange capacity was fairly good in RNA and K-2-N, satisfactory in Bentoclay and K-1 and poor in select Grill and CH. *p*H values of all the bentonite samples fell between 8·5 and 9·5. D.T.A. tests showed that all bentonites predominantly contained montmorillonite.

(iv) "*Hathi-Ki-Dhani*" *Bentonite sent by Mining Engineer, Jodhpur* — The sample was a fine variety of bentonite containing low SiO_2 and high Al_2O_3 and TiO_2. It had low swelling index and gel strength values. Although it contained montmorillonite predominantly, it was not free from other non-swelling impurities. Its *p*H value was 9. It had a satisfactory base-exchange capacity. D.T.A. tests showed it contained montmorillonite as a major constituent. The bentonite had sodium base. Good bonding properties were obtained with 3 per cent addition of high silica sand.

(v) *Rajula Bentonite sent by Ambica Mine Chem. Industries, Bhavnagar* — The sample

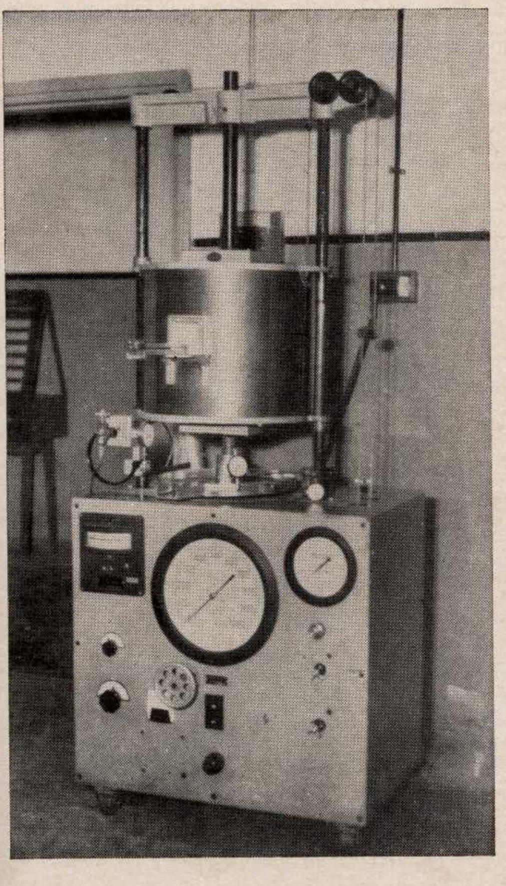


FIG. 29 — A VIEW OF THE APPARATUS FOR STUDYING THE PROPERTIES OF FOUNDRY MOULDING SANDS AT ELEVATED TEMPERATURES

possessed good swelling index and gel strength values. The sample was capable of being powdered, very fine. Its *p*H value was 9·9. It contained 60 per cent SiO_2 and 12 per cent Al_2O_3. It had a poor base exchange capacity. It sintered between the temperatures of 1025°-1075°C. The sample was calcium base montmorillonite and contained above 90 per cent montmorillonite. 5 per cent addition of the bentonite to a standard sand showed that it did not have good bonding characteristics.

(vi) *English Bentonite sent under the Scheme of International Working Group on Bonding Clays* — The bentonite had a low gel strength and a low swelling index value. The sample did not have very fine particle size. It had a *p*H value of 9·8. It contained about 51 per cent SiO_2 and 15 per cent Al_2O_3 with a slightly high percentage of Fe_2O_3 of about 7 per cent. Sintering range of the sample was 990°-1040°C. It had a low total base exchange value. The major content of the bentonite was safronite followed by sodium base montmorillonite. Good moulding characteristics were obtained when 6 per cent bentonite was used with a standard sand.

(vii) *Spanish Bentonite sent under the Scheme of International Working Group on Bonding Clays* — The sample possessed a high swelling index but only satisfactory gel strength value. It had fine particle size. *p*H value of 9·9 was obtained for this sample. SiO_2 content of the sample was 56 per cent, while Al_2O_3 was 14 per cent, which had a sintering range of 1000°-1050°C. Total base exchange capacity of the bentonite was good. Its major constituent was sodium base montmorillonite. Fairly satisfactory moulding characteristics were obtained when 6 per cent bentonite was used for bonding a standard sand.

(viii) *Poland Bentonite sent under the Scheme of International Working Group on Bonding Clays* — The bentonite possessed a very low swelling index and low gel strength value. The sample did not have a very fine particle size. It had a *p*H value of 5·8 only. It had a high SiO_2 content of 73 per cent and comparatively low Al_2O_3 content of 10 per cent. It indicated a sintering range of 1290°-1340°C. It showed a poor base exchange capacity. The sample contained calcium base montmorillonite as a major constituent with a high amount of impurities. Six per cent of bentonite, when mixed with a standard sand, showed fairly good moulding characteristics.

35.0 Solidification of Cast Iron

Apart from the well established effect of magnesium and cerium on the structure of

TABLE 26 — CHEMICAL ANALYSES OF THE CAST IRON SAMPLES

CHEMICAL CONSTITUENTS %	HEAT No. 1	HEAT No. 2			HEAT No. 3								
		Cu	Mo	Al	Cu	Mo	Al	Ti	V	Ca	Zn	Pb	Sn
C	3·25	3·14	3·13	3·18	3·27	3·3	3·85	3·84	3·42	3·42	3·15	3·23	3·46
Si	1·23	1·5	1·8	1·5	1·0	1·99	1·70	2·2	1·87	1·98	1·90	1·7	1·88
Mn	0·65	0·8	0·82	0·80	1·01	1·03	0·99	1·13	1·4	1·08	2·08	2·15	2·07
S	0·02	0·08	0·07	0·06	0·03	0·02	0·022	0·02	0·015	0·022	0·008	0·017	0·015
P	0·27	0·098	0·14	0·14	0·26	0·26	0·24	0·33	0·33	0·30	0·30	0·31	0·33
Inoculant as indicated	nil	0·50	0·20	0·30	0·19	0·29	—	0·089	0·012	Trace	nil	0·079	0·17

cast iron, many other elements are known to alter the graphite structure of cast iron to some extent. A study was taken up to study the effects of inoculants on the structure of cast iron.

It is well known that by increasing the number of nuclei, there is a possibility of decreasing the malleabilizing period. In this study, melts were fast cooled and inoculants added to increase the number of nuclei. Studies were made to note the effect on the micro-structure of cast iron after annealing for different lengths of time and at various temperatures. With the above objectives the following cast iron heats were made. In all the cases foundry pig No.: 2 was taken as the raw material.

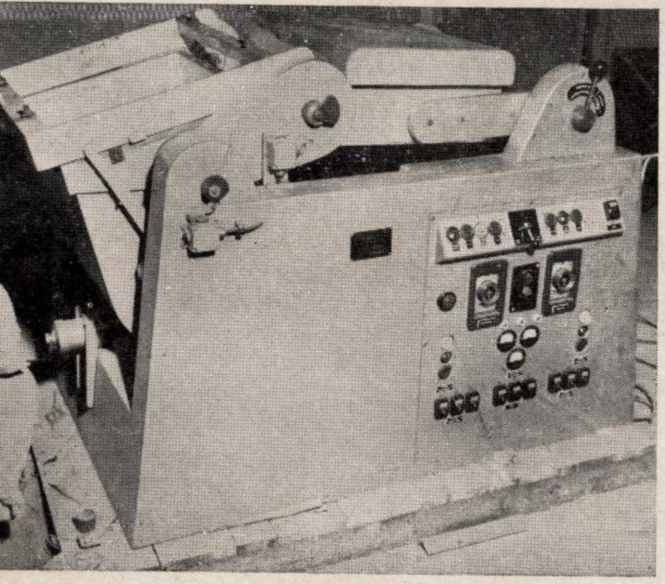


FIG. 30 — A VIEW OF THE SHELL MOULDING UNIT INSTALLED AT THE NATIONAL METALLURGICAL LABORATORY

Heat No. 1 — A thirty lb. heat was made in which oxygen was passed for approximately one minute at a pressure of 60 lb.

Heat No. 2 — A thirty lb. heat was melted and oxygenated as in heat No. 1 and then the melt was equally divided in three heated crucibles. Each was separately inoculated with Cu, Mo and Al and designated as No: 2 Cu, No: 2 Mo and No: 2 Al respectively.

Heat No. 3 — Three thirty lb. heats were made but were not oxygenated. These were inoculated with Cu, Mo, Al, Ca, V, Ti, Sn, Pb and Zn as in heat No. 2.

In all cases, the melt was poured in ingot moulds which were quenched in running water as soon as the solidification started.

Chemical analyses of the heats made are given in Table 26, while the heat treatments and subsequent results obtained on heat No. 2, are given in Table 27. All experiments were performed on Heat No. 3, which consisted in annealing the specimens for 12 hours at 900°C. and subsequently at 800, 850, 900 and 950°C., for time intervals of 40 hours and 60 hours at each temperature. In every case, the furnace was brought to room temperature by slow cooling. So far, microstructure studies on specimens treated with Cu were completed, studies on other specimens are in progress. The specimens revealed that at 900°C. when annealed for

TABLE 27 — HEAT TREATMENT AND MICROSTRUCTURE AND HARDNESS OBTAINED IN HEAT NO. 2 SPECIMENS

Specimen No.	As-cast Structure Obtained from a Chip of the Ingot	Microstructure and Hardness after 12 hrs Annealing at 900°C. and Subsequently Slow-cooling	Microstructure and Hardness after further 12 hrs (24 hrs total) Annealing at 900°C. and Subsequently Slow-cooling	Microstructure and Hardness after further 12 hrs (36 hrs total) Annealing at 900°C. and Subsequently Slow-cooling	Microstructure and Hardness after further 12 hrs (48 hrs total) Annealing at 900°C. and Subsequently Slow-cooling	Microstructure and Hardness after further 12 hrs (60 hrs toal) Annealing at 900°C. and Subsequently Slow-cooling
Heat No. 2 Cu	Typical massive cementite + ferrite + under-cooled graphite flake, graphites fine	Ferrite matrix, few pearlite islands, under-cooled graphite flake, graphites fine V.P.H.N. 149	Ferrite matrix, few pearlite islands, under-cooled graphite, V.P.H.N. 149	Ferrite matrix, few pearlite islands, under-cooled graphite and breakdown of dendritic structure marked, V.P.H.N. 145	Ferrite matrix, spheroidal pearlite, further breakdown of dendrites, V.P.H.N. 144	Ferrite matrix, spheroidal pearlite, further breakdown of dentrites, V.P.H.N. 144
Heat No. 2 Mo	do Flake graphite fine, V.P.H.N. 147	do V.P.H.N. 146	do V.P.H.N. 146	do V.P.H.N. 143	do V.P.H.N. 141	do V.P.H.N. 141
Heat No. 2 Al	do Flake graphite coarse, V.P.H.N. 151	do Flake graphite coarse, V.P.H.N. 149	do Flake graphite coarse, V.P.H.N. 147	do Flake graphite coarse, V.P.H.N. 146	do Flake graphite coarse, V.P.H.N. 145	do Flake graphite coarse, V.P.H.N. 145

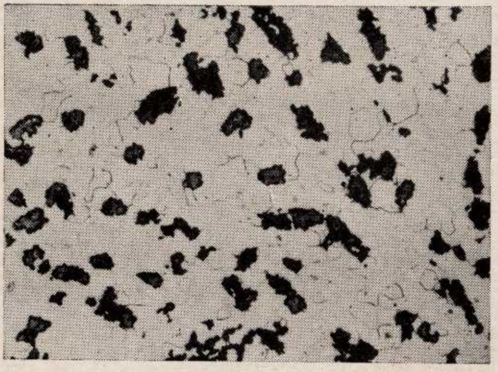


FIG. 31 — PHOTOMICROGRAPH OF CAST IRON INOCULATED WITH COPPER AND ANNEALED FOR 40 HR AT 850°C. × 250

60 hours and at 950°C. for 40 hours or 60 hours, there was complete disappearance of dendritic structure; this structure was observed in other cases more especially with lower soaking time at lower temperatures. In all cases, ferrite matrix was obtained with some irregular pearlite grains. There was, however, no tendency of globulization of the pearlite constituent even when annealed at 950°C. for 60 hours. In most cases, graphite nodular had the rosette type of structure.

The specimens annealed at 850°C. and 900°C. for 40 hours showed malleabilized structure on the edges of the specimen, as shown in Figs. 31-34. Further study was in progress to study the effects of other inoculants on the structure of cast irons, without annealing and after annealing for different periods.

36.0 Aluminium-Silicon Alloys

Work was undertaken to develop a new modifying agent capable of refining both the primary silicon crystals as well as the eutectic matrix of the aluminium-silicon alloys. These alloys are extensively used in the automotive field and their application to other engineering fields is fast increasing. Hitherto, no single modifying agent has been suggested which is effective in both the hypo- and hyper-eutectic ranges of the alloys. The modifying agent developed in this laboratory covered both the ranges with remarkable improvements in microstructure, fluidity, machinability, tensile strength etc.

Applications for obtaining patents on the new process were field in India, U.S.A., Great Britain, West Germany and France.

37.0 Powder Metallurgy

Work was taken up with a view to (i) produce copper powder continuously by electro deposition; (ii) make metal powders of aluminium and tin by atomizing; and (iii) make porous bronze bearings.

A successful method was developed on continuous electro-deposition of copper powder, which involved the treatment of the copper-sulphate electrolyte with an organic substance, and assisted in the self-regulated removal of copper powder from the cathode. It was found possible to control within wide limits the production of copper powder of required quality. The process was tested experimentally on the basis of continuous electro-deposition of copper over a two-months period, and proved successful.

The utility of the use of the organic addition was also tested at higher bath tempera-

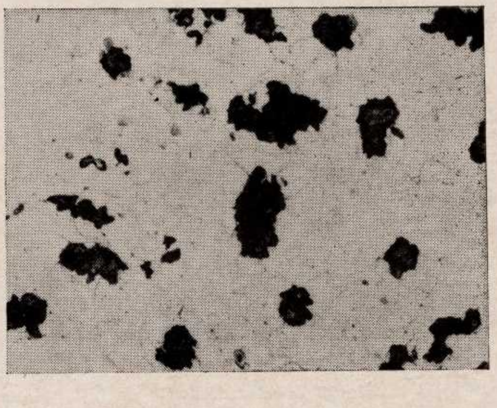


FIG. 32 — PHOTOMICROGRAPH OF CAST IRON INOCULATED WITH COPPER AND ANNEALED FOR 40 HR AT 850°C. × 450

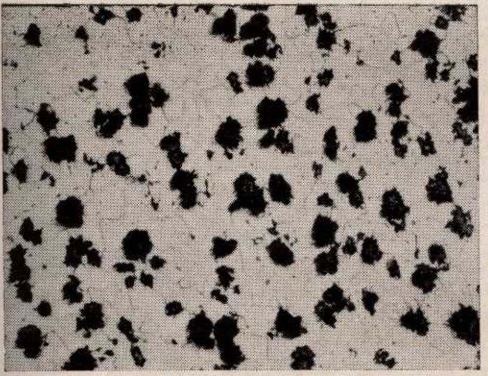


FIG. 33 — PHOTOMICROGRAPH OF CAST IRON INOCULATED WITH COPPER AND ANNEALED FOR 40 HR AT 900°C. × 250

tures up to 60°C., and found beneficial which had the advantage of requiring lower cell voltages and the powder obtained was of a higher apparent density. Continuous deposition of copper powder under controlled conditions could be carried out even at a cell voltage of 0·6 V. Some of the results obtained during this study are summarized in Table 28. A Project Report on the commercial production of copper powder by this method was prepared.

The work already underway on making porous bronze bearings by the technique of powder metallurgy was continued. Simple tools, required for making self aligning bearings of spherical shape were developed. With the aid of these tools uniform bearings with accurate dimensions could be manufactured without the necessity of maintaining a critical control over weight of charge, moulding pressures and sintering temperatures. The procedures of manufacturing was simplified to a large extent to make it suitable for industrial scale application. It consisted of simple operations as:

(a) Charging the die with a powder mixture,
(b) Moulding by applying pressure,
(c) Removal of the moulded green bearing from the die,
(d) Sizing of the moulded green bearing,
(e) Sintering of the green bearing,
(f) Resizing of the sintered bearing.

Results obtained are given in Table 29. Samples of porous bronze bearings, sent to table fan manufacturing firms for trials, were reported to be highly satisfactory in service trials.

Due to non-availability of tungsten ore concentrate as a starting material for the manufacture of ammonium paratungstate, ferro-tungsten was used and oxidized with commercial sodium nitrate. It was then purified by repeated precipitation with hydrochloric acid and ammonia. The ammonium paratungstate formed required to be powdered to a few microns size before it was ready for use. Experiments were carried out to study the suitability for making machine components by iron powder metallurgy. Suitable mixtures of iron powder with copper and zinc stearate were prepared and a machine component was made by moulding followed by sintering at proper temperature. The studies revealed that, while there was no difficulty in obtaining the uniform size, very high moulding loads were necessary. However, since iron powder had very poor compressibility, certain special techniques were developed for the successful operation of the process. Machine components which had their top and bottom surfaces parallel to each other were successfully made on a

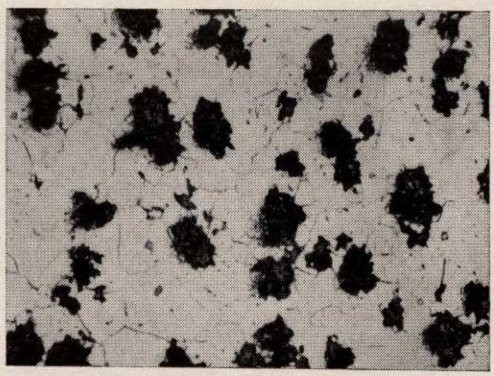


FIG. 34 — PHOTOMICROGRAPH OF CAST IRON INOCULATED WITH COPPER AND ANNEALED FOR 40 HR AT 900°C. × 450

TABLE 28 — DATA OF MANUFACTURING CONDITIONS AND RESULTS OBTAINED FOR MAKING COPPER POWDER BY CONTINUOUS ELECTRO-DEPOSITION

COMPOSITION OF THE ELECTROLYTE			TEMP. OF ELECTROLYSIS °C.	VOLTAGE OF THE CELL VOLTS	SIEVE U.S.S. −100 +140	ANALYSIS OF THE POWDER MADE			APPARENT DENSITY OF THE POWDER MADE g/c.c.
Copper g/l initial	Sulphate g/l final	Sulphuric acid g/l				Sieve Fraction % by wt. −140 +200	−200 ++270	−270	
40·0	73·0	170	30	1·0-1·1	—	10·7	30·9	58·5	2·36
40·0	—	140	45 to 50	0·6-0·7	3·8	10·2	17·1	68·9	—
30·0	—	140	60	0·7-0·8	—	8·5	23·7	67·8	2·50

TABLE 29 — DATA ON VARYING MOULDING AND SIZING CONDITIONS, VARYING CHARGES AND SINTERING TEMPERATURES FOR MAKING SPHERICAL POROUS BRONZE BEARINGS UNIFORM IN DIMENSIONS AFTER FINAL SIZING

WEIGHT OF CHARGE gm.	DIA. OF SINTERED BEARING mm.	LENGTH OF SINTERED BEARING mm.	MOULDING LOAD USED lb.		INITIAL SIZING LOAD USED lb.	SINTERING TEMP. °C.
			For sph. part	For cyl. part		
16·85	20·00	21·90	5000	5000	3000	840
16·86	19·88	22·20	6000	5000	3000	840
16·40	19·86	21·68	5000	5000	2500	840
16·37	19·46	21·72	6000	5000	2500	840
16·35	19·76	21·70	5000	5000	2000	830
15·85	19·46	21·20	5000	5000	2000	830

laboratory scale. It was found that since iron powder was a hard material, tools for its powder metallurgy have to be made from highly wear resistant materials.

38.0 Production of Iron Powder from Blue Dust

The project was taken up with a view to produce iron powder utilizing fine iron ore that could not be successfully utilized in blast furnace. Pockets of fine iron powders known as ' Blue Dust ' often found in the iron ore mines and the ores of these pockets are so fine as to pass through 200 mesh mostly. Iron powder could be produced by direct reduction of these ores with coke breeze. The reduction characteristics of Noamundi blue dust is shown in graph (Fig. 35). Iron powder made from Noamundi blue dust after reduction and milling had a chemical analysis of 93 per cent Fe with 5 per cent FeO and 0·8 per cent C. The iron powder of this grade could be upgraded by annealing the powder in an atmosphere of hydrogen, wherein carbon in the powder reacted with the oxide of iron giving a higher final iron content. Hydrogen was passed in a slow stream and also to drive out the product of the reaction. Hydrogen annealing at various temperatures and lengths of time was carried out by taking the powder sample on a porcelain boat heated in a tube furnace over which dry hydrogen was passed. Annealing at 900°C. for 1 hour gave the powder of following analyses:

Fe-96·5%; FeO-0·4%; C-0·11%

About 5 lb. of powder could be hydrogen annealed in one batch by placing the reduced mass in a stainless steel vessel which was heated in an electric furnace and over which dry hydrogen was passed.

Suitability of blue dust received from Hindustan Steel Ltd, Rourkela, was investigated for the manufacture of iron powder. The ore had a composition: Fe, 63·0 per cent,

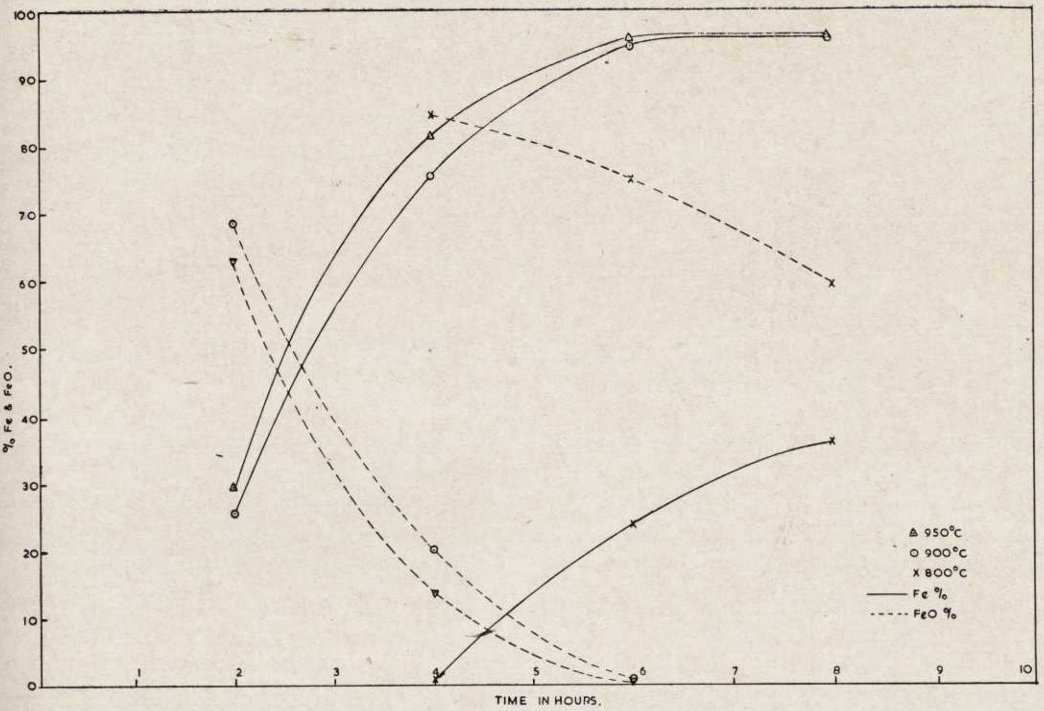


FIG. 35 — REDUCTION CHARACTERISTICS OF NOAMUNDI BLUE DUST AT VARIOUS TEMPERATURES

Al_2O_3, 0·69 per cent, SiO_2, 0·86 per cent. On reduction of this ore at 1000°C. for 7 hours with coke breeze, the powder obtained had the composition: Fe-83·6 per cent, FeO-6·12 per cent. After magnetic separation, an iron content of Fe-87·1 per cent and FeO-3·6 per cent were obtained. An iron ore sample was received from Orissa Mining Corporation for investigating the suitability of the ore for the production of iron powder. Investigation with this ore was in progress.

A preliminary project report on the preparation of iron powder was prepared and circulated to interested industries for undertaking commercial production of iron powder by solid state reduction.

39.0 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 content of non-metallic inclusions in a low and a medium alloy steel.

(i) *Alcoholic-iodine Method* — Several steel samples were examined for their non-metallic inclusion contents by the alcoholic iodine method. The residue of oxide inclusion thus obtained was examined by X-ray, spectrographic and chemical methods. The inclusions residue showed lines pertaining to the following constituents using Co α radiation — FeO, Fe, MnO_2, αAl_2O_3 and aluminium silicate. Results are shown in Table 30.

After chemical analyses of the inclusion residue, total oxygen was calculated from the total oxides and compared with total oxygen obtained by vacuum fusion method, in Table 31.

(ii) *The Chlorine Method* — The chlorine method is valuable for materials containing carbide forming elements which vitiate the alcoholic-iodine method. Work was carried out by the chlorine method on the determination of non-metallic inclusions in plain carbon

TABLE 30 — X-RAY EXAMINATION OF THE INCLUSION RESIDUE

LINE	d-Å VALUE	DUE PROBABLY TO
1	3·18	Aluminium silicate Al_2O_3, SiO_2
2	3·10	MnO_2
3	2·91	—
4	2·71	γAl_2O_3
5	2·49	FeO and γAl_2O_3
6	2·39	MnO_2
7	2·12	FeO
8	2·03	Fe
9	1·98	γAl_2O_3
10	1·88	Aluminium silicate
11	1·86	—
12	1·69	—
13	1·59	MnO_2 and Al silicate
14	1·52	FeO
15	1·45	Fe
16	1·38	MnO_2 and Al silicate
17	1·25	FeO
18	1·221	—
19	1·16	Fe

steels. The apparatus for chlorination consisted essentially of a gas train, chlorination furnace, and ferric chloride collecting vessels. Steels for chlorination were about 5-8 gm. and chlorination time was about 8-12 hours. Generally the sample was taken in the form of a disc 1/10 in. thick. Samples were pickled in 50 per cent HCl, washed in ammonia and then hot water and rinsed in alcohol. There should be minimum delay between preparing the sample and introducing it into the chlorinating apparatus. Chlorination was carried out at a temperature of 400°C. The residue was extracted with a little cold water, filtered under slight suction through a paper pulp pad and washed in the following sequence:

1. 100 c.c. boiling water
2. 100 c.c. hot 3 per cent sodium carbonate
3. 100 c.c. boiling water
4. 100 c.c. cold water
5. 100 c.c. cold 5 per cent hydrochloric acid
6. 100 c.c. cold water

The sodium carbonate wash was used to remove hydrolysis products of any silicon compounds and the hydrochloric acid to remove ferric chloride and sodium compounds. After washing, the residue was ignited at 1000°C. to constant weight.

Two samples of steel were studied for chlorination process. Results are given in Table 32.

Both the above steel samples showed high percentage of inclusions, which by the alcoholic iodine method had given much lower percentage of inclusions (0·06 per cent). It appeared that the inclusions in the above experiments were contaminated with FeO presumably formed due to oxidation of iron

TABLE 31 — SHOWING THE RESULTS OF CHEMICAL ANALYSES OF THE INCLUSION RESIDUE, TOTAL OXYGEN, CALCULATED FROM THE SUM OF OXIDES AND TOTAL OXYGEN BY THE VACUUM FUSION METHOD

CONSTITUENTS	MILD STEEL 1		MILD STEEL 2		MILD STEEL 3		MILD STEEL 4	
	Test 1	Test 2	Test 1	Test 2	Test 1	Test 2	Test 1	Test 2
Total ignited residue %	0·0484	0·0484	0·0502	0·051	0·0859	0·0846	0·105	0·101
Silica (SiO_2)	0·0138	0·0098	0·0090	0·0098	0·0328	0·0513	0·0109	0·0110
Iron oxide (FeO), %	0·0162	0·0194	0·0276	0·0254	0·0130	0·0155	0·0249	0·0250
Alumina (Al_2O_3), %	0·0138	0·0151	0·0110	0·0119	0·0373	0·0143	0·0293	0·0300
Manganese oxide (MnO), %	0·0024	0·0031	0·0027	0·0025	0·0016	0·0017	0·0090	0·0090
Total	0·0461	0·0474	0·0503	0·0496	0·0906	0·0828	0·0741	0·075
Total oxygen, %								
(a) Calculated from the sum of SiO_2, FeO, Al_2O_3, MnO	0·018	0·017	0·017	0·017	0·038	0·038	0·027	0·027
(b) By vacuum fusion method	0·019	0·018	0·019	0·019	0·039	0·039	0·028	0·028

TABLE 32 — SHOWING THE RESULTS OF CHEMICAL ANALYSES OF THE INCLUSION RESIDUE, OBTAINED BY CHLORINATION METHOD

CONSTITUENTS	M.S. I					M.S. II				
	Test No. 1	Test No. 2	Test No. 3	Test No. 4	Test No. 5	Test No. 1	Test No. 2	Test No. 3	Test No. 4	Test No. 5
Ignited residue, %	1·32	1·40	4·7	3·5	2·78	2·9	1·2	1·3	1·1	1·3
Silica (SiO_2), %	12·5	12·3	7·9	4·3	15·5	5·7	4·2	4·5	5·0	4·6
Iron oxide (FeO), %	68·8	46·5	78·2	70·10	63·1	81·0	85·5	80·3	81·1	80·3
Alumina (Al_2O_3), %	6·8	11·5	2·7	2·50	8·4	3·7	2·9	2·1	2·0	1·9
Manganese oxide (MnO), %	5·3	2·1	1·4	1·90	2·1	2·5	1·9	1·8	1·7	1·6

or contamination of chlorine with oxygen. The chlorination arrangement was slightly modified to prevent oxidation and further experiments are in progress in order to standardize the chlorination technique and get uniform results.

The alcoholic-iodine method had given satisfactory results with mild steel samples as shown in Table 31. Arrangements were made to procure special steels deoxidized with known deoxidizers for studies of inclusions in various parts of the ingot.

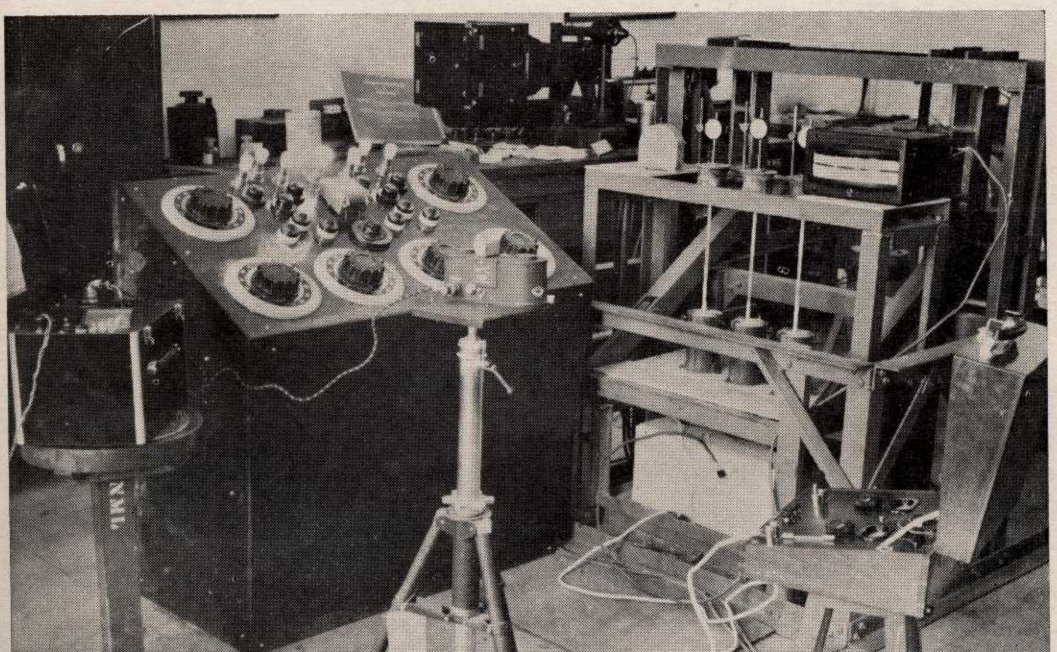


FIG. 36 — SET-UP FOR DETERMINATION OF ISOTHERMAL TRANSFORMATION CHARACTERISTICS OF PLAIN CARBON AND ALLOY STEELS BY DILATOMETRIC METHOD

40.0 Study of Isothermal Transformation Characteristics of Indian Steels

The project was taken up with a view to prepare an Atlas on isothermal transformation characteristics of Indian plain carbon and alloy steels. Dilatometric, metallographic and hardness techniques were followed for the determination of the diagrams. Dilatometric and metallographic method were followed for fast transforming steels. For slow transforming steels, only metallographic method is used.

A new quenching dilatometer designed and fabricated in the laboràtory was set up. In this apparatus three specimens could be simultaneously examined. Quenching of the specimens from the austenitizing furnace to the quenching bath was done automatically. T.T.T. curves of the steels (see table on p. 85) were completed during the year (Figs. 37-41).

Jominy end-quench tests were performed to assess the hardenability of the alloy steels. Steels of the compositions given on p. 86 (upper table) were tested (Figs. 42-45).

At present the steels given on p. 86 (lower table) are under study.

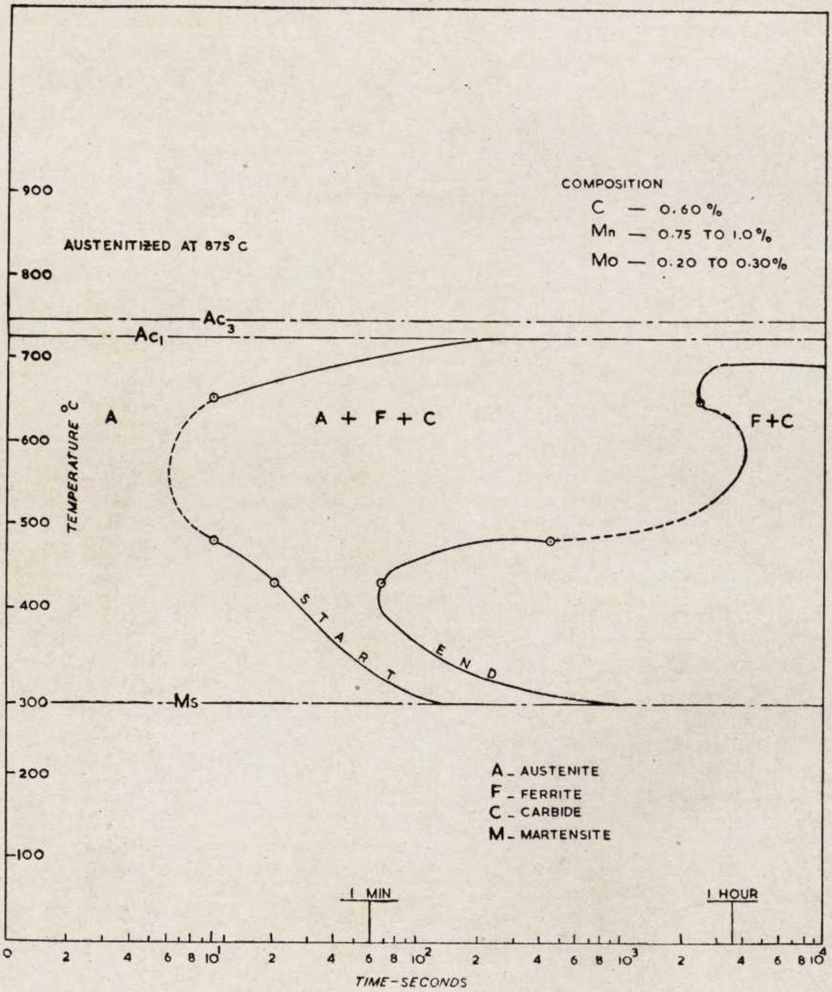


FIG. 37 — T.T.T. CURVE OF MOLYBDENUM STEEL

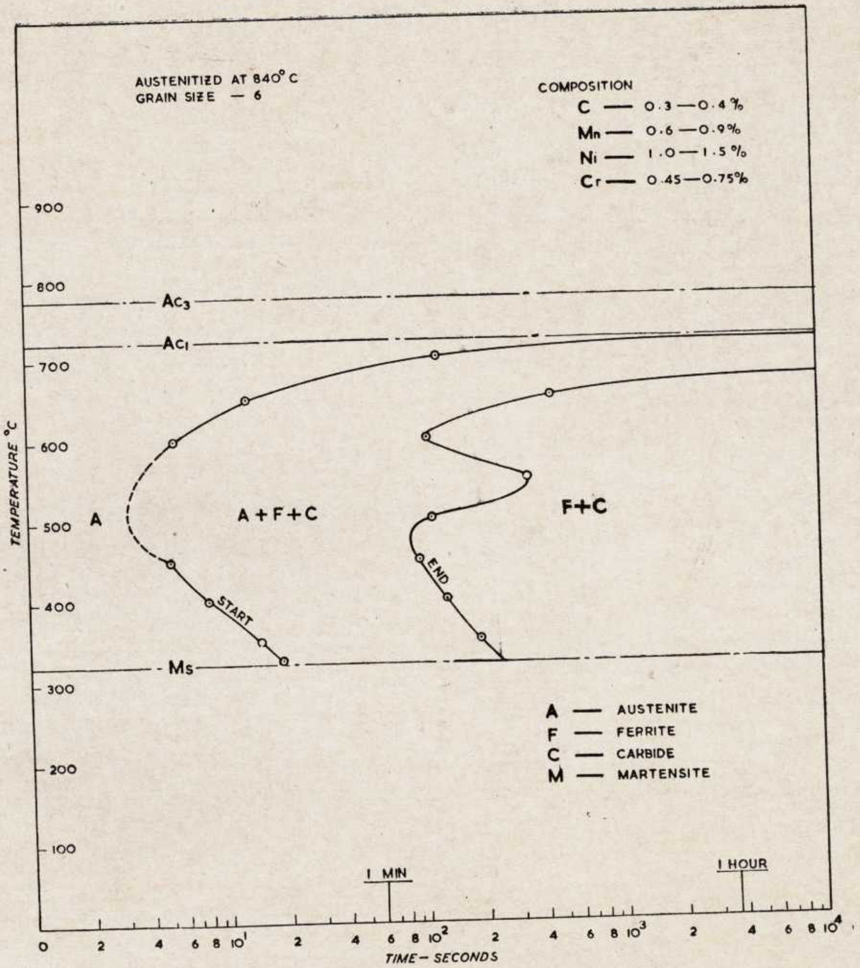


FIG. 38 — T.T.T. CURVE OF Ni-Cr STEEL (A11·5)

40.1 Continuous Cooling Transformation Study of Indian Alloy Steels

Isothermal transformation diagrams have contributed to a better understanding of transformation characteristics of steels at constant temperature and found very useful for defining special heat treatments. However, the data obtained are not readily applicable for formulating heat-treatments involving transformation under continuous

	C	Si	Mn	Ni	Cr	Mo
1) Mo steel	0·60	—	0·75/1·0	—	—	0·2/0·3
2) Ni-Cr steel (A 11·5)	0·3/0·4	0·1/3·35	0·6/0·9	1·0/1·5	1·45/0·75	—
3) Ni-Cr-Mo steel (A 12·5)	0·31	0·12	0·48	2·66	0·61	0·44
4) Ni-Cr-Mo high tensile steel (A 12·4)	0·40	0·19	0·59	1·65	1·21	0·44
5) Carbon tool steel	1·32	0·24	0·42	—	—	—

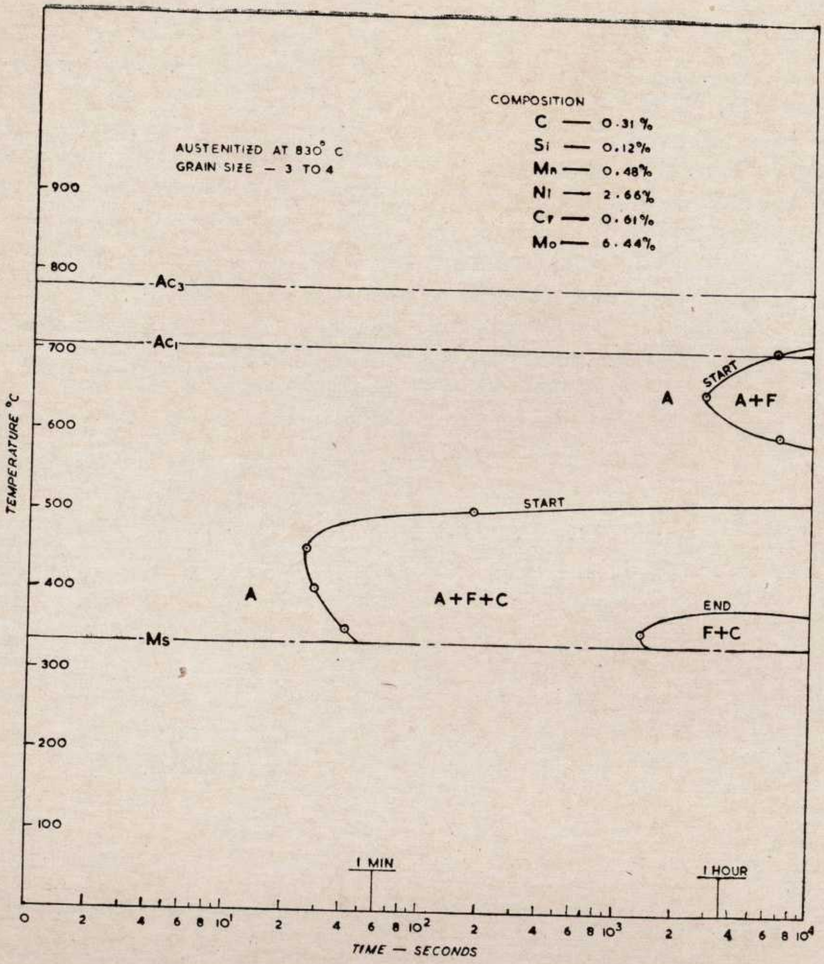


FIG. 39 — T.T.T. CURVE OF Ni-Cr-Mo STEEL (A12·5)

	% Composition					
	C	Si	Mn	Ni	Cr	Mo
1) Mo steel	0·60	—	0·75/1·0	—	—	0·2/0·3
2) Ni-Cr-Mo high tensile steel	0·40	0·19	0·59	1·65	1·21	0·44
3) Ni-Cr-Mo steel	0·31	0·12	0·48	2·66	0·61	0·44
4) Ni-Cr steel	0·3/0·4	0·10/0·35	0·6/0·9	1·0/1·5	0·45/0·75	—

	% Composition					
	C	Si	Mn	Ni	Cr	Mo
1) 1½% Mn steel (A 2·5)	0·42/0·52	0·10/0·35	1·3/1·7	—	—	—
2) Mn-Mo steel (A 3·2)	0·30/0·40	0·10/0·35	1·3/1·7	—	—	0·35/0·55
3) Cr-steel (A 5·5)	0·35/0·45	0·10/0·35	0·60/0·90	—	0·90/1·20	—
4) Cr-Mo steel (A 7·2)	0·35/0·45	0·10/0·35	0·50/0·80	—	0·90/1·20	0·20/0·35
5) Ni-Cr-Mo steel (A 12·3)	0·35/0·45	0·10/0·35	0·40/0·70	1·2/1·6	0·9/1·3	0·10/0·20

cooling conditions. Moreover, most of the practical heat treating processes of steel consisted of austenetizing the specimen followed by continuously cooling in the conventional media. The work was undertaken to study the transformation characteristics of Indian alloy steels under continuous cooling conditions. Liedholm method was followed to study the transformation under continuous cooling condition. Cooling rates at different positions of the standard Jominy specimen was observed during end quenching. In the second set of experiments, Jominy specimens were end quenched for a predetermined time, followed by immersion quenching in the brine water. Metallographic examination and hardness survey were done along the length of the Jominy specimen to draw the curve. Continuous cooling transformation diagrams of the alloy steels given on p. 88 are being made at present.

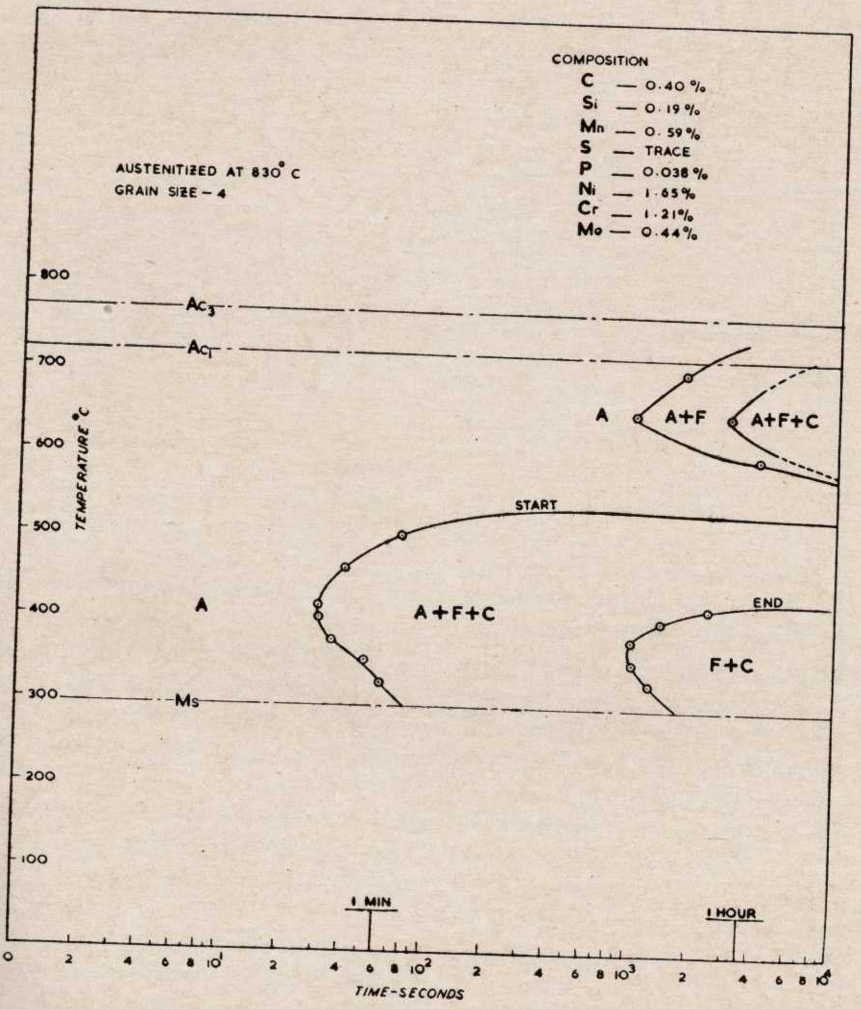


FIG. 40 — T.T.T CURVE OF Ni-Cr-Mo-HIGH TENSILE STEEL (A12·4)

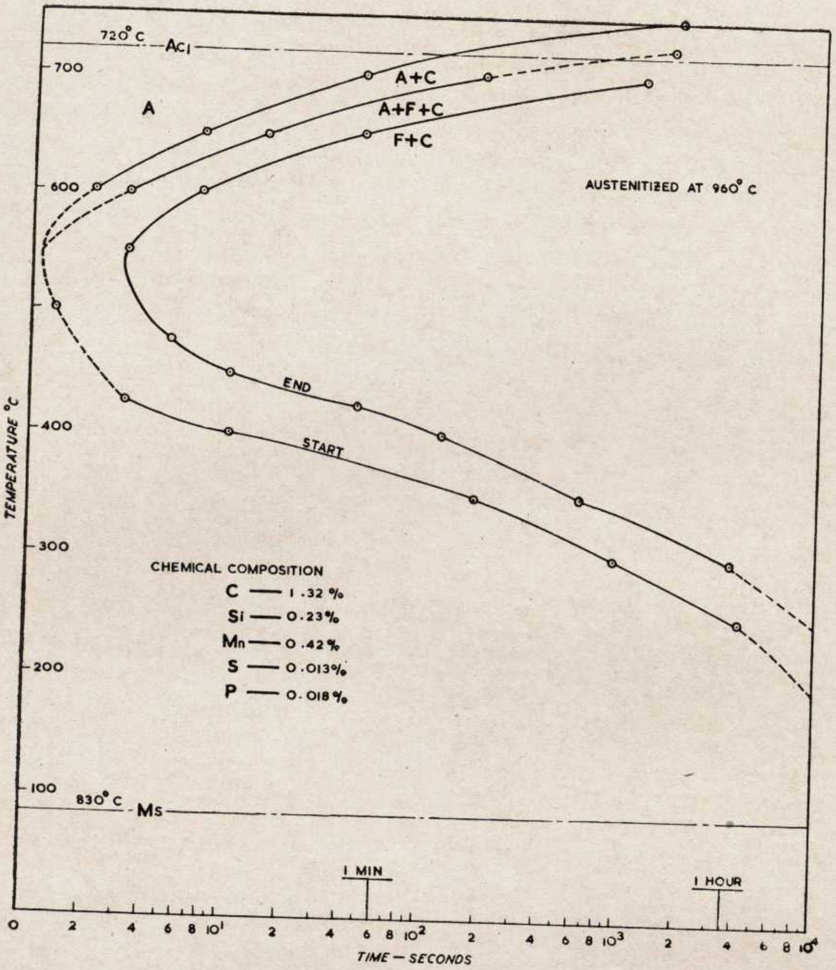


FIG. 41 — T.T.T. CURVES OF HIGH CARBON TOOL STEEL

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

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

The effect of boron on quench-ageing and strain-ageing characteristics was studied. Work was taken up to confirm the influence of boron on the results presented by Morgan

	% Composition						
	C	Si	Mn	Ni	Cr	Mo	V
1) 1½% Mn steel (A 2·5)	0·42/0·52	0·1/0·35	1·3/1·7	—	—	—	—
2) Mn-Mo steel (A 3·2)	0·30/0·40	0·1/0·35	1·3/1·7	—	—	0·35/0·55	—
3) Cr- steel (A 5·5)	0·35/0·45	0·1/0·35	0·6/0·9	—	0·9/1·2	—	—
4) Cr steel (A 6·1)	0·45/0·55	0·1/0·35	0·5/0·8	—	0·9/1·2	—	0·15 min.
5) Ni-Cr-Mo steel (A 12·4)	0·35/0·45	0·1/0·35	0·4/0·7	1·25/1·75	0·9/1·3	0·20/0·35	—

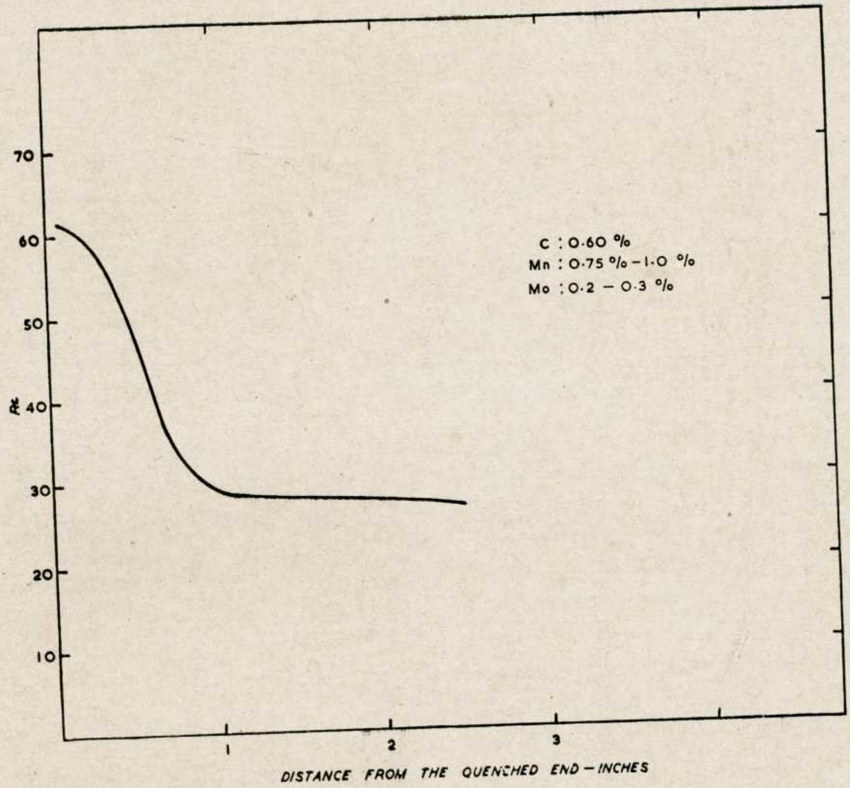


Fig. 42 — Jominy end quench hardenability curve of Mo steel

and Shyne by using ferro-silicon as deoxidizer in place of aluminium. Morgan and Shyne were able to get non-ageing steel with the additions of boron after deoxidizing with small quantity of aluminium. Nine heats were made with different additions of ferro-boron and ferro-silicon in melts of Low-moore iron. The metal was treated in the ladle and poured into ingot moulds where capping device was applied. The ingots were forged and rolled at 1100-1150°C. and homogenized at 930°C. for 4-5 hours and cooled slowly thereafter. Specimens were machined out from the homogenized sheets, the dimensions of which are shown below:

(i) *For Strain Ageing*

(a) Tensile test specimens — flat, $\frac{1}{8}$ in. thickness with 2 in. gauge length.

(b) Electrical conductivity specimens — $\frac{1}{8}$ in. dia. × 6 in. length

(ii) *For Quench Ageing*

Specimens — $\frac{1}{2}$ in. × $\frac{1}{4}$ in. × $\frac{1}{8}$ in.

All the above specimens were annealed at 930°C. for half an hour.

Chemical analyses of the heats are shown in Table 33.

Strain Ageing: (a) *Tensile Test*

The tensile test specimens were prestrained to 5 per cent elongation after which at the intervals of 4 hours. 24 hours. 1 week, 1 month, 2 months, 3 months and 6 months, yield stress and maximum stress values were taken as shown in Table 34.

(b) *Electrical Conductivity*

By straining, the diameters of the specimens were reduced by 5 per cent. The specimens were then aged and conductivity readings were obtained after ageing them for 1 hour, 4 hours, 24 hours, 48 hours, 1 week, 1 month, 2 months, 3 months and 6 months.

TABLE 33 — CHEMICAL ANALYSES OF THE BORON-TREATED STEELS

Heat No.	C %	N %	B %	Mn %	Al %	Si %
1	0·035	0·011	0·0027	0·035	Trace	0·060
2	0·040	0·012	0·0035	0·026	Trace	0·047
3	0·035	0·009	0·0035	0·023	Trace	0·067
4	0·040	0·010	0·0034	0·020	Trace	0·023
5	0·045	0·010	0·0039	0·010	Trace	0·033
6	0·030	0·010	Not determined	0·010	Trace	0·05
7	0·040	0·011	0·0021	0·016	Trace	0·047
8	0·035	0·010	0·0023	0·020	Trace	0·079
9	0·030	0·10	0·0026	0·014	Trace	0·079

The results obtained are shown in Table 35.

Quench Ageing — Specimens were solution treated at 930°C. for 15 minutes and were quenched in ice cooled brine solution after which these were aged at 100°C. The hardness values obtained after ageing them for a period of 1 hour, 4 hours, 24 hours, 48 hours, 1 week, 1 month, 2 months, 3 months and 6 months are shown in Table 36.

The work was further extended by semi-killing the melt by aluminium and then adding ferro-boron. Six heats were made in this series. The ingots were forged, rolled and homogenized as previously. Work on these heats was in progress.

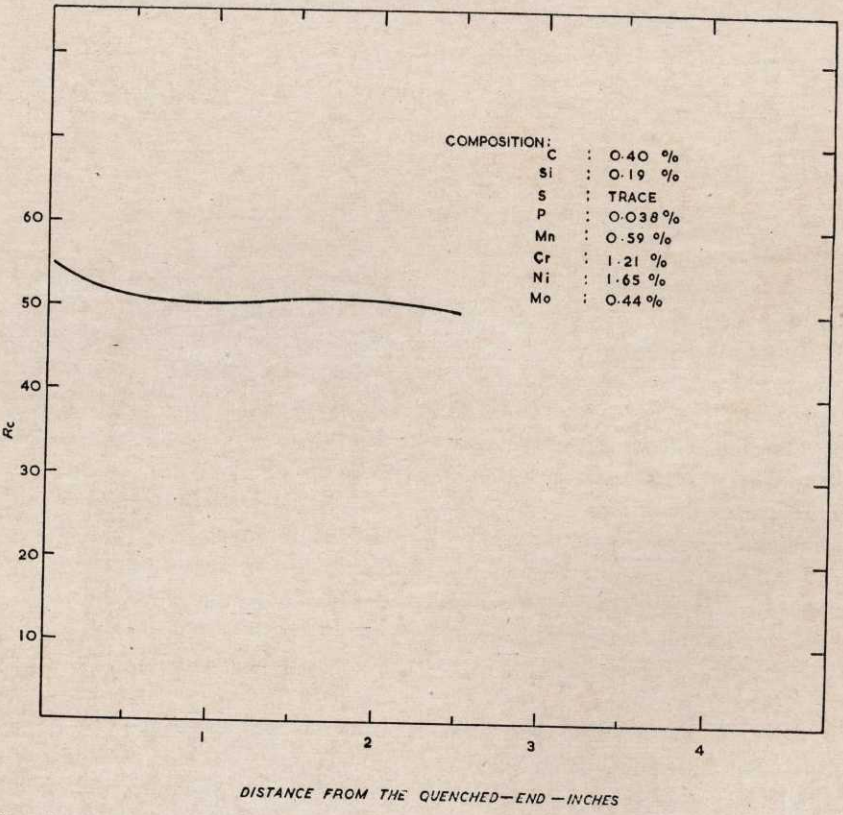


FIG. 43 — JOMINY END QUENCH HARDENABILITY CURVE OF Ni-Cr-Mo HIGH TENSILE STEEL

TABLE 34 — RESULTS OF TENSILE TESTS ON BORON-TREATED STEEL

HEAT No.	MAX. AND YIELD STRESS OF PRESTRAINED (TO 5% ELONGATION) SPECIMENS AFTER THE AGEING PERIOD OF													
	Nil		24 hrs		One week		One month		2 months		3 months		6 months	
	Yield stress TSI	Max. stress TSI	Yield stress TSI	Max. stress TSI	Yield stress TSI	Max. stress TSI	Yield stress TSI	Max. stress TSI	Yield stress TSI	Max. stress TSI	Yield stress TSI	Max. stress TSI	Yield stress TSI	Max. stress TSI
1	22·34	24·3	—	—	—	—	LY-27·11 UY-27·39	29·03	—	—	LY-25·44 UY-25·97	30·53	LY-24·64 UY-26·16	29·76
2	LY-20·59 UY-20·97	23·7	—	—	—	—	LY-22·57 UY-23·41	28·43	—	—	LY-26·04 UY-27·07	29·11	LY-24·85 UY-26·02	29·67
3	LY-22·54 UY-23·54	25·16	—	—	—	—	LY-22·54 UY-22·82	26·00	—	—	LY-25·42 UY-25·88	27·76	LY-22·29 UY-23·32	26·40
4	LY-22·84 UY-23·18	26·04	—	—	—	—	LY-25·24 UY-25·99	27·84	—	—	LY-24·08 UY-24·45	27·79	LY- UY-	—
5	LY-21·58 UY-22·01	24·12	—	—	—	—	LY-23·30 UY-23·78	27·58	—	—	LY-23·83 UY-24·31	28·12	LY-23·74 UY-24·23	28·10
6	LY-22·48 UY-23·02	25·43	LY-23·44 UY-23·83	27·55	LY-24·17 UY-24·53	29·43	LY-24·04 UY-25·36	30·09	—	—	LY- UY-	—	LY- UY-	—
7	LY-20·46 UY-21·13	23·35	LY-23·31 UY-23·69	26·73	LY- UY-	—	LY-22·14 UY-22·74	26·77	LY-22·17 UY-22·69	27·19	LY-22·58 UY-22·94	28·24	LY-23·10 UY-23·49	28·72
8	LY-24·06 UY-24·46	27·28	LY-24·79 UY-25·67	29·66	LY-29·32 UY-29·81	35·18	LY-25·08 UY-25·71	30·24	—	—	LY- UY-	—	LY- UY-	—
9	LY-22·18 UY-22·43	24·90	LY-23·46 UY-23·49	27·05	LY- UY-	—	LY-22·55 UY-22·79	27·83	LY-23·57 UY-24·54	28·53	LY-23·35 UY-23·84	28·28	LY-22·53 UY-22·78	27·78

TABLE 35 — ELECTRICAL CONDUCTIVITY OF THE SPECIMENS OF BORON-TREATED STEEL

HEAT No.	ELECTRICAL CONDUCTIVITY IN MHO/CM. AFTER PRESTRAINING AND SUBSEQUENT AGEING FOR A PERIOD OF								
	1 hr	4 hrs	24 hrs	48 hrs	1 week	1 month	2 months	3 months	6 months
1	76040	—	76381	76510	—	79984	—	81476	79800
2	78091	—	80741	82428	—	82427	—	84737	82427
4	61952	—	62372	61952	—	63958	—	64717	63980
4	74133	—	75171	75684	—	76572	—	77285	77102
5	76741	—	77625	78530	—	79642	—	80194	81102
6	52610	52610	52480	—	53440	52810	53370	83430	53800
7	78450	76400	72300	77780	57820	78630	78760	79000	79110
8	55520	55250	55280	—	55860	54670	55400	55520	55540
9	73310	64220	65670	68530	69020	70080	72800	72610	73580

42.0 Study on Austenitic Grain Size Control in Steels

The mechanism of grain size control by aluminium has attracted the attention of many investigators in this field. The influence of Al present as aluminium oxide or residual aluminium or as aluminium nitride has been a subject of considerable controversy. In recent years, however, it has been

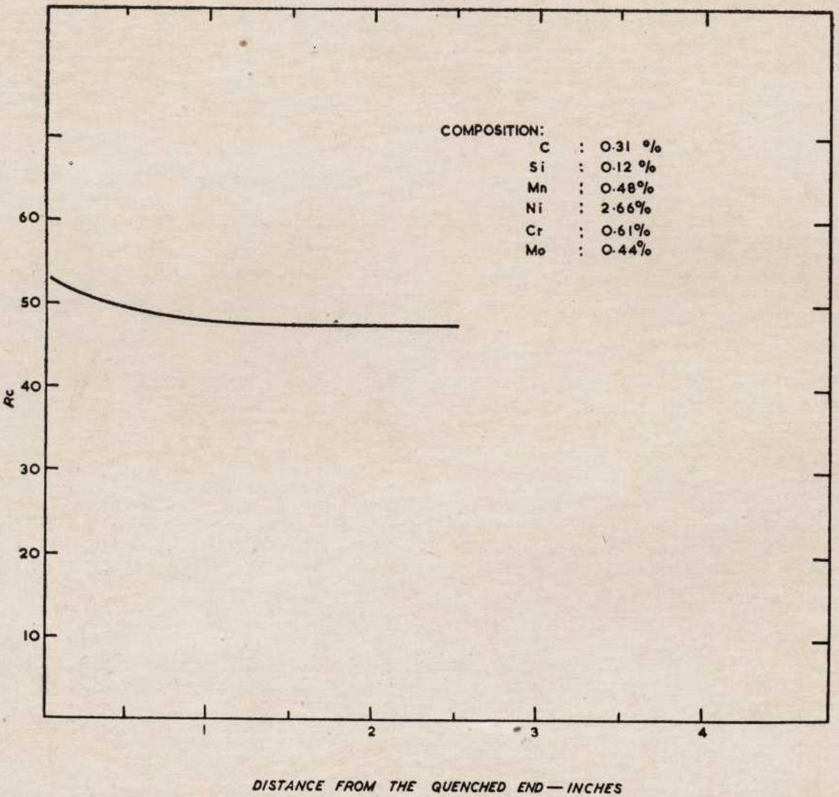


FIG. 44 — JOMINY END QUENCH HARDENABILITY CURVE OF Ni-Cr-Mo STEEL

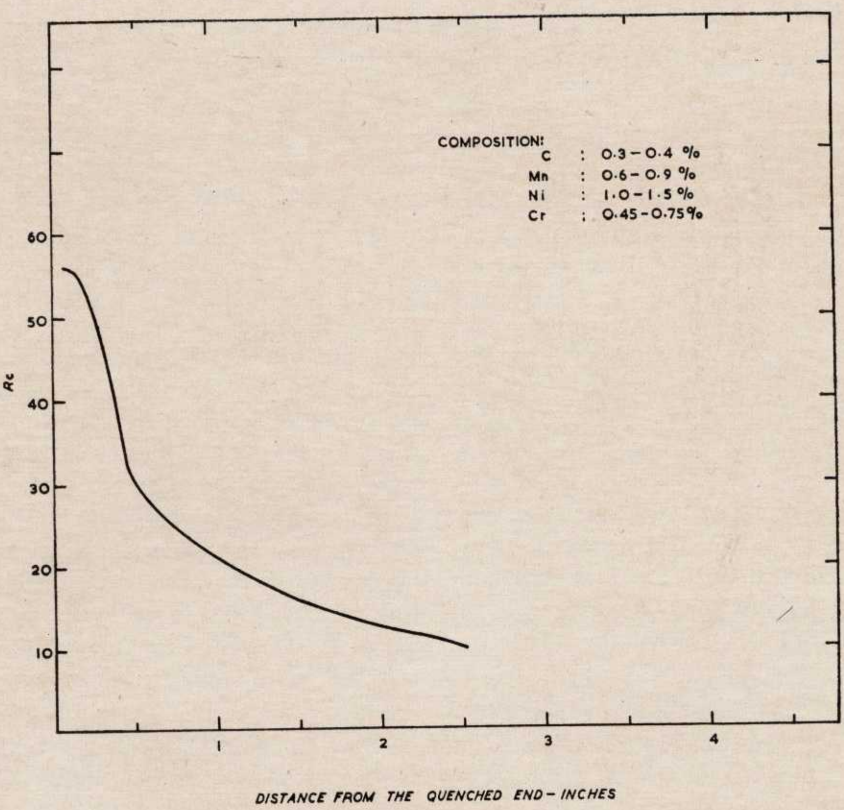


FIG. 45 — JOMINY END QUENCH HARDENABILITY CURVE OF Ni-Cr STEEL (A11·5)

proved that the influence of aluminium nitride as a grain growth inhibitor is the most conclusive one. Some anomalies have been reported by certain workers which required experimental classification.

During the period under review, work was conducted on the air-melted Al-killed steels. The experimental aluminium-killed steels were prepared. Desired Al contents in this melt were reached by additions of Fe-Al

TABLE 36 — HARDNESS (V.P.N.) VALUES OF STRAIN-AGED BORON-TREATED STEEL SPECIMENS

Heat No.	Hardness Values (V.P.N.) of the Quench-aged Specimens after Ageing them for a Period of								
	1 hr	4 hrs	24 hrs	48 hrs	1 week	1 month	2 months	3 months	6 months
1	139	138	137	—	133	135	—	133	133
2	122	124	127	—	125	128	—	128	134
3	132	130	130	—	132	133	—	136	133
4	128	129	130	—	130	133	—	141	138
5	126	126	128	—	127	130	—	132	132
6	127	130	132	133	135	130	126	126	126
7	116	123	118	118	119	119	120	115	118
8	120	123	124	125	125	122	120	120	120
9	128	129	126	124	124	125	123	121	121

TABLE 37 — Al CONTENTS OF THE DIFFERENT EXPERIMENTAL STEELS

Steel No.											
1	2	3	4	5	6	1A	2A	4A	1B	2B	3B
0·147	0·195	0·137	0·152	0·16	0·4	0·052	0·05	0·075	0·028	0·06	0·033

master alloys previously prepared for this purpose, after the melts had first been deoxidized with ferro-silicon. Table 37 shows the Al contents of various heats investigated.

Determinations of the grain sizes were made by McQuid-Ehn test in the temperature range of 800°-1050°C. The maximum grain growth inhibition was exhibited by the steel No. 2B containing 0·06 per cent Al.

Studies were also made on the solubility equilibrium of AlN in both ferritic and austenitic temperatures of Al-killed steels which showed maximum grain growth inhibition. Samples of these steel were nitrided (by soaking at 1050°C. in pure and dry N_2 atmosphere for 24 hours) to ensure the presence of AlN phase in the steel. AlN remaining insoluble at various temperatures from 200° to 1100°C. was estimated by soaking the specimens at these temperatures for 5 hours followed by quenching in water, and subsequently extracting the phase by Beeghly's method of separation of AlN phase from the steel. The extracted residues were analysed for N_2 after carefully dissolving them in H_2SO_4 solution.

From 200°C. to 600°C. (i.e. in the ferritic range), the nitrogen content of the extracted residues had more or less a constant value (the value being very low between 0·002 per cent and 0·004 per cent). From 600°C. to 800°C., the nitrogen content increased very rapidly and then remained constant up to 900° or 950°C., decreasing thereafter and ultimately attaining the original values between 1100°C. and 1200°C. This suggested that the precipitation of AlN took place at initial temperatures in the austenitic range which subsequently decomposed promoting thereby grain-coarsening at these elevated temperatures.

The results obtained so far indicated indirectly that AlN played an important role in grain growth inhibition. Future work will be directed towards elucidating the mechanism of grain growth inhibition. The presence and distribution of AlN were under examination with an electron microscope and by electron diffraction technique after subjecting the specimens to various heat-treatment cycles.

43.0 Study on Age Hardening

(i) *Effect of Specimen Size and Isothermal holding at Elevated Temperature on Room Temperature ageing of Al-Cu Alloys* — The investigation was taken up to study the effects of isothermally holding a specimen at elevated temperatures on the resulting room temperature ageing of Al-3·8 per cent copper alloy of varying thickness. The thicknesses of the samples employed were 0·05 in., 0·1 in., 0·2 in. and 0·5 in. The specimens were isothermally held at 40°, 100° and 170°C. for durations of 1, 10, 30 and 60 minutes after solution treatments at 520°C. for 24 hours. After isothermal holding, the specimens were quenched in iced water and aged at room temperature. From the ageing curves, quenched hardness values, incubation periods and rate of hardening were determined and plotted against thickness and time of holding. Full study of the results was under progress. The results showed that after holding the samples at 40°, 100° and 170°C. for 1 minute, ageing in all the samples proceeded at a fairly rapid speed showing that growth of Guinier-Preston zones took place through vacancies. Vacancies were not completely annihilated by the isothermal holding treatment either at 100° or 170°C. In thick specimens, the ageing rate was comparatively slow. For holding times of 10-60 minutes, at 40° and 100°C., the ageing rate was still appreciable for all the thicknesses studied. At 170°C. for holding periods of 10-60 minutes, the age hardening was not appreciable and conclusion was that most of the vacancies were annihilated during isothermal holding at 170°C. The incubation periods, quenched hardness values and ageing rate were correlated with holding time, holding temperature and specimen size.

(ii) *Reversion in Age Hardening Alloys* — When a fully aged alloy was heated to about 200°C., a decrease in hardness was observed in the first two or four minutes of annealing; the alloy thereafter reverted to its original quenched hardness value. Recently Mondolfo pointed out that the alloy may not revert completely to its original quenched hardness value provided it was aged over a prolonged period. The reason for this behaviour given by Mondolfo was that transformations kept on proceeding in the aluminium-copper alloy that changed the structure towards that corresponding to high temperature ageing. Mondolfo's alloys were comparatively richer in solute atoms and it was thought necessary to investigate this behaviour in alloys having somewhat less copper content. Further, if initial quenching conditions of such alloys were altered prior to long time ageing, what would be their reversion characteristics ? To study these aspects, specimens with different thicknesses but of the same composition (3·8 per cent Cu) were employed to get different cooling rates. The alloys were aged for five years at room temperature and then reverted at 170°C. From this study, following conclusions were derived:

(i) Specimens with different thicknesses reverted to different hardness values. Thin specimens resisted reversion whilst thick specimens did not.

(ii) A specimen of the same thickness but treated differently showed different reversion hardness.

(iii) A specimen of the same thickness but plastically deformed at room temperature before ageing showed a considerable resistance to reversion. The sample which was not deformed reverted completely to its original hardness values.

A complete report on reversion results was under preparation.

44.0 Study on Temper Brittleness

Retrogression is shown by precipitation hardening systems and took place during early stages of precipitation. If any system showed ' retrogression ', it can be taken to show precipitation. Retrogression in temper brittleness was reported in literature but with some ambiguity. It was the object of this investigation to study if it was shown by steels susceptible to temper brittleness.

The wire wrap bend test apparatus used by Kaddow and Rosenthal for temper brittleness was an adaptation of the apparatus constructed by Zappfee and Haslem for investigating embrittlement of steel due to hydrogen. The apparatus constructed at the National Metallurgical Laboratory was based on the description of the apparatus by Kaddow and Rosenthal and consisted of a semi-circular sheet of perspex with graduations in degrees marked along the periphery and mounted suitably on a stand. At the centre of the semi-circle, a fixed pin 0·1 in dia. was placed so that the test wire when mounted was at a tangent to the pin. The test wire specimen was mounted along the diameter of the semi-circular sheet by two rectangular perspex blocks. One of the blocks was fixed to the perspex sheet whilst the other block was movable. The rotating block was moved by a string attached to the shaft of an electric motor rotating at a slow speed. When the shaft was rotated, the movable block moved over the plate. A longitudinal hole was drilled in the moving arm in which the specimen was inserted whilst a groove housed the specimen in the stationary arm. The specimen was a 16 swg. wire with a notch made in the centre; this notch, when the specimen was mounted in position, would correspond to the position of the pin round which the specimen was wrapped during test. The test was conducted by moving slowly the rotating arm so that the wire specimen was moved round the pin till it broke and the angle at which the specimen broke was noted. The test was carried out at various temperatures and a graph of angle of failure vs temperature of test was plotted and transition temperature was thus determined.

To carry out tests at various temperatures, liquid air or any other low melting point liquid was allowed to fall on the specimen in the region of the notch and the test was thereafter carried out. To ensure uniformity of temperature whilst the test was carried out, a chamber to hold the low melting point liquid was added to the set-up and with this, it was considered possible to carry out the test with temperatures remaining uniform.

A heat of Ni-Cr steel was made and wires were drawn therefrom to make specimens for the wire wrap bend test apparatus. After heat-treatment, tests were carried out in the wire wrap bend test apparatus and it was found that more tests were necessary to standardize the apparatus. A set of impact specimens was also tested to determine their transition temperature. Work was under progress.

45.0 Study on Deformation of Single Crystal

This project was taken up with a view to find relation, if any, between strain hardening capacity due to solute and its valency. It was proposed to investigate this aspect by preparing single crystals of alloys of copper or aluminium with solutes of differing valencies and studying their deformation characteristics. The method was being standardized for such a study.

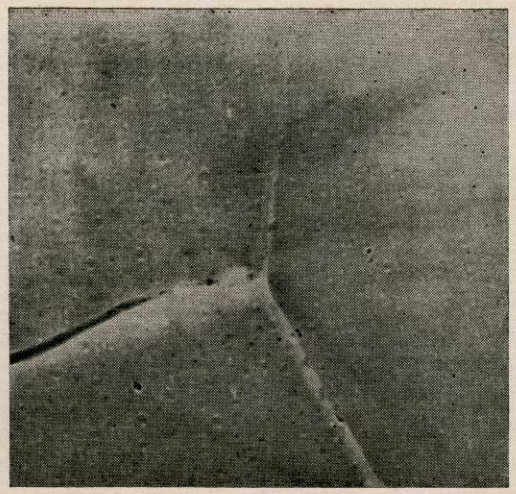


FIG. 46 — ELECTRON MICROGRAPH OF NITRIDED NEEDLES IN M.S. × 6000. FORMER REPLICA (UNSHADOWED)

46.0 Curie Temperature of Iron-Chromium Alloys

This project was taken up with a view to determine the electron distribution of iron contributing to ferro-magnetism. There was a difference in the ferro-magnetic behaviour of iron on the one hand and that of cobalt and nickel on the other. It was felt that curie temperature which was taken to be an index of ferro-magnetism should reflect this difference. The experimental procedure contemplated was to prepare alloys of the required composition, homogenize these alloys and determine their curie temperatures.

47.0 Preferred Orientation in Rolled Sheets

The usual method of determining the preferred orientation produced in cold-rolled sheet was 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 rolling direction.

(i) *Anisotropy of Electrical Resistivity in Cold-rolled* 60: 40 *Brass* — The electrical resistivity of the annealed unworked 60: 40 brass was determined by both the Kelvin bridge and ballistic methods and was found to be 6·84 μ ohm-cm. The percentage increase of the resistivities of the 60: 40 brass sheet cold-rolled to 98 per cent at angles variously inclined to the direction of rolling was calculated from the ballistic values of the resistivities which were almost identical to the Kelvin bridge values at the corresponding angles inclined to the direction of rolling.

The anisotropy of electrical resistivity of 98 per cent cold-rolled 60: 40 brass shown on an arbitrary scale was found to be as in the curve joining the points inside the triangles in Fig. 48, the increase in resistivity in any direction (hkl) being taken as proportional to $(h^2 + k^2 + l^2)^{\frac{1}{2}}$.

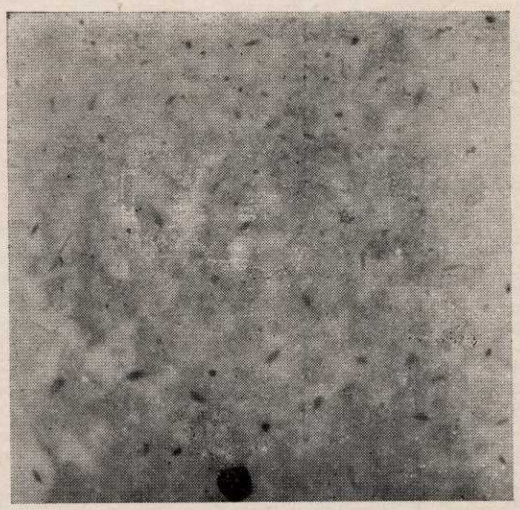


Fig. 47 — Electron micrograph of nitrided needles in M.S. × 10,000. Former replica (unshadowed)

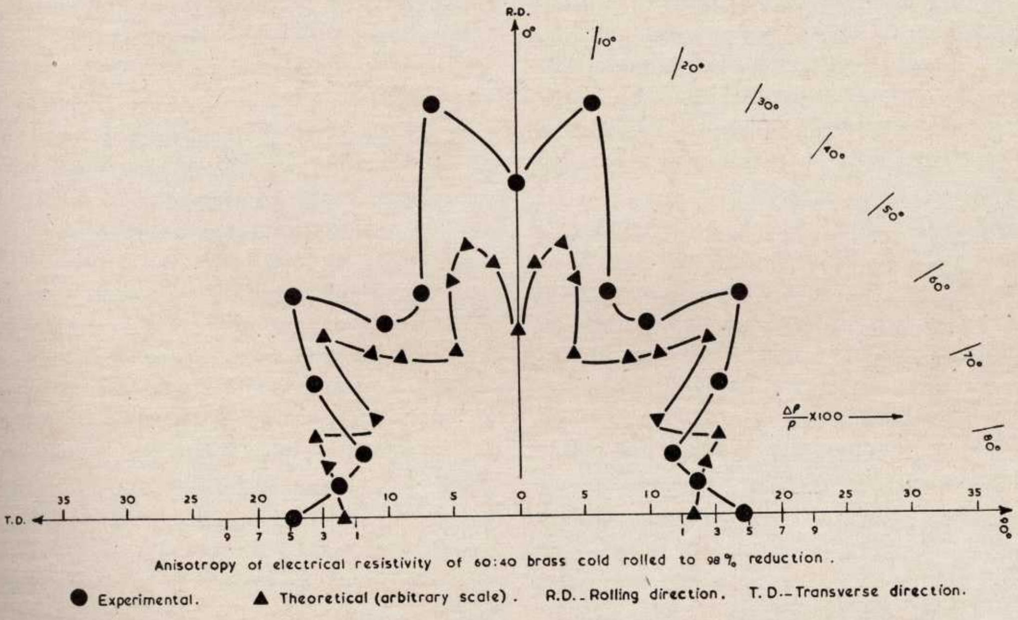


Fig. 48 — Anisotropy of electrical resistivity of 60: 40 brass cold-rolled to 98 per cent reduction

The agreement between the theoretical and experimentally obtained polar plots is worth noting. A somewhat better agreement was possible, if the slight scatter in rolling direction, as indicated by Brick, was also taken into consideration. An exact coincidence was, however, hardly to be expected, since the texture of the β phase, as well as the presence of textures other than (110) [112] in the α phase, were ignored.

It was also interesting to note that the degree of anisotropy in cold-rolled 60:40 brass was not as great between the rolling and transverse directions as between some other directions. The difference between the percentage increase in resistivity in the rolling and transverse directions was 8 per cent whereas at angles of 11° and 22½° and 11° and 56° respectively to the rolling direction, the differences were ~ 13 per cent. The relatively smaller difference between the resistivities in the rolling and transverse directions may also probably explain why cold-rolling was not generally supposed to result in considerable anisotropy, most measurements being apparently confined to these two directions. These directions, in rolled copper, for example, corresponded to directions of almost equal ease of slip, more particularly if the textures (112) [111], (100) [001] considered to be present to some extent along with (110) [112] were also taken into account.

(ii) *Pole Figures for Randomly Oriented Sheets of Silicon Steel and Aluminium* — The pole figures for the polycrystalline randomly oriented sheets of silicon steel and aluminium were drawn for reflections of Mo-Kα radiation from the (110) planes in silicon steel and of Cu-Kα radiation from the (111) planes in aluminium with the calculated intensities of emergent X-rays after varying absorption by the metallic sheets themselves at different angles of the azimuth over the entire (110) Debye cone for the former and (111) Debye cone for the latter at the various angles of tilt of the specimens. The intensities at various azimuth angles were calculated based on a graphical evaluation of the lengths within the specimen itself of the diffracted beams.

Decker had drawn the pole figure for polycrystalline and randomly oriented silicon steel sheet by a method based on the mathematical calculation of the intensities of the X-rays diffracted through the specimen. The X-rays which considered by Decker to be diffracted from each point, across the entire thickness of the sheet, were considered to be diffracted from the centre of the specimen only. It was also shown that the intensities of the emergent X-rays considered to be diffracted in this way did not differ by even 1 per cent from values obtained if scattering was assumed to occur from various points along the thickness.

The pole figures showing clearly the effect of varying absorption were constructed using angles of tilt α = 11°, 22·5°, 34°, 45°, 57° and 67·5° with silicon steel and α = 10°, 20°, 30°, 40°, 50°, 60° and 65° with aluminium where 'α' is the angle between the sheet normal and the incident X-ray beam.

The emergent intensities for each were classified into four groups as follows:

(a) 75 per cent to 100 per cent of maximum intensity — strong
(b) 50 per cent to 75 per cent of maximum intensity — medium
(c) 25 per cent to 50 per cent of maximum intensity — weak
(d) below 25 per cent of maximum intensity — very weak

The necessary curves for the variation of the ratio of minimum to maximum intensity with the angle 'α' and the variation with 'ϕ' the azimuth angle, of the ratio of the intensity at an angle 'ϕ' to the maximum intensity for a given 'α' were drawn for the first order (110) ring for silicon steel and first order (111) ring for aluminium.

(iii) *Preferred Orientation in Cold-rolled Aluminium Sheets* — This work was taken up with a view to study in detail of the preferred orientation developed during cold-rolling commercial aluminium sheet under various rolling procedures using the conventional X-ray method. The detailed study included: (i) the details of the scatter about the ideal orientations developed during cold-rolling, (ii) the difference if any between the preferred orientation developed on the surface and interior layers of the cold-rolled sheets, (iii) the relation, if any, between the electrical resistivities in various directions with reference to the rolling directions of the rolled sheets and the preferred orientations developed during cold-rolling.

Commercial aluminium sheet of 0·131 in. thickness in the as-received condition was annealed at 550°C. for one hour so as to homogenize the grain size and to eliminate the effects of previous cold working if any. The annealed sheet was then cold-rolled under the following rolling procedures:

(i) To keep the direction of rolling the same in each of the successive passes.
(ii) To cross-roll the specimen after each pass.
(iii) To reverse the direction of rolling after each pass.
(iv) To combine reverse and cross-rolling after every successive pass.

In the first procedure, the thickness of the sheet was reduced from 0·131 in. to 0·010 in. in 19 successive passes resulting in 92·3 per cent reduction; in the second, from 0·131 in. to 0·012 in. in 18 successive passes giving 90·8 per cent reduction and in the last two, from 0·131 in. to 0·11 in. in 18 successive passes resulting in 91·6 per cent reduction.

Various X-ray transmission patterns necessary for drawing the pole figures were taken with the interior and surface layers of the rolled sheets placed respectively with their rolling directions vertical and horizontal and tilted to various angles with respect to the incident X-ray beam. A specimen for interior layer was obtained by dissolving to the requisite extent both the surfaces of the rolled sheet while for one for surface layer was obtained by dissolving from one surface only of the rolled sheet, the other surface being protected with a layer of paraffin wax.

The variation of intensity over the (111) and (200) Debye rings of the X-ray transmission photograph was measured as a function of the azimuth angle. The intensity was classified into three groups — (i) strong, (ii) medium, and (iii) weak.

The (111) and (200) pole figures both for the surface and inside textures developed during cold-rolling under the rolling conditions for the first three mentioned above were drawn. The drawing of the pole figures for the sheet cold-rolled under the last rolling procedure and the interpretation of the pole figures were undertaken. The determination of electrical resistivities at angles variously inclined to the rolling directions of the above cold-rolled sheets was in progress.

48.0 Measurement of Carbon Activity in Iron and Its Alloys

Under the C.S.I.R. Research Fellowship Scheme work was initiated on equilibrium study between carbon dissolved in iron and definite mixtures of carbon monoxide with carbon dioxide which was 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 gamma iron, heat of transformation of the different forms of iron and nature of the crystal structure, etc.

It was proposed to study the carbon activity in iron and its alloys at different temperatures. The mechanical pump used in the set-up was found to have developed leaks and the gas mixture in the system was always contaminated with oxygen and nitrogen. The mechanical pump was replaced by a magnetic diaphragm pump and the system was found to work satisfactorily.

The flow meters were also replaced by more sensitive flow meters and butyl phthalate was used in the flow meters. Different types of tubes were also used in the furnace. It was observed that the used silica tubes gave the best result and no carbon deposition was observed in the system. Studies were also carried out to determine the optimum circulation rate for preventing any carbon deposition. Experiments were in progress for determining the carbon absorption, to arrive at the maximum time required for each reaction. Some of the typical results are indicated in the following table.

TABLE 38 — EXPERIMENTAL RESULTS OF EQUILIBRIUM OF CO-CO_2 MIXTURES WITH AUSTENITE AT 1000°C.

Expt No.	P_{CO} = Vol. % CO	P_{CO_2} = Vol. % CO_2	P_{CO}/P_{CO_2} in Atmos.	Carbon in wt %
31	68·88	32·8	1·244	0·02
37	70·97	25·72	1·958	0·06
35	78·56	19·15	3·223	0·22
28	91·31	8·69	9·921	0·25
26	90·29	9·71	8·472	0·25
27	93·33	6·97	12·42	0·28
29	98·0	1·8	53·34	1·12

The activity of carbon in iron will be measured at different temperatures and thereafter the binary alloys like iron-silicon, iron-chromium, iron-manganese will be taken up.

49.0 Solubility of Nitrogen in Plain Carbon and Alloy Steels

Under the C.S.I.R. Research Fellowship Scheme a study was taken up with a view to determine the solubility of nitrogen in nickel-free austenitic Cr-Mn-N-Cu stainless steel in solid and liquid state.

The preliminary experiments carried out on the Sievert's type apparatus consisted in the determination of hot volume of the closed system, and the volume of nitrogen absorbed by the system sample. It was observed that the solid sample was invariably coated with the oxygen and the volume measurements could not be used to determine the nitrogen solubility. Moreover, the oxide coating interfered with the nitrogen solubility. These experimental difficulties were due to presence of even traces of oxygen in the nitrogen. The stainless steel sample was thereafter surrounded by chromium powder and no oxidation of the sample was observed. The experimental procedure was modified so that the sample was kept in between but away from two capsules containing chromium powder. The sample was equilibrated with purified nitrogen gas at the desired temperature. After the reaction time, the sample was quickly removed from the hot zone, quenched in water and analysed for the nitrogen content.

The solubility of nitrogen at 1 atmospheric pressure on commercial 18/8 stainless steel at 900°-1000°C. was obtained as follows:

Temperature	*% by weight*
900°	1·61
1000°	1·42 1·29

The samples were equilibrated for 50 hours each. Further experiments are in progress to check the attainment of equilibrium and reproducibility of the results and to measure the solubility of nitrogen in nickel free austenitic Cr-N-Mn-Cu stainless steels.

50.0 Solubility of Carbon in Fe-Cr-Si Alloy

High carbon ferro-chrome is produced by smelting chrome ore with carbon, while low carbon ferro-chrome is produced by two stage process comprising the production of an intermediate low carbon Fe-Cr-Si alloy, by reduction of chrome ore and quartz with carbon and its subsequent desiliconization. The extent of carbon pick up depends on the nature and concentration of the alloying constituents and the operating temperature. The present investigation was undertaken under the C.S.I.R. Research Fellowship Scheme to determine the solubility of carbon in the iron-chromium-silicon alloys as a fraction of silicon-content and to elucidate the thermodynamic state in which carbon and silicon co-existed in the metallic solution.

An experimental set up for determining solubility of carbon in metallic melts was assembled utilizing a molybdenum wound furnace. Temperature measurements were made with a calibrated optical pyrometer. Solubility of carbon in liquid iron at different temperatures as determined followed a linear relationship of the type

$$\log N_c^c = \frac{560}{T} - 0{\cdot}375$$

Solubility of carbon was also determined in iron-chromium melts containing up to 30 per cent chromium at various temperatures up to 1600°C.

Solubility of carbon in Fe-Cr-Si alloys containing lower proportions of chromium and silicon were determined. Further experiments on the measurement of carbon solubility in Fe-Cr-Si alloys containing higher percentages of chromium and in Fe-Cr-Si alloys of various compositions would be carried out. These experiments were expected to yield quantitative relationships for the solubility of carbon as a function of the concentration of alloying constituents and temperature.

51.0 Development of Thermostatic Bimetals

Thermostatic bimetals 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 bimetal was increasingly felt, but still the entire requirements were met by imports. This project was, therefore, undertaken to develop suitable manufacturing techniques for different types of bimetals.

During the year under review, considerable progress was made to achieve the following objectives:

(a) Development of suitable melting and casting technique for high expansion Mn-Cu-Ni alloys.

(b) Improvement in the working technique for Mn-Cu-Ni alloys, Fe-Mn-Ni alloys and invar alloys.

(c) Study of physical and mechanical properties of Mn-Cu-Ni alloys.

(d) Bonding of all-ferrous bimetals by using bigger-slabs than done earlier.

(e) Bonding of Mn-Cu-Ni alloy with invar.

(f) Bonding of brass and invar.

(i) *Development of Suitable Melting and Casting Technique for Mn-Cu-Ni Alloys* — High expansion Mn-Cu-Ni alloy, owing to its very high thermal expansion, high electrical resistivity and good mechanical properties, was eminently suitable for making high sensitivity bimetals. But these properties combined with high solidification shrinkage, poor thermal conductivity, high viscosity of the molten metal and thick scum formation over the melt, were mainly responsible for the difficulties encountered during melting and casting of these alloys. Appearance of cold shots, hot tears, lapping, hairline cracks, unweldable secondary piping and oxide inclusions were some of the difficulties encountered. After a number of trials, following measures were found adequate for eliminating the defects:

(a) proper control of casting and teeming temperature,

(b) proper deoxidation of the melt,

(c) use of ingot mould with suitable diameter to length ratio, and

(d) use of suitable size hot top.

(ii) *Improvement in the Working Technique for Various Alloys* — For invar and high expansion ferrous alloys, suitable hot working temperature was worked out. Ingots were soaked initially at a predetermined temperature in electric furnace for a period depending on thickness and later transferred to gas furnace at higher temperature, where it was

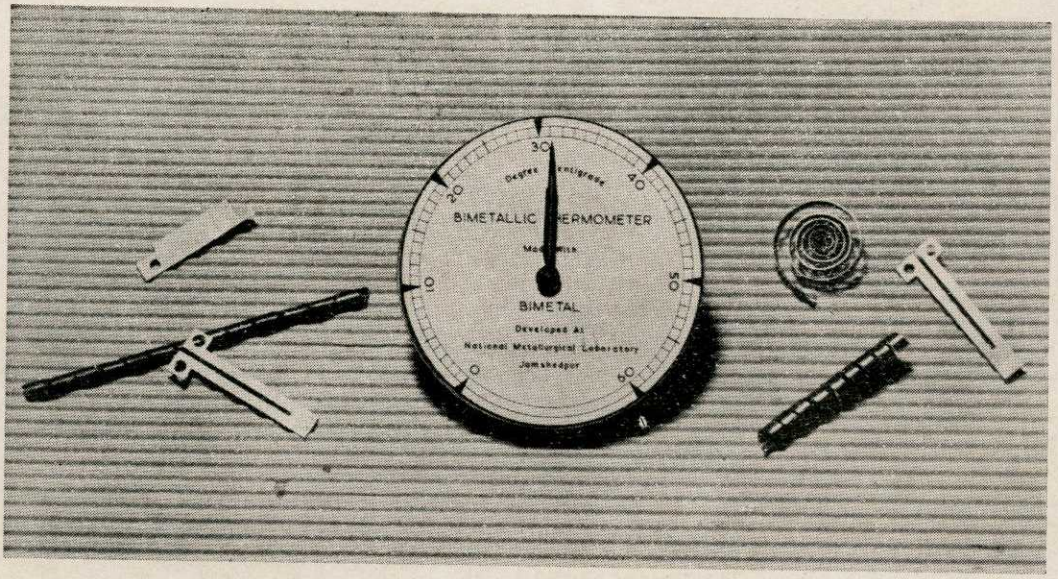


FIG. 49 — BIMETALLIC THERMOMETERS FABRICATED WITH BIMETALS DEVELOPED AT THE NATIONAL METALLURGICAL LABORATORY

held for a small interval of time prior to working. For high expansion Mn-Cu-Ni alloys hot working temperature was found to be 880°-850°C.

(iii) *Study of Physical and Mechanical Properties of High Expansive Mn-Cu-Ni Alloys* — Coefficient of thermal expansion vs temperature curve was drawn for five alloys of this group; and the compositions 75 per cent Mn, 15 per cent Ni, 10 per cent Cu and 72 per cent Mn, 10 per cent Ni, 18 per cent Cu were selected. For all these alloys thermal expansion showed a stiff rise at a temp. of 300°C. This sudden change is supposed to be due to order-disorder transformation. Mechanical properties determined are given as follows:

Composition: 72 per cent Mn, 18 per cent Cu, 10 per cent Ni

Coeff. of expansion (30°-100°C.)	26 cm × 10^{-6}/°C.
Tensile strength	1,10,000 p.s.i.
Proof stress	95,000 p.s.i.
Hardness after 50 per cent cold roll	240 V.H.N.
Softening temp. for 50 per cent cold work and one hr annealing	650°C.

Hardness of an annealed specimen was 120 V.H.N. After ageing at 400°C. for one hour, hardness value rose to 170 V.H.N. Further studies were made to obtain a full understanding of the thermal and mechanical stability of these alloys.

(iv) *Bonding of All-ferrous Bimetals by Using Bigger Slabs* — Some attempts were made to bond 20 per cent Ni, 6 per cent Mn alloy with invar by using slabs of the size 5 in. × 5 in. × 0·3 in.-0·4 in. The reduction attainable with smaller rolling mill was insufficient to effect any bonding in slabs of this thickness. Slabs of maximum thickness up to 0·2 in. could be bonded. However, good bonding with thicker slabs could be obtained by pressing the pack (between a pair of syndiana board 1 in. thick) to a pressure of 5 tons/in.^{2} at a temperature of 1150°C. followed by subsequent rolling by the smaller rolling mill. Attempts were made to bond slabs of the size 5 in. × 8 in. × 0·5 in. by using bigger rolling mill, so that pressing operation might be avoided.

(v) *Bonding Mn-Cu-Ni Alloys with Invar*— The chief difficulty encountered in bonding Mn alloys with invar by hot rolling technique was the problem of welding the periphery, as iron formed a brittle phase with Mn alloy during welding, resulting in the failure of the weld during first stage of hot rolling. Use of suitable electrodes improved the situation. In most experiments, perfect bonding was not obtained. Formation of Mn oxide during welding was suspected to be the main cause. Attempts were made to overcome this difficulty by taking nickel-plated Mn alloy. Experiments were undertaken to obtain a suitable bond by casting Mn alloy over invar.

(vi) *Bonding of Brass-Invar and Bronze-Invar Bimetals*—Several attempts were made to obtain bond between brass and invar by hot rolling technique by using 4-ply pack. It was possible to obtain brass-invar bimetal by hot rolling at 820°C. But brass surface was very badly damaged owing to working at such high temperature. It may be possible to obtain brass-invar bimetal at lower temperature if heavier reduction could be given. Hot rolling technique for bonding brass-invar is not considered suitable for industrial use. Attempts were made to obtain brass-invar and bronze-invar bimetal by fusion or casting method.

52.0 Development of Clad Metals

The process of cladding aluminium on mild steel and the optimum conditions required for obtaining good bond strength had been earlier described. 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 bimetallic bearing. In this connection, attempts were made in the first instance to make a suitable aluminium-base bearing material with improved bearing properties. Several heats were made in the gas-fired furnace. Various mechanical tests, viz. tensile,compression,antiseizure, etc., were carried

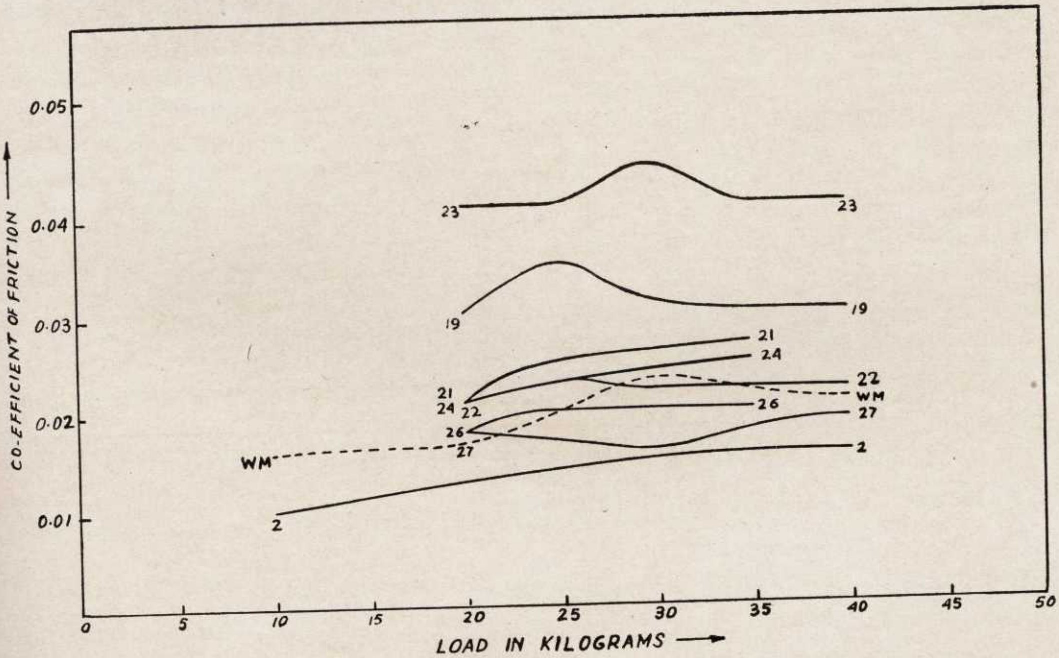


FIG. 50 — COMPARISON OF COEFFICIENT OF FRICTION AT DIFFERENT LOADS OF COMPOSITIONS MADE WITH THAT OF WHITE METAL

TABLE 39 — RESULTS OF MECHANICAL TESTS OF VARIOUS HEATS

Heat No.	Ultimate Tensile Strength tons/sq. in.	Elongation %	Young's Modulus ×10^6 lb./sq. in.	Hardness in B.H.N.	Coefficient of Friction (under Lubrication)	Remarks
AB 14	23·94	1·56	10·5	114	0·0236	
AB 15	15·11	10·00	10·8	78	0·0247	
AB 16	15·84	2·50	10·0	78	0·0237	
AB 17	—	—	—	95	0·026	No further tests have been carried out as the ingot was full of blow holes
AB 18	—	—	—	—	—	do
AB 19	21·44	3·12	10·6	104	0·03	
AB 20	10·43	3·12	8·4	60·6	0·05	
AB 21	13·94	3·12	6·4	81·3	0·025	
AB 22	10·36	18·75	7·9	25	0·022	Hot-rolled at 250°C. Further it was cladded with steel at 450°C.
AB 23	—	—	—	—	0·042	After observing high frictional values, further tests were abandoned
AB 24	4·69	12·5	6·7	25	0·023	Hot-rolled at 250°C. Further the material was cladded with steel at 450°C.
AB 25	6·85	10·93	7·0	38·8	—	
AB 26	5·04	31·25	6·9	24·2	0·02	Hot-rolled at 250°C. Further the material was cladded with steel at 450°C.
AB 27	10·00	11·00	8·0	40	0·018	

out to determine the frictional and other properties of the alloys. The results obtained in mechanical tests are given in Table 39. Coefficient of friction at different loads obtained from the alloys made, was compared with the well established bearing alloy i.e. white metal and shown in Fig. 50.

Metallographic examination of the alloys revealed heterogenous structures. The alloys AB 22, 24 and 26 showed uniform dispersion of lead and antimony in the white etched matrix of aluminium. Dendritic structure showing as cast condition was observed for the alloys AB 23, 25 and 27. The structure

TABLE 40 — CRUSHING STRENGTH OF THE GRAPHITE AND P.T.F.E. BUTTONS

S.P. No.	Percentage Composition of the Composite Buttons	Tons/ sq. in.
1	Graphite, 20% ; rest, bakelite powder	6·5
2	Graphite, 30% ; rest, bakelite powder	6·1
3	P.T.F.E., 20% ; asbestos, 5% ; rest, bakelite powder	5·8
4	P.T.F.E., 30% ; asbestos, 5% ; rest, bakelite powder	4·2
A	Graphite, 20% ; PbO, 5% ; asbestos, 1% ; rest, bakelite powder	4·7
B	Graphite, 30% ; PbO, 5% ; rest, bakelite powder	3·5

TABLE 41 — FRICTION TESTS OF THE BAKELITE BUTTONS

Materials	Percentage Composition of the Composite Buttons	Matting Metallic Material	No. of Hours	Coefficient of Friction
Bakelite buttons	Graphite, 20% ; rest, bakelite powder	Grey cast iron	10	0·18-0·23
Bakelite buttons	Graphite, 30% ; rest, bakelite powder	do	8	0·17-0·23
Bakelite buttons	P.T.F.E., 20% ; asbestos, 5% ; rest, bakelite powder	do	20	0·13-0·16
Bakelite buttons	P.T.F.E., 20% ; asbestos, 5% ; rest bakelite powder	do	4	0·15-0·19

consisted of deep etched black-patches probably lead and antimony over a solid solution of copper in aluminium. The alloys AB 20 and AB 21 exhibited heterogeneous structure together with the eutectics of aluminium and silicon. AB 17 and AB 18 were full of micro porosity. The alloys AB 22, 24 and 26 satisfied all the requisite properties of a soft bearing material; in addition they could also be forged, rolled and bonded with steel following the above-mentioned technique. Further work in standardization of a technique to obtain the uniform product and also to develop alloys of the requisite bearing properties was in progress.

53.0 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.

Due to failure of stearic acid lubricants in the service condition as encountered in railways, the work was redirected to use a solid lubricant which is expected to be stable under the service condition. As reported earlier, use of firstly the P.T.F.E. powder or graphite in various proportions with the thermosetting bakelite powder and secondly the $PbO + SiO_2 + FeO$ coating on mild steel base bonded by sintering at temperature of 900°F. under a neutral atmosphere were resorted to. The second method did not prove satisfactory whereas the first method gave desirable coefficient friction. Further work was done regarding the composite bakelite compacts using graphite and P.T.F.E. powder along with a little of asbestos to toughen the compact. Some experiments were conducted with the whole compact made of a given composition and some were carried out only with the surface layer of the given composition, the remaining portion being of bakelite. Results of compressive strength and friction tests are given in Tables 40 and 41 respectively. Since PbO was also expected to show good lubricating quality under conditions of the experiment, it was also incorporated in some of the experimental buttons, prepared by sintering under pressure the mixture of bakelite plus PbO plus graphite with and without asbestos. These were again tested for frictional property at 300 lb./sq. in. normal pressure and the results are given in Table 42.

From the results obtained it was seen that the combination of 5 per cent PbO, 20 per cent graphite gave reasonably low value of eoefficient of friction which remained steady even after long time of run. Increase of graphite resulted in some small increase in friction at the initial stage. Incorporation of P.T.F.E. powder gave lower value of coefficient of friction in all cases. Further work was carried out for making larger compacts of the most suitable composition, so that the same could be tested under actual service.

54.0 Study on Internal Friction of Metals

The aim of the project is to study the phenomena of strain ageing, temper embrittlement and the various interactions of

TABLE 42 — FRICTIONAL PROPERTIES OF COMPOSITE BUTTONS

Materials	Percentage of Composite Buttons	Matting Metallic Materials	No. of Hours	Coefficient of Friction
A	Graphite, 20% ; PbO, 5% ; rest, bakelite powder	Grey cast iron	50	0·19-0·18
B	Graphite, 30% ; PbO, 5% ; rest, bakelite powder	do	40	0·20-0·18
C	Graphite, 20% ; PbO, 10% ; rest, bakelite	do	35	0·20-0·18
D	Graphite, 20% ; PbO, 15% ; rest, bakelite	do	50	0·19-0·18

TABLE 43 — TEMPERATURE DEPENDENCE OF INTERNAL FRICTION FOR BISRA IRON

TEMP. °C.	TAN δ $\times 10^{-3}$
22·5	1·68
27·5	1·72
29·7	1·89
31·7	2·10
33·2	2·14
35·2	2·07
42·0	1·95
43·7	1·77
47·0	1·72
55·5	1·39
61·7	1·00
64·0	0·94
68·7	0·95
71·0	0·90

TABLE 44 — VALUES OF S.C.F. AT DIFFERENT DEPTH OF STRESS RELIEVING NOTCHES

DEPTH OF THE LOAD RELIEVING NOTCHES	S.C.F.	% CHANGE IN S.C.F.
0 (single notch)	3·23	—
$\frac{3}{8}''$	3·09	4·3
$\frac{1}{4}''$	3·04	5·8
$\frac{5}{8}''$	2·99	7·4
$\frac{6}{8}''$	2·88	10·8
$\frac{7}{8}''$	2·35	27·2
1″ (three notches of same depth)	1·78	44·9

interstitial solutes, viz. C or N in α-iron through the measurements of internal friction.

For this purpose, a simple apparatus of the Kê type torsion pendulum of 1-2 cps. was constructed for ease of visual observations. During initial working of the apparatus, difficulties were experienced about the working of the wire specimen grip. Later the grip was redesigned and use of split ' V ' groove with screw tightening system showed improved performance. Some other parts were also modified for ease of setting of the specimen. The furnace control was set for manual operation so as to regulate the temperature gradient in the test zone of the furnace within allowable limit of about 1°C.

The high purity iron kindly supplied by BISRA was hot forged and cold drawn to 19 swg. wire with interstage annealing and taking proper care to avoid any contamination. The wire was straightened and finally annealed at 700°C. for about an hour in hydrogen atmosphere and subsequently quenched in water. The internal friction was measured. The values of a set of readings are given in Table 43 showing the nitrogen peak at about 36°C. for 1·8 cps. frequency. Further work was carried out to remove nitrogen to considerably low value by wet hydrogen treatment and measuring internal friction after introducing known amount of nitrogen in solution by ammonia treatment.

55.0 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 in view the work on photoelastic stress analysis was started at the National Metallurgical Laboratory.

Initially work was conducted on a beam stressed under pure bending. Stress concentration factor (S.C.F.) due to a sharp and deep notch ($r - \frac{1}{8}$ in., depth $h - 1$ in., minimum section of the beam $d - 2$ in., $h/r - 8$ in., $r/d - 0{\cdot}062$) in a beam under pure bending was determined. Later, two more notches of the same radius were machined on either side of the central notch, the centre of the three notches being $\frac{1}{2}$ in. apart; and the S.C.F.'s were determined for different depths of the stress relieving notches. The results are given in Table 44.

Further work for arriving at general conclusion regarding nature and characteristic of stress relieving effect of notches was done by varying the distances of the notches and also with notches of varying sharpness and depth.

56.0 Study on Welding

Work on ' Cold pressure welding ' was undertaken to study the mechanism of metal

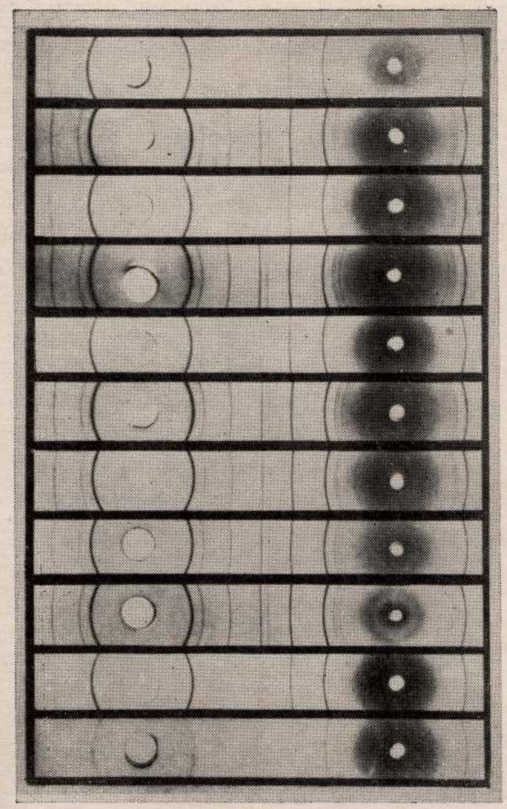


Fig. 51 — X-ray diffraction patterns for the samples fired at different temperatures ranging from 600°C to 1700°C.

bonding, particularly in the case of metals difficult to weld by usual fusion welding methods. The work was started with aluminium and copper.

Pairs of aluminium strips $\frac{1}{8}$ in. thick and 1 in. wide were bonded using an indenting tool $\frac{1}{8}$ in. thick. Aluminium strips were first degreased, surface oxide layer ground off and subsequently washed with trichloroethylene. Bonding was carried out soon after to prevent the formation of oxide layer thick enough to hinder bonding. Bonding was carried out by applying optimum reduction in steps. Bond strength was observed to increase linearly with increase in percentage of reduction.

Copper strips were likewise bonded. Effects of post bonding heat-treatment were determined. For aluminium, post bonding heat-treatment had a detrimental effect on the bond strength, whereas in the case of copper no change in bond strength was noticed.

Metallographic examinations of the bonded section was also carried out. A new tool with smaller indentors was prepared for carrying out investigation on bonding of thin gauge sheets.

57.0 Production of Basic Refractories from Indigenous Resources

(i) *Studies on Almorah Magnesite* — Magnesite bricks are indispensable for lining basic open hearth furnaces. Recently new deposits of magnesite have been located in Almorah (U.P.) Laboratory scale study conducted earlier on these samples indicated that Almorah magnesite was similar to Austrian magnesite and could be used for the production of basic magnesite bricks. It was observed that the presence of about 5 per cent lime in the magnesite was responsible for the low refractoriness under-load of refractories made from Almorah magnesite. From D.T.A. studies, it was found that lime was present as dolomite. Efforts were made to beneficiate the magnesite by low and high temperature calcination. A process was evolved whereby the excess lime could be removed and chemical composition of the beneficiated magnesite brought within the limits specified for manufacture of basic bricks for use in steel plants. Mineralogical changes occuring on firing the magnesite from 600°C. to 1700°C. at intervals of 50° to 100°C. were studied in detail by X-ray diffraction and petrographic methods. Fig. 51 shows the X-ray photograph of a specimen fired at different temperatures. Between 800°C. and 900°C., periclase formation started.

(iii) *Chrome-magnesite Refractories* — In view of expansion of Indian Iron and Steel Industry during the Second and Third Five Year Plans, demands for basic refractories using chrome ore are expected to steadily increase. Recent trends in steel making and in refractory practice in all-basic open-hearth furnaces with chrome-magnesite roofs have established their superiority of performance in respect of lining life, operational control and increased productivity in relation to acid roofs.

Trials were carried out with batches of different compositions using a mixture of Byrapur chromite and raw magnesite and calcining it above 1600°C. With the pre-calcined material as grog and making a grading of very high packing density, 4½ in. × 2¼ in. × 2 in. quarter size bricks were pressed and fired at a temperature of 1600°C. These bricks had very low bursting expansion in contact with iron oxide (about 4 per cent) in comparison with earlier values obtained approaching 20 per cent. Other refractory properties such as porosity, bulk density, refractoriness-under load etc. were as follows:

Bulk density		2·9 to 2·8
Porosity		20 to 25 per cent
Refractoriness-under-load:	'TA'	1640°C.
	'TE'	1790°C.

(iii) *Studies on Tata-chemical Magnesite* — A sample of pea-size magnesite was sent by Tata Chemicals Ltd, Bombay, for testing and evaluation for use as a refractory material. The source of this magnesite was said to be sea water.

A detailed study of the sample of dead burnt sea water magnesite was carried out. It was found that in spite of its higher percentage of CaO, its refractoriness-under-load, spalling resistance, bulk density etc. were very good, which may be due to the fact that though CaO content was high, the SiO_2 was low. Hence the lime formed tricalcium and dicalcium silicates with high melting points. It was shown that though this sea water magnesia may not be within the I.S.I. specifications for dead burnt magnesites for steel plant applications, it was good refractory material for making basic refractory bricks suitable for use in steel-making. Investigation was completed and the full Report sent to Messrs. Tata Chemicals.

(iv) *Sintering of Dolomite in Shaft Kiln of Hindustan Steel Plant, Rourkela* — Investigations were taken up to study the sintering characteristics of dolomite since it was reported that the dolomite shaft kiln installed at the Rourkela plant of Hindustan Steel Company was not at all working satisfactorily. A detailed investigation was therefore taken up.

Dolomite required for making sintered dolomite should have the following properties:

(a) It should not 'dust' during cooling before discharge.

(b) It should yield a good sinter having a high bulk density and capable of withstanding atmospheric hydration during the intervening period of discharge from the kiln and processing the sinter into bricks or blocks which may range from 7 to 15 days.

(c) It should not cause hanging, bridging and scaffolding in the shaft kiln caused by undue sticking of the dolomite pieces.

Considered on the basis of the above criteria, Hirri dolomite appeared, to be unsuitable for use in a shaft kiln, due to non-uniform distribution of silica in the dolomite matrix. Unless, selective mining was adopted, separation of high silica dolomitic material prior to the sintering would not be practicable.

Hardi dolomite yielded a very good sinter and was free from the heavy dusting observed in the case of Hirri samples; but it had a slight tendency to stick at high temperatures in the range of 1620°-1720°C. and above. Whether such sticking in actual shaft kiln operation, would cause hanging required to be determined by full scale shaft kiln operations. Except for such slight sticking, Hardi dolomite should be an excellent raw material for use in shaft kilns.

Baraduar dolomite is free from all such troubles of dusting and sticking and hence appear to be the best of the three dolomites. However, on firing to 1650°C. it did not yield as dense a sinter as the Hardi dolomite and

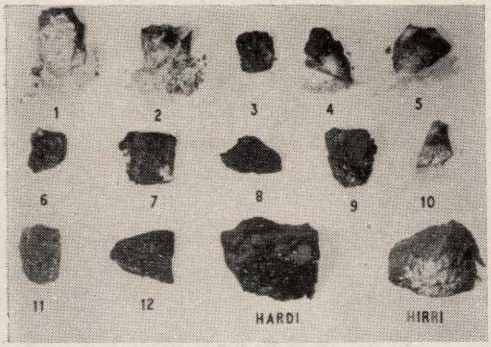


FIG. 52 — SAMPLES OF DOLOMITE CALCINED AT 1600°C.

consequently higher firing temperature would be needed to get an equally good sinter. Fig. 52 shows samples of dolomite calcined at 1600°C. some of which have crumbled and dusted.

(v) *X-ray diffraction Study of Firing Behaviour of Chrome-Magnesia Mixes* — Magnesia was added in varying percentages to the chromite spinels isolated from Tullochs' and Byrapur ores. After thorough pulverizing and pelletizing of the mixture, the pellets were fired at 1650°C. This process was repeated twice to ensure complete reaction between magnesia and chromites. The lattice parameters of the mixture after firing were measured by X-ray diffraction powder method. The results indicated that the magnesia had reacted with chromite. The lattice parameter of the chromite increased to a maximum due to formation of magnesium spinels and then remained more or less constant. It was thus possible to judge the extent of reaction of magnesia with chromite at high temperatures. An X-ray diffraction study of the fired chrome magnesite brick indicated that reactions involving magnesia and chromite had been completed.

58.0 Development of High Alumina Refractories

High alumina refractories are required to meet a wide variety of service requirements in metallurgical furnaces and cement kilns. The presence of high titania in many 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 therefrom.

(i) *Studies on Kashmir Bauxite and Bauxite Clay* — Samples of a bauxite and a bauxitic clay were received from the Director of Geology & Mining, Jammu and Kashmir for assessing the suitability of these raw materials for refractory manufacture. From the chemical analysis given below, it appears that the bauxite was highly diasporic which was also confirmed by differential thermal analysis.

	Bauxite	*Bauxitic clay*
Loss on ignition, %	15·12	14·84
Al_2O_3, %	65·29	53·53
SiO_2, %	12·98	25·25
Fe_2O_3, %	1·28	0·99
TiO_2, %	5·09	4·47
MgO, %	Traces	Traces
Alkalies, %	Trace	0·75
P.C.E. (Orton cone)	38	35

Preliminary trials carried out with the sample of bauxite indicated that due to its high refractoriness, it did not sinter well unless the bauxite was crushed to —80 mesh and the ore mixed with not less than 15 per cent plastic clay. The clay sample also showed a porosity of 40 per cent when fired to 1550°C. indicating its highly refractory nature. As these appeared to be the only diaspore and diasporic clay deposits known so far in India and as there was a dearth of high alumina clays in the country, these deposits could serve as potential reserves for future use and thus warrant further investi-

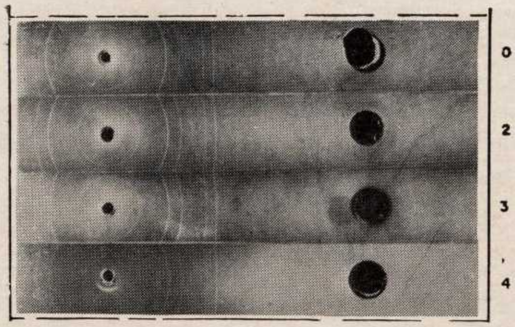


Fig. 53 — Powder X-ray photograph: O. Raw ore; 2. Byrapur ore fired with 20 per cent MgCo ; 3. Byrapur ore fired with 34 per cent MgCo , 4. Byrapur ore fired with 40 per cent $MgCO_3$

gation. With this end in view, representative samples were collected for a comprehensive study on the subject.

(ii) *Development of Calcium-aluminate Cement* — Calcium aluminate and calcium di-aluminate cements were prepared using very pure crystalline limestone and alumina. An X-ray diffraction study of the cement corresponding to calcium di-aluminate composition was made. It was found both by microscopic and X-ray studies that for the preparation of di-aluminate cement, a higher firing temperature was required. Normal consistency and setting time of these cements were determined as per I.S.I. Specifications for portland cement. The modulus of rupture and tensile strength of neat cements as well as mixture of cement and alumina were determined. These results indicated that both the cement compositions were satisfactory. The differential thermal analysis of the hydrated CA and CA_2 cements indicated that the maximum water loss took place between 280° and 325°C.

Preliminary experiments indicated that the CA and CA_2 cements could be used for tube furnaces wound with nichrome or kanthal. Internally wound furnaces with CA cement and sillimanite were successfully operated. A mixture of pure CA_2 cement and pure alumina was used for a refractory coating over a platinum winding and raised to high temperatures. No reaction between the cement and platinum wire was observed. The pure CA and CA_2 cements had higher P.C.E. values compared to commercial 'Cement Fondu' and application thereof with aggregates for service at higher temperatures would be recommended.

(iii) *Development of Foamed Alumina Bricks* — Sintering characteristics of Bayer's alumina were studied in great detail by D.T.A. and thermogravimetric methods. It was observed that combined water of aluminium trihydrate was lost in three distinct stages. Corrundum formation appeared to take place in the temperature range of 1140°-1240°C. After several trials with different mineralizers such as rutile, red mud, ilmenite, china clay, dolomite, chromite, etc. a combination of some of these oxides was found to give a very hard and dense sinter with alumina. Foaming agents such as a carbonate with a mineral acid and a metal powder with an organic acid were tried for getting minute bubbles. Starch and dextrine gave promising results as foam stabilizers. Further trials to produce evenly distributed bubbles in the brick were continued.

(iv) *Abrasion Resistant High Alumina Bricks* — With highly ferruginous bauxites from Katni as well as Shevaroy, it had earlier been observed that though the bricks made

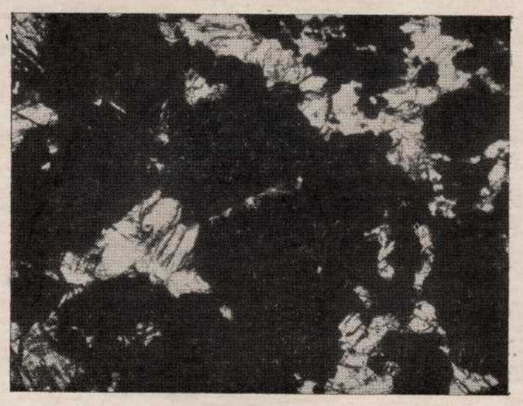


Fig. 54 — Photomicrograph of Salem chrome ore, polarized light × 50

from such material were not highly refractory due to the presence of excess iron oxide, they were very hard, dense and abrasion resistant, and could not be cut even with diamond tools. As such hard and dense bricks would be ideal for use as skid rails in reheating furnaces, where costlier silicon carbide bricks were currently used.

Ferruginous Katni bauxite with more than 18 per cent Fe_2O_3 and 9 per cent TiO_2 was fired to grog at 1500°C. and using 5 per cent plastic clay as bond along with crushed and graded grog, $9 \times 4\frac{1}{2} \times 3$ in. bricks were pressed at 10,000 lb./sq. in. in a hydraulic press. After drying, the bricks were fired at 1450°C. The porosity of the brick was 15 per cent and cold crushing strength 6000 lb./sq. in. A trial batch of 45 full size bricks was supplied for service trials as skid rails to Tata Engineering & Locomotive Co., Ltd. The bricks were reported to have given good service for the past one year by this firm.

59.0 Studies on Carbon

This study concerned carbon refractories and other carbon products for diverse applications in metallurgy. At this stage, it was proposed to study carbonaceous materials from the point of view of their ultimate utilization as raw materials for the production of carbon products, which involved examination of their pyrolytic characteristics with and without pretreatment as well as additions in the first instance, to be followed up by their graphitization characteristics. Some of the carbonaceous materials readily available in India such as low ash coals of Assam, washed coals of the Jharia & Raniganj area and lignite from Neyveli, and petroleum coke from Digboi were initially taken up for research studies.

Materials under study in this investigation were Assam Coal No. 1 and Assam Coal No. 2, Neyveli Lignite and Giridih Coal (washed).

(i) *Dynamic Test on Volatile Evolution* — The samples were submitted to thermogravimetric (weight loss) study in an inert atmosphere and temperature steadily increased from room temperature to 950°C. at 10°C. per minute.

(ii) *Isothermal Weight Loss Studies by Gravimetric Method* — Samples of above coals were submitted to weight loss study at certain specified temperature maintained with an accuracy of ±5°C.

(iii) *Differential Thermal Analysis*—D.T.A. was done up to 600°C. with temperature rising steadily at 10°C. per minute. A number of types of specimen holders were made for this purpose. The most suitable one was a stainless steel block. This apparatus was undergoing standardization test.

(iv) *Agglutinating Value Determination* — The apparatus was standardized and materials were prepared.

(v) A giesler plastometer was fabricated and was undergoing standardization tests.

60.0 Study on Refractory Raw Materials from Travancore

Under the Council of Scientific & Industrial Research Fellowship Scheme, research work was initiated on the study of refractory raw materials from Kerala State.

(i) *Kumblam Clay* — A sample of clay from Kumblam was studied for its suitability for refractory purposes. The clay was very plastic, more than 67 per cent of the particles being less than 0·5 in. diameter. Its high water of plasticity and Atterberg's number as well as X-ray, D.T.A. and base exchange capacity indicated that it consisted of kaolinite mixed with disorded kaolinite known as "levisite". It resembled English ball clays in many respects. It had a P.C.E. of Orton cone 32 and specimens made with 50 per cent grog and 50 per cent raw clay had a refractoriness under load of 1550°C. These experiments proved that the clay could be used for refractory purposes as well as for slip casting etc.

(ii) *Moulding Characteristics of Beach Sands of Travancore* — A representative sample of quartz sand from Trivandrum beach was studied for its use in foundries as a moulding sand. Chemical and petrographic analysis, fineness test, moulding characteristics like green strength permeability etc., when mixed with bentonite as well as high temperature tests and casting characteristics were studied as per standard A.F.S. methods.

The sand was found to consist mainly of quartz grains with impurities such as ilmenite, leucoxene, sillimanite and zircon which could be easily separated by tabling. The upgraded sand was found to possess good moulding characteristics when admixed with optimum quantities of bentonite, dextrine, oil etc., for making moulds as well as cores. Actual castings made with cast iron as well as steel, using moulds and cores made out of this sand were satisfactory. A comparison of the properties of the sand with some of the other well known foundry sands of India showed that it was as good as some of the best foundry sands in India.

(iii) *Effect of Rare Earth Chlorides on Sintering of Zircon Sand* — The action of rare earth chlorides obtained as a byproduct from the treatment of monazite sands of Travancore by lowering the sintering temperature of zircon sand was investigated. Zircon sand as received and after ball milling was treated with 1 to 2 per cent of rare earth chlorides and buttons heated to 1400°C. gave fairly well sintered products. The high temperature properties of these products are under study.

(iv) *Effect of Calcium Oxide and Calcium Chloride Treatment on Rare Earth Oxides* — Cerium, lanthanum, praseodymium and samerium oxides were heated to 800°C. separately prior to X-ray diffraction studies. These oxides were individually treated with a calculated mixture of calcium oxide and calcium chloride and heated to different temperatures. After removal of excess calcium chloride by washing, X-ray diffraction photographs were taken and d-values were calculated, which were found to be quite different from those of the original oxides indicating that some reaction had taken place. The possibility of utilizing this technique of treating with a mixture of calcium oxide and calcium chloride to separate individual rare earth oxides from a mixture was under investigation.

61.0 Studies on Clays

(i) *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 affected 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 decided to carry out some specific cation exchange on some Indian ceramic clays and study its influence on green and fired properties of clays. The work will cover differential thermal analysis and X-ray diffraction analysis as supplementary to base exchange in identifying the clay minerals present.

Rajmahal china clay, Travancore china clay and a Ball clay were selected for the study. The fractions passing through 300 mesh B.S.S. were used in the case of Rajmahal and Travancore clays and the fractions finer than 2 microns was chosen in the case of Ball clay with a view to retain different proportions of crystalline impurities in the different clays and study their effect besides the exchangeable cations on the thermal behaviour of kaolinite. The silica/alumina ratio of Rajmahal, Travancore and Ball clays studied were 2·15, 1·98 and 2·12 respectively.

The cation exchange capacity (c.e.c.) of these clays was determined by three different methods : (1) Leaching of the clay with 1N ammonium acetate and distillation, (2) Potentiometric titration of the hydrogen form of the clay with N/10 sodium hydroxide and (3) Saturation of the hydrogen clay with 1N KCl and estimating the liberated acid. The results are given in Table 45.

TABLE 45 — CATION EXCHANGE CAPACITY OF THE CLAYS

	Ammonium Acetate Leaching	Potentiometric Titration of H. Clay	Saturation with 1N KCl.
Rajmahal china clay — 300 B.S.S.	10·1 at *p*H 7	11 at *p*H 7	10·84 at *p*H 7
Ball clay	14·17 at *p*H 7	12·3 at *p*H 7	10·8 at *p*H 7
Travancore china clay — 300 B.S.S.	4·0 at *p*H 7	6·8 at *p*H 8·8	5·6 at *p*H 7

Mono cationic forms (Li^+, Na^+, K^+, Ca^{++}, Ba^{++}) of the above clays were obtained from corresponding H-forms, by treating with the hydroxides and Mg-forms were obtained by treating the H-clays with 1N $MgCl_2$ and washing the clay free of chloride with 95 per cent alcohol.

The thermal behaviour of the above clay salts was studied up to 1400°C. by differential thermal analysis using a platinum wound furnace and Pt foil cups as containers. It was observed that hydrogen clays exhibited highest amplitude of the dehydration peak. The order of cations with regard to (A/W) ratios in Rajmahal and Ball clays so far studied is as follows :

Rajmahal china clay : $H^+ > Na^+ > Li^+ > K^+ > Mg^{++} > Ca^{++} > Ba^{++}$

Ball clay : $H^+ > Na^+ > K^+ > Li^+ > Ca^{++} > Ba^{++} > Mg^{++}$

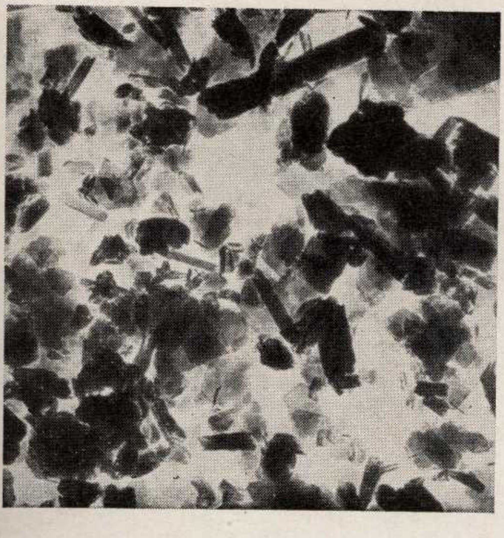


FIG. 55 — EECTRON MICROGRAPH OF RAJMAHAL CHINA CLAY. × 20,000

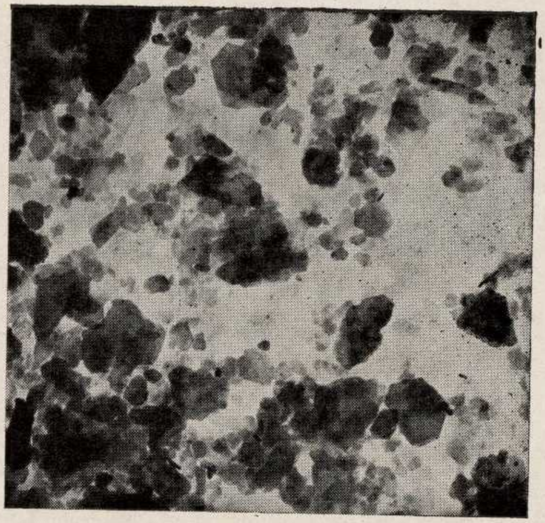


FIG. 56 — ELECTRON MICROGRAPH OF BALL CLAY. × 20,000

The most interesting observation was that monovalent cations give a higher ratio than divalent ions. Correlation with regard to the peak amplitude was also obtained by thermogravimetric studies of the monocationic forms.

Work so far done with Rajmahal and Ball clays revealed that the temperatures of the primary mullitization peaks were not much affected by the type of cation but the amplitudes were very much influenced. Hydrogen gave the highest peak and sodium the smallest, the other cations falling between the two limits. This peak being associated with the simultaneous formation Al_2O_3 and mullite, work was done on the measurement of relative intensities of Al_2O_3 and mullite phases by X-ray diffractometer with a view to examine whether the change in amplitude was associated with the relative proportions of the two phases formed. There was also some evidence that changes in surface areas

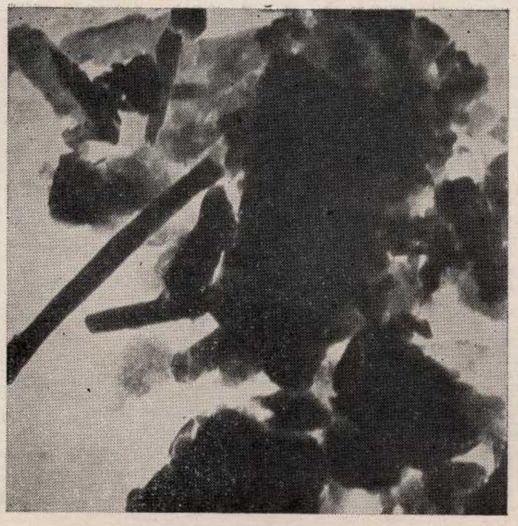


FIG. 57 — ELECTRON MICROGRAPH OF RAJMAHAL CHINA CLAY. × 20,000

would occur due to cationic fixation in heating. In order to isolate the effect of surface from phases formed during the endothermic reaction, surface area measurement of the clays heat treated under different conditions were carried out. Measurement of specific surface was being done by stearic acid adsorption from methonol and in this connection a conductometric assembly for measuring stearic acid before and after adsorption was set up.

The cations were found to have no appreciable influence in the secondary mullitization peak of kaolinite as seen by differential thermal analysis since no difference in temperatures or shapes of the peaks were noticed.

(ii) *Studies on Clays from South India* — This study was taken up on behalf of the Director of Industries & Commerce, Madras State, to catalogue the characteristics as well as possible refractory applications of clays from various localities in Madras State.

A number of samples were received and studied. Sampling, size analysis, chemical analyses, differential thermal analyses, etc., were completed. Firing characteristics, shrinkage, apparent porosity, bulk density, modulus of rupture and refractoriness under load properties were determined. From a study of the various properties of the clays the following conclusions were drawn :

(a) The clays were kaolinitic. The nature of the iron impurities was, however, not established. Much of it was present in the coarse fraction and was removable by washing.

(b) Washing did not show appreciable improvement in Sivaganga and Panruti clays but Tindivanam and Neyveli clays showed a remarkable improvement in refractoriness.

(c) Sivaganga and Tindivanam clays were coarse grained while Panruti and Neyveli clays are fine grained. Tindivanam clay contains free quartz.

(d) Thermal behaviour and colours obtained on firing the clay bars indicated that the clays were suitable for refractory usage. The refractoriness-under-load of Sivaganga Clay indicated that high heat duty refractories could be easily manufactured out of these clays. Shrinkage of the unwashed clays were well within controllable limits, and should not pre-

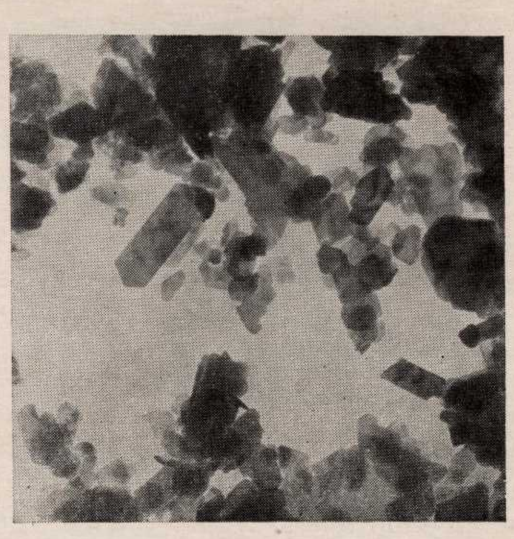


FIG. 58 — ELECTRON MICROGRAPH OF TRAVANCORE CHINA CLAY. × 20,000

sent any manufacturing difficulties. Tindivanam clay when unwashed was very promising as raw material for semi-silica refractories and when washed became quite a good clay for refractories of 40 per cent alumina grade.

(e) Binding power and workability experiments were not done on all the clays. These data would give a complete picture of their suitability for brick manufacture. However, the results so far obtained indicated that these clays were suited for the manufacture of high grade 35-40 per cent Al_2O_3 refractories.

62.0 Suitability of Indigenous Asbestos for Manufacture of Asbestos Cement Products

Ten more samples of asbestos in larger quantities were procured through Geological Survey of India from different parts of the country, for further studies.

Suitable grade of amphibole samples were obtained by crushing and sieving. Chrysotile samples found associated with serpentine in ultrabasic rocks were processed by crushing followed by magnetic separation and air classification. Standard screen test as per Quebec system was done on all samples.

After preliminary trials, the following compositions and optimum conditions were fixed :

Asbestos fibre ($-8+32$ mesh)	25 parts
Portland cement	50 parts
Silica sand	25 parts
Water	25 % of the mix.

Pressure 200 lb./sq. in.

Curing under wet cloth — 4 weeks. Modulus of rupture tests were conducted on test bars 6 in. × 1 in. × $\frac{1}{2}$ in. size and the results are given in Table 46.

Specimens of flats, 'S' shaped corrugations and pipes were made by pressing of hand ramming and subjected to curing for one month under wet conditions. These are shown in Fig. 59.

Average modulus of rupture values ranged from 919 to 1333 psi for amphiboles and from 1371 to 1815 psi for chrysotile samples. The chrysotile samples gave comparatively better results than amphiboles. Though the indigenous resources of asbestos of amphibole variety may be somewhat inferior to the chrysotile variety of asbestos, it was possible to use these in cement asbestos products.

63.0 Plasticizing and Colloidizing of Non-plastic Refractory Materials

An important and major advance in ceramic technology is that of producing artefacts

TABLE 46 — MODULUS OF RUPTURE TESTS

SAMPLE No.	SOURCE	TYPE OF ASBESTOS	AVERAGE M.O.R. lb./sq. in.	MAX. M.O.R. lb./sq. in.
1	Seraikela	Amphibole	919·0	1028·0
2	Chaibasa R	do	1289·0	1433·0
3	Chaibasa S	do	1050·0	1242·0
4	Rajasthan	do	1150·0	1362·0
5	Rajasthan B	do	1248·0	1514·0
6	Mysore H	do	1333·0	1540·0
7	Mysore	do	1055·0	1162·0
8	Chaibasa N	Chrysotile	1315·0	1914·0
9	Chaibasa B	do	1371·0	1553·0
10	Chaibasa U	do	1627·0	1885·0

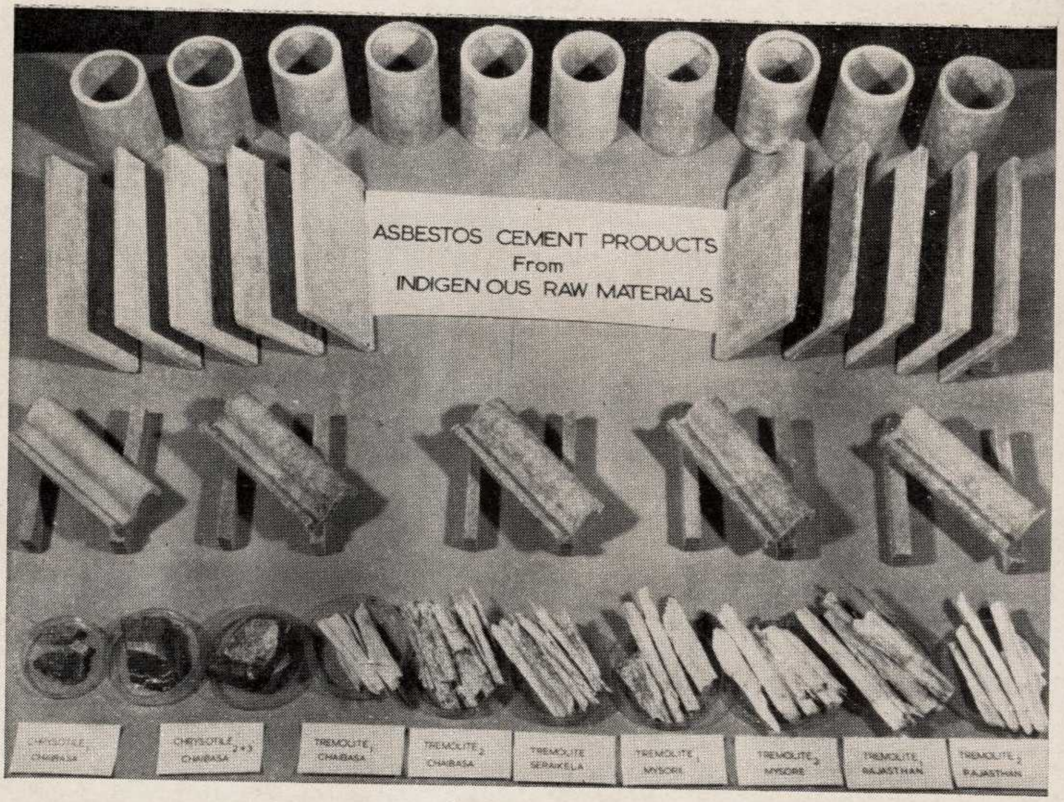


FIG. 59 — ASBESTOS CEMENT PRODUCTS PRODUCED FROM INDIGENOUS RAW MATERIALS AT NATIONAL METALLURGICAL LABORATORY

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 therefore initiated with sillimanite from the beach sands of Travancore and its grindability under certain predetermined conditions was assessed.

During the period under review the following studies were carried out :

(i) Base exchange capacities of the sillimanites ground for various durations of time.

(ii) Effect of electrolytes like sodium pyrophosphate on *p*H and viscosity of the suspensions of sillimanite.

TABLE 47

Extrusion Charteristics of Sivaganga Clay

Clay taken, 50 *gm.*

WATER ADDED cc.	PRESSURE OF EXTRUSION IN LB./SQ. IN.
13	450
15	225
17	160
19	100
21	75
23	45

Extrusion Characteristics of Tindivanam Clay

Clay taken, 50 *gm.*

WATER ADDED cc.	PRESSURE OF EXTRUSION IN LB./SQ. IN.
10	515
11	412
12	310
13	260

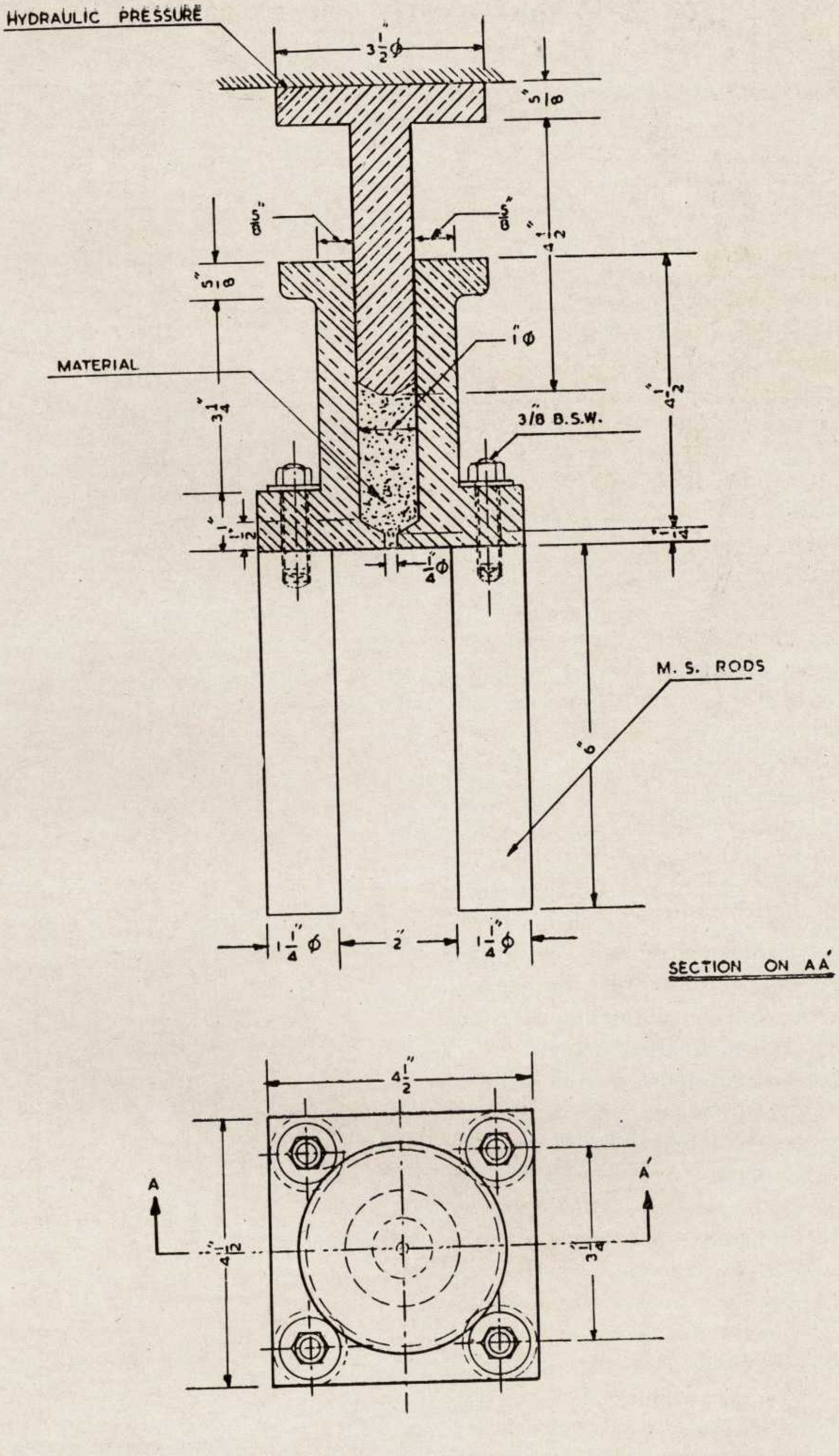


Fig. 60 — Extrusion apparatus for ceramic pastes

TABLE 48 — VISCOSITY AND pH VALUES

ELECTROLYTE					
Sodium Pyrophosphate		Hydrochloric Acid		Liquor Ammonia	
*p*H	Viscosity	*p*H	Viscosity	*p*H	Viscosity
7·2	4·5 mts.	7·2	4·5 mts.	7·2	4·5 mts.
7·25	56·0 sec.	4·05	18·5 sec. (settling slip)	—	—
7·8	18·0 sec.	—		—	—
8·0	18·0 sec.	3·55	15·0 sec.	7·7	62·0 sec.
8·1	18·0 sec.	—	—	8·9	19·5 sec.
8·2	A very thixotropic type of slip	—	—	(Slip was setting)	—
				9·2	18·0 sec.
				9·6	18·0 sec.
8·7	—	—	—	9·8	—

(iii) The type of charge, positive or negative, on the sillimanite when it was ground and suspended in distilled water.

An extrusion apparatus based on the principle of the flow of a plastic material through an orifice under steadily increasing pressure was designed. Amsler Universal testing machine was used as pressure generating equipment. Fig. 60 is the sketch of the plastometer. Results of a few trials with plastic clay water mixes are given in Table 47.

An U-tube electrophoresis apparatus was made in order to determine the nature of charge as well as the rate of migration of colloidal particles. Fig. 61 is sketch of the apparatus. Experiments were conducted under a potential of 220 volts d.c. using a current of the order of 0·9 milliamps. The apparatus is under standardization.

The particle size of more than 50 per cent by weight of the various samples of sillimanite and per cent by weight of particles finer than 1 micron are given in Fig. 62. Base exchange capacities of the various samples of sillimanite determined by the ammonium acetate leaching method are also given in Fig. 62. Sample of sillimanite ground for 96 hours was taken for studying the variation in fluidity of the sillimanite with *p*H. The electrolyte studied was a saturated solution of sodium pyrophosphate besides diluted hydrochloric acid, and liquor ammonia solutions. Fifty per cent solids was used and the suspension after the addition of electrolyte was rotated in a pot mill rack overnight and the viscosity and *p*H were determined the next day. The results of these experiments are given in Table 48. From the results of the experiments, Na-pyrophosphate appeared to be more effective. To find out the nature of charge on the

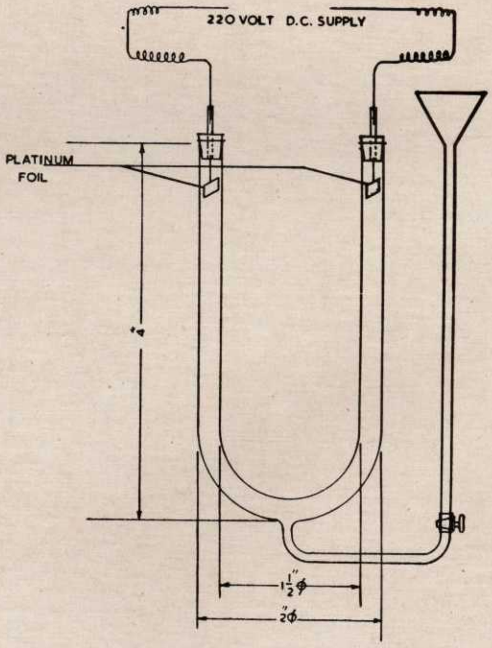


FIG. 61 — SKETCH OF U-TUBE ELECTROPHORESIS APPARATUS

particles in suspension an U-tube catophoretic cell was made for electrophoresis experiments. Some preliminary experiments with clay and suspension of sillimanite were made. The results obtained indicated that sillimanite fines were negatively charged.

64.0 Correlation of Chemical and Physical Properties of Blast Furnace Refractories with their Resistance to Carbon Monoxide Disintegration

Investigation work was taken up to collect data on blast furnace refractories from different sources, both indigenous and foreign to arrive at optimum figures for desirable physical properties. These data would be used in collaboration with Indian Standards Institution in formulating suitable specifications for blast furnace refractories. Study of physical properties and chemical analysis of blast furnace refractories from indigenous and foreign sources were continued. The data so obtained are given in Table 49.

65.0 Thermal Conductivity of Insulation Refractories

Insulating bricks from different Indian firms as well as samples from a foreign source were collected for the determination of their thermal conductivity values using A.S.T.M. Thermal Conductivity Apparatus.

Samples of insulation refractories were received through the Indian Standards Institution. Their thermal conductivity values as well as other physical properties were tested. The physical properties of all the insulation bricks studied are given in Table 50.

66.0 Investigation on Some Blast Furnace Stove Refractories

Messrs Hindustan Steel Ltd, reported failure of some of their blast furnace stove

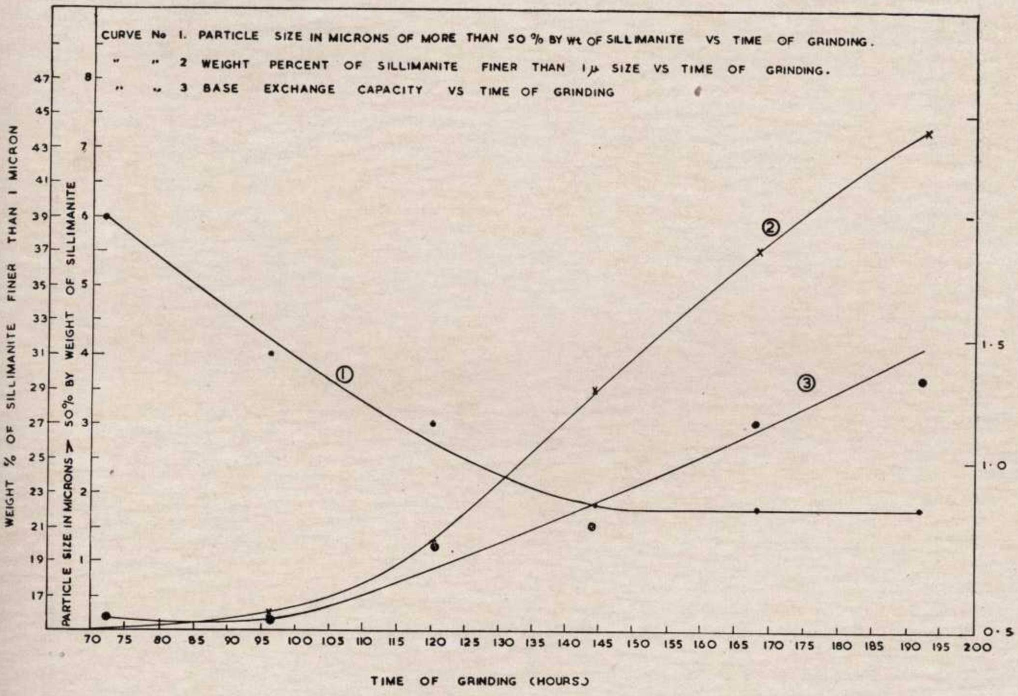


FIG. 62 — PARTICLE SIZE OF SILLIMANITE SAMPLES

TABLE 49 — PHYSICAL AND CHEMICAL PROPERTIES OF BLAST FURNACE BRICKS

PROPERTY	INDIGENOUS							FOREIGN						
	1(a)		1(b)			2		English		German				Russian
	H. & B.	Inwall	H. & B.	K	T	SI	H. & B.	H. & B.	Inwall	PFR 1488 G	Max. D 1068	Max. D	Max. HH	D-2
Apparent porosity, %	26·1	26·0	21·5	23·0	24·1	14·1	18·9	20·0	14·0	13·65	14·92	16·46	12·08	15·49
Bulk density, gm./cm.³	2·132	2·093	1·992	2·027	1·964	2·261	2·163	—	—	2·132	2·095	2·14	2·148	2·188
Permeability, C.G.S. units	0·18	0·17	0·74	0·67	0·68	0·23	0·40	0·103	0·02	0·10	0·11	0·37	0·14	0·25
% reheat change after 2 hrs. at 1450°C.	1·07	−1·30	—	—	—	0·09	0·16	—	0·0-0·5	−0·12	−0·29	−0·12	−0·26	−0·45
Cold crushing strength, kg./sq. cm.	102·9	79·6	131·3	—	121·8	674·8	558·6	—	—	262·7	351·8	235·8	225·2	294·8
Carbon monoxide disintegration test, 100 hrs.	Unattacked		Unattacked			Unattacked		Out of 5 samples two cracked and fell apart after 80 hrs. passage of CO. Rest withstood 100 hrs. run		Unattacked				Out of 4 samples kept one sample cracked after 58 hrs. passage of CO. Rest were intact up to 100 hrs. run
Chemical analysis:														
Loss at 110°C.	—	—	0·17	0·20	—	—	—	—	—	—	—	—	—	
L.O.I., %	—	—	0·04	0·02	0·02	0·16	0·26	0·68	0·26	0·28	0·26	0·16	0·10	
SiO_2, %	55·52	60·48	60·53	60·75	58·2	56·9	55·66	53·46	56·29	57·69	60·89	56·20	55·63	
Al_2O_3, %	39·52	34·36	33·83	34·07	33·48	34·33	36·94	40·70	37·44	36·83	33·64	38·18	38·47	
Fe_2O_3, %	1·20	0·52	1·10	1·40	2·48	2·72	1·92	1·76	2·68	2·31	2·47	2·50	2·47	
CaO, %	Trace	0·64	0·55	0·76	0·35	0·4	1·47	1·58	0·89	1·01	0·83	0·84	0·58	
MgO, %	0·98	1·25	1·39	1·09	1·7	1·9	0·49	0·41	0·47	0·49	0·42	0·86	0·97	
TiO_2, %	2·88	2·24	1·57	1·42	2·04	2·15	1·38	1·32	1·07	1·19	0·98	1·88	2·08	
Alkalies	nil	nil	—	—	—	—	1·89	0·35	—	—	—	—	—	

TABLE 50 — PHYSICAL PROPERTIES OF INSULATION BRICKS

SL. NO	APPARENT POROSITY %	B.D. lb./c. ft	THERMAL CONDUCTIVITY IN B.T.U./HO/SQ. FT/ IN./°F.		P.C.E.	COLD CRUSHING STRENGTH IN LB./SQ. IN.	% REHEAR CHANGE AT	
			B.t.u.	Mean temp. °F.			850°C. (24 hrs)	1500°C. (24 hrs)
Foreign								
1	72	46·9	3·66	729	34·35	252·2	0·2	3·14%
Indian								
2	73	45·0	3·0	586	31·32	528·6	0·3	
3	77	38·0	1·87	544	28·29	98·37	0·37	
7	71	48·8	5·47	746	31	250·2	1·25	
5	79	36·3	0·84	532	9·10	158	0·91	
6	66	56·3	3·48	739	27·28	233·1	0·08	After 3 hrs at 1500°C. 0·43. After 24 hrs at 1500°C. badly shrunk
7	65	58·8	2·56	708	28	321	0·17	
8	59	60·3	3·23	750	27·28	536·4	0·03	
9	65	60·3	2·91	700	32·33	130·5	0·04	
10	74	42·5	2·56	700	18·19	256·0	0·19	
11	67	52·5	1·14	700	9·10	116·2	0·1	

refractories and desired investigations on the causes which led to the premature failure of these refractories after a service life of only three years. The failed refractory bricks were taken from two adjacent zones from the top portion of the checker-work of the blast furnace stove. Fig. 63 shows the two sets of refractory bricks received for testing.

Chemical and microscopic analysis were carried out and physical properties such as porosity, bulk density, reheat shrinkage, P.C.E. and refractoriness under-load of the unused and used brick specimens were determined. Failure of the bricks could be attributed to the following:

(i) Apart from containing large amount of fluxing oxides, the bricks used were not up to the standards generally used in blast furnace stove checker-work, having only 32-36 per cent Al_2O_3 as against 39-42 per cent normally recommended.

(ii) During service, the bricks had suffered attack of alkali bearing vapours and/or dusts in the blast furnace gas which had caused vitrification and consequent deformation.

Present trend in blast furnace stove checker work refractory practice was to use well fired clay refractories of much higher alumina content (50 per cent and above) since alkali attack and failure due to vitrification is minimum with high alumina refractory.

67.0 Studies on Corrosion of Metals and Alloys

Long-term studies on various aspects of corrosion research under Indian tropical weather conditions have been taken up at the National Metallurgical Laboratory. The scheme for setting up the marine corrosion research unit at Digha on the sea coast of West Bengal has been finalized. The marine corrosion research unit at Digha will be provided with facilities for sea water corrosion study under partially and fully immersed condition, soil corrosion, corrosion due to micro-organism, cathodic protection, etc. During the period under review considerable progress was made on long-term studies on the atmospheric corrosion of metals under industrial atmospheres as well as a number of corrosion problems.

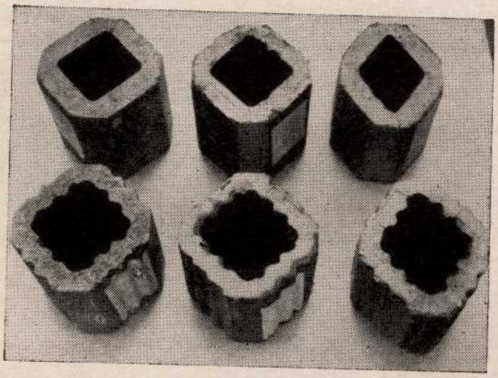


FIG. 63 — BLAST FURNACE STOVE REFRACTORIES SHOWING UNUSED, PARTLY DEFORMED AND MORE DEFORMED BRICKS

(i) *Studies on Atmospheric Corrosion of Metals and Alloys* — Studies on atmospheric corrosion of various metals and alloys exposed to industrial atmosphere of Jamshedpur was continued. Following metals and alloys exposed after 1956 were removed in 1961:

Material	*Period of Exposure Years*
Nickel	4
Copper	4
Brass	5
Zinc	5
Mild steel exposed horizontally	2
Mild steel exposed at 45°	2
Mild steel exposed vertically	2
Tiscor exposed horizontally	2
Tiscor exposed at 45°	2
Tiscor exposed vertically	2

The samples were removed and cleaned of corrosion products to determine loss in weight. During this period samples of low alloy high tensile steel of twelve compositions developed at the National Metallurgical Laboratory, were removed, a few after one and others after two years exposure. From the weight loss data obtained copper and chromium were observed to be highly beneficial in reducing corrosion. To study the behaviour of coupled metals and alloys in atmosphere, samples of aluminium, zinc, mild steel and copper wire in galvanic contact with mild steel, brass and stainless steel were exposed in July 1960. The bolt wire types of samples were removed after 1½, 3, 6 and 12 months' exposure. Corrosion rate of wire and bolts were separately determined. These tests showed marked increase in corrosion rate of aluminium and zinc when coupled to mild steel, brass and stainless steel. It was noted that type and texture of rust on steel exposed to atmosphere varied with time and composition of steel. To study the changes in properties of rust samples of mild steel and Tiscor were exposed vertically at 45° and horizontally, in the month of January 1961. The samples were removed after 2, 6 and 12 months exposure. In addition to the determination of loss in weight, rust collected from the samples was analysed for total sulphate, total iron, ferrous iron and moisture content; porosity of rust in situ was measured by putting a drop of water on the surface and then measuring the area and time of absorption of water. Anodic polarization characteristics of the rusted samples with rust in situ were also determined. The results indicated considerable variations in the properties of rust on mild steel and Tiscor but would require further studies to draw definite conclusions.

Detailed monthly record of sulphur pollution of atmosphere and meteorological data e.g. maximum and minimum temperature, maximum and minimum percentage relative humidity, rain fall etc. were obtained for a proper assessment of the effects of climatic and pollution factors on corrosion resistance.

(ii) *Mechanism of Chromate Passivation* — Treatment of zinc, aluminium, etc., in acid chromate solution improved their resistance to corrosion in a stagnant layer of water. The mechanism of film formation and its relation to improved resistance to corrosion were studied.

Studies on chromate films formed on zinc, galvanized steel, and zinc plated steel to define conditions necessary for the formation of chromate film were continued. Nature of film formed was determined in solutions containing 200 gm. per litre of sodium dichromate and 40-60 cc. per litre of sulphuric acid by varying the immersion time from 5 to 60 seconds. The results obtained by using varying amount of acid indicated that rate of attack of zinc increased with increase in acid content and that the amount of film formed was not proportional to the loss of zinc. Effect of acid content and time of immersion on corrosion resistant properties of chromate film was under study. Study on mechanism of chromate film formation on aluminium and its alloys was taken up. Some preliminary experiments were carried out with chromate fluoride bath.

(iii) *Studies on Corrosion Resistance Properties of Adivasi Steel* — The tribal communities in India crudely smelt high grade iron ore in small mud clay furnaces (about 30 in. high) for the production of agricultural implements required by them e.g. spears, axes, etc.

A detailed study on the composition, structure and corrosion resistance properties of this steel was carried out. Chemical analyses of both metal and scale were carried out. The metal was found to contain very high carbon (1·3 per cent) with traces of other element e.g. silicon, manganese, sulphur and phosphorus. The scale contained in addition to oxides of iron, about 2·4 per cent SiO_2. Studies on microstructure showed that composition of steel varied considerably from place to place. Salt spray test and atmospheric corrosion test of samples of mild steel and adivasi steel with and without scale showed considerable protective properties of scale on adivasi steel when compared to that of mild steel. It was probably due to the fact that the viscous slag formed during crude smelting operation was worked into the metal during crude smelting and hot-forging, thus reducing the number of breakdown areas where the corrosion could be initiated. Comparison of corrosion resistance properties of adivasi steel and mild steel without scale did not show much differences in corrosion rates.

(iv) *Corrosion of Monel Tubes in Transformer Oil Cooling Systems* — Monel tubes are used for heat-exchangers in electrical transformer for cooling transformer oil as the alloy possesses excellent resistance to corrosion. However, premature failure of monel tubes is sometimes reported. Detailed investigation was carried out to find out the factors responsible for this type of attack. It was established that corrosion started from the water side of the tubes, and extended outwards resulting in a deep and shallow pit. Product of corrosion was mostly reddish in colour (cuprous oxide) and also contained considerable amount of bluish green salt of copper and nickel. On analysis, the deposits showed presence of Cu, Ni and Ca. Possible causes of corrosion may be stated as follows:

(a) Deposit attack: If the circulating water contained considerable amount of undissolved solids, these may be deposited on the tube causing rapid attack of the metal below the deposit due to formation of an oxygen concentration cell.

(b) Film of carbon derived from lubricant, used in the drawing process and deposited on the surface of the tube, during annealing may initiate attack as the carbon film acts as an active cathode.

(c) Pits once formed may progress further at an accelerated rate at hot spots, created due to uneven transfer of heat through the exchanger. Tests carried out indicated that hot spots accelerated pitting considerably.

68.0 Preparation of Plating Salts

The electro-plating industry in India has so far been using imported plating salts, polishing compositions and plating equipment. Due to strict import restrictions, these proprietory plating salts are not available in the market and the electro-plating industries in India are experiencing serious difficulties in procuring plating materials. Some of the Indian products have marketed plating goods including plating salts but these products have in most cases failed to produce satisfactory results. To assist the plating industry in India, development work on the formulation of common plating salts such as cyanide copper plating salt, dull and bright nickel plating salt, etc., was taken up, based essentially on the use of raw materials and chemicals made in India. Addition of special ingredients beneficial in increasing conductivity, good throwing power, brightness and related properties for good plating were also investigated from indigenous sources.

Formulae for satisfactory nickel-plating salt equivalent in service performance to imported plating salts were successfully worked out. To assess the feasibility of NML formulated salt in commercial plating practice, 3 cwt. of the salt for large scale experimental trials were supplied to the firm. Highly satisfactory reports on the performance tests of NML plating salt were also received from the Defence Establishments.

68.1 Hard Chromium Plating on Aluminium

The use of hard chromium plating on aluminium is common for many engineering applications in producing a hard, wear-resistant and oil-retaining surface in components such as cylinders, pistons, bearings and brake drums. Hard chromium plating on aluminium was successfully done from chromic acid bath using the interlayer of (i) iron and (ii) nickel by immersion process from the respective solutions. The chromic acid bath used for chromium plating has the following compositions:

CrO_3 250 gm./L.; H_2SO_4 2·5 gm./L.

The aluminium specimens were properly etched before giving an immersion deposit of nickel or iron.

(i) *Nickel-interlayer* — Experimental results observed on hard-chromium plating on etched aluminium surface with an interlayer of nickel are given in Table 51; nickel layers are removed before putting in electro-plating vat.

(ii) *Iron-interlayer* — Following experimental results were obtained in the case of aluminium specimens using an interlayer of iron after etching. Plating conditions on the etched aluminium after removal of interlayer once formed are given in Table 52.

Further investigations on the deposition of hard chromium on aluminium with an interlayer of zinc and without interlayer (simply by use of suitable etching agent) were in progress.

In the investigations of zinc-interlayer, it was observed that there should be an optimum concentration of caustic soda in the zincate solution used for immersion deposition of an interlayer of zinc. Hard chromium deposits on the zinc-interlayer medium were not sufficiently adherent, which needed investigation for establishing the method on a commercial basis.

TABLE 51 — RESULTS OBSERVED ON HARD CHROMIUM PLATING ON ETCHED ALUMINIUM SURFACE

Expt No.	Aluminium Specimen and their Area	Duration of Plating hrs	Current Passed amp.	C.D. amp./ft²	Amount of Cr Deposited	Thickness of the Deposit	Hardness in V.P.N.	Adhesion
1	3″×1″×2″	3	10	240	2·0246	0·00283″	950	Good
2	3″×1″×2″	4	12	288	2·8072	0·0039″	1161	do
3	3″×1″×2″	4	15	260	1·9850	0·00282″	1067	do

TABLE 52 — PLATING CONDITIONS ON ETCHED ALUMINIUM AFTER REMOVAL OF INTERLAYER

EXPT No.	ALUMINIUM SPECIMEN	DURATION OF PLATING hrs	CURRENT PASSED amp.	C.D. amp./ft^2	AMOUNT OF DEPOSIT	THICKNESS OF THE DEPOSIT	HARDNESS IN V.P.N.	ADHESION (Bending Test)
1	4″×1″×2″	2	15	270	2·075	0·002″	1081	Good
2	4″×1″×2″	2	10	180	1·4996	0·0016″	950	do
3	4″×1″×2″	2	12	216	1·523	0·00162″	1242	do

69.0 Determination of Gases in Metals

The vacuum fusion gas analyses apparatus designed and fabricated at the National Metallurgical Laboratory was used for estimation of gases in different metals and alloys. Determination of hydrogen in metals and alloys by carrier gas technique was taken up to avoid the complication in maintaining a steady high vacuum. The method was based on the principle that when an inert gas such as argon or nitrogen was passed around a steel sample at 650°-750°C. hydrogen was diffused out of the sample and swept by the carrier gas. Hydrogen evolved from the sample was oxidized to water by passing it over heated copper oxide and iron oxide for measurement by Karl Fischer reagent. Hydrogen values obtained by this method showed good agreement with values obtained by conventional vacuum heating method.

Study of hydrogen pick up by steel samples during pickling in presence of different inhibitors in the pickling bath was carried out with a view to evaluate the conditions under which hydrogen pick up was inhibited. Addition of pickling inhibitors prevented vigorous acid attack of the matrix without appreciably affecting the rate of descaling. The pickling bath consisted of 2N and 4N sulphuric acid and pickling was carried out at room temperature for different periods. The hydrogen evolved during pickling was measured in an inverted burette. Samples 1 in. long, ½ in. wide were cut from normalized sheet which had been cold reduced by 10 per cent. It was observed that nearly 0·1 per cent of the hydrogen discharged during pickling diffused into the metal when no inhibitors were employed. Inhibitors from urea group such as urea, thiourea and di-*o*-toluence thiourea were studied. In case of urea rate of hydrogen pick up by the sample was proportional to the rate of descaling. When thiourea was used as inhibitor hydrogen pick up was initially more in relation to urea. Later, it became proportional to the rate of descaling in both cases. Whereas with di-*o*-toulene thiourea initially hydrogen pick up was more but gradually it decreased with time.

Possibility of determination of the hydrogen present in steel in molecule and atomic form was examined by the electrochemical method.

70.0 Spectrographic Analyses

(i) *Rapid Method of Spectrographic Analysis of Slags* — Work was taken up to develop a suitable routine process for spectrographic analyses of steel making slags as well as blast furnace slags. In the manufacture of steel, importance is attached to analytical control of furnace slag. Samples taken at intervals throughout the process require analysis as quickly as possible. Conventional wet chemical methods are too slow to be of direct value to the melt being made. With a view to improve both the speed and accuracy of the analysis, attempts are being made to use the spectrograph as a tool for analysing steel making slag as well as blast furnace slag. Use of untreated slag sample has the advantage of speed but in view of the possibilities of loss of accuracy in the result due to the fact that slag structure varies greatly and may have different excitation characteristics for similar composition, it is necessary to treat the slag samples chemically for uniformity of composition and accuracy of results.

To start with, basic open hearth slag was taken up. The slag sample was fused with a mixture of sodium carbonate, 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. of hydrogen peroxide solution. A very clear solution of the sample was obtained. Large quartz spectrograph was used. Few drops of the solution were dried on a graphite electrode and A.C. intermittent arc was used for excitation taking specific pure carbon as counter electrode. For estimation of CaO and SiO_2 neutral filter having 10 per cent transmission of radiation was used. Prominent analytical lines of Si and Ca were obtained with satisfactory reduction of intensity of the lines suitable to be measured by microphotometer. For estimation of Al_2O_3, MnO and MgO, however, a filter need not be used. Since it was extremely difficult to prepare standard synthetically where Si is to be included, actual slag having variable known compositions were taken as standards. Attempts were made to prepare standards synthetically by taking known amounts of different constituents present in the slag by accurate weighing. These mixtures were to be fused and finally the mass was to be extracted into a clear solution. Optimum conditions for fusing them, and subsequently extracting them in solution were under study.

(ii) *Spectrographic Estimation of Phosphorus in Steel* — The objective of this study is to develop a method to estimate phosphorus in steel spectrographically using Hilger large quartz spectrograph. Spectrochemical analysis of phosphorus in steel is troublesome in the near ultraviolet and visible region. In order to overcome this trouble, instruments such as fluorite polychrometer-vacuum spectrograph have been developed which make use of the resonance lines of the elements in the far ultraviolet region. Attempts were made to make use of existing ' Hilger ' large quartz spectrograph in estimating phosphorus in steel. In case of large spectrograph, P 2136·2 A line was selected for analysis. Cu 2135·98 was an interfering line with P 2136·2 A line. This difficulty would not arise at all if the steel was absolutely free from copper. Exposures of standards samples of various phosphorus and copper contents were made (up to 0·46 per cent Cu). In case of 0·46 per cent Cu only, line of Cu 2135·98 A has come along with P 2136·2 A. Spectrographic plate of high sensitivity (Kodak B_{10}) was used. To have better sensitivity and excitation, rotating electrode was employed with a satisfactory result. Intermittent A.C. arc of 15Kv current 6 amps was used. Phosphorus line upto 0·02 per cent P contents is distinctly observed.

PILOT PLANTS

71.0 Low-shaft Furnace Pilot Plant Project

It is well known that due to the absence of metallurgical coking coals, establishment of iron and steel industry based on the conventional smelting in a blast furnace is not practicable in certain regions of India. Smelting trials of the Low-shaft Furnace Pilot Plant during the period under review were conducted with raw materials from Andhra Pradesh, Punjab and Maharashtra. The frequent change of physical and chemical characteristics of these raw materials employed for smelting called for considerable flexibility of approach and operational skill in maintaining continuous operations. It perhaps needs no emphasis that organized working of an integrated pilot plant with a consumption magnitude of over 100 tons of raw materials per day by a research laboratory demanded consistently systematic and methodical approaches for developing metallurgically feasible and commercially acceptable smelting techniques for the regional establishment of iron and steel plants in different parts of India. The iron ores of Andhra Pradesh have been successfully

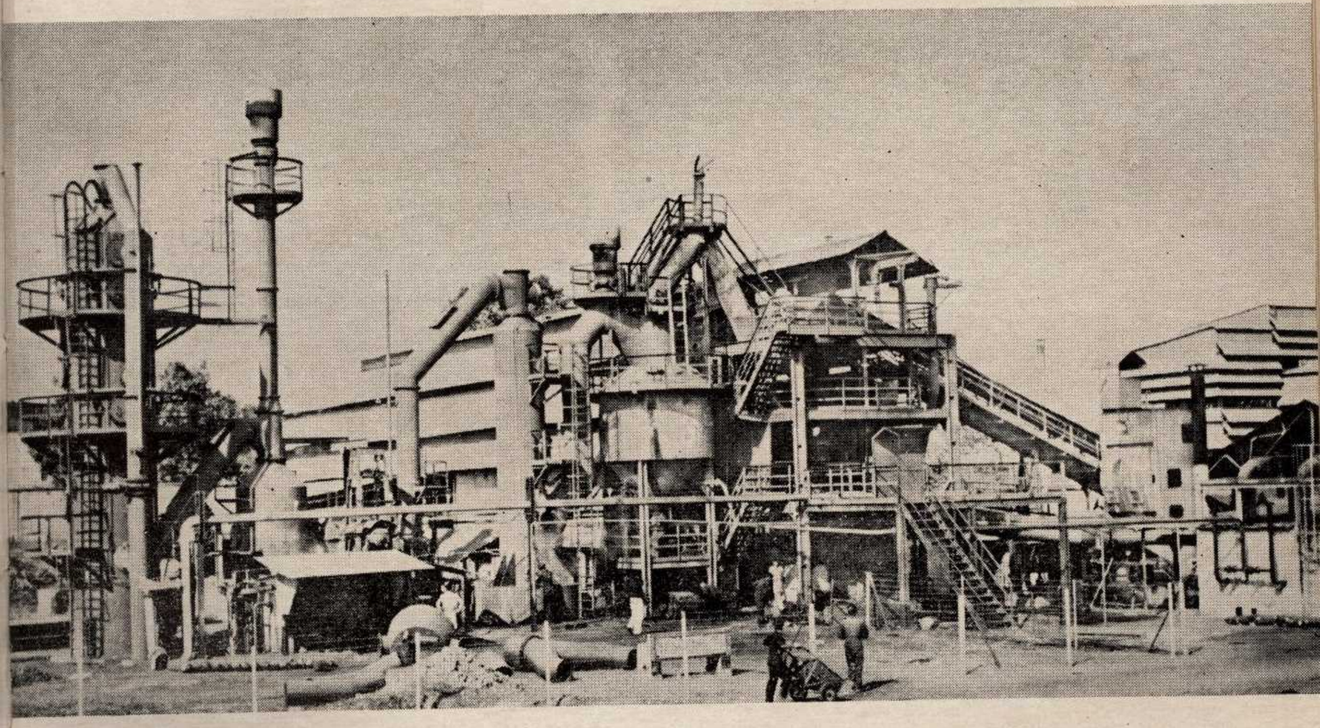


FIG. 64 — A PANORAMIC VIEW OF THE LOW-SHAFT FURNACE PILOT PLANT OF 15 TONS/DAY CAPACITY WITH ANCILLARY PLANTS

smelted both with direct use of non-coking coal from Singareni collieries, or with low-temperature carbonized coke made from Singareni non-coking coal, using dolomitic limestone from Andhra Pradesh. Although substantial quantity of pig iron was produced by the direct use of large varieties of non-coking coals, their industrial-scale application in a low-shaft furnace was more or less completely ruled out. Besides, in the utilization of non-coking coals directly, valuable byproducts are lost, which can be recovered by carbonization of non-coking coals at suitable temperatures. It was, therefore, considered that utilization of low-temperature carbonized coke with the elimination of moisture and recovery of volatile matter with consequent increase in its fixed carbon, better metallurgical reactivity and physical strength will not only improve the smelting characteristics but will also improve the overall economics of iron smelting.

The single-stage process developed by the Demag-Humboldt Niederschachtofen Gessellschaft, West Germany, depends on carbonization and smelting in a low-shaft furnace of self-fluxing briquettes containing iron ore, flux and non-coking coal in optimum proportions suitably bonded. The raw materials (0-5 mm.) are briquetted with 4 per cent coal-tar pitch and 4 per cent sulphite lye as binders. After ageing at room temperature for at least 24 hours the briquettes should not burst on sudden heating from the atmospheric temperature to 350-400°C. maintained at the top of the furnace and they should retain their shape until they reach the tuyere region to reduce unnecessary loss as dust. The caking index of coal and the reducibility of iron ore greatly influence the smelting process. Very little reduction of the iron oxide occurs in the upper part of the furance, but rapid reduction begins at a height of about 1 m. from the tuyere level, and 80 per cent of reduction occurs during the passage of the briquette to the tuyere level.

At room temperatures, coal-tar pitch binder provided the strength to the briquettes for transportation whilst sulphite lye on decomposition at 160-180°C. into intergranular envelopes strengthened the briquettes. At elevated temperatures the bonding strength due to binders disappeared and high-temperature strength of the briquettes was related to fusion of coal particles. Systematic investigations indicated that non-coking coals of extremely poor caking index did not provide such a fusion of coal particles or the strength to the briquettes for smelting. As the primary objective of smelting in a low-shaft furnace was to utilize non-coking coals, it became obvious that the briquetting process provided no means for the utilization of non-coking coals. Apart from the technological drawback it was found that to make 100 tons of briquettes, 4 tons of sulphite lye and 4 tons of coal-tar pitch are required as binders; with the prevailing market price of the former at Rs 175 per ton and the latter at Rs 280 per ton, the cost of binders will be Rs 1820 excluding the other raw materials, labour charges, depreciation, maintenance charges, etc. In other words, the cost of binders alone per tonne of pig iron would amount to Rs 121 which precludes the commercial use of the briquetting process for iron production. The smelting of briquettes, therefore, did not show any attractive metallurgical or operational features. As such, the single-stage process of smelting of briquetted burden proved unpractical and uneconomical under India's conditions and had to be discarded.

In order to economize fuel consumption and save metallurgical coke, preliminary investigations were conducted on the injection of coke-oven gas in an experimental furnace with simultaneous enrichment of the blast with oxygen during smelting trials. Based on these results, another low-shaft furnace with arrangements for the injection of the clean low-shaft furnace top gas through auxiliary tuyeres was designed and fabricated. (Fig. 65). Washed iron ore fines and beneficiated limestone were also briquetted. The beneficiated limestone

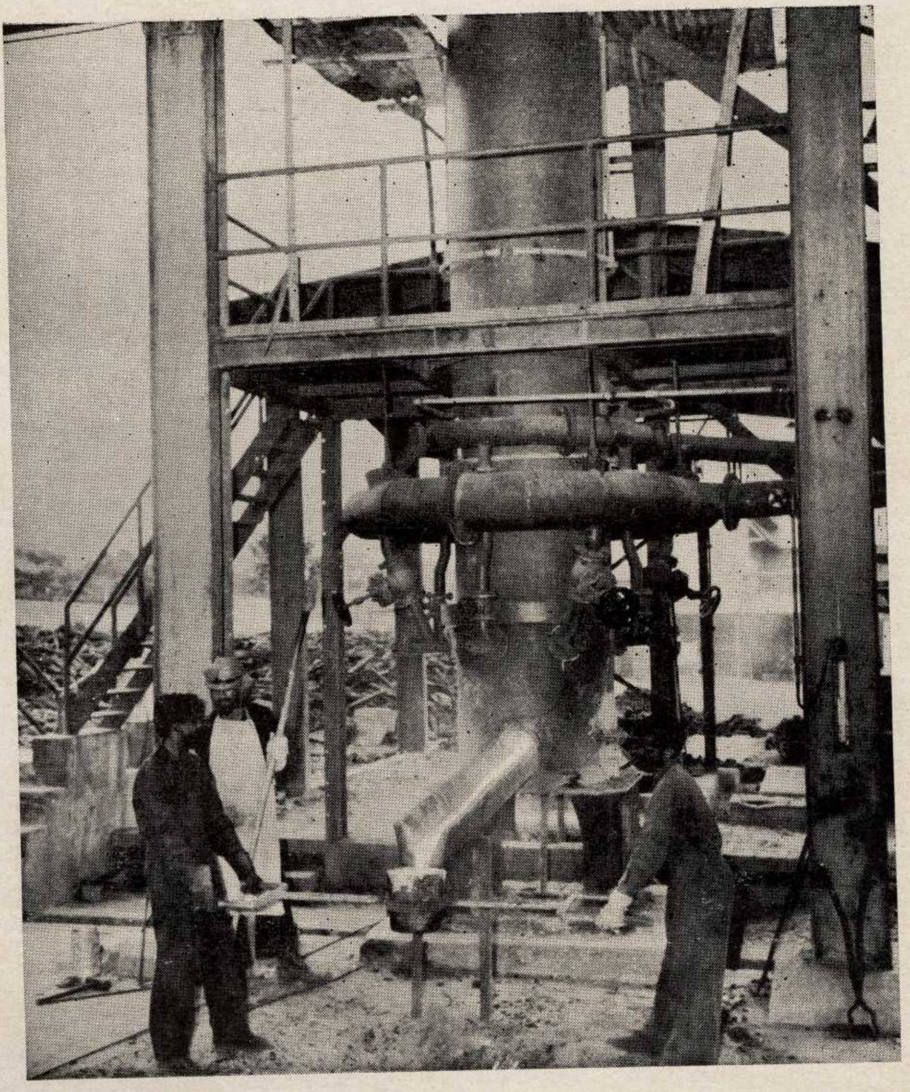


FIG. 65 — A VIEW OF TAPPING OF PIG IRON FROM THE 5-6 TONS/DAY CAPACITY LOW-SHAFT FURNACE HAVING PROVISION FOR FUEL INJECTION — DESIGNED AND FABRICATED AT THE NATIONAL METALLURGICAL LABORATORY

briquettes were successfully employed in steel making. The iron ore briquettes were smelted in a baby low-shaft furnace. Preliminary investigations were conducted to study the smelting characteristics of briquettes made of Salem magnetite concentrates with carbonized Neyveli lignite. Investigations were conducted on malleabilization of low-phosphorus pig iron produced in low-shaft furnace.

Programme of Work

As indicated earlier, the projected programme of work was based on the following phases:

Phase I

(1) Iron ores from Orissa and Bihar with non-coking coals from the Raniganj and Dishergarh coal either in bedded charge or by briquetting.

(2) Iron ores from Chanda district and non-coking coals from Ballarpur, Kamptee, and Wardha Valley, Bombay State.

(3) Iron ores from Chapra Antribeharipur in Mohindergarh, Punjab, with non-coking coals.

(4) Iron ores from Anantpur, Warangal, etc., and non-coking coals from Kothagudium, Yellandu, etc., in Andhra Pradesh.

(5) Iron ore from Katni in Madhya Pradesh with non-coking coals from adjacent coalfields (Kanhan and Pench Valley).

(6) Iron ores from Morinza near Jaipur and Udaipur, Rajasthan, initially with non-coking coals and then high-temperature carbonized Palana lignite [*vide* Phase III (i)].

(7) Iron ores from Siddhi in Uttar Pradesh and non-coking coal from Singrauli (Rewa Belt) etc.

Phase II — Smelting of self-fluxing briquettes made from iron ore fines, limestone and non-coking slack coals.

Phase III

(i) Utilization of lignite after its high-temperature carbonization for iron smelting.

(ii) Utilization of Salem magnetite after its beneficiation and briquetting in conjunction with carbonized lignite coke.

Phase IV — Utilization of low-temperature coke made from non-coking coals and other fuels.

Phase V — Research and development investigations on oxygen-enrichment of air blast, injection of oil and gas through auxiliary tuyeres, such as of low-shaft furnace clean gas, for uniforming fuel consumption and overall economics of smelting operations.

Phase VI — Production of ferro-alloys such as ferro-manganese with or without oxygen injection in the low-shaft furnace pilot plant.

In the period under report investigations were conducted under Phases I — (2), (3), (4) and Phase IV.

Furnace Campaigns

Depending on the characteristics of the raw materials employed, the smelting operations of the furnace in the period under review are divided into four campaigns, viz.

Campaign XI — Non-coking coals from the Singareni colliery with iron ores from Orissa.

Campaign XII — Burnt coal (jhama coal) from Jamadoba colliery with iron ores from Orissa.

Campaign XIII — Non-coking coal from Singareni collieries and low-temperature carbonized coke (Kolsite) made from it with iron ores and dolomitic limestone from Andhra Pradesh.

Campaign XIV — A. Smelting of iron ores from Mohindergarh district of Punjab initially with nut-coke and low-temperature carbonized coke, and later wholly with low-temperature carbonized coke made from totally non-coking coals from Singareni.

B. Smelting of iron ore and non-coking coal from Maharashtra.

Eleventh Furnace Campaign

The eleventh furnace campaign was conducted with non-coking coals from the Singareni colliery in Andhra Pradesh with iron ore and limestone used earlier and whose smelting behaviour was fully known thereby, followed by exclusive use of all the raw materials from Andhra Pradesh; this method of processing fairly obviated difficulties expected when all the raw materials of unknown smelting characteristics were employed.

The object of this campaign was primarily to assess the possibilities of exclusive utilization of non-coking coals from Singareni colliery, whilst adjusting the operational techniques to attain minimum fuel consumption.

The analyses of the raw materials employed are given on p. 131.

Furnace Operations

Tuyere diameter (mm.)	75
Hot blast volume (Nm.^{3}/hr)	2600-2800
Hot blast pressure (mmWC.)	2100-2600

Proximate Analyses of Non-coking Coal on Air-dried Basis

LOCATION	MOISTURE, %	V.M., %	F.C., %	ASH, %
1. Singareni Colliery (Andhra Pradesh)	7·1	26·1	49·5	17·4

Ash Analyses of Non-coking Coals

COLLIERY	SiO_2, %	Al_2O_3, %	CaO, %	MgO, %	Fe, %
Singareni	65·6	22·8	1·3	1·4	6·3

Analyses of Iron Ore

LOCATION	Fe, %	SiO_2, %	Al_2O_3, %	CaO, %	MgO, %
Barbil (Orissa) M/s Bird & Co.	61·1	2·2	9·5	Trace	Trace

Analyses of Limestone

LOCATION	CaO, %	MgO, %	SiO_2, %	Al_2O_3, %
1. Rajur (Maharashtra)	47·3	3·5	6·7	0·9
2. Birmitrapur (Orissa, Bisra Limestone)	44·8	3·3	6·96	1·6

Hot blast temperature (°C.)	590- 600
Fuel consumption (fixed carbon/ton of pig iron)	1·85
Slag volume in ton/ton of pig iron	1·8

The average composition of pig iron smelted was as follows:

	Per cent
Total carbon ...	2·5-3·5
Silicon ...	2·5-4·0
Manganese* ...	0·2
Sulphur ...	0·1-0·9

The average analysis of the slag was as follows:

	Per cent
CaO ...	37-43
SiO_2 ...	35-37
MgO ...	2·5-3·5
Al_2O_3 ...	18-20
FeO ...	0·5-1·0

Sulphur was effectively partitioned between the slag and the metal; the basicity and the high volume of the slag both contributed towards effective desulphurization of the pig iron smelted.

The top gas temperature varied from 300° to 430°C., while the CO/CO_2 ratio of the top gas varied from 5 to 6. An increase in the height of the burden column enabled lowering of the CO/CO_2 ratio of the gas — it reduced the lower limit of the top gas temperature below the optimum necessary for the prevention of condensation of tar from the top gas. Under these operational conditions the top gas analysed:

	Per cent
CO ...	21-23
CO_2 ...	3·5-4·5
CH_4 ...	6-9
C_nH_{2n+2} ...	0·3-0·5
H_2 ...	0·2-0·8

There was considerable generation of flue dust. The particle size of the dust was extremely fine. Summarizing the smelting performance in this campaign, it may be mentioned that the operation was characterized by irregular descent of the burden, and constant vigilance over the smelting operation was required throughout the campaign.

Twelfth Furnace Campaign

This campaign was conducted to find out the feasibility of utilization of burnt coke (15 seam jhama coal) from Digwadih colliery of the Tata Iron & Steel Company Limited. The analyses of raw materials employed in this campaign were as given in the upper table on page 132.

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*No manganese ore was added in the burden, and as such the manganese content of the pig iron was low.

Proximate Analyses of Jhama Coal on Air-dried Basis

LOCATION	MOISTURE, %	V.M., %	F.C., %	ASH, %
Digwadih (Jharia)	1·0	14·0	69·8	15·2

Analyses of Ash of Jhama Coal

COLLIERY	SiO_2, %	Al_2O_3, %	CaO, %	MgO, %	Fe, %
Digwadih (Jharia)	52·8	34·2	3·4	4·7	4·1

Analyses of Iron Ores

LOCATION	Fe, %	SiO_2, %	Al_2O_3, %	CaO, %
1. Barajamda (Orissa)	61·1	2·2	3·93	0·04
2. Barbil (Orissa)	61·4	1·52	3·93	—

Analyses of Limestone

LOCATION	CaO, %	MgO, %	SiO_2, %	Al_2O_3, %
Birmitrapur (Orissa)	44·8	3·3	6·96	1·6

The burnt coke (jhama coal) was received in big lumps of 4 in. to 6 in. each, unsuitable for direct charging to the low-shaft furnace. It was crushed to −2 in. to + ¾ in. size for smelting. The nut-coke in the fuel burden was gradually replaced exclusively by this burnt coke (jhama coal) and the furnace was blown under the following operational conditions:

Tuyere diameter (mm.)	75
Hot blast volume (Nm.^{3}/hr)	2000-2100
Hot blast pressure (mmWC.)	2300-2900
Top gas temperature (°C.)	420-460
Fuel consumption (fixed carbon/ ton of pig iron)	2·6-3·0
Slag volume tons/ton of pig iron	1·2

Under these operational conditions, the analysis of pig iron, slag and top gas was as follows:

PIG IRON		SLAG	
Carbon, %	2·8-3·0	CaO, %	32-36
Silicon, %	1·0-2·0	SiO_2, %	32-34
Sulphur, %	0·12-0·15	FeO, %	2
		Al_2O_3, %	25-27
		MgO, %	5

TOP GAS	
CO, %	24
CO_2, %	4
CH_4, %	4·5

The smelting operation was extremely irregular causing frequent tendency towards chilling of the hearth yielding cold metal and slag as evidenced by high sulphur contents of the metal and high iron oxide contents of the slag. Frequent blank charges of nut-coke and limestone had to be fed additionally to provide requisite smelting heat. Despite these burden adjustments and the extremely high fuel rate of 3 tons of fixed carbon per ton of pig iron, two opposite tuyeres became blocked due to deposition of a pasty mass and further penetration of the air blast became progressively more difficult. Ultimately, the burden did not descend due to extremely slow rate of combustion around the tuyere region and the smelting was discontinued.

It was concluded that exclusive use of burnt coke (jhama coal) was not possible. It may, however, be possible to incorporate a certain amount of burnt coke (jhama coal) with nut-coke or non-coking coals for smelting in a low-shaft furnace.

Thirteenth Furnace Campaign

With a view to establish a small integrated iron production unit, the State Government of Andhra Pradesh sent iron ore, limestone, non-coking coal (Singareni collieries) and low-temperature carbonized coke made from the same non-coking coal for investigations. Successful smelting trials were conducted with low-temperature carbonized coke made from Singareni non-coking coal. In this campaign, therefore, comprehensive investigations were conducted for the development of a suitable smelting technique for the exclusive utilization of these regional raw materials for the production of acceptable foundry grades of pig iron. The smelting operation can broadly be subdivided into two categories depending on the nature of the fuel employed, namely — (a) non-coking coal from the Singareni collieries, and (b) low-temperature carbonized coke made from the same non-coking coal termed as ' Kolsite '.

The analyses of the raw materials employed were as follows:

Analyses of Non-coking Coal

Location	Moisture, %	V.M., %	F.C., %	Ash, %
Singareni Colliery (Andhra Pradesh)	7·1	26·1	49·5	17·4

Ash Analyses of Non-coking Coals

Colliery	SiO_2, %	Al_2O_3, %	CaO, %	MgO, %	Fe, %
Singareni (Andhra Pradesh)	65·6	22·8	1·3	1·4	6·3

Analyses of Low-temperature Carbonized Coke

Analyses	General Range		Typical Individual Sample			
	Coal (Air-dry basis), %	Kolsite, %	Coal (Air-dry basis), %		Kolsite, %	
			I	II	I	II
Moisture	5-7	2-3	6·80	5·1	2·1	2·8
Ash	18-20	21-23	16·04	19·6	21·5	22·6
V.M.	25-28	8-9	26·15	26·4	8·4	8·1
F. carbon	45-50	65-67	51·01	48·9	68	66·5
Calorific value, B.t.u./lb.	9,800-10,500	11,000-11,400	—	10,500	—	11,400

Kolsite — Ash Analyses

	% (General range)	% (Typical Individual Sample)
Sio_2	63-65	62·07
A_2lO_3, TiO_2	20-23	22·72
CaO	2·5-3	1·59
MgO	1·5-2·5	2·18
Fe_2O_3	10-15	10·98
SO_3	0·5-0·7	0·36
Alkali (by difference)	Less than 1	0·10
Fusion (under mildly reducing and mildly oxidizing atmosphere, Leitz, Heating microscope method) 1500-1500°C. 1555°C.		
Source of Coal	Singareni Collieries Co. Ltd, Kothagudem	

Analyses of Iron Ore

Fe, %	60-64	SiO_2, %	3·8-4·6	Al_2O_3, %	2-5·92

Analyses of Limestone

Location	SiO_2, %	Al_2O_3, %	CaO, %	MgO, %
Andhra Pradesh limestone	0·3	0·56	32·2	25·0
Bisra limestone (Orissa)	8·7	1·6	44·8	3·3

In view of the earlier observed position that the use of $-\frac{1}{4}$ in. fraction of non-coking coal or of Kolsite led to irregularities for the descent of the burden, both Singareni coal and Kolsite were screened through $\frac{1}{2}$ in. screen and $-\frac{1}{2}$ in. portion was not used. The furnace was blown under the following operational conditions.

OPERATIONAL VARIABLES	CAMPAIGN 13A	CAMPAIGN 13B
Tuyere diameter (mm.)	75	75
Hot blast volume ($Nm.^3/hr$)	2600-2800	2500-2700
Hot blast pressure (mmWC.)	1800-2000	1800-2100
Hot blast temperature (°C.)	590-600	590-600
Top gas temperature (°C.)	325-450	270-350
Fuel consumption (fixed carbon/ton of pig iron)	1·8-1·95	2·03-2·3
Slag volume per ton of pig iron	1·3-1·5	1·8-2·0

The average analyses of pig iron were as follows:

	%
Total carbon	2·7-3·5
Silicon	2·75-4·5
Manganese*	0·2-0·4
Sulphur	0·01-0·10
Phosphorus	0·14-0·17

*No manganese ore was added which explained the low manganese content of the pig iron.

The average analysis of slag was as follows:

CONSTITUENTS %	CAMPAIGN 13A	CAMPAIGN 13B
CaO	30	32
SiO_2	35	35
Al_2O_3	17	16
MgO	18	17
FeO	0·8	0·7

The average analysis of top gas was as follows:

CONSTITUENTS %	CAMPAIGN 13A	CAMPAIGN 13B
CO	25	24
CO_2	5·0	4·5
CH_4	7·0	8·5
H_2	0·8	1·0

Operations with low-temperature carbonized coke made out of totally non-coking coals, containing 8-9 per cent volatile matter and 65-67 per cent fixed carbon, were, however, characterized by exceedingly regular descent of the burden resulting in suitable end product, i.e. foundry grade pig iron of desired analyses. The furnace top temperatures could also effectively be regulated between the desired level of 350° and 400°C. The CO/CO_2 ratio varied from 3·5 to 4·5 and a minimum fuel rate of 1·28 tonnes of fixed carbon/tonne of pig iron was also obtained; the low fuel rate can be attributed to much higher metallurgical reactivity of the low-temperature carbonized coke (Kolsite). Dust formation was within tolerable limits and continuous furnace operations were fully feasible.

Fourteenth Furnace Campaign

A. The fourteenth furnace campaign was conducted with iron ore from Mohindergarh in East Punjab. This ore on average contains about 0·7 per cent phosphorus which will cause high phosphorus contents in the pig iron. Due to non-availability of regional coal resources the smelting of iron ore from Mohindergarh district, Punjab, was conducted with non-coking coal, low-temperature carbonized coke (Kolsite and C.F.R.I.) and nut-coke with variations introduced in particle sizes of the iron ore. It was observed that smelting of this ore was fully feasible as also highly successful with either low-temperature carbonized coke or nut-coke. For smelting operations with nut-coke, the furnace stack height will have to be somewhat raised for ensuring effective heat exchange between the descending burden and ascending gases and promotion thereby of adequate metallurgical reaction between the two. As both these factors greatly affect the fuel rate and thereby reflect on the cost of iron production, stack height of the furnace for smelting operations with nut-coke will have to be somewhat suitably increased.

The analysis of raw materials employed in this campaign was as follows :

Analysis of Low-temperature Carbonized Coke

	General Range		Typical Individual Sample			
	Coal (Air-dry basis) %	Kolsite %	Coal (Air-dry basis) %		Kolsite %	
			I	II	I	II
Moisture	5-7	2-3	6·80	5·1	2·1	2·8
Ash	18-20	21-23	16·04	19·6	21·5	22·6
V.M.	25-28	8-9	26·15	26·4	8·4	8·1
F. carbon	45-50	65-76	51·01	48·9	68·0	66·5
Calorific value (B.t.u./lb.)	9,800-10,500	11,000-11,400	10,500		11,400	

Analysis of Iron Ore

Location	Fe %	SiO_2 %	Al_2O_3 %	P %	S %
Nizampur (Punjab)	62·6	6·65	2·0	0·4-1·2	0·03

Analysis of Limestone

Location	CaO %	SiO_2 %	Al_2O_3 %	MgO %	Fe_2O_3 %
1. Andhra (M/s. Singareni Collieries Co.)	32·2	0·03	0·56	25·0	—
2. Birmitrapur (Orissa)	44·8	6·96	1·6	3·57	—
3. Madras	54·31	0·88	1·23	1·01	0·33

After the initial operation of the furnace with nut-coke, the latter was gradually replaced by non-coking coal. It was, however, found that on increasing the percentage of Singareni coal in the burden beyond 50 per cent, various operational difficulties such as irregular descent of the burden, difficulties in the penetration of the blast, generation of abnormal dust inside the furnace resulting in frequent opening of the explosion doors were experienced. The amount of dust carried over was also substantial which showed poor utilization of the fuel for smelting.

The previous trials with fresh coal, however, were more practicable. It was concluded that the weathering of the non-coking coal due to its long exposure to atmosphere might have affected its physical properties leading to its immediate disintegration into fines on entry into the furnace. As such, the non-coking coal was ultimately replaced by low-temperature carbonized coke made out of the same non-coking coal and ultimately the furnace was operated with 100 per cent low-temperature carbonized coke (Kolsite) comprising the fuel burden.

Fig. 66 — A view of the side-blown basic converter of $\frac{1}{2}$ ton capacity in operation. The converter has been designed and fabricated at the National Metallurgical Laboratory

The furnace was operated under the following conditions:

Tuyere diameter (mm.)	75
Hot blast volume (Nm.^{3}/hr)	2000-2200
Hot blast pressure (mmWC.)	2150-2400
Hot blast temperature (°C.)	590
Top gas temperature (°C.)	350-400
Slag volume (per ton of pig iron)	1·65 tons

During smelting operations with Kolsite, different burden schedules with variations in the fuel rate and basicity ratio of the slag aiming at low slag volume were investigated. Fluxes containing Bisra limestone and Andhra dolomitic limestone were employed; the latter was added primarily to give a MgO content of 8-10 per cent in the slag. It was found that a burden with a calculated slag composition: CaO 43 per cent, SiO_2 36·5 per cent, MgO 9 per cent, Al_2O_3 12 per cent having a calculated lime basicity CaO/SiO_2 gave very satisfactory results. The high fuel consumption has to be reckoned due to high slag volume. Operations with low-temperature carbonized coke made out of totally non-coking coals containing 8-9 per cent volatile matter and 60-62 per cent fixed carbon were characterized by regular descent of the burden and no difficulties were experienced in the smelting operation. The composition of slag and that of pig iron and gas under these conditions were as follows :

Composition of Slag :	*Per cent*
CaO	43·2
SiO_2	36·0
FeO	11·32
Al_2O_3	17·4
MgO	5·5

Composition of the Metal :	*Per cent*
Total carbon	3·4-4·0
Si	1·78-2·9
S	0·02-0·08
P	0·21-0·37
Mn	0·24-0·48

Top Gas Analysis :	*Per cent*
CO	22·4-24·3
CO_2	4·2-3·5
CH_4	7·7-7·2
CO/CO_2	5·3-6·8

In order to obtain adequate manganese content in pig iron, a small quantity of manganese ore assaying: Mn, 50·7; Fe, 5·6; SiO_2, 11·2; and Al_2O_3, 10·3 per cent, was added to each individual charge due to which the Mn content in the metal was raised from the normal of 0·3 to nearly 1·0 with a manganese recovery of over 50 per cent.

On the completion of the investigations with low-temperature carbonized coke, investigations were conducted with nut-coke and varying particle size of iron ore primarily to assess the influence of particle size of ore on the operational characteristics. In view of the low and restricted throughput time in a low-shaft furnace, the proper matching of the particle size of various materials is highly significant from the point of view of heat-exchange criterion and is thus very important. It was previously experienced that the particle sizes of the raw materials greatly influence the temperature of the top gas, the CO/CO_2 ratio of the top gas, the fuel rate and the productivity of the furnace. It was considered that suitable adjustments of the particle size of the iron ore will not only promote indirect reduction leading to better CO/CO_2 ratio and fuel economy, but will also lower the top temperature of the gas, thereby reducing the loss of sensible heat. Both these factors will help in fuel economy. With these objects in view, the iron ore was crushed and screened into four different sizes and investigations were conducted under identical conditions with the only exception of the variation of the particle size of the ore.

The operational data under the four different size classifications of the iron ore are summarized in Table 53.

An examination of the data revealed that the fuel rate decreased from 2·09 to 1·54 tonnes of fixed carbon per tonne of pig iron, with simultaneous decrease in the flux rate from 0·96 to 0·81 tonnes and decrease in the slag volume from 1·18 to 0·97 tonnes per tonne of pig iron. The CO/CO_2 ratio decreased from 6·4 to 5·4 which showed improvements in the indirect reduction. The top gas temperature dropped from 410° to 360°C. The employment of fine-grained material obviously increased the dust production which increased from 6 per cent to about 10 per cent of the weight of raw material charged. It was, therefore, concluded that there was a limitation to the use of fine-grained material, since highly fine-grained material led to various operational difficulties such as irregular descent of the burden, and difficulties in the blast penetration due to the reduced void space in the burden column, bridge formation in furnace stack causing frequent opening of the explosion doors for the release of excess pressure developed inside the furnace. The degree of fineness of the ore or limestone suitable for smelting also

TABLE 53 — OPERATIONAL DATA OF SMELTING PROCESS UNDER VARIOUS SIZES OF IRON ORE

PARTICULARS	INVESTIGATION 1	INVESTIGATION 2	INVESTIGATION 3	INVESTIGATION 4	INVESTIGATION 5	INVESTIGATION 6
Raw materials employed	Lumpy Punjab ore + Nut-coke	Medium size ore + Nut-coke	Medium 50%, Fine 50% size ore + Nut-coke	Fine size + Nut-coke	L.T.C. (F.R.I.) + Medium size ore	L.T.C. (F.R.I.) + Fine ore
Fuel rate/tonne of P.I.	2·09	1·98	1·65	1·54	2·03	1·81
Slag volume/tonne of P.I.	1·18	1·16	1·04	0·97	1·65	1·52
Iron ore/tonne of P.I.	1·47	1·47	1·47	1·47	1·47	1·47
Flux rate/tonne of P.I.	0·96	0·96	0·88	0·81	1·32	1·25
Average analysis of metal:						
C,%	2·9	3·1	2·52	3·2	3·4	3·75
Si,%	2·6	2·9	3·0	3·7	2·6	3·27
S,%	0·07	0·11	0·06	0·066	0·10	0·11
P,%	0·66	0·58	0·63	0·68	0·87	0·64
Mn,%	0·25	0·32	0·26	0·38	0·53	0·80
Average analysis of slag:						
CaO	39·84	38·1	41·2	37·90	40·4	35·3
SiO_2	33·40	38·48	32·6	34·20	37·64	42·4
FeO	0·88	1·0	0·77	0·67	1·1	0·55
Al_2O_3	17·00	18·0	19·78	20·30	15·9	18·05
MgO	8·30	5·3	5·6	6·40	4·8	5·0
S	1·07	0·84	0·95	1·2	0·71	0·62
P_1 (C)	1·29	1·40	1·50	1·47	1·22	1·38
P_1 (A)	1·19	0·9	1·22	1·10	1·07	0·8
Average gas analysis:						
CO	2·50	24·0	24·0	26·8	23·0	24·0
CO_2	3·8	3·8	4·0	4·6	3·8	4·2
CH_4	3·6	3·4	3·50	3·0	4·2	4·5
CO/CO_2	6·4	6·3	6·0	5·4	6	5·7
Blast volume (Nm.^{3}/hr)	2700-3000	2500-2800	2200-2500	2200-2600	1900-2300	1900-2100
Blast pressure (mmWC.)	2000-2300	2100-2500	2100-2300	2000-2200	2100-2500	2100-2500
Hot blast temp. (°C.)	580-600	575-590	560-580	580-590	580-600	580-590
Top gas temp. (°C.)	380-440	350-420	350-400	320-380	350-420	340-400

depended on the fuel employed. It was found that while ore fines containing 55 per cent below —⅛ in. would be compatible with nut-coke for attaining minimum fuel rate, a similar combination with non-coking coal would not be amenable to smelting due to the various operational difficulties indicated earlier. The generation of a large amount of dust from the non-coking coal itself in the furnace will not permit the use of iron ore or flux fines to a large extent.

This investigation was followed by a series of investigations in which two different sizes of iron ore (Punjab) were smelted with low-temperature carbonized coke made from non-coking coals obtained from the Central Fuel Research Institute, Dhanbad. The smelting operation was quite smooth and regular and there were conspicuous absence of hanging or bridge formation. These investigations again confirmed earlier observations that the fuel consumption considerably decreased with the fineness of the iron ore. The larger particle size of the fuel led to high top gas temperature and lower CO/CO_2 ratio compared to the operation with nut-coke. The consumption of fuel in this case was higher than that obtained with nut-coke which was presumably due to the above factors and high ash content of the low-temperature carbonized coke leading to a high slag volume of 1·6 tonnes/tonne of pig iron. The operational data under these conditions have also been recorded in Table 53 for comparison.

B. Government of Maharashtra had sent their regional raw materials, namely iron ore, limestone and coal, for conducting necessary trials and the results based on the trials with non-coking coal from Ghughus colliery in Wardha Valley had been reported earlier. Further trials were conducted with non-coking coal from Kanhan-Kamptee colliery during the period under review.

After stabilization of the smelting conditions using low-temperature carbonized coke, several concentrated attempts were made for the gradual replacement of this fuel by non-coking coal from the Kanhan-Kamptee colliery. The analysis of raw materials employed are given below.

It was considered that a straight forward replacement of the low-temperature carbonized coke by Kanhan-Kamptee non-coking coal merely on the basis of their fixed carbon contents will lead to severe chilling of the furnace hearth. Adequate precuations were taken to replace one part of fixed carbon of low-temperature carbonized coke by 1·5 parts of fixed carbon of the non-coking coal which was expected to take care of the additional heat requirements for the dehydration and carbonization of non-coking coal in the higher stack regions of the furnace. It was noticed that regularity of the smelting operations could be had with a maximum of only 32 per cent of the non-coking coal in the fuel burden. But even under these conditions, apart from the difficulties mentioned earlier, tuyere openings were blocked at frequent intervals and as such frequent additions of blank charges consisting of nut-coke and limestone as corrective dozes were necessary whilst adequate hearth heat appeared to be lacking. This resulted in the cold metal containing high sulphur (0·12 per cent) and the slag containing high FeO (5-8 per cent). The operational conditions of the furnace with 32 per cent Kanhan-Kamptee coal and 68 per cent low-temperature carbonized coke in the fuel burden were as given on p. 140.

Analysis of Iron Ore

LOCATION	Fe %	SiO_2 %	Al_2O_3 %	P %	S %
Chanda (Maharashtra)	64·3	3·1	2·5	0·83	—

Analysis of Non-coking Coal

COLLIERY	H_2O %	V.M. %	F.C. %	ASH %
Ghughus	9·7	34·4	42·8	13·1
Kanhan-Kamptee	5·0	34·0	37·6	23·4
Hindustan Lalpeth	5·3	32·7	45·8	16·2

Analysis of Limestone

LOCATION	CaO %	MgO %	SiO_2 %	Al_2O_3 %
Rajur (Maharashtra)	47·28	3·45	6·68	0·35

Analysis of Ash of the Non-coking Coals

COLLIERY	SiO_2 %	Al_2O_3 %	CaO %	MgO %	Fe_2O_3 %
Ghughus	57·8	34·95	3·7	2·8	0·85
Kanhan-Kamptee	55·0	34·9	2·7	3·2	4·1
Hindustan Lalpeth	42·8	47·0	4·8	2·3	2·6

Fuel consumption F.C., tonnes/tonne of pig iron	2·5
Volume of blast (Nm.^{3}/hr)	1800-2000
Pressure of the blast (mmWC.)	2200-2400
Blast temperature (°C.)	580-600
Top gas temperature (°C.)	300-350
Flux rate, tonnes/tonne of pig iron	1·5
Slag volume/tonne of pig iron	2·0

Compositions of Slag:

	Per cent
CaO	33-34
SiO_2	34-35
FeO	0·8-2·0
Al_2O_3	18-22
MgO	8-9

Composition of Pig Iron:

	Per cent
C	2·75-3·0
Si	3·0 -3·5
S	0·04-0·10
Mn	0·6 -0·8
P	0·4 -0·6

Gas Analysis

	Vol. per cent
CO	20-22
CO_2	4-5
CH_4	5-7
CO/CO_2 ratio	4·5

During the investigations with non-coking coals, high volume of dust, 10-12 per cent of the weight of raw materials, was generated.

Proximate Analysis of Burnt Coke, Low-temperature Carbonized Khaskenda Coals

LOCATION	MOISTURE %	V.M. %	F.C. %	ASH %	S %
Burnt coke	1·0	14·0	60·8	15·2	—
Low-temperature carbonized coke	5·8	4·62	61·58	28·0	—
Khaskenda	2·8	30·2	49·86	17·14	0·5
Singareni	7·1	26·1	49·5	17·4	—

Ash Analysis of Burnt Coke, Low-temperature Carbonized Khaskenda Coals

LOCATION	SiO_2 %	Al_2O_3 %	CaO %	MgO %	Fe %	P_2O_5 %	SO_3 %	P %
Burnt coke	52·8	34·2	3·4	4·7	4·1	—	—	—
Low-temperature carbonized	56·31	21·99	3·45	2·20	10·87 (as Fe_2O_3)	1·83	0·73	—
Singareni	65·6	22·8	1·3	1·4	6·3	—	—	—
Khaskenda	49·04	24·03	2·44	1·35	4·37	—	—	0·59

Analysis of Iron Ore

LOCATION	Fe %	SiO_2 %	Al_2O_3 %	CaO %	MgO %	S %	P %
Nanalal Varjang	61·4	1·52	3·93	—	—	—	—

Analysis of Limestone

LOCATION	CaO %	SiO_2 %	Al_2O_3 %	MgO %
Bisra	44·8	6·96	1·6	3·3
Bombay	47·28	6·68	0·85	3·45
Madras	54·31	0·88	1·2	1·0
Andhra	32·2	0·3	0·56	25

At the end of either slagging and tapping operations, large amount of ignited fuel pieces were ejected. Based on pilot plant trials in the low-shaft furnace of the National Metallurgical Laboratory, it was concluded that direct utilization of non-coking coals from Kanhan-Kamptee, Hindustan Lalpeth and Ghughus collieries of Maharashtra will not be possible for iron making in a low-shaft furnace charged either directly in lumpy bedded form or through one component burden as self-fluxing briquettes.

Fifteenth Furnace Campaign

During this campaign, iron ore from Orissa (Nanalal Varjang) was smelted with burnt coke from Digwadih colliery in gradual steps up to 32 per cent of the total fuel, balance being nut-coke. An unsuccessful attempt was made previously to utilize burnt coke (Campaign XII). It was considered that prior crushing and screening of burnt coke might lead to improvement in smelting. However, due to the development of inadequate hearth heat due to high ash contents, the smelting trials were not successful. Further trials included smelting of iron ore with low-temperature carbonized coal (from Fuel Research Institute) and replacing low-temperature carbonized coal by non-coking coal (Singareni) in gradual steps. But even 25 per cent of the Singareni coal used in the burden led to serious furnace operational difficulties like hanging etc. Trials were then conducted with non-coking coal from Khaskenda colliery and ultimately 100 per cent of this coal was employed.

The analyses of raw materials utilized in this campaign are shown in the table on the previous page.

Furnace Operation

Exclusive employment of the burnt coke was not possible due to its ash contents which contained very high amounts of alumina.

The furnace was blown in under the following operational conditions :

Tuyere diameter	75 mm.
Hot blast pressure	2500 (mmWC.)
Hot blast temperature	590°C.
Hot blast volume	2000 Nm.^{3}/hr
Fuel rate/tonne of pig iron	2·5 tonnes
Slag volume/tonne of pig iron	1·15 tonnes

Under these conditions, the analyses of pig iron, slag and the furnace top gas were as follows :

PIG IRON		SLAG	
Carbon	2·7-3·0%	CaO	43·00-39·4%
Silicon	3·1-5·0%	SiO_2	31·88-29·3%
Sulphur	0·14-0·009%	FeO	0·7-0·5%

TOP GAS	
CO	22·3
CO_2	3·8
CH_4	3·6
CO/CO_2	5·8

Under the same operational conditions, the fuel was gradually changed to non-coking coal (Khaskenda collieries). Although exclusive employment of it was possible, the usual difficulties like development of high pressure inside the furnace with irregular descent of the burden were noticed. The effect of change in the degree of basicity of 0·8, 1·0, and 1·2 on smelting operation of the furnace, particularly the fuel rate and desulphurization, was studied. The fuel consumption on average amounted to 2·3 tonnes of fixed carbon per tonne of pig iron, while the corresponding slag volume was 1·5 to 1·65 tonnes per tonne of pig iron.

		I STAGE	II STAGE
Basicity		1·0-1·1	1·2-1·3
Fuel rate		2·34	2·45
Slag volume		1·42	1·58
Average analyses of metal:	C,%	3·15	3·0
	Si,%	4·10	2·9
	S,%	0·10	0·08
Average analyses of slag:	CaO,%	37·8	41·4
	SiO_2,%	33·8	32·0
	FeO, %	1·11	1·21
Top gas analyses:	CO,%	22·8	23·6
	CO_2,%	3·4	2·0
	CH_4,%	7·4	8·2
	CO/CO_2	6·7	7·9

In general it was found that the fuel rate increased with slag basicity degree and its volume.

71.1 Investigation on Briquetting

(i) *Briquetting of Iron Ore Fines* — Kiriburu iron ore was subjected to beneficiation treatment at the National Metallurgical Laboratory including washing, scrubbing and other treatments which yielded washed iron ore concentrate assaying 64·2 per cent Fe, 1·89 per cent SiO_2, and 1·1 per cent Al_2O_3; this iron ore was screened and minus 5 mm. fraction excluding very fine ' sand ' fraction was employed for briquetting tests. Water, bentonite, sulphite lye, totanin, cement and hydrated lime were used as binders either singly or jointly in different proportions.

Briquettes were made with additions of 2, 4 and 6 per cent water, and were tested either after drying at room temperature or at 200°C. The crushing strength of these briquettes was very poor, and on dropping these briquettes from a height of 6 ft and above (Shatter Test) the briquettes crumbled to pieces. It was, therefore, concluded that in view of the absence of inherent binding properties, the iron ore washed fines cannot be agglomerated merely by the addition of water.

Briquettes were made with the additions of 2, 4 and 6 per cent bentonite as binder with the addition of 4-6 per cent water. These briquettes were subsequently dried at 200°C. The room temperature crushing strength was satisfactory. The shatter test characteristics of these briquettes were as follows :

FRACTION	2% BENTONITE	4% BENTONITE	6% BENTONITE
−2″ to +1″	0·0	0·0	47·4
−1″ to +½″	21·5	51·5	22·8
−½″ to +¼″	18·3	12·1	11·7
−¼″ to +⅛″	18·9	14·6	8·4
below ⅛″	41·3	21·8	9·7

With the exception of briquettes with 6 per cent bentonite as binder, shatter test values of briquettes made with smaller quantities of bentonite as binder were poor and unsuitable for furnace smelting.

Briquettes with Sulphite Lye — Briquettes were made with the additions of 2-4 per cent sulphite lye as binder with some water. The room temperature compressive strength of the briquettes was satisfactory. The shatter test characteristics of the briquettes with 4 per cent sulphite lye after ageing at room temperature followed by drying at 200°C. were as follows :

FRACTION	BRIQUETTES WITH 4% SULPHITE LYE	
	Dried in air %	Dried at 200°C. %
−2″ to +1″	92·0	91·0
−1″ to +½″	6·9	7·2
below ½″	1·1	1·8

Briquettes with Slaked Lime — Briquettes were made with the additions of 2, 6, 4, 8, 10, 15, 20 and 25 per cent slaked lime as binder. Although the shatter index attained a high value with 20 per cent binder (slaked lime) additions, strength of the briquettes was not high enough to warrant smelting; this was attributed to the poor quality of the hydrated lime. It was also felt that drying in an atmosphere of CO_2 available from waste flue gases may improve the bonding. For satisfactory briquettes, a compressive strength of about 60 kg./cm.^{2} and 70 per cent fraction above 10 mm. on shatter tests after a fall from 2 m. was necessary.

Briquettes with ' Totanin ' as Binder — Briquettes were made with the addition of ' totanin ' as a binder — a by-product from tanning industry. The proportion of the binder was gradually increased from 2 to 6 per cent in steps of one per cent, keeping the moisture content constant at 4 per cent. The briquettes were aged in atmosphere for 48 hours, and then dried at 200°C. for 45 minutes. The results are shown in the table on top of page 143.

The addition of 4-6 per cent totanin resulted in a crushing strength of 700 lb. and on shatter test 86 to 97 per cent of broken

QUANTITY OF BURDEN	FRACTION, %				
	−2″+1″	−1″+½″	−½″+¼″	+¼″+ − ⅛″	⅛″
2% binder	43·3	29·0	9·8	5·8	14·1
3% binder	72·9	5·2	8·6	7·5	5·8
4% binder	85·9	8·2	1·9	1·8	2·2
5% binder	94·8	5·2	—	—	—
6% binder	97·5	2·5	—	—	—

briquettes were in 2-1 in. fraction. The briquettes were suitable for transport handling and for subsequent smelting. Crushing strength of these briquettes was found to be poor at elevated temperature of 600°-800°C., evidently due to the decomposition of the ‘totanin’ binder.

High temperature optimum crushing strength of briquettes was an important criterion. In view of the fact that no binder provided adequate high temperature crushing strength, non-coking coal was added to the iron ore fines to impart high temperature maximum crushing strength which rose to 168 lb. at 10 per cent addition of non-coking coal.

Effects of other binders such as cement and a mixture of cement with other binders did not give encouraging results to warrant detailed description thereof.

(ii) *Self-fluxing Briquettes — Maharashtra Raw Material* — In view of the fact that direct utilization of non-coking coals from Maharashtra was not practicable, investigations were conducted for making self-fluxing briquettes with these coals by the addition of 4 per cent sulphite lye and 4 per cent coal-tar pitch as binders. The investigations were classified to study the following:

(a) Effects of variations in grain sizes of the ingredients on the room temperature and high temperature crushing strength of the briquettes.

(b) Effects of addition of different amounts of coking coal on furnace stability of the briquettes.

(c) Effects of variations in grain size of coking coal on high temperature crushing strength and stability of the briquettes for smelting operation.

To find out optimum conditions for making self-fluxing briquettes, briquettes were prepared with variations in composition and grain size, which can be broadly classified into the groups given in the table below.

It was noticed that briquettes prepared exclusively with non-coking coals from Maharashtra State did not have the requisite physical characteristics suitable for furnace smelting and necessitated additions of coking coal. The amount of coking coal was, therefore, gradually increased, but it was

CONSTITUENTS OF THE BRIQUETTES		VARIABLES STUDIED
I	Kanhan-Kamptee coal, dust iron ore, dust limestone	The effect of variation of grain size of coal −6, −16, −30 mesh (B.S.S.) on briquettability
II	Hindustan Lapeth coal, dust iron ore, dust limestone	do
III	Kanhan-Kamptee coal, dust iron ore, dust limestone	Addition of 20 per cent washed coking coal, coal −6, −16 and −30 mesh B.S.S.
V	do	Kanhan-Kamptee coal −6 mesh, addition of 40 per cent washed coking coal −6, −16, −30 mesh B.S.S.
VI	do	Kanhan-Kamptee coal −6 mesh, addition of 50 per cent washed coking coal −6, −16, −30 mesh B.S.S.
VII	do	Kanhan-Kamptee coal −6 mesh, addition of 60 per cent washed coking coal −6, −16, −30 mesh B.S.S.

only after addition of 60 per cent briquettes that satisfactory furnace smelting was obtained. However, apart from the unfavourable economics of smelting with briquettes, the necessity of addition of a large amount of washed coking coal would defeat the very purpose of iron making with non-coking coal whilst heavy quantities of washed coking coal will have to be transported to the iron smelting site in Chandha area, the cost of which will be prohibitive.

It was concluded that the direct utilization of non-coking coals such as from Kanhan-Kamptee, Hindustan Lalpeth and Ghughus of Maharashtra will not be possible for iron-making in low-shaft furnace either by charging them in lumpy bedded form of burden charge or for employing them as ' one component ' burden in the form of self-fluxing briquettes.

(iii) *Study on the Smelting Characteristics of Beneficiated Salem Magnetite Briquettes with Carbonized Neyveli Lignite Briquettes in a Laboratory Shaft Furnace* — Initial investigations were conducted at the National Metallurgical Laboratory to study the smelting characteristics of beneficiated Salem magnetite in the form of briquettes by using Neyveli lignite coke in a small laboratory shaft furnace, designed and fabricated at the National Metallurgical Laboratory, employing cold air blast enriched with oxygen gas.

Analyses of Raw Materials:

Iron Ore (Beneficiated)

	Per cent
Fe	63·5
SiO_2	9·5

Limestone

	Per cent
CaO	47·2
SiO_2	6·7
MgO	3·2
Al_2O_3	0·85

Neyveli Lignite Coke

H_2O, 8·1 per cent; ash, 14·6 per cent; V.M., 8·75 per cent; F.C., 68·55 per cent.

Composition of Briquettes

The Salem magnetite concentrate was prepared by the National Metallurgical Laboratory based on grinding, wet magnetic separation, drying and briquetting of the magnetite concentrate. The binders used in the briquettes were 6 per cent molasses and 2 per cent lime. Smelting operations were smooth; the fluidity and separation of the metal and slag were optimum.

Rate of air blast	35-38 cu. ft/min.
Pressure of the blast	1-3 lb./sq. in.
Degree of oxygen enrichment	4-6·5 per cent of the total volume of air blast

The figures are applicable for the laboratory shaft furnace used by the Laboratory and would naturally be different for industrial plant installations.

The average analyses of the metal and slag obtained in these investigations were as follows :

Pig Iron

	Per cent
Carbon	2·6-3·3
Silicon	1·6-2·4
Phosphorus	0·26-0·29
Sulphur	0·04-0·07
Manganese	0·35-0·55

Slag

	Per cent
CaO	35-38
SiO_2	33-36
MgO	8-10

From these preliminary trials it was concluded that the carbonized lignite possessed adequate reactivity for iron smelting.

(iv) *Briquetting of Beneficiated Limestone* — Briquettes were made of the beneficiated limestone (from TISCO) in the roller briquetting press at low-shaft furnace using 6 per cent molasses as binder. Investigations were undertaken to determine the hygroscopic characteristics of these beneficiated limestone briquettes to assess their use in steel making on industrial scale. A study of both green and dried briquettes revealed that there was gradual loss in weight with time. The crushing strength was adequate to permit handling during transport and during charging in the open-hearth furnace for steel making.

Industrial scale steel making trials with beneficiated limestone concentrate briquettes were made in 250 tons and 90 tons basic open-hearth steel making furnaces of the Tata Iron & Steel Works to assess the suitability of these briquettes for steel making purposes. The briquettes, in general, were found to be more efficacious in relation to unbeneficiated limestone apart from the position that much smaller quantities of limestone briquettes were required for effecting dephosphorization reaction necessary for making quality steel.

72.0 Manganese Metal Pilot Plant

During the year under review, regular production of manganese metal in the small pilot plant was continued for various research projects in National Metallurgical Laboratory such as nickel-free austenitic stainless steel, nickel-free coinage alloy, manganese brass, bimetallic elements, etc. The fabrication of 100 lb. manganese metal cell which incorporated a newly designed false bottom for efficient separation of anolyte and catholyte was completed. Besides the production of electrolytic manganese metal, the 100 lb. per day electrolytic manganese pilot plant will treat different low-grade manganese ores (ferruginous, siliceous and other complex varieties) from various parts of India to investigate the amenability of each ore for the production of manganese metal, in order to determine percentage recovery of each class of low-grade manganese ore including economics of production.

72.1 Manganese Dioxide Pilot Plant

The 100 lb. per day manganese dioxide cell was operated successfully along with the production of electrolytic manganese; leaching and purification vessels were common for both the plants. Manganese dioxide produced was tested to conform to battery active grade. Construction of the underground spent liquor tank, installation of the lead pipes and pump motor to take spent liquor to the leaching tank were completed.

72.2 Battery Activity of Manganese Dioxide

Several sets of 'D' size dry cells were prepared with electrolytic manganese dioxide made from West African ore, Tirodi ore, Shivrajpur ore and imported manganese dioxide, and their discharge characteristics and shelf life were studied after various periods of ageing. Studies were also made on cells prepared with mixtures of electrolytic manganese dioxide and natural ores in various proportions. The manganese dioxide produced in National Metallurgical Laboratory was found to be an efficient depolarizer when used alone and also when mixed in suitable proportions with natural ores. The results of investigation were in agreement with the test results submitted by M/s Ray-O-Vac Company of U.S.A. who had tested the NML-made manganese dioxide. This investigation was completed and a report has been prepared.

72.3 Study of the Effect of Trace Elements

A number of samples of manganese dioxide were deposited from baths containing various trace elements. Effects of the trace elements on the current efficiency of deposition were studied. The battery activity of the samples was evaluated by an electrochemical method consisting of two parts: (a) *p*H potential relationship of the MnO_2 electrodes and (b) utilization test involving direct evaluation of depolarizing capacity by discharging manganese dioxide electrode under specified conditions. A blank experiment was carried out with three different dioxides of known

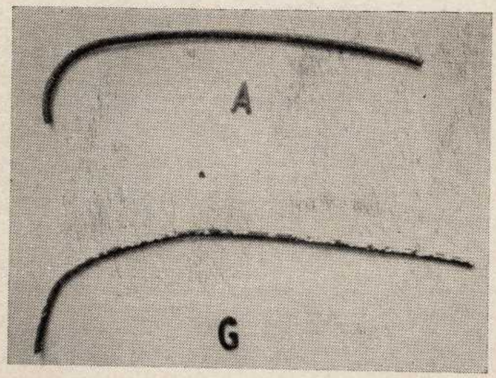

FIG. 67 — A, ALUMINIZED AND G, GALVANIZED WIRE SAMPLES AFTER SIX MONTHS' EXPOSURE AT VISHAKHAPATNAM

activity. The position of the *p*H potential curves showed that the method could be relied on. *p*H potential relationships of the sample deposited from baths containing trace elements were also studied. The experiments were repeated to check the reproducibility. The crystal structure of the samples are being studied by means of X-ray diffraction.

73.0 Pilot Plant on Hot-dip Aluminizing of Steel

Samples of hot-dip aluminized wire exposed for six months at Beach Road, Visakhapatnam, in an experimental telephone line erected in December 1960, were received from the Additional Chief Engineer, Posts & Telegraphs, Jabalpur (M.P.), in August and December 1961 for examination.

Fig. 67 shows the representative samples of A (aluminized) and G (galvanized) wire. A was uniformly coated with a film of protective reddish brown product while G has flaky deposits at places with considerable rusted spots. The deposits on galvanized wire were easily peeled off.

Stripping test on the exposed aluminized wire showed that 147·3 mgm. of metallic aluminium per foot was still available out of 238 mgm. for sacrificial protection and 87·2 gm. of iron oxide was adhering to the wire at the end of this field exposure. The saline atmosphere contained chlorides and sulphates of sodium, magnesium and calcium. Whereas the corrosion products of aluminized wire were adherent and remained intact with the wire, those of galvanized wire were flaky and were removed from the wire by atmospheric phenomena of wind, dew, rain, etc. Metallographic examination conclusively proved that in the period of six months all zinc was consumed while quite a good amount of metallic aluminium remained on the wire and would afford further sacrificial protection.

Aluminizing of Pole-line Hardware — 1400 each of stalks and U-Backs were aluminized for Posts and Telegraphs Department, Calcutta.

Fig. 68 shows an assortment of hardware aluminized with a view to replacing galvanized hardware.

Promising results were obtained in this investigation. Equipment and raw materials for aluminizing were indigenously available. Aluminium coating was superior to zinc coating in many ways, as indicated by humidity and salt spray tests. The appearance of the product was better and the lustre was retained even after long exposure unlike galvanized coating.

It has been recommended that Post and Telegraphs Department should aluminize the

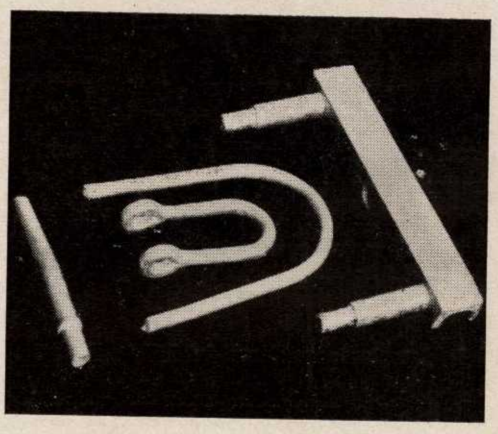

FIG. 68 — HOT-DIP ALUMINIZED POLE-LINE HARDWARE

pole-line hardware instead of galvanizing with imported zinc.

Aluminizing of H.T. Transmission Tower Members — Messrs Kamani Engineering Corporation Ltd., Bombay, approached the National Metallurgical Laboratory to investigate the feasibility of hot-dip aluminizing of structural members of H.T. transmission towers, sub-station structures nuts and bolts, etc.

Fig. 69 shows the hardware samples in the as-coated condition which was quite satisfactory as good, lustrous, uniform and adherent coatings are obtained.

Hot-dip Aluminizing of Buckets — Several firms approached the National Metallurgical Laboratory for technical know-how on aluminizing of buckets to replace galvanizing process thereof. Some samples sent by these were successfully aluminized (Fig. 70). Negotiations are now in progress with some of the firms for granting licenses to them for exploitation of processes developed.

Hot-dip Aluminizing of Auto-mufflers and Silencers — Auto-mufflers and silencers were aluminized for E.M.E. Technical Services Group. Samples were received from different Army Base Workshops and coated. It was expected that the superior corrosion and heat resistance of aluminized mufflers could give a long service life to the auto-mufflers.

74.0 Continuous Strip Aluminizing

With requisite modifications to wire aluminizing pilot plant, viz. installation of feed reel in horizontal axis, change of rolls and modification of exit end after hot-dipping, it was possible to conduct preliminary trials on continuous hot-dip aluminizing of mild steel strip. Three, 6 and 9 in. wide strips varying in thickness from 0·6 to 3 mm. in the as-annealed and cold-rolled finish were used as the basis material for aluminizing.

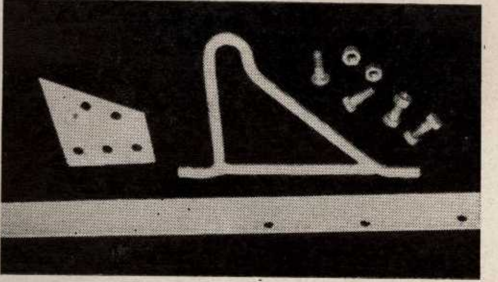

Fig. 69 — Aluminized high tension transmission tower

Fig. 70 — Aluminized buckets

In a typical run the strip coil was first loosened and batch degreased in I.C.I. degreaser No. 1, washed thoroughly and put on to the feed reel. In the continuous line it passed through 1 : 1 hydrochloric acid, swill tank, spray wash, flux bath, drier and molten aluminium. Annealed strip was first batch descaled in 8 per cent sulphuric acid. After hot-dipping the strip came out either through a system of rolls or asbestos wipes to control the thickness and finish of coating.

Coatings obtained were lustrous, uniform and sound as ascertained by metallographic examination, Preece test, and nitric acid soundness test. Based on the initial experiments, a strip aluminizing pilot plant was being designed and fabricated at the National Metallurgical Laboratory.

75.0 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 indigenous ores for drawing up industrial beneficiation flowsheets for commercial plants, a pilot plant capable of treating over 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. These systematic research investigations have clearly established that many of the ores can be effectively upgraded to the required rigid specification. The projected integrated 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 at the National Metallurgical 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.

Considerable progress had already been made for setting up the pilot plant. Over three-fourths of the machinery and equipment were procured and their installation work was progressing more satisfactorily. Full plant operations were expected to start early in 1963, whilst flotation, gravity treatments, pelletizing and agglomeration operations would commence at the end of 1962.

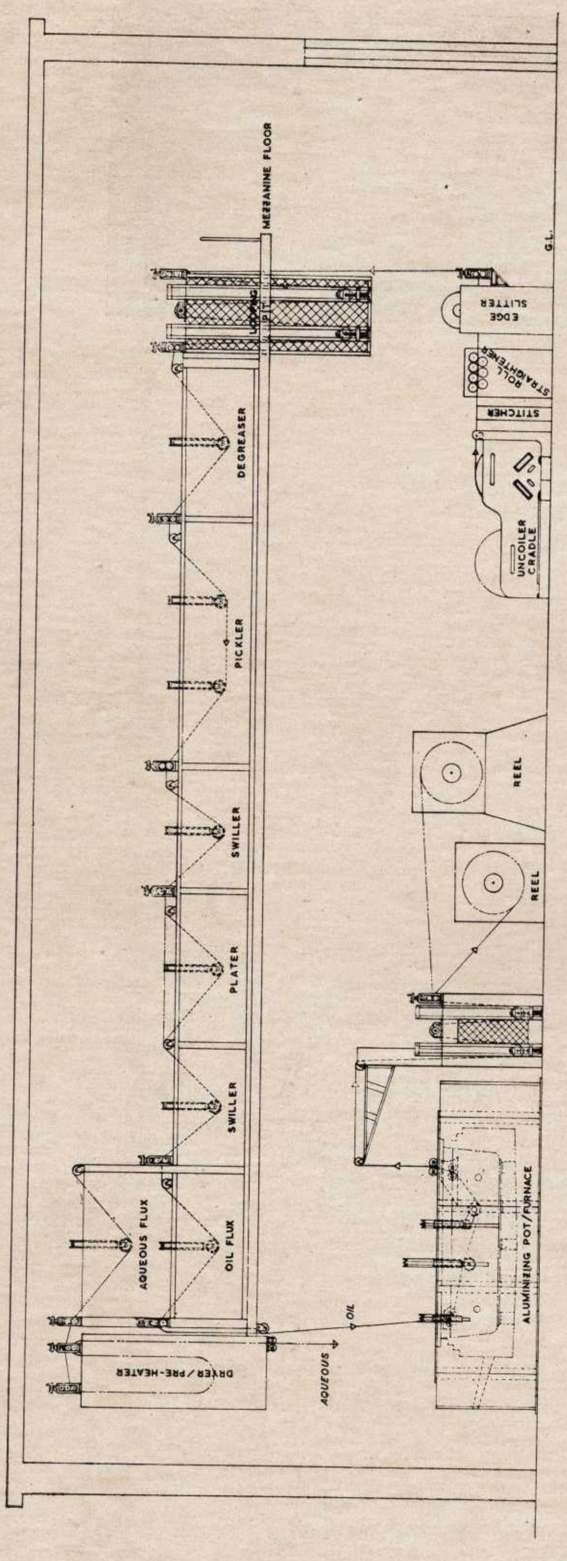

FIG. 71 — STRIP ALUMINIZING PLANT (SCHEMATIC LAYOUT)

76.0 Pilot Plant for Refractories

The following equipment for the pilot plant were fabricated and installed:

- (i) Down-draft kiln of 7 tons nominal capacity going up to 1600°C:
- (ii) Dryer of 15 tons total capacity and a ton per day running capacity.
- (iii) A complete crushing plant including primary and secondary crusher, sieve-shaker, storage bins and conveyors.

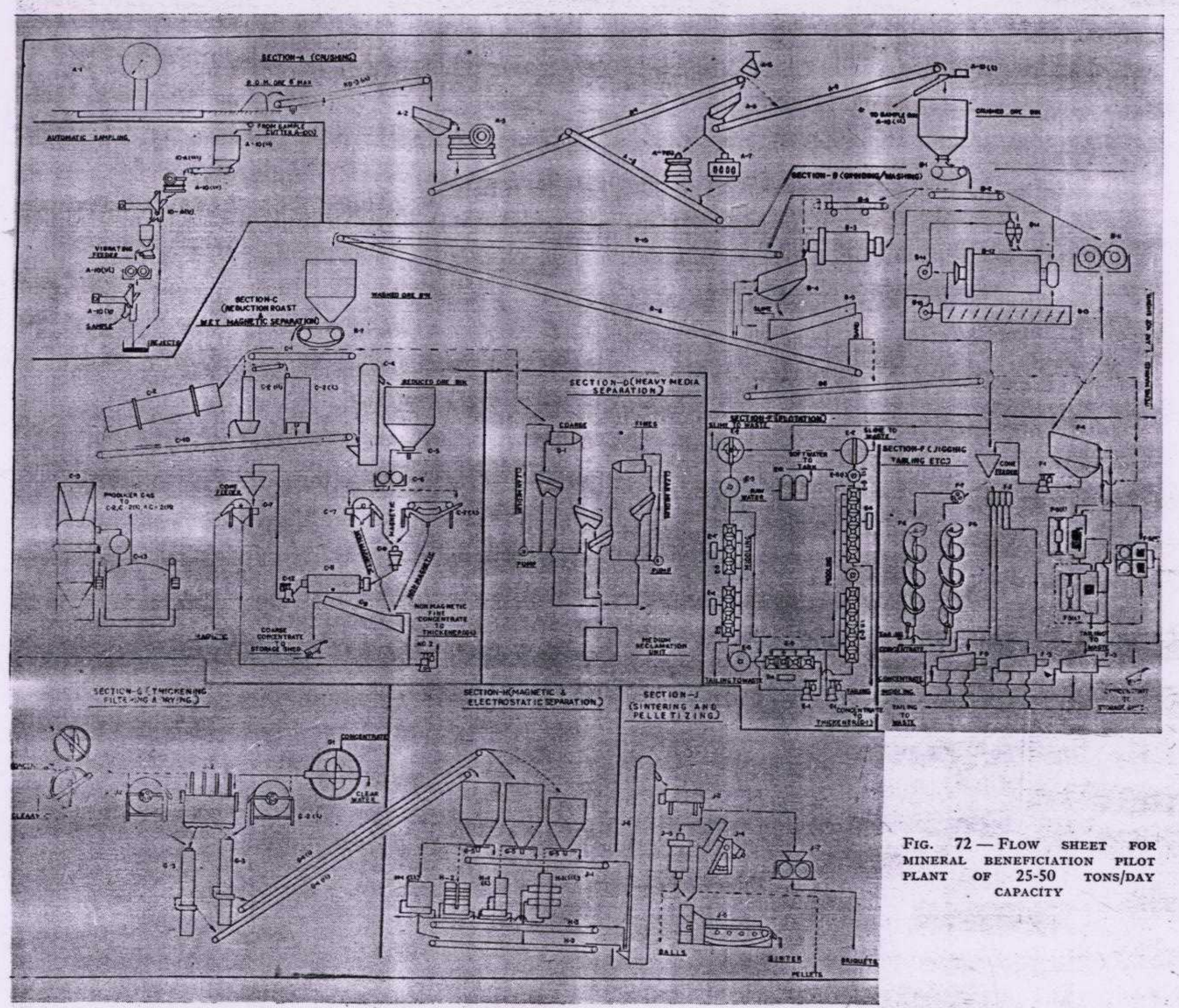

FIG. 72 — FLOW SHEET FOR MINERAL BENEFICIATION PILOT PLANT OF 25-50 TONS/DAY CAPACITY

LIST OF EQUIPMENT

Section A:	Qty.
1. Weighing Platform 24′×10′	1
2. Vibrating Grizzly 2′×5′	1
3. Jaw Crusher 10″×16″	1
4. Belt Conveyor 116″×15′	1
5. Guard Magnet 0·24″	1
6. Vibrating Screen 2′×4′	1
7. Symons Cone Crusher 2′	1
7(i) Gyratory Crusher	1
8. Belt Conveyor 16″×50′	1
9. Belt Conveyor 16″×75′	1
10(i) Sample Cutter	1
10(ii) Sample Bin	1
10(iii) Ore Feeder	1
10(iv) Jaw Crusher 5″×6″	
10(v) Sample Splitter (Synder)	2
10(vi) Roll Crusher 10″×6″	1
Section B:	
1. Constant Weight Feeder	1
2. Belt Conveyor 16″×25′	1
3. Blade Mill 3′×4′	1
4. Vibrating Screen 2′×4′	1
5. Spiral Classifier 12″×9′-6″	1
6. Belt Conveyor 16″×130′	1
7. Apron Ore Feeder 18″×6′	1
8. Belt Conveyor 12″×32′	1
9. Belt Conveyor 16″×18′	1
10. Belt Conveyor 16″×120′	1
11. Roll Crusher 16″×10″	1
12. Ball Mill 3′×6′	1
13. Rake Classifier 18″×14′-8″	1
14. Cyclone Classifier	1
15. Sand Pump 2″	1
Section C:	
1. Belt Conveyor 16″×25′	1
2. Rotary Kiln 3″×30′	1
2(i) Multiple Hearth	1
2(ii) Vertical Furnace	1
*3. Gas Producer	1
4. Bucket Elevator 35′	1
5. Disc Feeder 2′	1
6. Roll Crusher 12″×8″	1
7. Electromagnetic Separator	2
7(i) Electromagnetic Separator	1
8. Demagnetizing Coil	1
9. Spiral Classifier 9″×5′-8″	1
10. Belt Conveyor 16″×65′	1
11. Rod Mill 16″×4″	1
12. Vertical Sand Pump 1½″	1
13. Gas Holder 20,000 cu. ft	1

Section E:	Qty.
1. Vertical Sand Pump	2
2. Hydro Classifier 4′	2
3. Conditioner 3′×4′	2
3(i) Conditioner 18″×18″	2
4. Wet Reagent Feeder	4
5. Flotation (4) Cell Unit	3
5(i) Flotation (6) Cell Unit	2
6. Automatic Pulp Sampler	2
7. Dry Reagent Feeder	1
8. Water Softening Unit	1
Section F:	
1. Vertical Sand Pump 1½″	1
2. Hydrosizer 4″×4″-4 Com.	1
3. Shaking Table 7′-6″×3′-6″	3
4. Vibrating Screen 2′×4′	1
5(i) Jog (Harz Type) 18″×32″	2
5(ii) Mineral Jig 8″×12″ Duplex	1
6. Spiral Concentrator	2
7. Pulp Distributor 4 Com.	1
Section G:	
1. Thickener 16′×8′	1
1(i) Thickener 9′×6′	1
2. Disc Filter 4′×4 Disks	1
2(i) Drum Filter 4′×2′-6″	2
3. Rotary Dryer 18″×10′	2
3(i) Rotary Dryer 18″×15′	1
4(i) Belt Conveyor 12″×105′	1
4(ii) Belt Conveyor 12″×110′	1
5. Disc Feeder 18″	3
6. Hydro Classifier 6′	1
Section H:	
1(i) Magnetic Separator	1
1(ii) Magnetic Separator (Duplex)	1
1(iii) Magnetic Separator (Cross Belt)	1
2. Electrostatic Separator	1
3. Belt Conveyor 12″×25′	2
Section J:	
1. Belt Conveyor 12″×25″	1
2. Paddle Mixer 3′×8′	1
3. Balling Drum 2′×4′	1
4. Pelletizing Disc 5′	1
5. Sinter Strand	1
6. Bucket Elevator 38′	1
7. Briquetting Press	1

76.1 Pilot Plant Studies on Refractory Projects

(i) *Studies on Refractory Brick Forming* — Some results on the single-stage compaction of ceramic mixes as also on compaction under blows of varying intensities had earlier been reported. During the year under review, work was carried a stage further and experiments were conducted with specimens of full brick size. Salient features of this work are given below :

Experimental Mix : The mix consisted of clay grog and water in the proportion 10 : 90 : 8. The clay was a plastic fireclay from the Rajhara area and the grog, also made of burnt fireclay, had the physical characteristics as given in Table 54.

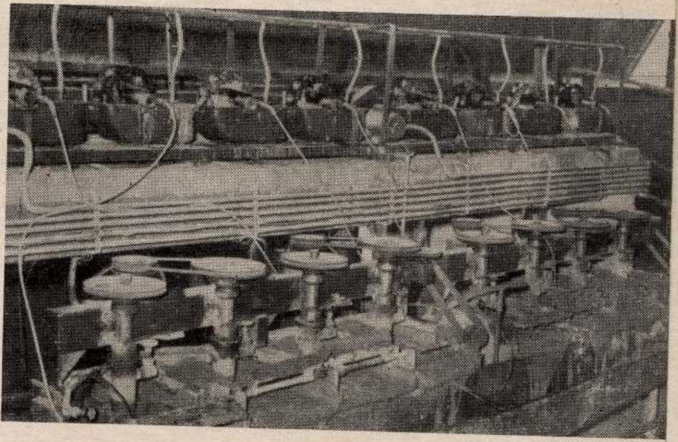

FIG. 73 — A VIEW OF THE FLOTATION BATTERY OF THE PILOT PLANT FOR BENEFICIATION OF LOW-GRADE ORES (25-50 TONS/DAY CAPACITY) INSTALLED AT THE NATIONAL METALLURGICAL LABORATORY

Experiments and Results

Compacts made under various number of blows were sectioned and the water, clay and grain size distributions in the various regions of the compact were determined. Typical results are given in Table 55. Variations in compaction with increasing number of blows are given in Table 56 and Fig. 75.

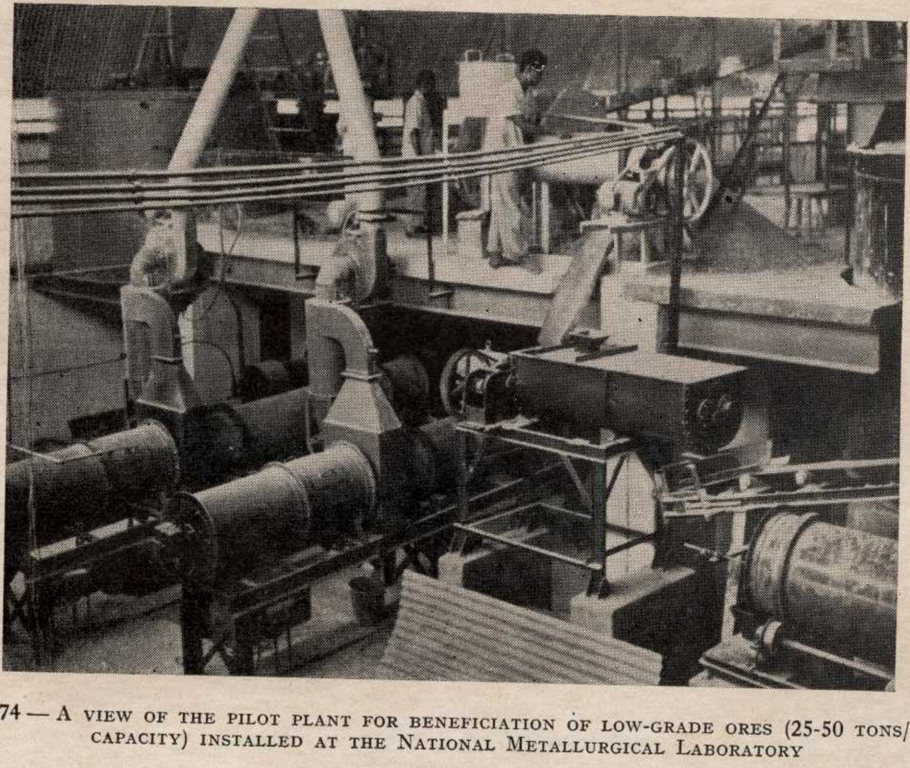

FIG. 74 — A VIEW OF THE PILOT PLANT FOR BENEFICIATION OF LOW-GRADE ORES (25-50 TONS/DAY CAPACITY) INSTALLED AT THE NATIONAL METALLURGICAL LABORATORY

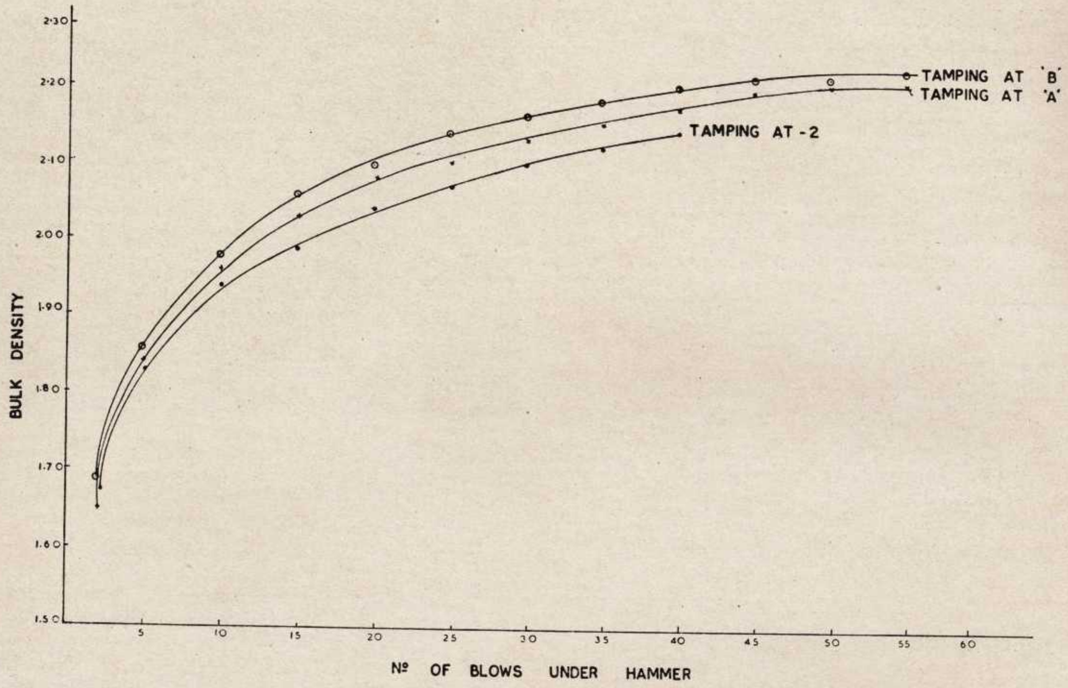

FIG. 75 — BULK DENSITY VERSUS NUMBER OF BLOWS IN FULL BRICKS UNDER PNEUMATIC HAMMER

TABLE 54 — PHYSICAL PROPERTIES OF THE GROG

Apparent porosity	16%	Packing density gm./c.c.	1·83
Bulk density gm./c.c.	2·27	Ree's Sp. Gravity gm./c.c.	2·67
App. Sp. Gravity	2·70	Sieve analyses:	
Crushing strength p.s.i.	14,200	−5+7	19·2
		−7+14	27·00
Modulus of rupture p.s.i.	4,100	−14+25	21·00
		−25+52	14·80
		−42+72	4·20
		−72+100	3·60
		−100	10·20
		Total	100·00

(ii) *Forsterite Project* (Based on Indian Patent No. 57884) — This patent is based on the process developed at the National Metallurgical Laboratory for the production of forsterite refractories from magnesium silicate rocks which at present find no use in industry. These refractories find applications in steel plant practice and the Bhilai Steel Plant of Messrs Hindustan Steel Ltd use them in their open-hearth checkers; the latter have offered to undertake long-term service trials on N.M.L.-developed refractories.

The trials planned were intended to undertake the following :

(i) Assess the suitability of laboratory scale process on an industrial scale.

(ii) To examine the possibility of converting it into a more economical one-stage process instead of the two-stage process as stipulated at present.

(iii) Work out optimum conditions for getting a uniform and good serviceable end product.

(iv) To fix the variables for various types of magnesium silicate rocks available — particularly those coming out as waste products in chromite mining.

(v) To produce requisite tonnage of refractory bricks of various shapes for long-term trials under actual service conditions.

TABLE 55 — MOISTURE, GRAIN SIZE AND CLAY DISTRIBUTION INSIDE A TAMPED BRICK

(*Each specimen cut perpendicular to long axis in three equal pieces, Nos.* 1, 2 *and* 3. *Each such piece cut parallel to long axis into three, top, middle, bottom*)

A. Moisture Distribution (in wt. per cent)

	Top	Middle	Bottom
1	6·93	7·09	7·51
2	7·00	7·08	7·33
3	6·97	7·00	7·37

B. Grain size distribution:

Sieve Nos.	Sieve Analysis Original mix.	% Sieve Analyses					
		1 Top %	1 Middle %	1 Bottom %	2 Top %	2 Middle %	2 Bottom %
−5+7	30·5	18·3	17·4	16·3	17·5	18·0	17·3
−7+14	25·5	19·5	21·9	18·9	10·9	18·7	17·0
−14+25	10·5	14·0	12·6	16·3	14·4	16·4	17·0
−25+52	8·9	12·9	11·3	12·0	11·9	11·0	12·5
−52+72	2·6	4·0	3·5	5·3	5·4	3·7	4·2
−72+100	2·6	22·3	3·0	2·4	2·3	3·2	2·5
−100+200	2·3	4·9	4·8	3·8	4·0	4·5	4·5
−200	7·1	24·1	25·5	25·1	23·7	24·5	25·0

TABLE 56 — EFFECT OF COMPACTION OF STANDARD MIX. UNDER HAMMER (FULL BRICK) UNDER VARIOUS NUMBER OF BLOWS

No. of Blows	Hammering Position No. 2		Hammering Position "a"		Hammering Position "te"	
	Bulk* density	Void† Percentage	Bulk density	Void Percentage	Bulk density	Void Percentage
2	1·67	37·4	1·65	38·2	1·69	36·7
5	1·83	31·5	1·84	31·1	1·86	30·3
10	1·94	27·3	1·96	26·6	1·98	25·8
15	1·99	25·4	2·03	24·0	2·06	22·8
20	2·04	23·7	2·08	22·1	2·10	21·3
25	2·07	22·5	2·10	21·3	2·14	19·8
30	2·10	2·13	2·14	20·3	2·16	19·1
35	2·11	21·0	2·15	19·4	2·18	18·4
40	2·14	19·8	2·17	18·8	2·20	17·6
45	—	—	2·19	18·0	2·21	17·2
50	—	—	2·20	17·6	2·21	17·2
55	—	—	2·20	17·6	2·22	16·9

*Bulk density calculation on dry basis.
†Void percentage calculated on Ree's Specific Gravity basis.

The work was divided into two stages. In the first stage a serpentine rock from Ramla chromite mines, which had been used in the laboratory scale experiments, would be handled on a pilot scale to study the optimum grain size to which it had to be crushed to ensure its full conversion to forsterite, nature and type of magnesia additions that gave best results, optimum binder content, pressure and other factors that were necessary to produce good refractory bricks on a large scale.

In the second stage, a readily available and industrially feasible deposit will be chosen. Data obtained during the first stage would be applied to produce bricks and shapes for service trials. The economics of the process would also be studied in detail and a suitable flow-sheet will be drawn up.

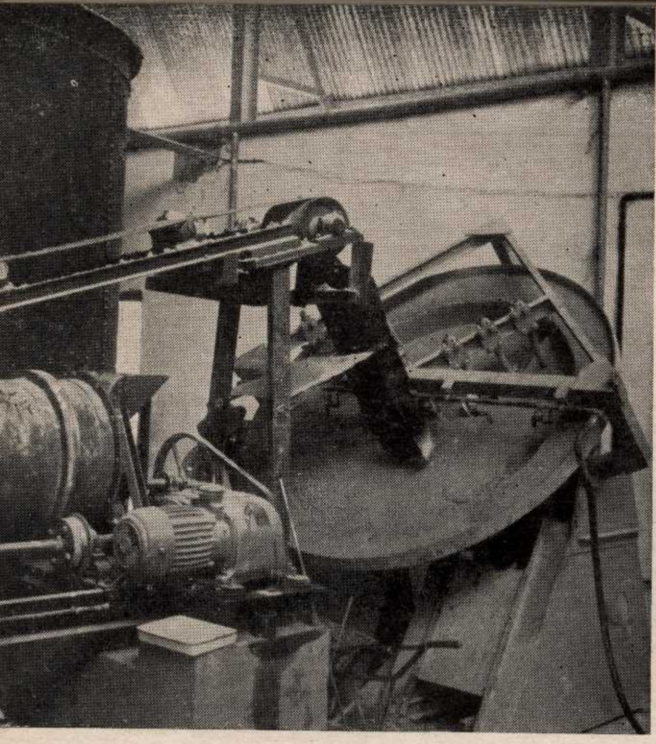

FIG. 76 — A VIEW OF THE DISC PELLETIZER OF THE PILOT PLANT FOR BENEFICIATION OF LOW-GRADE ORES (25-50 TONS/DAY CAPACITY) INSTALLED AT THE NATIONAL METALLURGICAL LABORATORY

Experiments conducted up to date included studies on crushing characteristics of serpentine from Ramla mines and fabrication of $9 \times 4\frac{1}{2} \times 3$ in. straights of various compositions covering several grain sizes of the rock and magnesium oxide additions. Determination of the physical properties was in progress.

77.0 Pilot Plant for the Production of Magnesium Metal

Erection of a bay for the installation of the magnesium pilot plant was completed. Detailed specifications for machinery, equipment and other accessories required in connection with the installations were drawn up. Design of the plant, fabrication and procurement of different items of equipment were in progress. Meanwhile, it was proposed to install a small furnace unit in order to produce the metal in regular batch operations.

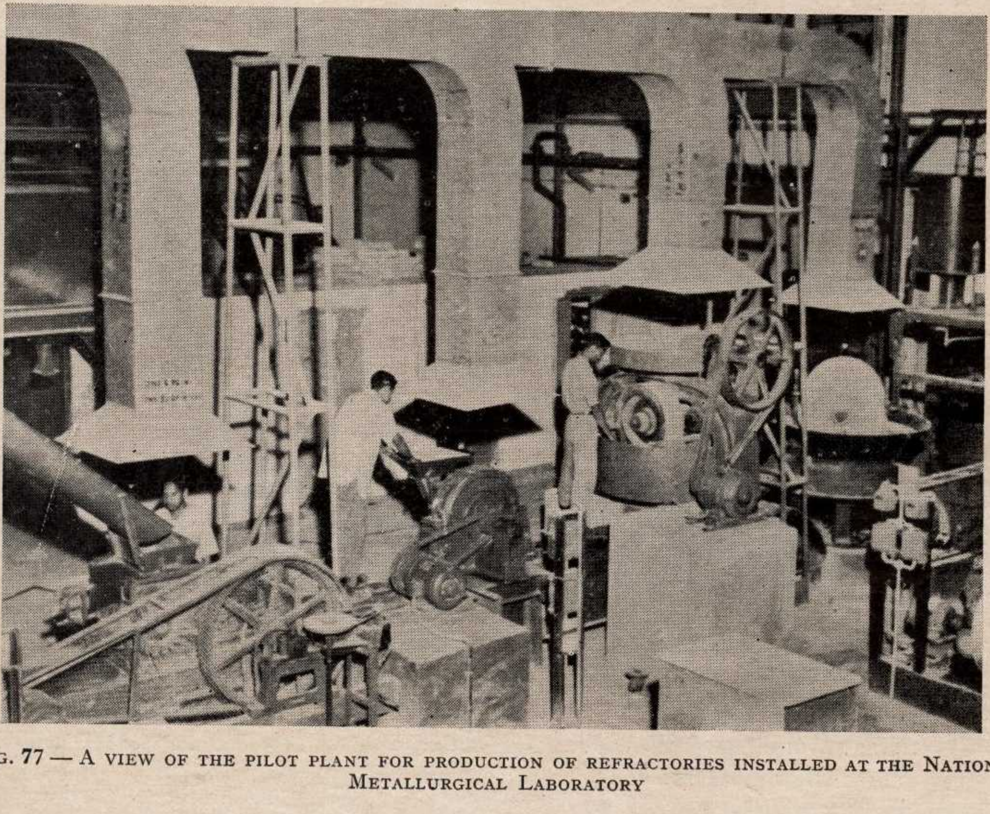

FIG. 77 — A VIEW OF THE PILOT PLANT FOR PRODUCTION OF REFRACTORIES INSTALLED AT THE NATIONAL METALLURGICAL LABORATORY

ELECTRIC FURNACES

An Inter-divisional Committee was formed in November 1960 with the object of evolving suitable refractory cement compositions for use in electrical furnaces and also for making suitable designs of electric furnaces for specific research projects for their fabrication in the laboratory's mechanical, electrical and electronic workshops.

During the period under review, clay-kyanite cement mixture developed earlier was widely used for fabrication of laboratory furnaces with kanthal resistance element. The cement had given excellent performance and no trouble was reported on account of any failure of the cement material. The aluminous cement, containing calcium aluminate as binding material had also been similarly used for fabrication of electric resistance furnaces. No failure of the cement had so far been reported.

A third cement with phosphoric acid as the binding component was elaborately examined during the year under review. Optimum ratio of the binding component and the grog material was determined. A curing temperature of 400°-500°C. was found to be sufficient for furnace bodies moulded from this cement composition. The moulded bodies did not show any perceptible deterioration even after use at 1200°C. for 500 hours. A new ceramic composition was developed from the aluminous cement and aluminous grog materials for making cast furnace bodies. Three compositions were investigated. Shrinkage during setting was found to be less than 2 per cent. It was possible to obtain the casts to close tolerances. The casts were dried at 150°C. and did not require high temperature curing except drying at 150°C. before mounting in furnace shell. Work was continued to study the performance of the cast as a furnace member in the temperature range 1000°-1400°C.

Design of Electric Furnaces for Specific Jobs—The technique of making internally wound tubular furnaces was systematically applied to meet all diverse requirements of tubular furnaces of the laboratory. The technique was now effectively standardized and regularly used for diverse jobs. The gradientless furnaces were made with secondary end windings, separated from the main central winding. One of the furnaces had already worked for more than 2000 hours in the temperature range 700°-1100°C. and continued to give good service. The other furnace had been recently commissioned. An experimental, internally wound tubular furnace, designed to operate at 1100°C., was fabricated from National Metallurgical Laboratory developed heat-resistance element and refractory materials also developed at the laboratory. The furnace had already given satisfactory service for over 1000 hours and was still in service.

Work was undertaken to make platinum-rhodium (10 per cent) wound furnace for operation at 1500°C. The design essentially consisted of a primary winding for 1500°C. on the furnace tube itself with platinum-rhodium wire with a secondary winding of Kanthal resistance wire located at 1000°C. isothermal plane of the primary winding. The secondary winding was an internally wound cylindrical element fabricated with clay-kyanite cement. The object of providing the secondary winding was to compensate the dissipation of heat from the primary winding and thus to reduce current load on the costly platinum-rhodium element. The primary winding was suitably designed to compensate end losses and to provide a uniform temperature zone of 4 inches. The two elements were coaxially mounted in a furnace shell of 18 in. × 18 in. × 18 in. Design had also been completed for a silicon carbide element furnace for long service operations at 1450°C. The heating space of the furnace was 4 in. dia. × 6 in. The complete ceramic structure for mounting 12 separate heating rods had been cast into two halves with the ceramic compositions developed at the National Metallurgical Laboratory by blending aluminous hydraulic cement and aluminous grog materials. The furnace was nominally rated at 4 KW. and was under assembly stage. Design was completed for a tubular furnace for service at 1800°C., with molybdenum tape as the heating element. The winding was on porous recrystallized alumina tube. The element was designed to remain in an atmosphere of cracked ammonia. Provision was also made for using a second coaxial ceramic tube to permit the facility of carrying on experiments under gas atmospheres other than cracked ammonia. Heating space available was about 140 cubic inches.

ENGINEERING SECTIONS

Design Engineering

During the year, the development of suitable designs for a complete plant for the continuous hot-dip aluminizing of steel strips was undertaken. Out of a total of 72 detailed engineering drawings, more than 32 were completed during the year, the balance being scheduled for completion by December 1962. Among equipment developed and detailed: Uncoiler Cradles, Pre-treatment Line Structures, Drag Bridles, Pinch Rolls, Guide Rolls, Continuous Vapour Degreaser, Batch and Continuous Picklers, Aluminizing Melt Pot and Furnace complete with Jockey Rolls and Dross Shields, etc.

Figure 71 depicts schematically the proposed layout of the strip aluminizing plant. This prototype plant was being

FIG. 78 — PHOTOELECTRIC TEMPERATURE CONTROLLER, DESIGNED AND FABRICATED AT THE NATIONAL METALLURGICAL LABORATORY

designed for strips up to 300 mm. wide and up to 16 swg. thick. Since the plant was an industrial prototype, it was not designed primarily for high throughputs but for variable throughput to determine optimum process conditions and eventual scale up to a production unit. Maximum design throughput for the plant will be 5 meters per minute utilizing primary pickled skelp.

The strip would be continuously processed commencing at the uncoiler cradle and followed by a welder/stitcher, roll straightener, and edge slitter. The strip then entered the pre-treatment line located on a mezzanine floor. This line included a slack pit or looping tower, a vapor degreaser, a muriatic acid pickler for secondary in-line pickling, a swiller-cum-scrubber, a plating tank and washer for oil flux processes, and a composite flux unit for alternate oil or aqueous fluxing. A dryer/preheater follows the fluxes when aqueous flux was used. The fluxed strip then entered the aluminizing line consisting of a aluminizing melt pot and furnace provided with dross shields and stationary jockey rolls. The aluminized strip left the melt pot vertically via the exit pinch rolls and dross shield and after traversing a looping tower, it was coiled into rolls of suitable diameter. Pinch rolls and motorized drag bridles at suitable locations conveyed the strip through the lines at controlled tension and also prevented strip recoil during threading or failure. Each process tank was provided with a motorized jockey roll for ease of threading and traverse control.

A primary pickling line consisting of a single uncoiler cradle, a pickler for sulphuric or muriatic pickling, and a coil reel, was also provided for heavy descaling etc. at controlled variable rates prior to insertion of the skelp coils into the continuous line.

Two major design work under consideration are a 'Continuous type Multistrand Patenting Furnace' for metallic wires and a 'High Temperature Chlorination Furnace' for the production of molten anhydrous magnesium chloride.

Electronics Engineering

(i) *Photoelectric Temperature Controller* — A photoelectric temperature controller for controlling temperature up to 1100°C. with an accuracy of ± 1½°C. was successfully developed. Thermocouple circuit, galvanometer, photo-cell unit and the d.c. amplifier were incorporated in one compact unit.

(ii) *Propositional Temperature Controller for High Temperature Use* — A thyratron-driven temperature controller was developed for controlling the temperature of a platinum furnace up to 1600°C. The furnace winding itself formed one arm of a wheatstone bridge. The unbalanced voltage was stepped up and applied to a grid-controlled thyratron. The accuracy obtained at present with the prototype was ± 5°C. With certain modifications the accuracy could be improved to ± 1°C. Further work is in progress.

(iii) *Electronic Titrimeter* — A tentative design was worked out and a prototype was under fabrication. An electronic voltmeter operating either from the alternating current or direct current gave the indication of end points of the titrated solution in a cathode ray tube indicator.

(iv) *Damping Capacity Meter* — Design for instrument for measurement of internal friction of metals was completed and instrument was being fabricated. The instrument enabled the decrement interval to be measured quickly and accurately by an electronic timer circuit between frequency 50 kc./s. and 10 kc./s.

(v) *Wide-band Stabilized Power Supply* — The need for a wide band stabilized power supply (0-500 volts; 300 ma.) was felt for various applications in the laboratory. As such a literature survey was carried out and suitable design was being worked out.

(vi) *Glass Solenoid Gas Pump* — A Glass-Solenoid Pump was fabricated based on BISRA design. This pump maintains gas circulation in a closed circuit. The electromagnet was entirely designed in the electronic section. Besides the above, several maintenance, installation and calibration jobs were completed. The major jobs handled were repair of various types of temperature controllers and recorders, magnetic amplifier and control equipment for 12 in. rolling mill, Lange Flame Photometer, Differential Thermal Analysis Instruments; Installation of six nos. of Smythe Proportional Temperatures Controllers for creep testing machines and calibration of number of temperature indicators and recorders.

Mechanical Engineering

During the year under review more than 1500 technical jobs were executed of various description from making of test pieces to some of the major repairs of machinery and fabrication of heavy equipment. A few of the important fabrications are mentioned below with their salient features :

Electrolytic Manganese Cell — Design and fabrication of the above 50 kg./day manganese cell suitable for 16 cathodes and capable of taking 3000 amp. were undertaken. Suitable non-corrosion inner lining, thermal insulation and cooling coils to keep the electrolyte at a low temperature were provided. The cell is nearing completion.

Continuous Casting Machine — A continuous casting machine for light metals and alloys was designed and being fabricated. The design of this machine was based on an experimental continuous casting machine, which has previously been built in the Laboratory, with suitable additions and modifications. A special type of cooling system to cool the die and to spray water on the outcoming cast metal was introduced. A positive drive to two sets of rolls was provided through reduction gear unit, speed variator, and machined teeth spur gears.

Vertical Reduction Kiln — The fabrication of this 1 m. dia. and 6 m. tall furnace was undertaken. Its outer shells, flanged angles, rotating bottom, feed hopper etc. were completed. The kiln was capable of reducing 1000 kg./hour of lumpy ferruginous ores.

Installation of Refractories Pilot Plant — Installation of Jaw Crusher, Bucket Elevator, Sieve Shaker, Belt Conveyors, Feeders, etc., was completed and all their connecting components such as tanks, chutes, launders, covers, platforms, railings, etc., were fabricated. An arrangement was made for continuous feeding of raw material from the ore lines to the crushers at a higher level than the lines made with the help of vibratory feeder and a hopper. The height of the feeder could be adjusted at various points. The plant on the whole was kept flexible to suit different types of flow-sheets.

Side-blown Converter — The fabrication of side-blown converter was completed. Its outer shell, blast pipes, butterfly valves etc. were fabricated and trunions and their bearings lined with aluminium bronze, rotary seal etc. were machined in the workshop of the laboratory. Wooden and steel formers and moulds for refractory lining were also supplied.

Giesler Plastometer — The design and fabrication of this instrument to find the softening point and maximum fluidity of coal was completed. The furnace can be heated electrically up to 900°C. and frictionless spindle and stirrer with four fingers rotated under a certain load. Provisions to record the rotation on a dial at different temperatures is made. The 20 mm. dia. 16 mm. deep retort is loaded with coal under a load of 10 kg. with the aid of a special loading device.

Disc Pelletizer — The design of 48 in. dia. Disc Pelletizer for making pellets with iron ore, coal, lime stone etc. was completed. Speeds of 10, 20 and 30 r.p.m. were attained by stepped pulleys and a speed reduction unit. The disc axis can be tilted between horizontal and vertical positions by means of levers and can be locked at intervals of 5°.

Electrical Engineering

During the year, following major electrical development and design projects were undertaken for various research schemes of the laboratory besides the repairs and maintenance of the electrical equipment.

Power Distribution for Manganese Beneficiation Pilot Plant — For manganese beneficiation pilot plant, power distribution and industrial lighting were designed and detailed. 1000 kVA. power at 6600 volts will be transmitted from existing Low-Shaft Plant substation by underground 3-core 0·12 sq. in. 550 meter long cable connected through H.T. oil circuit-breakers. This power by stepping down to 415 volts 3-phase 4-wire system will be distributed to the plant.

Power Supply to 500 *kVA. Submerged Arc Furnace* — For the installation of submerged arc furnace for ferro-alloy plant, 500 kVA. power at 6600 volt and 75 kVA. power at 400 volts were needed. Complete power distribution design, installation and commissioning were carried out. H.V. power was supplied from Pilot Plant Technological Substation by underground cables through suitable oil circuit-breakers. Besides, water recirculation system for cooling the furnace was designed and detailed.

Pilot Plant Substation Expansion — The existing 500 kVA. transformer of the Pilot Plant substation was not sufficient to meet the power demand of the Pilot Plant Technological Bays because of the installation of one very high power consuming electrode boiler which alone needed 450 kW. powder. Thus the necessity of expansion of the substation capacity arose. Design work to increase the power capacity by installing another 500 kVA. transformer and modifying the circuit was completed.

OPERATIONAL RESEARCH

The liaison and operational research work of the Laboratory increased manifolds covering a wide range of spectrum including dissemination of research results, publication of NML Technical Journal and Symposia Proceedings, Organization of Symposia; preparation of Project Reports, preparation and circulation of technical notes of the National Metallurgical Laboratory developed methods for commercial scale exploitation and holding practical demonstrations of these processes, translation, documentation and photographic services, etc. Furnishing of detailed technical advice and data to the industries and taking up operational research problems on their behalf to evolve effective means of solving their problems comprised another major aspect of the work of the National Metallurgical Laboratory.

During the year under review, several operational problems referred to by the industries were undertaken. A foundry in Calcutta was experiencing difficulties in their foundry in respect of low discharge of their cupola. On the spot investigation was made and concrete practical steps were introduced in improving the melting rate, methods of proper charging, etc. A non-ferrous metal organization in Banaras was experiencing difficulties in finding suitable aluminium alloy in the absence of aero-scrap, for moulding body cover and base castings of universal fractional motors. After a detailed investigation of their requirements, recommendations were made for a suitable composition and physical properties of aluminium alloys suitable for their purpose. A number of cases of failure of metals under service conditions were investigated and causes of the failures were determined.

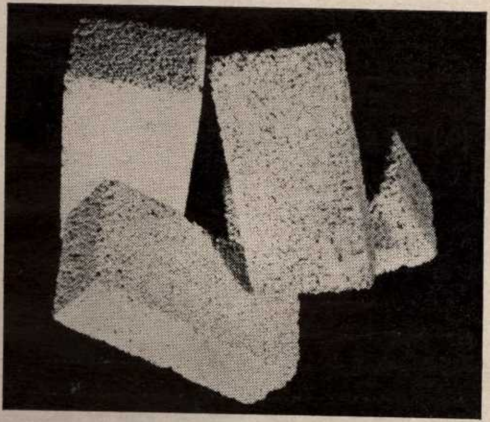

FIG. 79 — INSULATION BRICKS MADE OUT OF EXPANDED LOW-SHAFT FURNACE SLAG

Operational research relating to the utilization of low-shaft furnace slag was initiated to investigate its utility for slag cement and as light weight aggregates for insulating concrete, concrete mason units and structural concrete.

(i) *Slag Cement* — Slag cement prepared by mixing granulated slag and portland cement as activator in different proportions showed good compressive and tensile strength. The slag cement was used in actual construction work in place of pure portland cement. One part of slag cement (60 per cent portland cement and 40 per cent slag) mixed with six parts of sand gave satisfactory results. With a view to utilize slags containing more than 5 per cent MgO, work was undertaken to prepare synthetic slag containing different percentages of MgO containing more than 5 per cent and their hydraulic properties were also determined.

(ii) *Light-weight Aggregate* — Slag from the low-shaft furnace was granulated with controlled amounts of water and expanded slag was processed and crushed to the

TABLE 57 — PERCENTAGE (BY WEIGHT) PASSING SIEVES HAVING SQUARE OPENINGS

SIZE DESIGNATION	¾ IN.	½ IN.	⅛ IN.	No. 4 (4760 micron)	No. 8 (2380 micron)	No. 16 (1190 micron)	No. 30 (590 micron)	No. 50 (297 micron)	No. 100 (149 micron)
Fine aggregates: No. 4 to 0	—	—	100	85-100	—	40-50	—	10-35	5-25
Coarse aggregate: ½ in. to No. 4	100	90-100	40-80	0-80	0-10	—	—	—	—
⅜ in. to No. 8	—	100	80-100	5-40	0-20	—	—	—	—
No. 4 to 8	—	—	100	9-100	0-20	—	—	—	—
Combined fine and coarse aggregate: ½ in. to 0	100	95-100	—	50-80	—	—	—	50-20	2-15
⅜ in. to 0	—	100	90-100	65-90	35-65	—	—	10-25	5-15

gradings as shown in Table 57 according to A.S.T.M. specifications.

The unit weight of the processed slag was determined as per A.S.T.M. specifications in a 1/10 cu. ft. vessel. It was found that the aggregates produced from the low shaft furnace and expanded with pit method possessed lower unit weight as compared to the specifications prescribed for such purpose, as shown below:

Dry loose unit wt. (lb./cu. ft.)

Light wt. L.S.F. Slag.		*A.S.T.M. Specifications*
Fine aggregates	60	70
Coarse aggregates	42·24	55

The fine and coarse aggregates were mixed in different proportions with cement and water. The mix was then moulded in 9 in. × 4½ in. × 3 in. blocks and 4 in. cube blocks. For moulding the blocks the mix was transfered into the mould in three layers and each layer was tamped 50 times with the tamping rod having the dimension according to A.S.T.M. specification. The blocks thus made were removed from the moulds after 24 hours and cured for 28 days in water. After curing, the blocks were dried at 100°C. and weighed till the weight was constant. The following tests were then performed with the blocks:

(i) Compressive strength test
(ii) Unit weight of the block
(iii) Bulk density test
(iv) Apparent porosity test
(v) Thermal conductivity test

The results of the tests are furnished in Table 58.

Thermal conductivity of the bricks investigated so far ranged from 1·5 to 4 in most of the bricks.

Project Reports

Following preliminary project reports relating to research work of the Laboratory were prepared and circulated to industrial concerns for commercial exploitation through the National Research Development Corporation:

(i) Production of iron powder for autogenous cutting
(ii) Electrical resistance alloys for heating elements
(iii) Technology of production of thermostatic bimetals
(iv) Flux for submerged arc welding
(v) Manufacture of alnico type permanent magnet alloys
(vi) Production of ceramic magnets
(vii) Production of carbon-free-ferro-alloys by alumino-thermic reaction.

Besides the above, Project Reports are also prepared at the request of the industries. A Project Report for the establishment of 3000 tons/annum of malleable iron castings was prepared. The report furnished details of the raw materials, method of production,

TABLE 58 — RESULTS OF TESTS CONDUCTED ON THE BLOCKS

SL No.	PROPORTION OF THE MIX.			UNIT WT IN LB./C. FE	COMPRESSIVE STRENGTH IN P.S.I.	BULK DENSITY	APP. POROSITY IN %	THERMAL CONDUCTIVITY IN B.T.U. PER SQ. FT PER HR PER DEGREE FAHR. FOR A THICKNESS OF 1 INCH
	Cement:	Fine: Aggregates	Coarse Aggregates					
1	1 :	2½ :	7½	72·960	126	1·04	48·65	2·9
2	1 :	3 :	6	68·10	119	1·05	50·00	2·87
3	1 :	2 :	8	68·64	101	1·06	48·10	3·32
4	1 :	1 :	9	64·80	91	0·98	51·48	Being determined
5	1 :	0 :	5	73·08	630	1·03	46·53	1·463
6	1 :	0 :	6	72·00	462	1·04	48·31	2·49
7	1 :	0 :	8	70·74	185	0·99	52·60	Being determined
8	1 :	0 :	9	64·08	98	0·98	53·24	do
9	1 :	0 :	10	65·40	168	0·96	50·24	do
10	1 :	3 :	5	82·99	125	1·28	49·13	do
11	1 :	3 :	4	80·294	224	1·23	45·00	do
12	1 :	3 :	3	82·476	476	1·19	48·65	2·356
13	1 :	3 :	7	71·40	84	1·28	56·9	Being determined

A.S.T.M. Specifications: unit wt of the bricks: 50-90 lb./c. ft; Thermal conductivity: 1·5-3·00 B.t.u./hr/sq. ft/°F./inch.

plant and equipment, capital expenditure, recurring expenditure, cost analyses, profit and loss account, etc.

Publications

NML Technical Journal — The NML Technical Journal, a quarterly issue, has stepped into its fourth year of publication. It is gratifying to state that the Journal has attained a set-up at par with other leading technical journals in India and abroad. The papers published in NML Technical Journal are abstracted by leading Abstracting Services, like 'Chemical Abstracts', 'Journal of Iron & Steel Institute, U.K.' etc. As many as one hundred and seventy-five Indian and foreign technical journals, publication, etc., are obtained on free exchange basis with NML Technical Journal and there are one hundred sixty-five annual subscribers of the NML Technical Journal.

Monograph — The monograph on 'Foundry Moulding Sands of India' published earlier was exceedingly well reviewed by foundry technologists and foundrymen at home and abroad. Excerpts from comments received in connection with this publication are reproduced below:

> "It represents an excellent piece of work, and with the growth of the foundry industry in India, should be an outstanding contribution towards helping foundrymen in your country to avail themselves of suitable moulding and core sand . . ."
>
> S. C. MASSARI,
> Technical Director,
> American Foundrymen's Society,
> U.S.A.

> ". . . This excellent book describes the occurrence, composition and properties of known deposits of foundry sands and bonding clays in India. No similar work on this scale has been attempted in Britain since Boswell's monograph was published in 1918 and the Indian authors have the advantage that methods of testing sand

have been developed in the interval . . . Anyone who has had experience in the examination of raw sands to determine their suitability for foundry use will appreciate the enormous amount of work carried out by the authors, the thoroughness of the investigation and the way in which the results have been condensed without loss of clarity . . . "

E. B. PARKES,
British Cast Iron
Research Association,
Birmingham, U.K.

" The reader will find a most comprehensive survey of Indian sand and bonding clays, and from both test and tables, their suitabilities for almost any foundry purpose can be readily determined. The book presents in a most lucid manner the results of what must have been a monumental research programme. If a similar modern work for the sand of the United Kingdom were to be undertaken, it could, with profit, be modelled on the same line as this monograph. The sand described are first classified both chemically and mineralogically, and the results of tests based on the standards of the American Foundrymen's Society are described. Sintering tests and production of trial castings lead finally to recommended use for each sand in turn ".

O. P. EINERL,
Foundry Scientist,
United Kingdom

Proceedings of the Symposium

During the year under review, publication covering the proceedings of the Symposium on " Light Metal Industry in India " which was held under the auspices of the National Metallurgical Laboratory in February 1961, was brought out within a record time. The publication contained twenty nine authoritative technical papers, brought out with a full account of the discussions that took place.

Translation Service

Scientific and technical papers published in French, German, Russian and Spanish languages are translated into English by the Translation Section of the Laboratory to provide immediate references and information required for the research work in progress. These translations are fully edited and copies are widely distributed among the research staff. Nearly 200 technical papers published in French, German and Russian have been translated so far each paper being on an average of 4,000 words.

Besides attending to general technical correspondence, the Translation section of the National Metallurgical Laboratory also offered facilities for oral translations and oral abstracting of scientific papers to ensure a wider coverage of Technical Journals published in languages other than English.

Patents and Technical Information and Survey Work

Over two thousand Patents and technical articles were summarized and made available to research workers during the year under review. Thorough scrutiny of the patent applications on metallurgical subjects filed with Patents Office was regularly made and relevant technical data and particulars were passed on to the research workers. The patent and technical literature survey has been found to be immensely useful both from fundamental and applied angles relating to research projects under active study at the National Metallurgical Laboratory.

Library and Documentation Service

The Library of the National Metallurgical Laboratory maintained an increasing tempo in fulfilling its role of providing the research staff with the desired technical literature. Its collections reached the 30,000 mark including books, bound volumes, journals and patent specifications. During the year the patent specifications were systematically classified so that search for patents by staff of National Metallurgical Laboratory and other scientists who used the National Metallurgical Laboratory as a regional patent inspection centre was greatly facilitated. The patent index was maintained on linedex cataloguers which made it possible to readily trace relevant patents on diverse subjects and explore their fields of application.

Documentation service facilities of the Library supplementing the existing printed abstracting services such as chemical, physical and metallurgical abstracts and special abstracts on cards of the Iron and Steel Institute were further expanded. The library's specialized abstract service on metallurgy and allied subjects was circulated in the form of a fortnightly technical bulletin, IOTA (Index of technical articles) which runs to more than 50 pages. The cards of the IOTA abstracts were maintained in a classified form under approximately 200 subject headings evolved on the basis of the laboratory's specific needs. These classified cards giving the most up-to-date information, were effectively used by the staff of the Laboratory. The IOTA and other abstracts provided finger-tip service to the staff of the National Metallurgical Laboratory covering scientific and technical information on subjects of their choice. Technical material from IOTA also found its way into the News and Notes published in the NML Technical Journal.

The Library now subscribed to about 500 scientific and technical journals and received an additional 175 journals in free complimentary exchange with NML Technical Journal, making a total periodicals list of over 650 which provided an extensive coverage of technical fields of metallurgical sciences based on fundamental and applied themes.

Technical Aid to Industries

Technical advice rendered to various industrial organizations, semi-government and Government bodies increased several folds both in the quantum of technical advice rendered as well as in the number of adhoc short and long term investigations besides specifications tests undertaken. One hundred and fifty cases of technical advice were attended to and sixty eight short-term investigations besides specification tests on behalf of the industries were conducted during the period under review.

Processes Patented

The following patents were filed during the period under review:

1) *Indian Patent No.* 76415 — Improvement in or relating to the modification of aluminium base alloys containing silicon — S. S. BHATNAGAR, P. K. GUPTE, B. R. NIJHAWAN & G. G. NAIR (on 27-4-61).
2) *Indian Patent No.* 79598 — An improved device for the continuous vapour phase degreasing of metallic wire and strip — M. J. SHAHANI (on 1-12-61).
3) *Indian Patent No.* 79597 — Improvements in a continuous vertical counter current solid-gas reactor — M. J. SHAHANI (on 1-12-61).
4) *Indian Patent No.* 81402 — Improvements in or relating to electro-deposition of metals by direct current electrolysis of aqueous solutions containing metal ions — T. BANERJEE & N. DHANANJAYAN (on 26-3-62).
5) *Indian Patent No.* 82403 — Improvements in a pneumatic basic-lined side blown vessel for the conversion of phosphorus containing pig-irons into high-grade steels — M. J. SHAHANI & UPKAR SINGH (on 26-3-1962).
6) *Indian Patent No.* 76997 — Improvements in or relating to the production of copper powder by electrolytic Process — S. RANGANATHAN (on 5-6-61).

The following patents were accepted during the period under review:

1) *Indian Patent No.* 68171 — Compositions and method of making welding

flux — M. RAMAKRISHNA RAO, N. V. NAIDU & H. V. BHASKAR RAO (on 8-2-1961).

2) *Indian Patent No.* 68401 — A pneumatic or such other forge hammer adapted for the manufacture of bricks or blocks out of ceramic mixes — H. V. BHASKAR RAO, H. P. S. MURTHY & R. C. VERMA (on 15-3-61).

The following patents were sealed during the period under review:

1) *Indian Patent No.* 68171 — Compositions and method of making welding flux — M. RAMAKRISHNA RAO, N. V. NAIDU & H. V. BHASKAR RAO (on 16-8-61).
2) *Indian Patent No.* 68174 — Refractory compositions containing non-refractory chrome ore and refractory products made therefrom — M. RAMAKRISHNA RAO, N. V. NAIDU & H. V. BHASKAR RAO (on 19-6-61).
3) *Indian Patent No.* 68401 — A pneumatic or such other forge hammer adapted for the manufatcure of bricks or blocks out of ceramic mixes — H. V. BHASKAR RAO, H. P. S. MURTHY & R. C. VERMA (on 14-11-1961).

Practical Demonstration

The third demonstration of the process of plating on aluminium, released free of royalty and premia for the benefit of the small plating industries was arranged at the National Metallurgical Laboratory. A large number of technical representatives from plating firms as well as representatives from Small Scale Industries Service Institute of Government of India attended the demonstration.

Symposium

To focus attention on the latest technological trends and research developments in the production of ferro-alloys, a Symposium on 'Ferro-alloy Industry in India' was organized by the National Metallurgical Laboratory from February 12 to 15, 1962, to exchange technical know-how with the leading scientists and metallurgists from different parts of the world in the context of the inter-related problems facing the industry and its growth along scientific and economic lines.

The symposium drew a large gathering of top ranking scientists and technologists from all over the world like Mr. G. M. Holmes, Chief Chemists, London & Scandinavian Metallurgical Co. Ltd, U.K., — representing the British Iron & Steel Research Association, London; Dr. A. G. Wilson, Assistant Blast Furnace Manager, Appleby-Frodingham Steel Co., the United Steel Companies Ltd, Lincolnshire, U.K.; Mr. J. R. Borrie, Rolling Mill Manager, Workington Iron and Steel Co., The United Steel Companies Ltd, U.K.; Prof G. Reginald Bashforth, Unesco Expert, Professor of Ferrous Production Metallurgy, Banaras Hindu University; Mr. F. A. Lytton, Export Sales Director, Mr. J. G. Ryder, Works Director and Mr. A. B. Fitz John, Assistant Chief Engineer of Jessop-Saville Ltd, Sheffield — from U.K.; Prof. A. F. Fillippov and Mr. R. M. Sapozhnikov, Unesco Experts, Indian Institute of Technology, Bombay — from U.S.S.R., Dr. H. H. Koppers, Director, Heinrich Koppers G.m. b.H., Essen — from W. Germany; Mr. J. Westly, Metallurgical Engineer, Elektrokemisk A/S.— from Norway; besides a large number of distinguished delegates from India. Twenty nine technical papers covering the various aspects of research and development work on ferro-alloys and their production and properties were presented and discussed in six technical sessions. The subjects covered relate to (i) survey of raw materials, sampling methods and role of research; (ii) extraction and production technology; (iii) scope for development of ferro-alloy industry, utilization of by-products and methods of standardization (iv) physico-chemical principles involved in the ferro-alloy production; and (v) general aspects of ferro-alloy technology.

FIG. 80 — DR. B. R. NIJHAWAN, DIRECTOR, NATIONAL METALLURGICAL LABORATORY, WELCOMING THE DELEGATES TO THE SYMPOSIUM ON " FERRO-ALLOY INDUSTRY IN INDIA " ON THE INAUGURATION DAY

The Symposium was inaugurated by Prof. M. S. Thacker, Director-General, Scientific & Industrial Research; Sir Jehangir Ghandy, Chairman of the Executive Council, National Metallurgical Laboratory, presided.

Dr. B. R. Nijhawan, Director, National Metallurgical Laboratory welcoming the distinguished delegates from India and abroad, said that such international symposia offered stimulation of thought and practical initiation to important technical developments. In having attracted valuable technical contributions from specialists in their fields and assembled distinguished scientists, metallurgists and technologists from many parts of the world, such short and long term value of symposia could hardly be over-estimated as metallurgical summits in their respective fields.

Ferro-alloys, Dr. Nijhawan stated, have a key role to play for the pursuit of peace as also for the prosecution of conflict. Ferro-alloys represent the ' back-room boys ' at the alloy, tool, special and stainless steel industries because no steel can be made without these. And without steel, heavy industry could hardly be established. However, drain of foreign exchange expenditure for the import of different types of ferro-alloys will take place when the alloy, tool, special and stainless steel industries attain their production target figures projected during the Third Five Year Plan. In the above context, the National Metallurgical Laboratory had embarked upon some major research and development projects relating to raw materials and production of diverse range of ferro-alloys. In this connection, Dr. Nijhawan stressed the importance of the role of the National Metallurgical Laboratory in the development of mineral, ferrous and non-

FIG. 81 — SIR JEHANGIR GHANDY, CHAIRMAN, EXECUTIVE COUNCIL, NATIONAL METALLURGICAL LABORATORY, AND DIRECTOR-IN-CHARGE, TATA IRON & STEEL CO. LTD, DELIVERING THE PRESIDENTIAL ADDRESS AT THE SYMPOSIUM ON " FERRO-ALLOY INDUSTRY IN INDIA "

ferrous metal industries under the impact of successive Five Year Plans. Basic and fundamental studies, laboratory bench scale trials, pilot plant scale investigations, prototype industrial runs and preparation of Project Reports were all devoted to these objectives. However, the one basic theme linking all these activities, Dr. Nijhawan said, is training with its implications of chain reactions of thought and action for which symposia like this provided an ideal background.

Concluding, Dr. Nijhawan expressed his sincere appreciation of the very hearty cooperation and full assistance given by Mr. C. G. W. Overton, Manager, Electric Furnace Division of Associated Electrical Industries (India) Pvt. Ltd and his staff who have expeditiously supplied the pilot submerged arc smelting furnace and the ancillary equipment and erected the furnace, in conjunction with the National Metallurgical Laboratory staff, in a record time.

Sir Jehangir Ghandy, Chairman, Executive Council of the National Metallurgical Laboratory, in his presidential address stated that the growth of ferro-alloy industry in India was slow and quite disproportionate in relation to the projected developments in alloy, tool, special and stainless steel industry and the production targets set forth in the Five Year Plans. Except for the indigenous production of ferro-silicon and ferro-manganese and sporadic production of some other ferro-alloys by the Tata Iron & Steel Co. during the last World War, there had been no integrated industrial unit producing the different ferro-alloys which have to be imported. The Symposium on ' Ferro-alloy Industry in India ' was, therefore, most timely and important in order to focuss attention on the development and production procedures.

In this connection, he commended the good work done by the National Metallurgical Laboratory on the beneficiation of raw materials and smelting of special ferro-alloys on laboratory and pilot plant scale. He expressed on behalf of the Executive Council of the National Metallurgical Laboratory their thanks and sincere appreciation to Dr. B. R. Nijhawan, Director, National Metallurgical Laboratory and his colleagues for the valuable research and development work being done by them at the National Metallurgical Laboratory and also for the most adequate arrangements made for the Symposium. He further asserted that the tempo of research had to be maintained to ensure progress in the ever-changing pattern of smelting technology and felt confident that the technical papers contributed for the Symposium and their discussions would be most useful to provide impetus to laboratory investigations and pilot plant work.

Prof. M. S. Thacker, in his inaugural address stated that the honour and privilege given to him every year for inaugurating the NML Symposium were a great source of inspiration to him as it provided the opportunity of studying the excellent progress in research

FIG. 82 — A VIEW OF THE DELEGATES WHO ATTENDED THE SYMPOSIUM ON "FERRO-ALLOY INDUSTRY IN INDIA"

FIG. 83 — A GROUP PHOTOGRAPH OF SOME OF THE DISTINGUISHED DELEGATES WHO ATTENDED THE SYMPOSIUM ON "FERRO-ALLOY INDUSTRY IN INDIA"

and development work maintained by the National Metallurgical Laboratory as well as meeting a large number of scientists and technologists specialized in the particular field of study, from all over the country and abroad. Continuing, Prof. Thacker said that the developments on research and industry which had taken place in the country during the last decade cannot only be considered as a measure of excellence of economic progress obtained but also as a measure of what could be done and achieved by will, determination and devotion. He mentioned that the part played by India's National Laboratories is by no means small judged from any standards. In 1947, the Indian Scientific and Industrial Research made a beginning in planning research and training of personnel. To-day there are 28 laboratories and research institutions besides those engaged in the co-operative research numbering about 12. Taking this opportunity, Prof. Thacker said that he was most happy to note the reference made by Sir Jehangir Ghandy, Chairman, Executive Council, National Metallurgical Laboratory, in his Presidential Address about the excellent progress of major research and development projects maintained by the National Metallurgical Laboratory and devotion to research shown by the staff of the National Metallurgical Laboratory under the leadership of Dr. B. R. Nijhawan. Prof. Thacker further said that he would like to add his own compliments to those of Sir Jehangir and would like to join him in expressing his appreciation and felicitations to Dr. Nijhawan and his devoted band of workers and would give them his sincere best wishes for the future progress of the laboratory.

FIG. 84 — DR. B. R. NIJHAWAN, DIRECTOR, NATIONAL METALLURGICAL LABORATORY, EXPLAINING TO MR. J. R. D. TATA, CHAIRMAN, TATA INDUSTRIES LTD, SOME OF THE PRODUCTS AND PROCESSES DEVELOPED AT THE NATIONAL METALLURGICAL LABORATORY. SIR JEHANGIR GHANDY, DIRECTOR-IN-CHARGE, TISCO AND CHAIRMAN, EXECUTIVE COUNCIL, N.M.L. AND MR. J. D. CHOKSI, VICE-CHAIRMAN, TATA INDUSTRIES LTD, ARE ALSO IN THE PICTURE

Dwelling upon the ferro-alloy industry in India, Prof. Thacker said that its progress during and prior to the Second Five Year Plan was slow and rather insignificant, but he hoped that it would make a headway during the Third Five Year Plan period based on scientific researches and pilot plant trials. In this context, Prof. Thacker expressed his deep appreciation and paid a warm tribute to Dr. Nijhawan, Director, National Metallurgical Laboratory, and his staff for the exhaustive, systematic and excellent work they were doing towards the development of metal and mineral industries of the country. Referring to the technical papers presented at the Symposium, Prof. 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 ferro-alloy industry in India. Prof. M. S. Thacker also inaugurated the ' Pilot Submerged Arc Smelting Furnace ' installed at the National Metallurgical Laboratory.

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. The supplement contained leading papers from several visiting scientists from abroad, including articles contributed specially for the Symposium. The paper also made daily coverage of the Symposium. ' Financial Express ' of Calcutta also brought a supplement out on the second day of the Symposium and made excellent coverage of the day to day proceedings of the Symposium. The inaugural session of the Symposium was covered by many leading news papers of the country.

GENERAL

Constructional work of the two side wings of the Main Building of the laboratory made considerable progress. The 'Wings' when completed would accommodate heavy equipments and other sections of the National Metallurgical Laboratory to meet the expanding activities of the laboratory. These wings will cover radio-active isotopes tracer technique section, research and investigation section for studies on corrosion and protection of metals and alloys, vacuum melting units, X-ray diffraction sections, heat-treatment units, hydro-metallurgical including pressure leaching sections and other speculated units of general and mechanical metallurgy divisions of the National Metallurgical Laboratory.

The Governing Body of the Council of Scientific and Industrial Research approved the establishment of a unit at the National Metallurgical Laboratory for the production of permanent magnets. The establishment of the four regional foundry stations made satisfactory progress.

Purchase and Stores sections of the National Metallurgical Laboratory kept unceasing activity for procurement of equipment, raw materials and supplies. Procurement of equipment for the Manganese Beneficiation Pilot Plant sanctioned against Exim Bank Loan was effectively completed. It was estimated that equipment/stores/raw materials valued at over Rs. 45 lakhs were handled during the year.

The administrative working of the laboratory was efficiently maintained. Heavy budgetary accounts of the laboratory were handled with efficiency and speed.

Since taking over the construction of staff quarters by Council of Scientific & Industrial Research from C.P.W.D. significant progress was made in the construction of staff quarters for the National Metallurgical Laboratory. DII type multistoreyed block consisting of 10 flats and E type multistoreyed block consisting of 30 flats were nearing completion and were expected to be ready for occupation in the near future. Besides two CII types, 3 F type and 6 H type quarters were completed and occupied. Constructional work for another DII type block of 10 flats, E type block of 30 flats, and 60 F, G & H type quarters was commenced.

APPENDIX I

SCIENTIFIC PAPERS PUBLISHED DURING THE YEAR 1961-62

The following papers were published in the *NML Technical Journal*, Volume III, No. 2, May, 1961.

1. Some Problems of Documentation in Scientific Research — Mrs. K. Banerjee (13-18)
2. Production of Low Carbon Ferro-chrome from High Carbon Ferro-chrome—V. S. Sampath and P. P. Bhatnagar (19-27)
3. Pole Figures for Randomly Oriented Sheets of Silicon and Aluminium — K. D. Maji and E. G. Ramachandran (28-35)

The following papers were published in the *NML Technical Journal* Volume III, No. 3, August 1961.

4. Some Aspects of Silicate Bonding with and without CO_2 — B. V. Somayajulu, Jatinder Mohan and S. S. Bhatnagar (15-19)
5. Non-metallic Inclusions — Their Quantitative Extraction from Steel — D. S. Tandon and J. R. Gupta (20-24)
6. Corrosion of Steam Turbine Blading — A Case Study — K. P. Mukherjee, A. K. Lahiri and T. Banerjee (25-28)
7. A Photo-electric Temperature Controller — K. Soma Kumar and A. P. Chowdhury (29-31)

The following papers were published in the *NML Technical Journal*, Volume III, No. 4, November, 1961.

8. Hot-dip Aluminizing of M.S. Wire on Pilot Plant Scale — S. M. Arora, A. N. Kapoor, P. K. Gupte and B. R. Nijhawan (4-9)
9. Spectrographic Analysis of High Chromium-Manganese Austenitic Stainless Steel by Porous Cup Technique Using Neutral Filter — M. K. Ghose, S. Pait and B. C. Kar (10-12)
10. Production of Dense Carbon Aggregate from Carbonaceous Materials for Varied Volatile Contents for Use in Carbon Products in General and Soderberg Paste in Particular — P. Prabhakaram and H. P. S. Murthy (13-15)
11. Failure of Boiler Tube by Differential Aeration Corrosion — B. K. Guha, P. K. Gupte and B. R. Nijhawan (16-20)
12. Corrosion of Nickel in Steam Condenser Containing Air and Carbon Dioxide — S. Rao Addanki, A. K. Lahiri and T. Banerjee (21-24)

The papers Nos. 13 to 15 were published in *Tisco Technical Journal*, Vol., 8, No. 1, 1961:

13. Service Behaviour of Metals in Transport — B. R. Nijhawan, (1-14)
14. Metals and Alloys in Air Transport — Ved Prakash, (35-44)
15. The Non-destructive Testing of Metals Components Used in Transport — E. G. Ramachandran, (57-65)
16. Phase — Transformation in Electrolytic Alloys with Special Reference to Copper-Zinc and Silver-Copper Systems — T. Banerjee and P. L. Ahuja; *Les Mémoires Scientifiques de la Revue de Métallurgie*, Paris, Vol. 58, No. 4, (309-316), April 1961.
17. Indian Mineral Wealth and its Utilization — B. R. Nijhawan, *Souvenir of the Maharashtra Mineral Industries Conference* 1961, (27-30)
18. Utilization of Sub-standard Grade of Raw Materials for Iron Production in Low Shaft Furnace — A. B. Chatterjea and B. R. Nijhawan, *Souvenir of the Maharashtra Mineral Industries Conference* 1961, (53-62)
19. Beneficiation of Low-grade Ores with Particular Reference to Ores of Western India — P. I. A. Narayanan and G. P. Mathur, *Souvenir of the Maharashtra Mineral Industries Conference* 1961, (65-69)
20. Studies on the Production of Porous Bronze Bearings — S. Ranganathan, *J. Sci. Industr. Res.*, Vol. 20D, No. 9 (341-347), Sept. 1961 and *Research & Industry*, Vol. 6, No. 10, (358-359), 1961.
21. Electrical Resistance Alloys — *Research & Industry*, Vol. 6, No. 1 (1-32), 1961.
22. Corrosion of C.I. Fusion Pots in Caustic Soda Manufacture : Its Causes and Prevention — *Research & Industry*, Vol. 6, No. 3 (84-85), March, 1961.
23. Production of Graphite Crucibles from Indigenous Materials — *Research & Industry*, Vol. 6, No. 4 (124-26), April 1961.
24. Bimetals for Thermostatic Use — *Research & Industry*, Volume 6, No. 7 (236-238), July 1961.
25. Aluminium Bronze Castings — *Research & Industry*, Volume 6, No. 9, (309-311), September 1961.
26. Production of Low Carbon Ferro-chrome — *Research & Industry*, Vol. 6, No. 11, (385-386), November 1961.
27. Synthetic Cryolite — *Research & Industry*, Vol. 6, No. 10 (368), 1961.
28. Towards Self-sufficiency in Metals and Alloys — B. R. Nijhawan, *Eastern Metal Review*, Annual Number, 1961, Vol. XIV, No. 28, (767-772)
29. Constitution and Properties of Stabilized Dolomite — M. R. K. Rao, P. C. Sen and H. V. Bhaskar Rao, *The Refractories Journal*, (304-308), October 1961.
30. Transformation of Cathodically Impregnated and Cooled Austenite — C. Dasarathy and E. G. Ramachandran — *Nature*, Volume 191, No. 4792 (998-1000), September 2, 1961 (Letter to the Editor)
31. Metallurgical Developments in India — B. R. Nijhawan and R. M. Krishnan — *Metal Progress*, Vol. 81, No. 1 (97-133), January 1962.
32. Present Status and Scope of Foundry Industry in India — B. R. Nijhawan, *Tisco Technical Journal*, Vol. 9, No. 1, (1-9), January 1962.

33. Non-ferrous Metal Industry in India — B. R. Nijhawan and Chintamani Sharma, *Eastern Metals Review*, Annual Number, 5 February 1962, Volume XV, No. 1, (59-69)

34. Corrosion of Nickel Tubes in Caustic Soda Evaporators — *Research & Industry*, Volume 7, No. 2, (46-48), February 1962.

35. Foundry Moulding Sands and Bonding Clays — R. M. Krishnan and B. V. Somayajulu, *Tisco Technical Journal*, Volume 9, No. 1, (24-33), January 1962.

36. Melting Techniques and Heat-treatment of Castings — A. B. Chatterjea, *Tisco Technical Journal*, Volume 9, No. 1, (81-91), January 1962.

37. Anisotropy of Electrical Resistivity in Cold Rolled 60 : 40 Brass — E. G. Ramachandran and K. D. Maji, *Journal of Institute of Metals*, London, 1961-1962, Volume 90, part 5 (172-175), January 1962.

The following papers were published in the *Proceedings of the Symposium on " Light Metal Industry in India "* held under the auspices of this Laboratory in 1961:

38. The Role of the National Metallurgical Laboratory in Research and Development of Light Metal Industry in India — B. R. Nijhawan (32-49)

39. A Study of the Production of Soderberg Paste from Indian Raw Materials — P. Prabhakaram & H. P. S. Murthy (108-111).

40. Development of Light Metal Industry in India — P. K. Gupta and K. N. Srivastava (150-159)

41. The Scope for Development of Magnesium Industry in India — R. N. Misra, V. S. Sampath and P. P. Bhatnagar, (200-205)

42. Electrolytic Production of Magnesium and Magnesium Alloys — P. K. Som, P. B. Chakravarti and T. Banerjee (206-211).

43. 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 and B. R. Nijhawan (222-230)

44. Recent Advances in the Technology of Al-Mg. Alloys — Y. N. Trehan and S. P. Bhadra (212-221)

The papers Nos. 45 to 48 were published in the special *Amrita Bazar Patrika* Supplement dated 12 February 1962 brought out during the Symposium on "Ferro-alloy Industry in India" held under the auspices of the National Metallurgical Laboratory:

45. Role of the National Metallurgical Laboratory in Research and Development of Ferro-alloy Industry — B. R. Nijhawan,

46. Ferro-alloy Industry in India — B. R. Nijhawan and P. P. Bhatnagar.

47. Electrolytic Preparation of Iron-chromium Alloys from Chromite Ore — S. C. Aush, P. B. Chakravarti and T. Banerjee.

48. Beneficiation of Indigenous Raw Materials — G. P. Mathur and P. I. A. Narayanan.

49. Ferro-alloys Industry in India : Greater Potentials Revealed — R. M. Krishnan, *Financial Express*, Dt. 12.2.62.

50. Design Factors in Basic Side Blown Converter Pneumatic Steelmaking — B. R. Nijhawan and M. J. Shahani, *Blast Furnace & Steel Plant*, Vol. 50, No. 1, (40-46), January 1962.

APPENDIX II

SCIENTIFIC INVESTIGATIONS COMPLETED AND REPORTS PREPARED

1. Moulding Characteristics of Moulding Sand received from the Foundry, Southern Railway Workshops, Mysore — K. K. Balasankar, Jatinder Mohan and S. S. Bhatnagar (IR202/61)
2. Investigation on the Core Properties of "Loudna Yellow Fine Core Sand" from Messrs Kirloskar Bros., Kirloskarvadi, Bombay-7--T. A. Beck, S. S. Bhatnagar & P. K. Gupte (IR203/61)
3. Crushing Tests with a Sample of Iron Ore from Kiriburu—S. B. Das Gupta, G. P. Mathur and P. I. A. Narayanan (IR204/61)
4. Beneficiation of Low Grade Garnet from Guda Area, Ajmer — P. K. Sinha, G. P. Mathur and P. I. A. Narayanan (IR205/61)
5. Preliminary Washing Test with an Iron Ore Sample from Kiriburu Iron Ore Project (N.M.D.C.) — S. B. Das Gupta, G. P. Mathur & P. I. A. Narayanan (IR206/61)
6. Investigation on the Moulding Characteristic of Silica Sand, Moulding Sand and Bonding Clay sent by Messrs Britannia Engg Co. Ltd, Titaghur, W. B. — T. A. Beck & S. S. Bhatnagar (IR207/61)
7. Studies on the Suitability of Water from Lakshmansagar in Dungarpur District, Rajasthan, for Flotation of Fluorspar from the nearby Deposits — P. V. Raman, G. V. Subramanyam and P. I. A. Narayanan (IR208/61)
8. A Study of Hirri, Hardi and Bara-duar Dolomites for Use in the Shaft Kilns at Hindustan Steel Ltd, Rourkela — M. R. K. Rao, P. C. Sen, & H. V. Bhaskar Rao (IR209/61)
9. Beneficiation of Low-grade Chromite from Vagda Deposits, Ratnagiri District, Maharashtra — R. Ganesh, G. V. Subramanyam and P. I. A. Narayanan (IR210/61)
10. Beneficiation of Calcitic Fluorspar from Matamagri Deposit, Rajasthan — P. V. Raman & P. I. A. Narayanan (IR211/61)
11. Reduction of Silica Content in Limestone Sample No. 1 from Purnapani Quarries of Messrs Hindustan Steel Ltd, Rourkela — A. Peravadhanulu, G. V. Subramanyam & P. I. A. Narayanan (IR212/61)
12. Reduction of Silica Content in Limestone Sample No. 2 from Purnapani, Sundargarh District, Orissa — M. V. Ranganathan & P. I. A. Narayanan (IR213/62)
13. Reduction of Silica Content in a Dead Burnt Magnesite Sample from Salem — A. Peravadhanulu, G. V. Subramanyam and P. I. A. Narayanan (IR214/61)
14. Studies on Sintering of Manganese Concentrate from Low-grade Manganese Ore from Siljora — Kalimati Mines, Orissa — B. L. Sengupta, G. V. Subramanyam and P. I. A. Narayanan (IR215/61)

15. Reduction of Silica Content in a Limestone Sample No. 3 from Purnapani, Orissa — P. K. Sinha, G. V. Subramanyam and P. I. A. Narayanan (IR216/61)

16. Reduction of Iron Content in a Rutile Sample from Travancore Minerals, Quilon — S. K. Banerjee and P. I. A. Narayanan (IR217/61)

17. Non-metallic Inclusions — Their Quantitative Extraction from Steel — D. S. Tandon and J. R. Gupta (IR218/61)

18. Reduction of Silica Content in a Limestone Sample (No. 4) from Purnapani Quarries, Orissa — R. Ganesh, G. V. Subramanyam and P. I. A. Narayanan (IR219/61)

19. Pilot Plant Studies on Beneficiation and Sintering of Bolani Iron Ores — S. B. Das Gupta, S. K. Banerjee, P. V. Raman, G. P. Mathur, G. V. Subramanyam and P. I. A. Narayanan (IR220/61)

20. Investigation into the Cause of Failure of Cold Rolling Roll of the Indian Tube Co. Ltd., Jamshedpur (IR221/61)

21. Basic Characteristics of Silica Sand for use in Foundries — R. M. Krishnan (IR222/61)

22. Investigation on Sabarmathy River Sands — K. K. Balasankar, Jatinder Mohan and S. S. Bhatnagar (IR223/61)

23. Investigation Report of the Bentonite sent by Messrs Machinery & Equipment Manufacturers Private Ltd., Ahmedabad — A. T. Chirayath, Jatinder Mohan & P. K. Gupta (IR224/61)

24. Beneficiation of Classifier Sand and Heavy Slime from the Noamundi Washing Plant, Tisco.— S. B. Das Gupta, G. P. Mathur & P. I. A. Narayanan (IR225/61)

25. Studies on the Decomposition Characteristics of Amjhore Pyrites — S. R. Srinivasan and P. P. Bhatnagar (IR226/61)

26. Beneficiation of Top Bench Iron Ore and Kanga Iron Ore from the Western Ridge, Noamundi — S. B. Das Gupta, G. P. Mathur and P. I. A. Narayanan (IR227/61)

27. Refractory Properties of Sea-water Magnesia Produced by Messrs Tata Chemicals, Mitthapur — P. C. Sen, M. R. K. Rao, & H. V. Bhaskar Rao (IR228/61)

28. Preliminary Flotation Studies on Fluorspar from Baroda District, Gujarat — P. V. Raman, G. V. Subramanyam, & P. I. A. Narayanan (IR229/61)

29. A Study of the Moulding Characteristics of the Beach Sands of Trivandrum (Kerala State) — K. N. Kartha & K. K. Balasankar (IR230/61)

30. An X-ray Study of the Reaction of Calcium Oxide and Calcium Chloride in the Decomposition of Monazite — K. N. Kartha (IR231/62)

31. Investigation of Suitability of Indigenous Asbestos for the Manufacture of Asbestos Cement Products (IR232/62)

32. Reduction of TiO_2 Content in a Sample of Iron Ore from Khursipar Area (Maharashtra) — M. V. Ranganathan, G. V. Subramanyam & P. I. A. Narayanan (IR233/62)

33. Investigations of the Rajula Bentonite received from Messrs Ambica Mine Chem. Industries, Nari Road, Bhavnagar — A. T. Chirayath, Jatinder Mohan and P. K. Gupte (IR234/61)

34. Pilot Plant Beneficiation Studies on a Limestone from Tisco and Proposals for Setting up a Treatment Plant of Capacity 250 Tons/day — P. V. Raman, S. K. Banerjee, G. V. Subramanyam, & P. I. A. Narayanan (IR235/61)

35. Pilot Plant Studies on Beneficiation and Sintering of Barsua Iron Ore for Rourkela Steel Plant of Hindustan Steel Limited — P. V. Raman, S. K. Banerjee, S. B. Das Gupta, G. P. Mathur, G. V. Subramanya & P. I. A. Narayanan (IR236/62)
36. Investigation into Oxygen Steel Making Process Employing Indian Raw Materials and Refractories — B. L. Sen, B. R. Nijhawan and A. B. Chatterjea (RR118/61)
37. Some Aspects of Silicate Bonding with and without CO_2 — B. V. Somayajulu, Jatinder Mohan and S. S. Bhatnagar (RR119/61)
38. Spectrographic Analysis of High Chromium-Manganese Austenitic Stainless Steel by Porous Cup Technique Using Neutral Filter — M. K. Ghosh, S. Pait and B. C. Kar (RR120/61)
39. Phase Transformation in Electrolytic Alloys with Special Reference to Copper-zinc and Silver-copper Systems — T. Banerjee and P. L. Ahuja (RR121/61)
40. Sintering of Iron-nickel-aluminium Alloys — S. K. Bose, Ved Prakash & E. G. Ramachandran (RR122/61)
41. Thermal Beneficiation of Low-grade Chrome Ores — Part II — Solid Reduction — R. N. Misra & P. P. Bhatnagar (RR123/61)
42. Studies on Fatigue Behaviour of Some Structural Steels — B. N. Das & G. D. Sani (RR124/61)
43. Thermal Beneficiation of Low-grade Ferruginous Manganese Ores — C. Sankaran, M. C. Sen & P. P. Bhatnagar (RR 125/61)