

ANNUAL REPORT

1962-1963



NATIONAL METALLURGICAL LABORATORY

COUNCIL OF SCIENTIFIC & INDUSTRIAL RESEARCH

JAMSHEDPUR, INDIA

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FOREWORD

IT gives me great pleasure to present the Report of the Director, National Metallurgical Laboratory, for the year 1962-63. I have had the opportunity of visiting the National Metallurgical Laboratory a number of times as also of visiting similar research institutions in India and overseas. I have watched the progress of National Metallurgical Laboratory with great interest. I am particularly gratified to note the high prestige and professional status with which the National Metallurgical Laboratory is held both at home and overseas.

Whilst inaugurating the International Symposium on Iron and Steelmaking organized by the National Metallurgical Laboratory in collaboration with the British Iron & Steel Institute and the Indian Institute of Metals, I had referred to the important and supreme role of research and development in the field of iron and steel production technology. I had also mentioned the importance of undertaking research with indigenous talents employing indigenous raw materials which have been one of the keystones of the excellent progress made by the National Metallurgical Laboratory. I was particularly gratified to listen to what Mr. Maurice Fiennes, President of the Iron & Steel Institute, had said during the Symposium that he was greatly pleased to see the National Metallurgical Laboratory which has set a very fine example of the determination of India to produce her own technology by the use of her own technologists.

Whilst, on the one hand, the National Metallurgical Laboratory has rightly earned high prestige in overseas circles, it has equally earned due recognition from leaders of private sector iron and steel plants, such as Mr. J. R. D. Tata who has often expressed his appreciation of the contributions of the National Metallurgical Laboratory to iron and steel industry in more than one platform. Equally so has been the experience of the Hindustan Steel Ltd. in relation to the National Metallurgical Laboratory which has spared no pains to undertake both short-term and long-range research and pilot plant projects on multitude problems both in the long-range study and appraisal of the vital raw materials as also undertaking development research projects on short-term themes. The National Metallurgical Laboratory has done valuable work on the establishment of small iron and steel plants on a dispersal basis including the establishment of South Indian iron and steel plants and has been an active champion for the installation of small pig iron production plants in the country.

I have also been impressed with the emphasis laid by the laboratory on large-scale industrial pilot plants and the notable progress made in the industrial scale implementation of the important processes developed by the National Metallurgical

Laboratory, which has resulted in the release of practically all the processes developed at the National Metallurgical Laboratory. The heavy integrated Mineral Beneficiation Pilot Plant, built at a cost of Rs. 5 million and which was inaugurated by Prof. Humayun Kabir, Union Minister for Scientific Research & Technical Education, early this year, at the time of last Symposium, has been acknowledged by the leading scientists as one of the best of its kind in the world. It is particularly pleasing to note that the entire planning, layout and engineering was done by the staff of the National Metallurgical Laboratory. Since its commissioning in February 1963, I find the plant is being operated round the clock and has already beneficiated hundreds of tons of iron ores and limestone for their utilization in public and private sector steel plants.

Although emphasis has been laid on operational and applied research projects, the laboratory has maintained equally good progress on a number of fundamental projects underway which have yielded fruitful results besides training a corps of research workers.

During the year, I find important pilot plant equipment and research apparatus have been designed and fabricated at the National Metallurgical Laboratory effecting considerable saving of foreign exchange. There has also been a substantial increase in the number of junior and senior research fellowships for those working for higher post-graduate degrees. I also commend the steps taken by the laboratory for imparting specialized training both at graduate and artisan levels in various metallurgical fields 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 laboratory completed the following project investigations and their reports have also been issued:

1. Report on the operation of Low-shaft Furnace Pilot Plant at the National Metallurgical Laboratory for the production of pig iron with East Punjab raw materials from Mahindergarh.
2. Report on the operation of Low-shaft Furnace Pilot Plant at the National Metallurgical Laboratory for the production of foundry pig irons with raw materials from Maharashtra State.
3. Report on the operation of Low-shaft Furnace Pilot Plant at the National Metallurgical Laboratory for the production of pig iron with iron ore and limestone from Nepal.
4. The operation of Low-shaft Furnace Pilot Plant at the National Metallurgical Laboratory for the production of pig iron with raw materials from Rajasthan.
5. Pilot Plant Studies on Beneficiation and Sintering of Kiriburu Iron Ore from National Mineral Development Corporation.
6. Pilot Plant Studies on Beneficiation and Sintering of Rajhara Iron Ore from Bhilai Steel Plant of Hindustan Steel Ltd.

The *NML Technical Journal* has now entered into the fifth year of publication and has established its place as a journal of repute in the field of metallurgy. The National Metallurgical Laboratory has brought out early this year another useful publication relating to the Proceedings of the Symposium on 'Ferro-alloy Industry in India'. As usual, the laboratory has maintained its high standard in the presentation and the technical value of the contents of the publication. The industrial liaison and scientific dissemination activities of the laboratory have given a great impact on the general level of technology of the country.

On my behalf and that of the members of the Executive Council, I would like to express our sincere appreciation of the sustained and devoted work that is being done under the able guidance of Dr. B. R. Nijhawan, Director of the Laboratory. I should like them to know that the members of the Executive Council of the National Metallurgical Laboratory deeply 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 an appreciation for the commendable success already achieved which augurs well for the future.

Sd/- C. SUBRAMANIAM

Chairman

Executive Council of the

National Metallurgical Laboratory

New Delhi

October 15, 1963

INTRODUCTION

DURING the year under review, research and development work in general and semi-industrial scale pilot plant trials at the integrated pilot plants of the National Metallurgical Laboratory in particular have continued to make steady progress. Almost all the processes developed at the National Metallurgical Laboratory have now been released to the industry on payment of handsome royalties, premia, etc., which is proof of acceptance by the industry of the fruits of research. Research and development work relating to the defence projects have continued to be given top priority and resources of this laboratory have been geared fully to meet the requirements of strategic metals and alloys needed for defence and ordnance organizations despite shortage of equipment and facilities.

The heavy integrated Mineral Beneficiation Pilot Plant, involving an outlay of Rs. 50 lakhs during the Third Five Year Plan, was commissioned early this year for round the clock operation for the beneficiation of raw materials particularly the steel quality flux limestone and its subsequent agglomeration for use in steel-making. The results obtained have been exceedingly useful. The mineral and metal industries have in recent years been increasingly appreciative of the immediate value and long-term benefits of research in the scientific growth and development of metallurgical industries.

Research work relating to agglomeration studies such as pelletizing, briquetting, sintering covers both fundamental and applied fields. A continuous sintering unit will be shortly installed which will further augment the facilities at the integrated

pilot plants in National Metallurgical Laboratory ranking amongst the best of their kind installed anywhere.

Likewise, operations of the Low-shaft Furnace Pilot Plant have maintained active progress during the year under review. The Foundry Regional Station in Batala is now functioning. The Corrosion Research Station at Digha will also start working by the end of this year.

The three side wings of the Main Laboratory Building have been completed and the main items of equipment relating to vacuum melting furnace, magnesium reduction furnace, installation of high-frequency melting furnaces, electronic instrumentation and X-ray fluorescence (Philips Autrometer) unit are now being installed in the side wings. Equipment relating to extraction metallurgy techniques and corrosion research at the National Metallurgical Laboratory is being housed in a new complete wing for undertaking corrosion research studies including surface passivity, electro-chemical factors involved in different types of corrosion attack, corrosion studies under different chemical media including stress corrosion, etc. The newly constituted Iron and Steel and Alloy Steel Divisions are moving into the respective side wings.

Early this year, the National Metallurgical Laboratory organized an International Symposium on 'Recent Developments in Iron and Steel Making with Special Reference to Indian Conditions' in collaboration with the Iron & Steel Institute, London, and the Indian Institute of Metals; this joint Symposium was unparalleled in India in many ways, not only in exchanging of technical views and ideas but

also in the opportunity it provided for the growth of professional trends within the Commonwealth and beyond. More than 300 delegates from home and abroad participated in the deliberations of the Symposium, including Sir Charles Goodeve, O.B.E., D.Sc., F.R.S., Director, British Iron & Steel Research Association, and Mr. Maurice Fiennes, President, Iron & Steel Institute, London. Fifteen authoritative papers on diverse aspects of iron and steel technology including preparation and use of Indian raw materials were presented and discussed. It is gratifying to report that the contributions made by the National Metallurgical Laboratory on the research and development work in relation to beneficiation studies of iron ores and iron production in Low-shaft Furnace were duly appreciated.

During the period under review, the National Metallurgical Laboratory also brought out an excellent publication containing the technical papers and Proceedings of the Symposium on 'Ferro-alloy Industry in India', held in February 1962 and which has been exceedingly well reviewed both at home and overseas. The *NML Technical Journal* has stepped into the fifth year of publication and its demand is increasing year by year.

Iron and steel industry both in the public and private sectors have continued to refer their long-range problems to the National Metallurgical Laboratory such as on the raw materials and finished products involving both short-term and long-term research projects. Several research and pilot plant investigations were undertaken and comprehensive investigation reports were issued to Hindustan Steel Ltd., Government of Rajasthan and His Majesty's Government of Nepal. During the period under review, successful smelting trials were conducted on the beneficiation and sintering of Rajhara iron ore for Bhilai Steel Plant of Hindustan Steel Ltd., and a detailed investigation project report incorporating economic evaluation of beneficiation has been issued

to the Hindustan Steel Ltd., as the basis for the installation of a beneficiation and sintering plant in consultation with the Soviet authorities. The systematic work of the National Metallurgical Laboratory has been acknowledged in the latest Annual Report (1961-62) of the Hindustan Steel Ltd.: "In particular the assistance given by the National Metallurgical Laboratory is gratefully acknowledged." Work on beneficiation of limestone has made notable progress. Several hundred tonnes of limestone concentrates, pellets and briquettes were prepared and large-scale steel-melting trials conducted in the 200-ton open-hearth furnace of Tata Iron & Steel Works have yielded highly promising data in respect of results to be expected.

Systematic researches on fundamental projects have yielded fruitful results apart from training a corps of scientific research workers. Fundamental problems under investigation have related to isothermal transformation studies of plain carbon and alloy steels, gas solubility studies in metals, carbon activity in iron and its alloys, studies on age-hardening, interaction between solute and solvent atoms in the liquid state, study on temper brittleness, phase studies on electro-deposited copper-tin alloys, studies on phase transformation in alloys by X-ray diffraction techniques, morphology of phosphorus in Indian manganese ores, etc.

Large reserves of low-grade Indian chrome ore having Cr:Fe ratio less than 3 cannot be directly used for the production of high-grade ferro-chrome. Conventional physical methods have failed to improve this ratio as FeO and Cr_2O_3 are in a chemically combined state in the chrome spinel. Investigations were undertaken on three low-grade chrome ores for upgrading by preferential reduction of FeO and its subsequent removal by acid leaching, yielding concentrates having Cr:Fe ratio 4, 8 and 12 with more than 90 per cent chromium recovery.

Investigation on the utilization of granulated low-shaft furnace slag for production of slag cement has yielded highly encouraging results. It was observed that granulated low-shaft furnace slag possesses good hydraulic properties. Slag cement was prepared by mixing granulated slag and Portland cement as activators in different proportions. The chemical composition, compressive strength and strength values of slag cement were determined in accordance with A.S.T.M. and B.S. specifications. The results obtained indicate that slag cement produced from low-shaft furnace slag with Portland cement as activator satisfies the A.S.T.M. and B.S. specifications. Work has also been done to find out the suitability of low-shaft furnace slag in expanded and porous forms, as aggregate for light-weight insulation concrete. No work has so far been done on the utilization of expanded slag from blast furnaces of iron and steel plants of the country. The trials made so far according to the A.S.T.M. specifications showed that the molten slag of proper composition when made porous by expanding with limited quantity of water yields a light-weight but strong aggregate which can serve as an ideal raw material for insulation concrete.

Electric smelting trials were completed on Salem magnetite concentrate briquettes in the 500 kVA. Submerged Arc Furnace of the National Metallurgical Laboratory and a detailed report was issued to the Government of Madras. The magnetite was beneficiated at the Pilot Plant of the National Metallurgical Laboratory. The chemical analysis of the pig iron obtained was excellent for foundry grades especially for malleable iron production for which it would fetch high premium owing to its exceptionally low-phosphorus content. The pig iron was equally suitable for steelmaking by conventional methods. These reports issued by the National Metallurgical Laboratory on the subject would, it is hoped, lead to establishment of an iron and steel plant in South India.

The Low-shaft Furnace Pilot Plant was continually in operation; two investigation project reports were issued during the period under review. Smelting trials were undertaken on iron production with Rajasthan raw materials, viz. iron ore and limestone employing nut coke as the reductant and the fuel. Due to the non-occurrence of coal in Rajasthan, the smelting of iron will have to be conducted with surplus nut coke procured from the nearest integrated iron and steel plant. Electro-thermal smelting with consequent reduction of fuel is equally feasible provided electric power is available. An investigation project report giving the results of pilot plant trials was issued to the Government of Rajasthan. Smelting trials for production were conducted at the request of His Majesty's Government of Nepal, in presence of their technical staff, with iron ore and limestone derived from Nepal. In view of the total absence of coal in Nepal, the trials were conducted with nut coke as the fuel and the reductant. A detailed investigation project report indicating the feasibility of a small iron-smelting unit was issued to the Government of Nepal.

With a view to utilizing surplus coke breeze available at the integrated iron and steel plants for iron smelting, trials were successfully conducted in the Shaft Furnace, designed and fabricated at the National Metallurgical Laboratory employing self-fluxing briquettes containing coke breeze, iron ore and limestone, bonded with 4-6 per cent bentonite as the binder. The Shaft Furnace has arrangements for pre-heating the blast in a recuperator system designed at the National Metallurgical Laboratory.

Pilot plant scale trials were also conducted on steelmaking in a basic-lined side-blown air converter (designed and fabricated at the National Metallurgical Laboratory) employing basic iron made at the Low-shaft Furnace Pilot Plant of the National Metallurgical Laboratory.

The results obtained are highly favourable for producing normal grades of steel in small steelmaking units.

The National Metallurgical Laboratory is today heavily engaged in giving maximum possible assistance to meet the requirements of the defence and ordnance establishments by undertaking production of essential products and short-term ad hoc research investigations and development work to meet the deficiencies in respect of special alloys. The National Metallurgical Laboratory has prepared and supplied the following ferro-alloys to the defence authorities conforming to rigid defence specifications to meet their urgent requirements:

High-carbon ferro-chrome

Low-carbon ferro-chrome

Ferro-tungsten

Ferro-titanium

Ferro-aluminium

In this connection, it is stated that ferro-tungsten has been produced and supplied for the first time in India from indigenous raw materials. Reconditioned magnesium powder has been supplied to the Ammunition Factory, Kirkee, and Ordnance Factory, Jabalpur, which prior to treatment at the National Metallurgical Laboratory contained 87.46 per cent magnesium and which was enriched to 99 per cent magnesium at the National Metallurgical Laboratory. The reconditioning capacity was stepped up by over fifty times from 250 lb. per month to 500 lb. per day. The defence authorities have expressed their sincere appreciation for the work undertaken at the National Metallurgical Laboratory on their behalf.

The laboratory has also continued to act as a central agency for long-range and short-term investigations connected with the development work of metals and alloys referred to by the National Mineral Development Corporation and Railway Designs and Standards Organization, Indian Standards Institution and other iron and steel organizations.

During the year the laboratory has taken the following patents:

Patents Filed

1. Indian Patent No. 82191 — An improved jacketted electrolytic cell for the electro-deposition of metals and metallic oxides in general and manganese dioxide in particular (on 10-5-1962).

2. Indian Patent No. 83652 — Improvements in or relating to magnesite refractories (on 10-8-1962).

3. Indian Patent No. 83968 — A method for reconditioning the coated magnesium powders (on 3-9-1962).

4. Indian Patent No. 84670 — Improvements in or relating to electrolytic cells (on 19-10-1962).

Patents Accepted

1. Indian Patent No. 76997 — Improvements in or relating to the production of copper powder by electrolytic process (on 28-11-1962).

2. Indian Patent No. 76415 — Improvements in or relating to the modification of aluminium alloys containing silicon (on 18-7-1962).

Patents Sealed

1. Indian Patent No. 76997 — Improvements in or relating to the production of copper powder by electrolytic process (on 18-5-1963).

2. Indian Patent No. 76415 — Improvements in or relating to the modification of aluminium alloys containing silicon (on 22-2-1963).

The process on aluminizing of ferrous materials developed at the National Metallurgical Laboratory has been leased out to over two dozen firms for commercial production. The technical know-how of the method is under demonstration to representatives of the firms.

A brief résumé of the progress of important projects under way at the National Metallurgical Laboratory during the period under review is given in the following pages.

RESEARCH PROJECTS

1.0 Pilot Plant Studies on Beneficiation and Sintering of Rajhara Iron Ore for the Bhilai Steel Plant of Hindustan Steel Ltd.

Systematic pilot plant studies were undertaken on beneficiation and sintering of iron ore from Rajhara for the Bhilai Steel Plant of Hindustan Steel Ltd., including the economics of iron production resulting from the use of self-fluxings inter. A 60-tonne representative sample of run-of-mine ore from Rajhara iron ore mines of Bhilai Steel Plant, Hindustan Steel Ltd., assaying Fe, 56.25; SiO_2 , 7.17; Al_2O_3 , 6.39; CaO, 0.68; MgO, 0.75; P, 0.076 per cent; and loss on ignition, 5.30 per cent, was received for beneficiation and sintering studies.

The sample contained predominantly hematite followed by goethite, lepidocrocite and magnetite. Quartz, calcite and clay were present as minor constituents. Red and yellow ochres or lateritic material and very fine siliceous material filled cavities and crevices in the porous and laminated type of ore. Screenability tests with different moisture contents in the ore indicated that maximum screening difficulty would be encountered when the moisture content of the ore was 5 per cent.

Detailed washing tests indicated that a clean sized washed product ($+\frac{3}{8}$ in.) free from adherent ore fines, suitable for direct charging into the blast furnace and a $-\frac{3}{8}$ in. classifier sand product free from undesirable slime could be obtained for sinter production. Washing of the ore after crushing it separately to -3 in., -2 in. and -1 in. sizes resulted in (i) improvement of iron grade of the washed products by

4.55-5.17 per cent and (ii) a decrease in the total insoluble contents by 5.14-5.92 per cent. The rejections of SiO_2 and Al_2O_3 in the slime were 28.7-30.4 and 17.6-10.3 per cent respectively for iron losses of only 3.3-4.6 per cent of the total values.

When washing was followed by heavy media separation, there was considerable improvement in the grades of the sink product and an appreciable decrease in its total silica and alumina contents. The results of washing and heavy media separation after prior crushing the ore to different sizes are furnished in Figs. 1, 2 and 3.

It was found that washing alone of the ore could yield (i) a washed, sized ore suitable for blast furnace charge assaying Fe, 60.75-61.56; SiO_2 , 3.05-3.40; and Al_2O_3 , 4.46-5.53 per cent, with iron recoveries varying from 59.2 to 78.2 per cent of the total; (ii) a $-\frac{3}{8}$ in. classifier sand assaying Fe, 50.77-54.94; SiO_2 , 8.93-13.08; and Al_2O_3 , 7.20-8.51 per cent, with iron recoveries ranging from 18.5 to 36.2 per cent of the total therein; and (iii) a slime rejecting 28.7-30.4 per cent of the total silica and 17.6-20.3 per cent of the total alumina present in the ore with iron losses of 3.3-4.6 per cent of the total. The $+\frac{3}{8}$ in. washed products were of good grade and may be used as such for blast furnace burdening. The $-\frac{3}{8}$ in. classifier sand fractions which can be used in the blast furnace only after sintering were of low grade containing high SiO_2 and Al_2O_3 contents and would need to be beneficiated before sintering to achieve the well-established requisite metallurgical advantages in iron smelting derived through the use of sinters.

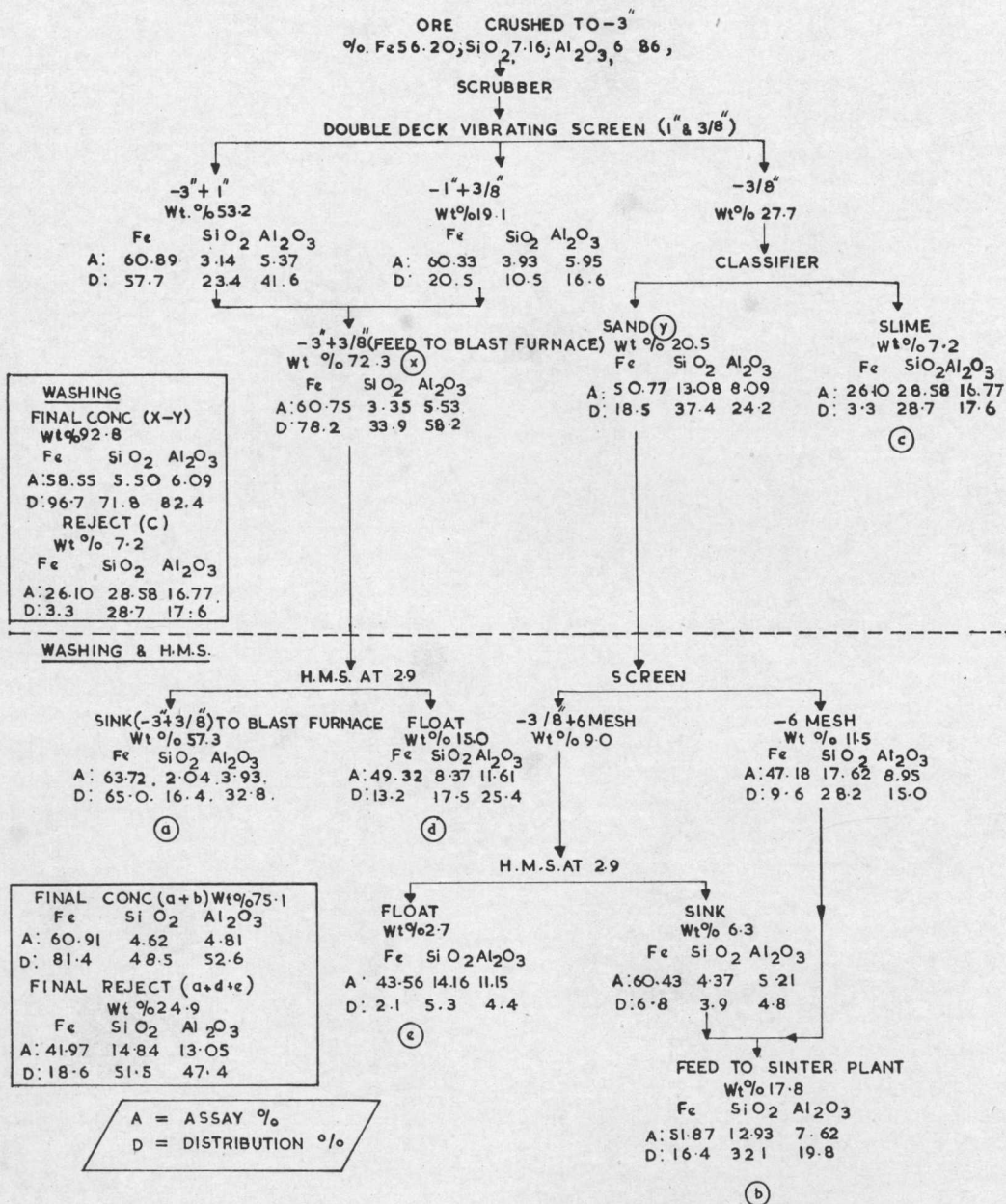


FIG. 1 — SUMMARY OF RESULTS OF BENEFICIATION OF RAJHARA IRON ORE AT -3 IN. SIZE

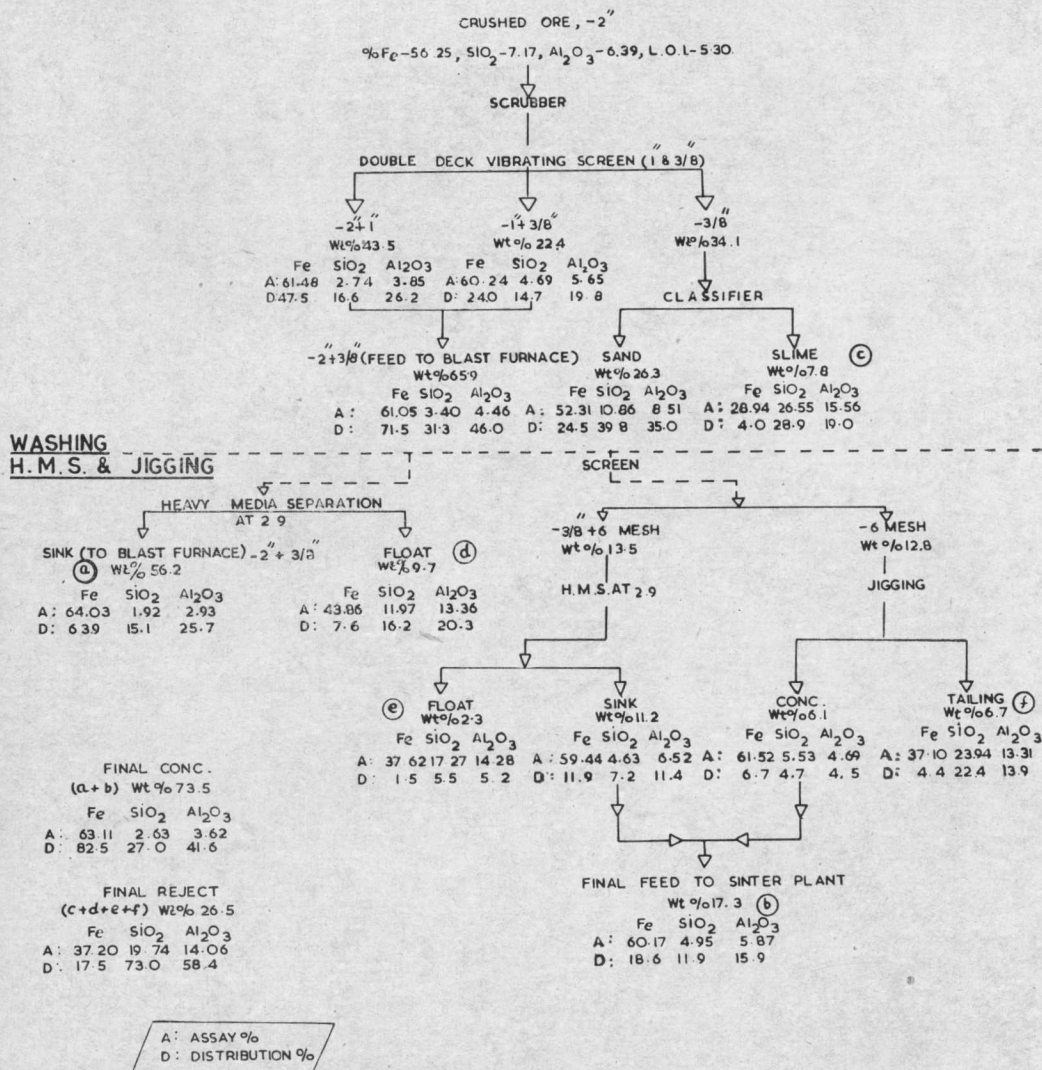
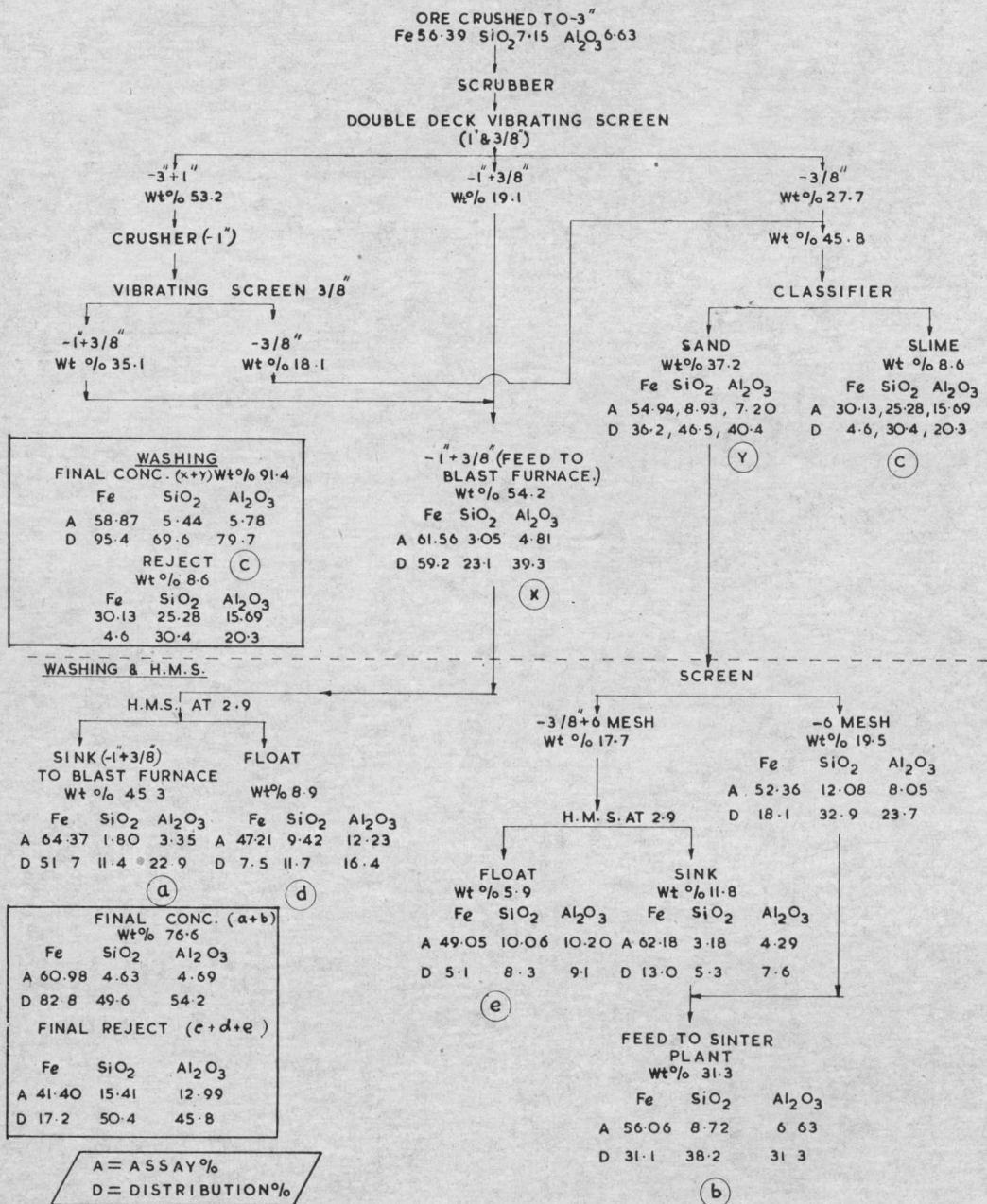


FIG. 2 — FLOWSHEET FOR THE BENEFICIATION OF RAJHARA IRON ORE AT -2 IN. SIZE

Heavy media separation could further reduce the insoluble contents of the $+ \frac{3}{8}$ in. washed products. It would be seen from the results summarized in the flowsheets that heavy media separation with sp. gr. of the medium at 2.9 produced sink products assaying Fe, 63.72-64.34; SiO₂, 1.80-2.04; and Al₂O₃, 2.93-3.93 per cent, with iron recoveries varying from 51.7 to 65.0

per cent of the total. Heavy media separation with sp. gr. of the medium at 2.9 of the $- \frac{3}{8}$ in. $+6$ mesh fraction of the classifier sand products produced concentrates assaying Fe, 59.44-62.18; SiO₂, 3.18-4.63; and Al₂O₃, 4.29-6.52 per cent, with iron recoveries of 6.8-13.0 per cent of the total in these products. The -6 mesh fraction of the classifier sand product was



of a low grade containing a major portion of the insoluble contents present in the classifier sand fraction. This -6 mesh fraction (obtained from washing at -2 in.) when subjected to jigging could produce a concentrate assaying Fe, 62.52; SiO_2 , 5.53; and Al_2O_3 , 4.69 per cent, with a recovery of 6.7 per cent Fe of the total in relation to 11.1 per cent Fe recovery obtained in the -6 mesh fraction; this jig concentrate when mixed with the heavy media concentrate (obtained from $-\frac{3}{8}$ in. $+6$ mesh fraction after washing at -2 in.) would together form the sinter feed constituting 17.3 per cent by weight of the total ore whilst assaying Fe, 60.17; SiO_2 , 4.95; and Al_2O_3 , 5.87 per cent.

Comprehensive studies on the sintering characteristics of (i) $-\frac{3}{8}$ in. classifier sand obtained from -2 in. washed ore and (ii) upgraded classifier sand, etc., were undertaken to determine the optimum conditions for producing good sinter. Optimum water content was found to be 7 per cent for fluxing and non-fluxing sinters from both classifier sand and upgraded fines. 4 and 4.5 per cent coke in the sinter feed with classifier sand and upgraded fines respectively produced fairly good fluxing sinters. Requirements of coke varied from 3.5 to 6 per cent as the basicity ratio varied from 1.0 to 2.5 in fluxing sinters. Good metallurgical quality sinter with a basicity ratio up to 2.5 could be produced by suitable additions of limestone and dolomite. Self-fluxing sinters showed a higher degree of oxidation than those of non-fluxing type.

It was observed that sinters made from fines obtained after ore washing only were of poor chemical composition though strong sinters could be produced. The upgraded fines, however, yielded sinters of good metallurgical grade and strength and also of fairly good chemical compositions.

Data regarding the economic evaluation of beneficiation of iron ore from Rajhara which was calculated on the following assumptions have been furnished in Table 1.

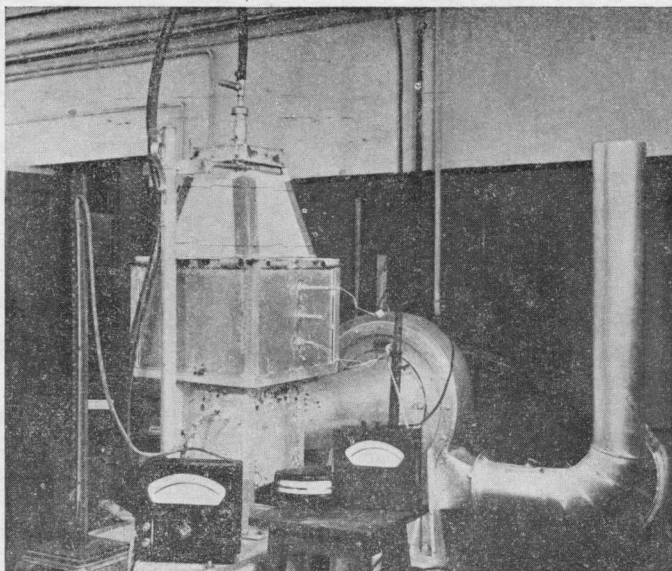


FIG. 4 — A VIEW OF THE SET-UP FOR SINTERING AS WELL AS HEAT-HARDENING OF IRON ORE PELLETS, DESIGNED AND FABRICATED AT THE NATIONAL METALLURGICAL LABORATORY

- (i) The cost of raw ore as mined was taken at Rs. 5.00 per ton. Transport charges for ore were not included.
- (ii) Beneficiation plant was assumed to operate with a capacity of 14,000 tonnes of run-of-mine ore per day.
- (iii) Capital cost of the plant did not include cost of mining equipment and township, but includes cost of machinery, erection, building, water and power supply to the plant.
- (iv) For every 1 per cent reduction in the alumina content of the ore achieved by beneficiation, the coke rate and flux rate would decrease by about 39 and 61 kg. respectively per ton of pig iron made. The increase in production of pig iron would be about 2.5 per cent. Due to these factors, the overall saving per ton of pig iron produced would be Rs. 4.00 after allowing for depreciation, etc.

Table 1 — Economic Evaluation of Various Processes Employed for the Beneficiation of Rajhara Iron Ore

	Crushing, dry screening and utilization of fines			Crushing, dry screening and rejection of — $\frac{3}{8}$ " size fines			Crushing, washing, wet screening, dewatering of fines (— $\frac{3}{8}$ ")			Crushing, washing, wet screening, concentration of only fines ($\frac{3}{8}$ ")			Crushing, washing, wet screening, concentration of both lumps and fines (— $\frac{3}{8}$ ")		
	Fe %	SiO ₂ %	Al ₂ O ₃ %	Fe %	SiO ₂ %	Al ₂ O ₃ %	Fe %	SiO ₂ %	Al ₂ O ₃ %	Fe %	SiO ₂ %	Al ₂ O ₃ %	Fe %	SiO ₂ %	Al ₂ O ₃ %
Analysis of final beneficiated iron ore	56.5	5.2	6.40	61.7	4.0	5.72	58.6	5.5	5.62	60.9	3.7	4.75	63.1	2.6	3.62
Yield, tonnes/day	(100%)			(70.7%)			(92.2%)			(83.2%)			(73.5%)		
Capital cost of the ore treatment plant	14,000			9,898			12,908			11,648			10,290		
Cost of treatment per tonne of beneficiated ore:	Rs. 2.5 crores			Rs. 2.50 crores			Rs. 3.36 crores			Rs. 4.50 crores			Rs. 5.40 crores		
(a) Capital cost	Rs. 0.894			Rs. 1.263			Rs. 1.301			Rs. 1.932			Rs. 2.625		
(b) Operating cost	Rs. 0.180			Rs. 0.255			Rs. 0.256			Rs. 0.434			Rs. 0.680		
(c) Cost of raw ore as mined	Rs. 5.000			Rs. 7.073			Rs. 5.424			Rs. 6.010			Rs. 6.803		
Total cost per tonne of prepared ore	Rs. 6.074			Rs. 8.591			Rs. 6.981			Rs. 8.376			Rs. 10.108		
Tonnes of ore required per tonne of pig iron	1.712			1.563			1.645			1.583			1.528		
Total cost of ore required per tonne of pig iron	Rs. 10.400			Rs. 13.430			Rs. 11.490			Rs. 13.260			Rs. 15.440		
Difference in alumina, per cent	—			0.67			0.77			1.64			2.77		
Expected saving in production cost per tonne of pig iron in blast furnace iron smelting due to decrease in alumina by beneficiation	—			3.35			3.85			8.20			13.85		

1.1 Pilot Plant Studies on Beneficiation and Sintering of Kiriburu Iron Ore for National Mineral Development Corporation

Further pilot plant investigations were continued on beneficiation of iron ore from Kiriburu, at the request of National Mineral Development Corporation, with a view to solving the practical difficulties of handling, crushing, screening, upgrading of the ore and the production of quality self-fluxing sinter from the ore fines as was reported in the 1961-62 Annual Report.

Since removal of gangue from an iron ore by physical methods wherever feasible is much cheaper as compared to slagging it off in the iron blast furnace, detailed study was undertaken on the possibilities of gangue removal by gravity methods subsequent to washing indispensable for eliminating handling and screening difficulties. Results obtained showed that heavy media separation and jigging of the washed products (lumpy ore as well as fines) could bring down the insolubles to low figures, but the extent to which such beneficiation is to be undertaken in practice will be governed by economics.

Goethite was the predominant mineral followed by hematite and lepidocrocite with traces of magnetite. A good amount of red ochery material was present as surface coating over the lumpy ore. This ochery material besides some kaolin present in pores and cavities contributed to the major part of the alumina in the sample. Very fine crystalline quartz was present in vugs enclosed by oxides of iron.

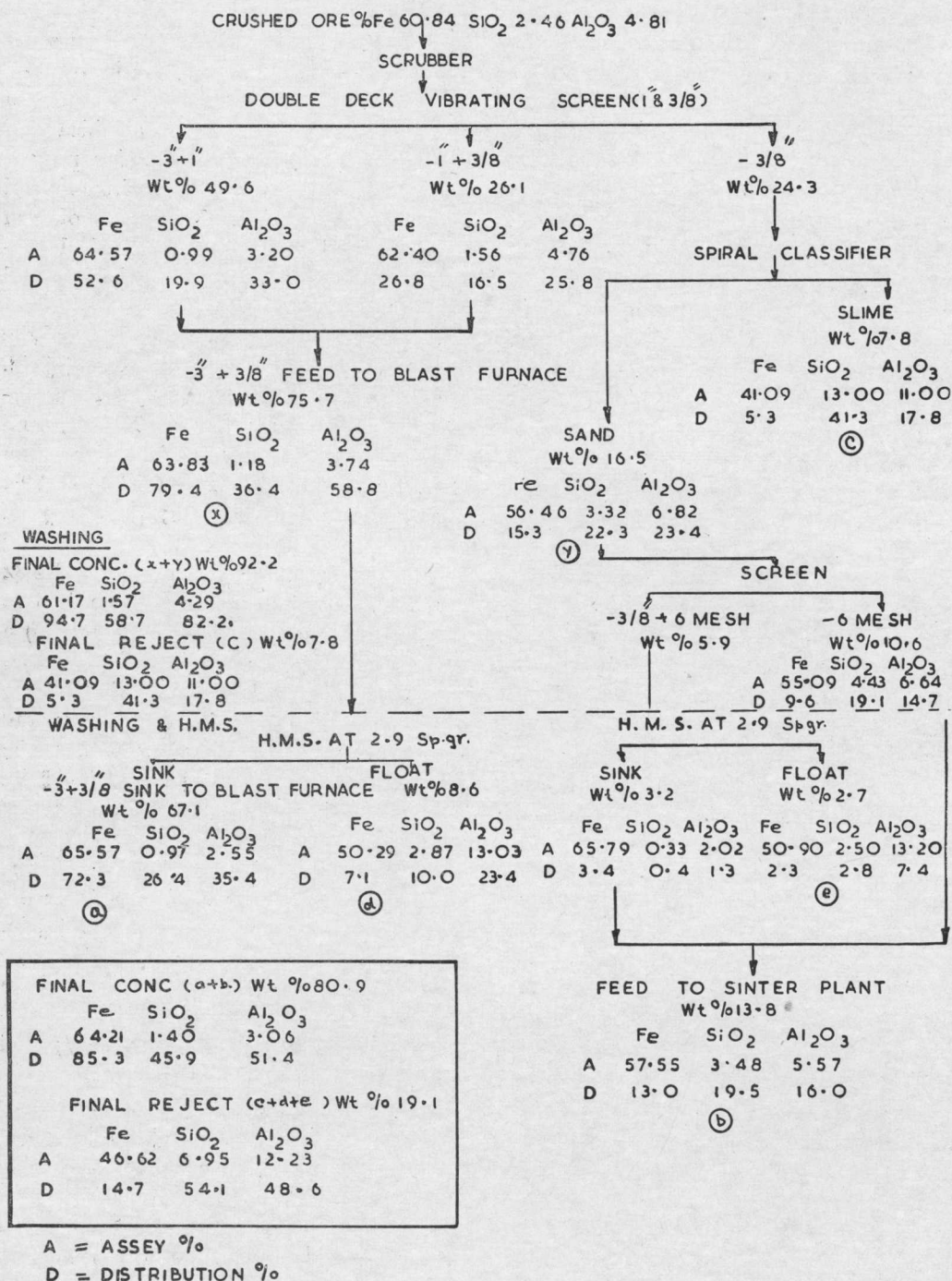
Washing tests on a 100-tonne representative sample of run-of-mine ore assaying Fe, 60.96; SiO_2 , 2.40; Al_2O_3 , 4.50; MgO , 0.15; CaO , 0.62; P, 0.08; and S, 0.03 per cent indicated that a clean sized washed product ($+\frac{3}{8}$ in.) free from adhered fines for direct charging to the blast furnace and $-\frac{3}{8}$ in. classifier sand product free from

undesirable slime for sinter manufacture could be obtained.

Washing of the ore after crushing it separately to -3 in., -2 in. and -1 in. resulted in the elimination through the slime of 41.3-50.6 per cent of the total silica and 17.8-23.6 per cent of total alumina present in the ore for a corresponding iron loss of only 5.3-6.3 per cent. The grade of iron also improved slightly in the washed products as a result of removal of silica and alumina.

It may be seen that washing alone of the ore could yield (i) a washed sized $+\frac{3}{8}$ in. ore suitable for blast furnace charge assaying Fe, 63.83-64.28; SiO_2 , 0.91-1.18; and Al_2O_3 , 3.24-3.74 per cent, with iron recoveries varying from 67.2 to 79.4 per cent, in these products; (ii) a $-\frac{3}{8}$ in. classifier sand assaying Fe, 56.46-59.07; SiO_2 , 2.40-3.67; and Al_2O_3 , 5.64-6.82 per cent, with iron recovery from 15.3 to 26.5 per cent in these products; and (iii) a slime rejecting 41.3-50.6 and 17.8-23.6 per cent of total silica and alumina respectively present in the ore with overall iron losses of 5.3-6.3 per cent. As the washed products both $+\frac{3}{8}$ in. and $-\frac{3}{8}$ in. were of good grades, further beneficiation thereof may not be necessary.

However, if further reduction of the alumina content is desired, heavy media separation can be employed with the $+\frac{3}{8}$ in. washed products. It can be also seen that heavy media separation at a medium sp. gr. of 2.9 produced sink products assaying Fe, 65.57-66.04; SiO_2 , 0.63-0.97; and Al_2O_3 , 2.0-2.62 per cent, with iron recoveries varying from 61.4 to 72.3 per cent. Heavy media separation of the $-\frac{3}{8}$ in. $+6$ mesh fraction of the $-\frac{3}{8}$ in. classifier sand product at medium sp. gr. of 2.9 produced concentrates assaying Fe, 62.90-66.07; SiO_2 , 0.33-1.07; and Al_2O_3 , 2.00-4.28 per cent, with iron recoveries of 3.4-6.3 per cent in these products. The -6 mesh fraction when mixed with the above heavy media concentrate ($-\frac{3}{8}$ in. $+6$ mesh) for sintering

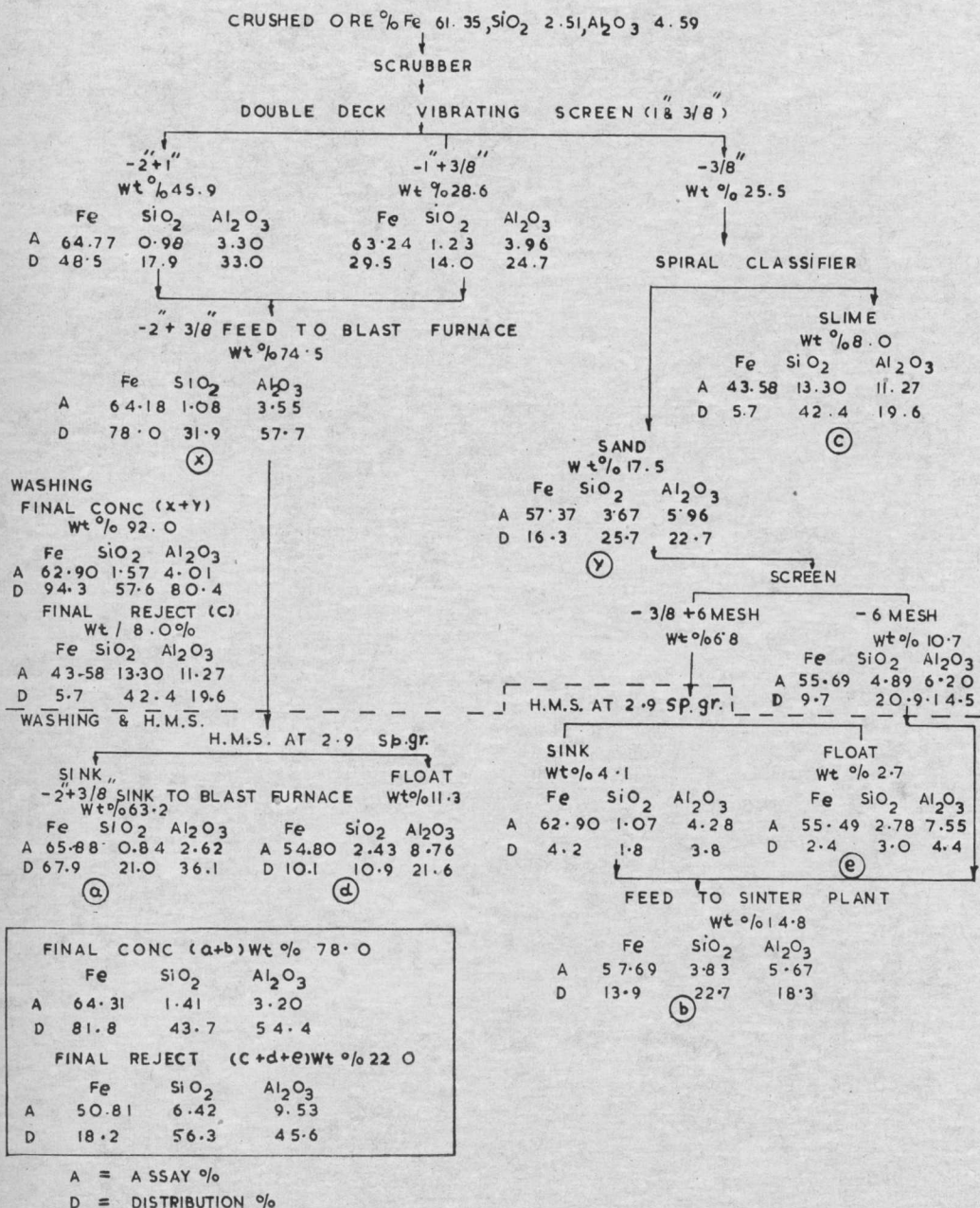


FINAL CONC (a+b) Wt % 80.9			
	Fe	SiO ₂	Al ₂ O ₃
A	64.21	1.40	3.06
D	85.3	45.9	51.4

FINAL REJECT (c+d+e) Wt % 19.1			
	Fe	SiO ₂	Al ₂ O ₃
A	46.62	6.95	12.23
D	14.7	54.1	48.6

A = ASSEY %
D = DISTRIBUTION %

FIG. 5 — SUMMARY OF RESULTS OF BENEFICIATION OF KIRIBURU IRON ORE AT -3 IN. SIZE



FINAL CONC (a+b) Wt % 78.0

	Fe	SiO ₂	Al ₂ O ₃
A	64.31	1.41	3.20
D	81.8	43.7	54.4

FINAL REJECT (c+d+e) Wt % 22.0

	Fe	SiO ₂	Al ₂ O ₃
A	50.81	6.42	9.53
D	18.2	56.3	45.6

FIG. 6 — SUMMARY OF RESULTS OF BENEFICIATION OF KIRIBURU IRON ORE AT -2 IN. SIZE

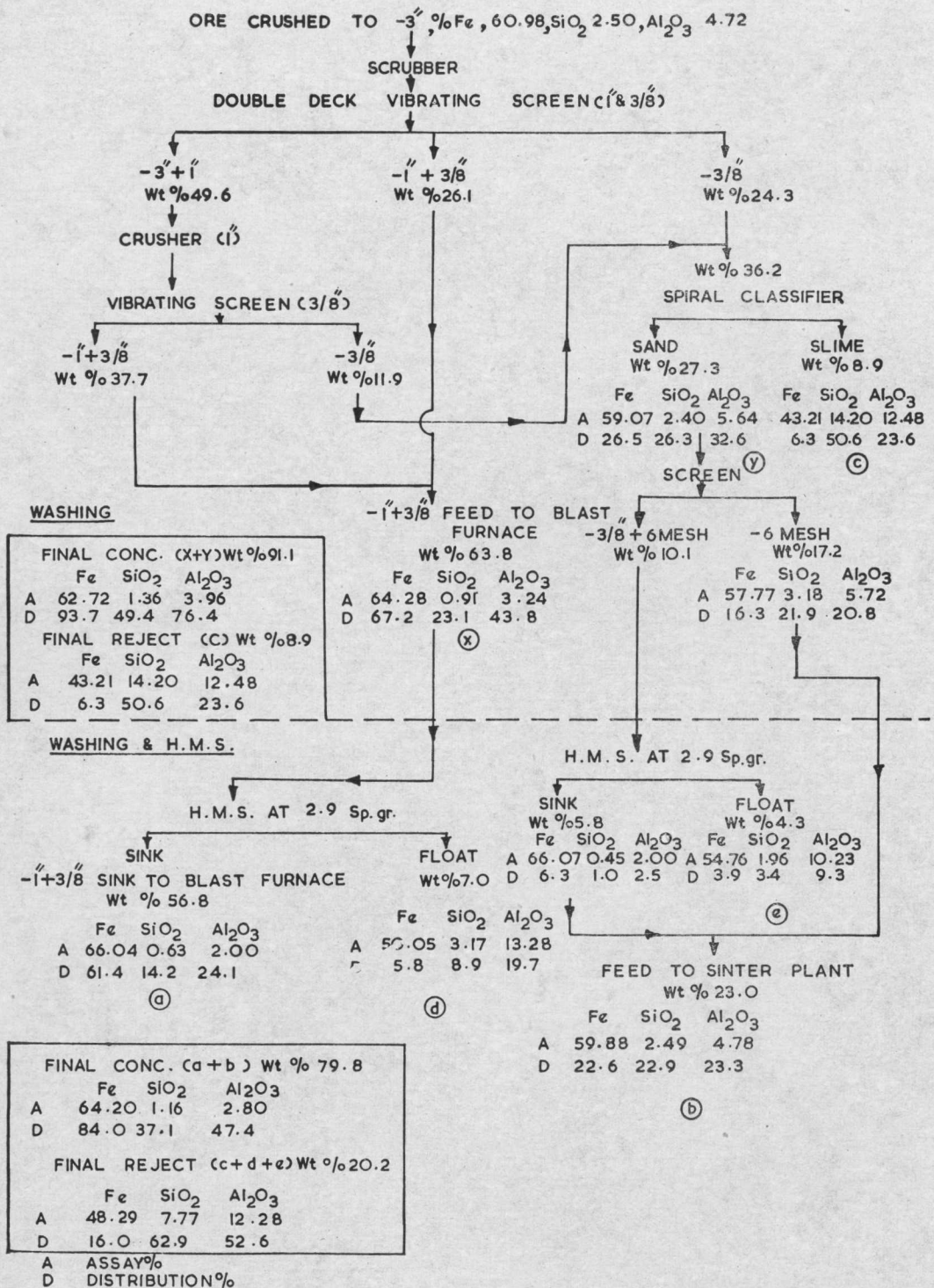


FIG. 7 — SUMMARY OF RESULTS OF BENEFICIATION OF KIRIBURU IRON ORE AT -1 IN. SIZE

would assay Fe, 57.55-59.88; SiO_2 , 2.49-3.83; and Al_2O_3 , 4.78-5.67 per cent. The total loss of iron in the floats at 2.9 and slime would be 14.7-18.2 per cent.

Alternatively, the -6 mesh fraction of the classifier sand could be subjected to jigging for further removal of alumina, yielding finally a sinter feed (consisting of heavy media concentrate - $\frac{3}{4}$ in. +6 mesh and -6 mesh jig concentrate) assaying Fe, 60.88; SiO_2 , 1.95; and Al_2O_3 , 4.54 per cent.

Sintering characteristics of the - $\frac{3}{8}$ in. classifier sand obtained after washing of -3 in. ore were studied. The optimum water content was 7 per cent for obtaining good fluxing and non-fluxing sinters and the coke content was found to be 3.3-5 per cent for the former and 4 per cent for the latter.

Good quality sinter with a basicity ratio up to 2.0 could be produced by suitable addition of limestone. Dolomite could also be added to increase MgO content of sinter without any deleterious effect in sinter quality. Self-fluxing sinters had higher degree of oxidation than those of the non-fluxing type.

The overall economics of beneficiation has also been evaluated taking into consideration the ultimate reduced cost of production of pig iron in the blast furnace by way of decreased coke rate and slag volume and increased production with the use of beneficiated iron ore.

Table 2 furnishes the economic evaluation of beneficiation of iron ore from Kiriburu which was calculated on the following assumptions:

- (i) The cost of raw ore as mined was taken at Rs. 5.00 per ton. Transport charges for ore were not included.
- (ii) Beneficiation plant was assumed to operate with a capacity of 14,000 tonnes of run-of-mine ore per day.
- (iii) Capital cost of the plant did not include cost of mining equipment and township, but includes cost of machinery, erection, building, water and power supply to the plant.

- (iv) For every 1 per cent reduction in the alumina content of the ore achieved by beneficiation, the coke rate and flux rate would decrease by 85 and 135 lb. respectively per ton of pig iron made. The increase in production of pig iron would be about 2.5 per cent. Due to these factors, the overall saving per ton of pig iron produced would be Rs. 4.00 after allowing for depreciation, etc.

Further samples of iron ore from the various mining benches in the two hills of Kiriburu mines have been received for conducting comprehensive beneficiation tests and to evaluate suitable flowsheets thereof depending upon the characteristics of the ore from the various localities. The work is under progress.

2.0 Pilot Plant Beneficiation

Studies on Limestone Sample from Tisco

Further pilot plant investigations were conducted with over 1000 tonnes of limestone sample received from the Tisco, with a view to reducing its silica content so that the beneficiated product can be advantageously and effectively utilized in steelmaking, and to formulate a suitable flowsheet for setting up a plant to treat 300 tonnes of low-grade limestone per day. The limestone sample assayed CaO , 46.7; SiO_2 , 7.1; and L.O.I., 38.2 per cent before upgrading. The sample was crushed to - $\frac{1}{2}$ in. size using jaw and cone crushers. The crushed sample was ground in a ball mill working in closed circuit with a rake classifier. The classifier overflow (80 per cent of -200 mesh) was conditioned with sodium silicate and an emulsion of oleic acid and sodium hydroxide. Rougher concentrate collected from eight cells was refloats in six smaller cells, whereas the tailing was floated in four scavenger cells

Table 2 — Economic Evaluation of Various Processes Employed for the Beneficiation of Kiriburu Iron Ore

	Crushing, dry screening and utilization of fines	Crushing, dry screening and rejection of $-\frac{1}{2}$ " size fines	Crushing, washing, wet screening, dewatering of fines ($-\frac{3}{8}$ ")	Crushing, washing, wet screening, concentration of only fines ($-\frac{3}{8}$ ")	Crushing, washing, wet screening, concentration of both lumps and fines ($-\frac{3}{8}$ ")
	Fe % SiO ₂ % Al ₂ O ₃ %	Fe % SiO ₂ % Al ₂ O ₃ %	Fe % SiO ₂ % Al ₂ O ₃ %	Fe % SiO ₂ % Al ₂ O ₃ %	Fe % SiO ₂ % Al ₂ O ₃ %
Analysis of final beneficiated iron ore	60.96 (100%)	62.45 (71.5%)	62.90 (92%)	63.78 (85.1%)	65.13 (73.8%)
Yield, tonnes/day	14,000	10,010	12,880	81,910	10,330
Capital cost of the ore treatment plant	Rs. 2.5 crores	Rs. 2.50 crores	Rs. 3.36 crores	Rs. 4.50 crores	Rs. 5.40 crores
Cost of treatment per ton of beneficiated ore:					
(a) Capital cost	Rs. 0.894	Rs. 1.249	Rs. 1.304	Rs. 1.889	Rs. 2.614
(b) Operating cost	Rs. 0.180	Rs. 0.252	Rs. 0.256	Rs. 0.424	Rs. 0.677
(c) Cost of raw ore as mined	Rs. 5.000	Rs. 6.993	Rs. 5.436	Rs. 5.876	Rs. 6.775
Total cost per ton of prepared ore	Rs. 6.074	Rs. 8.494	Rs. 6.996	Rs. 8.189	Rs. 10.066
Tons of ore required per ton of pig iron	1.581	1.544	1.532	1.512	1.480
Total cost of ore required per ton of pig iron	Rs. 9.607	Rs. 13.11	Rs. 10.73	Rs. 12.38	Rs. 14.89
Difference in alumina, per cent	—	0.4	0.49	0.83	1.60
Expected saving in production cost per ton of pig iron in blast furnace iron smelting due to decrease in alumina by beneficiation	—	Rs. 2.0	Rs. 2.45	Rs. 4.15	Rs. 8.00

producing a final tailing and a scavenger float for regrinding. The cleaner tailing was also recirculated in the circuit. The refloat concentrate assaying CaO , 50.3; L.O.I., 41.1; and SiO_2 , 2.0 per cent and with a CaO recovery of 94.7 per cent in it was thickened, filtered and dried. The dried concentrate was mixed with molasses and made into briquettes and pellets, a few hundred tons of which were supplied to Tisco. The full-scale steelmaking trials conducted in the 200-ton open-hearth furnaces of Tata Iron & Steel Works with limestone pellets have yielded highly promising results. The economics of beneficiation of limestone and agglomeration of the concentrate were also evaluated. Flow-sheet on pp. 149-150 has been employed for the pilot plant tests.

3.0 Batch and Pilot Plant Flotation Studies on Khetri Copper Ore

Batch and pilot plant flotation studies were undertaken on a 20-ton run-of-mine copper ore sample from Khetri, received from National Mineral Development Corporation. The ore sample assayed Cu , 0.8; Fe , 18.63; S , 3.08; SiO_2 , 54.65; Al_2O_3 , 9.62; MgO , 4.54 per cent; 2.37 dwt. Ag/ton ; 0.13 dwt. Au/ton and was essentially a garnetiferous quartz-chlorite schist. Quartz was the major transparent gangue followed by chlorite and garnet. The transparent gangue was liberated mostly at 65 mesh size. Pyrrhotite (6.7 per cent) and magnetite (5.6 per cent) were the principal opaque gangue minerals followed by small amounts of pyrite and marcasite (0.56-1.0 per cent) and traces of pentlandite. Chalcopyrite (2.0-2.3 per cent) was finely disseminated and was mostly free at about 150 mesh size. The very fine chalcopyrite-silicate interlocking, even though quite small in quantity, was expected to result in the loss of some copper values in the tailing.

Batch flotation studies employing potassium ethylxanthate as collector for chalco-

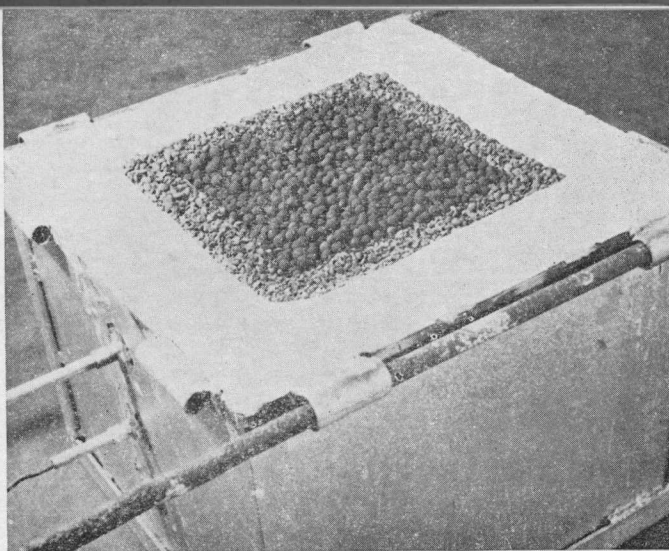


FIG. 8 — A VIEW OF THE FURNACE FOR HEAT-HARDENING OF IRON ORE PELLETS SHOWING THE FILLING ARRANGEMENT

pyrite and lime as a depressant for other sulphides indicated that with a coarse grind (51.9 per cent of -200 mesh) the tailing obtained assayed 0.16 per cent Cu which could be decreased to only 0.11 per cent using higher xanthates and high-pulp densities for flotation. Considering the cost of

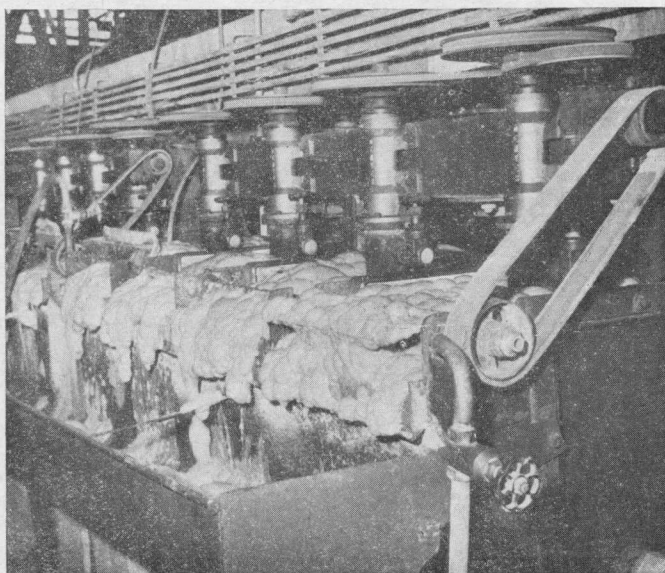


FIG. 9 — A VIEW OF THE FLOTATION CELLS IN MINERAL BENEFICIATION PILOT PLANT SHOWING THE LIMESTONE CONCENTRATE

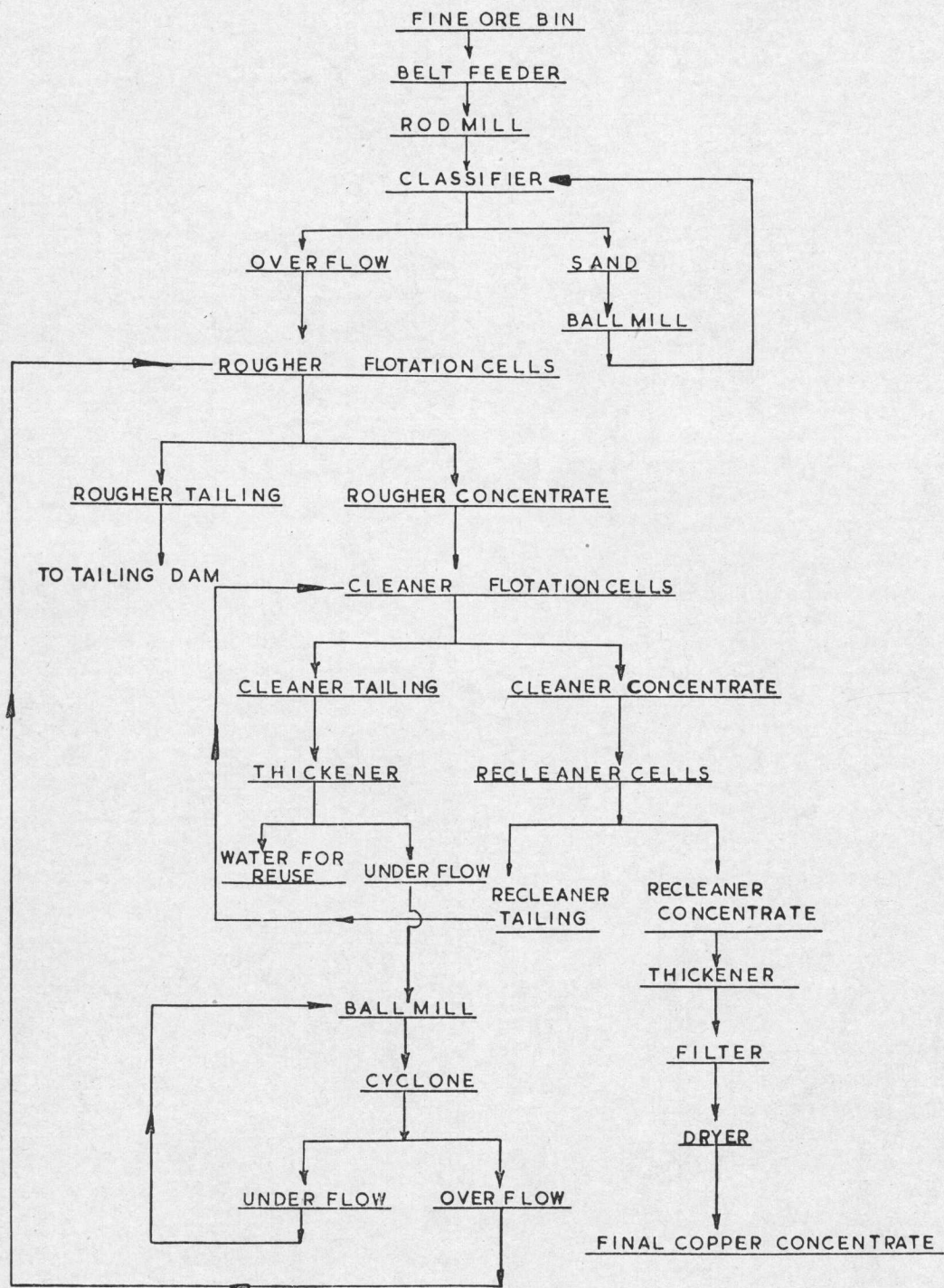


FIG. 10 — A GENERAL FLOWSHEET FOR THE FLOTATION OF KHETRI COPPER ORE

grinding and loss of copper values, a grind equivalent to 62.5-73.3 per cent -200 mesh was considered optimum. Two cleanings at higher pH values of 9.8-10 and 10.10.2 of the rougher float obtained at a pH of about 9 were found to yield copper concentrates assaying 25.1 per cent Cu and 24.6 per cent Cu with recoveries of 77.5 and 78.9 per cent Cu respectively. The loss of copper in the two cleaner and recleaner tailings was 11.3 and 13.0 per cent respectively. Recirculation of these tailings in practice after grinding was expected to yield better recoveries. It was found that by regrinding these tailings to -200 mesh and refloatation, only 3.9 and 2.8 per cent extra copper could be recovered giving final Cu recoveries of 81.4 and 81.7 per cent for concentrate grades of 22.0 and 24.5 per cent Cu respectively.

Regarding recovery of sulphur in the form of a pyrite-pyrrhotite concentrate, it was found that there was only 0.5-1 per cent pyrite and 6.7 per cent pyrrhotite in the sample. As such, the final pyrrhotite-pyrite concentrate could not be expected to be of a very high grade. Flotation performed with the copper tailings after acidification to a pH of 5.5 and employing potassium amylxanthate yielded a final pyrrhotite-pyrite concentrate weighing 5.6 per cent and assaying only 30 per cent S.

Continuous flotation studies more or less confirmed the results obtained by batch flotation. Flotation of the sample assaying 0.8 per cent Cu employing a grind of 64.5 per cent -200 mesh followed by two cleanings and recirculation of the middlings to the flotation circuit after regrinding yielded concentrates assaying 19.27-06 per cent Cu with recoveries ranging from 88 to 77 per cent. Regrinding of the entire rougher float was considered unnecessary.

It was found that all the concentrates contained the same precious metal content of 1.6 dwt. Au/ton and 22.8 dwt. Ag/ton with recoveries of about 30.0 and 25 per cent gold and silver respectively.

The comparatively low-copper recoveries in the concentrates were attributed to the loss in the tailings of (i) very fine chalcopyrite interlocked with the silicate gangue, and (ii) very fine chalcopyrite.

A general flowsheet drawn on the basis of experimental results obtained both on a batch as well pilot plant scale is given in Fig. 10 and can form a broad basis for the setting up of a flotation plant for the treatment of Khetri copper ore.

4.0 Beneficiation Studies on Lateritoid Manganese Ore from Malaya

Further studies on lateritoid manganese ore received from the Director, Eastern Mining & Metal Co. Ltd., Malaya, indicated that reduction roast treatment followed by wet magnetic separation at -28 mesh of the washed ore could yield a non-magnetic manganese concentrate assaying Mn, 48.3; Fe, 4.1; SiO₂, 7.3; Al₂O₃, 9; BaO, 9.7; and P, 0.049 per cent, with a recovery of 67.2 per cent Mn. Based on experimental results obtained, two flowsheets have been developed for the treatment of manganese ore from Malaya. The work has been completed and the report has been furnished to the Eastern Mining & Metal Co. Ltd., Malaya, embodying the results and recommendations made thereon.

5.0 Batch and Pilot Plant Beneficiation Studies on Low-grade Fluorspar from Rajasthan

A 50-ton representative low-grade run-of-mine fluorspar sample assaying CaF₂, 21.2; CaCO₃, 6.6; and SiO₂, 67.1 per cent, with traces of Pb, S, Zn and Ba was received from the Director of Mines and Geology, Government of Rajasthan, for pilot plant beneficiation and agglomeration studies. Fluorite in the sample was predominantly of greenish variety, followed by violet and

colourless varieties. Quartz, felspar and calcite constituted the gangue. Microscopic examination of the various sieve fractions of -7 mesh representative sample indicated that fluor spar was fairly free from the gangue at about 100 mesh size. Batch flotation tests, performed using an emulsion of oleic acid and NaOH as collector for fluor spar and sodium silicate and/or *katha*, indicated that a good grade of CaF_2 concentrate could be produced from the sample. Based on the batch tests, continuous pilot plant run was undertaken employing different combinations of sodium silicate and *katha* as depressant for gangue minerals and oleic acid emulsion as fluorite collector. The results indicated that use of *katha* alone as gangue depressant, during roughing and cleaning stages, could yield two concentrates, one suitable for acid manufacture and the other for metallurgical use. The acid grade product assayed CaF_2 , 97.3; SiO_2 , 1.1; and CaCO_3 , 1.2 per cent, with a recovery of 30.8 per cent CaF_2 , and the product of metallurgical grade purity assayed 83.1 per cent CaF_2 and represented an additional recovery of 48.7 per cent CaF_2 . The total recovery in both the products amounted to 79.5 per cent CaF_2 which should be considered satisfactory for a low-grade ore of this type containing high percentage of calcite. If only a concentrate of metallurgical grade purity is aimed at from the sample, two cleanings of rougher float would be sufficient and the recovery of fluorite in the concentrate also would be much higher. Soft water is also essential for low-reagent consumption during flotation. The work has been completed and a report has since been furnished to the Director of Mines and Geology, Rajasthan.

5.1 Beneficiation of Fluorspar from Ambadonagar, Baroda

A low-grade fluor spar sample weighing about a ton was received from Geological

Survey of India, Maharashtra-Gujarat Circle, Poona. It assayed CaF_2 , 34.07; SiO_2 , 56.0; CaCO_3 , 1.70; Al_2O_3 , 1.72; S, 0.30; Fe_2O_3 , 3.3; and L.O.I., 2.0 per cent. After preliminary crushing and grinding, a few flotation tests were performed employing feeds of different fineness ranging from 76 to 96 per cent -200 mesh size for flotation. Further studies are in progress.

6.0 Pre-concentration of Lead Zinc Ore from Zawar Employing Heavy-Media Separation

A sample of run-of-mine lead-zinc ore from Zawar Mine assaying Pb, 1.96; Zn, 4.34; CaO, 16.51; MgO, 11.99; SiO_2 , 28.45; Al_2O_3 , 1.83; and S, 2.81 per cent was received from the Metal Corporation of India Ltd. for pre-concentration using heavy media separation. Heavy media separation of the ore at sp. gr. 2.7, using galena as medium, rejected 34.5 per cent by weight of the material in the float with a loss of 10.03 per cent Zn and no loss of lead. The tests confirmed the results of earlier investigation at the National Metallurgical Laboratory that heavy media separation could reject about $\frac{1}{3}$ by weight of the material in the form of float with no loss of lead. Sphalerite being finely disseminated in the ore, some loss of zinc was inevitable irrespective of the size at which heavy media separation was carried out. The work has been completed and a report issued to the Metal Corporation of India Ltd.

7.0 Beneficiation of Lime-sludge from Titagurh Paper Mills Ltd.

A sample of lime-sludge obtained as a waste product from the Titagurh Paper Mills Ltd. was received from Messrs Bird & Co. (Pvt.) Ltd., Calcutta, for concentration studies so that the concentrated product could be utilized in the steel or other industries. The sample as received

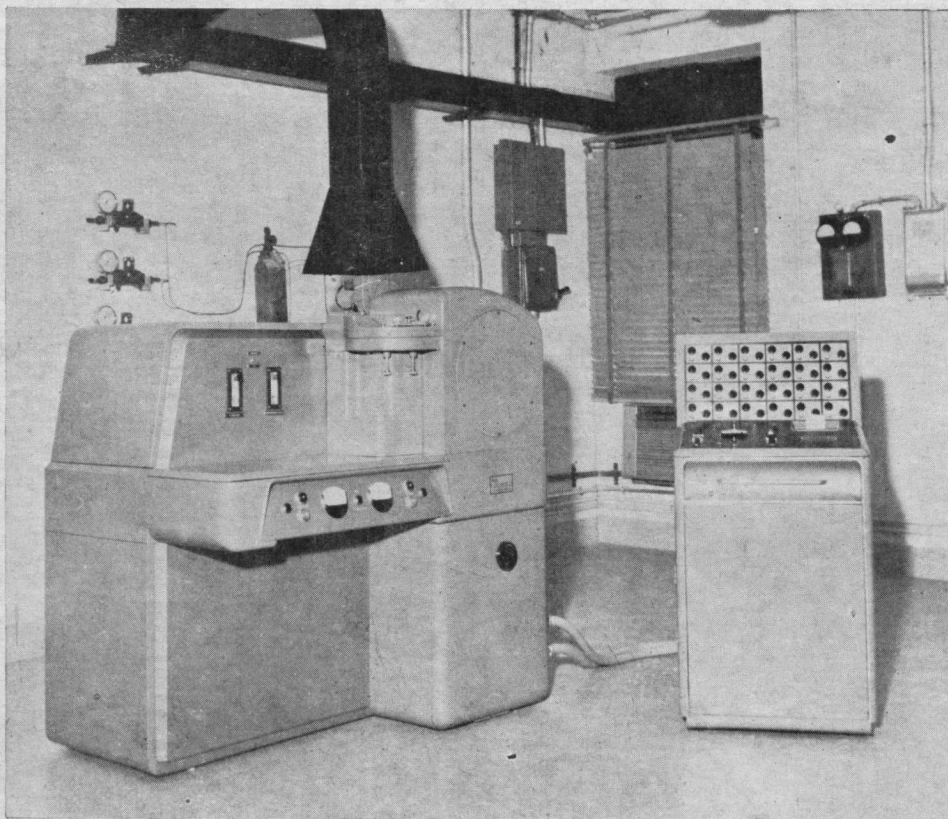


FIG. 11 — 'NORELCO' AUTROMETER FOR X-RAY FLUORESCENCE ANALYSIS, INSTALLED AT THE NATIONAL METALLURGICAL LABORATORY

assayed CaO , 44.1; MgO , 2.8; SiO_2 , 11.5; Al_2O_3 , 2.5; Fe_2O_3 , 0.9; CO_2 , 31.6; and Na_2O , 1.3 per cent. Calcite was the predominant carbonate mineral in the sample followed by minor amount of dolomite. The gangue minerals were quartz, diatomite and feldspars. Free lime and soda were also present in the sample. Flotation performed with the original sample as well as after grinding it to different periods did not yield satisfactory results. Flotation after grinding and removal of soluble alkalis also did not appreciably improve the grade. Intimate coating of precipitated carbonates over most of the gangue grains prevented selective flotation of carbonates from the siliceous gangue.

8.0 Flotation of Gypsum from Bikaner

A sample of gypsum, weighing about two tons, from Bikaner was received from Bikaner Gypsums Ltd., Rajasthan, for flotation studies. The sample as received assayed SO_3 , 32.98; CaO , 31.77; CO_2 , 7.68; SiO_2 , 7.74; Al_2O_3 , 2.84; Fe_2O_3 , 0.42; MgO , 1.58; L.O.I., 22.88; and 15.10 per cent combined water. Calcite and quartz were found to be the main gangue minerals in the sample which could be made free from gypsum at 100 mesh size. A batch flotation test using oleic acid and sodium hydroxide emulsion yielded after two cleanings a concentrate assaying 38.92 per cent SO_3 .

only with a recovery of 71.7 per cent SO_3 . Rougher flotation followed by six cleanings using sodium silicate and tannin extract as depressant for gangue yielded a final concentrate of 45.4 per cent SO_3 with a recovery of 41.0 per cent SO_3 .

9.0 Beneficiation of Dolomite from Tisco

A sample of dolomite, weighing about 100 tons, was received from Tisco for beneficiation studies on a pilot plant scale. Before taking up the pilot plant studies, a representative sample of the ore was taken to conduct laboratory scale experiments. The dolomite sample as received assayed CaO , 28.6; MgO , 20.0; insolubles, 6.8; and SiO_2 , 4.22 per cent. A number of flotation tests was performed with feeds ranging from 55 to 95 per cent -200 mesh fineness and the test using 82 per cent -200 mesh fineness gave the best results. The rougher dolomite float assayed 2.8 per cent insolubles and represented a percentage weight of 92.5 with respect to the original sample. Flotation experiments varying the quantity of sodium silicate indicated that 1.5 lb./ton was the optimum for the sample. Single cleaning of the rougher float yielded a refloat concentrate assaying 1.74 per cent insolubles and representing 81 per cent by weight of the original sample. Further tests using different collectors are in progress.

10.0 Studies on the Morphology of Phosphorus in Indian Manganese Ore

Under the Council of Scientific & Industrial Research Fellowship Scheme, further studies were continued on the morphology of phosphorus in Indian manganese ores. Detailed microscopic and petrological studies were carried out on manganese ore samples from Rajasthan, Maharashtra and Madhya Pradesh. Nearly 150 polished sections were studied under the ore-microscope and

whenever necessary etch tests, reflectivity measurements and X-ray studies were carried out. X-ray powder photographic studies and petrological studies had shown that the manganese ore from Maharashtra and Madhya Pradesh contain braunite as the most important and abundant manganese mineral with a good amount of jacobite; pyrolusite, psilomelane, hausmannite, sitaparite and hollandite were present in small quantities. But in the ore from Dongribuzurg, pyrolusite and psilomelane (cryptomelane) were seen to be in abundance compared to jacobite and braunite, indicating that they are low-temperature colloidal ores. In the case of Tirodi sample, hollandite and sitaparite were seen in abundance along with braunite. Most of the ores showed hematite as one of the important opaque gangue mineral associated with the manganese ores from Maharashtra and Madhya Pradesh. Goethite was seen only in the Rajasthan samples. Quartz, garnet, mica, rhodonite, feldspar, barite and apatite were observed to be the transparent gangue minerals present with the manganese ores.

Some samples indicated the presence of spessartite garnet in good amount. Garnets were separated from the ore in as pure a form as possible and were analysed for their Mn, Fe, SiO_2 and P content. The chemical analysis of five garnet samples have shown that the Mn content was between 29.02 and 37.16 per cent, Fe content was 5.02-9.01 per cent, SiO_2 varied from 31.61 to 46.66 per cent and phosphorus content varied from 0.11 to 0.62 per cent. The results indicated that garnet acts as the carrier of phosphorus and removal of these garnets will help in reducing the phosphorus content to some extent.

Though the Rajasthan samples showed an increase in the phosphorus with increase of iron, the samples from Maharashtra and Madhya Pradesh gave no such indication. Chemical analysis of the isodynamic separation products had shown that the phosphorus was maximum in the non-magnetic

fraction, which indicated that the phosphorus was mostly present as a non-magnetic mineral. Tests carried out in the isodynamic separation unit had shown that a good manganese concentrate with low-phosphorus content could be obtained as a magnetic concentrate with rejection of phosphorus in the non-magnetic tailing. Contact print studies were also carried out on polished ore sections and a few grain mounts. It was observed that in almost all the cases, phosphorus was uniformly distributed in the ore. Apart from apatite, no other mineral-carrying phosphorus could be identified. But it had been definitely established that the bulk of the phosphorus is present in the form of apatite and probably a small quantity of phosphorus is present in solid solution with iron and manganese minerals.

11.0 Flotation of Ferruginous Manganese Ore

Under the Council of Scientific & Industrial Research Fellowship Scheme, the project was continued on the separation of manganese minerals by flotation techniques.

Systematic studies were undertaken to determine the flotation behaviour of pure hematite and pyrolusite. For this purpose, large amounts of the two minerals were purified. A few tests were conducted in the Halimond tube, but pyrolusite produced lot of slimes, thus making it too difficult to observe the flotation. Hence, flotation experiments were carried out employing a sintered glass funnel which was similar to the Leaf & Knoll type cell. Effect of various reagents on the flotation behaviour of pyrolusite and hematite are furnished below.

Oleic Acid — Experiments were conducted varying the oleic acid concentration from 0.5 to 10.0 mg./100 cc. Both hematite and pyrolusite were found floating in the pH range of 2.3-11.9 studied. Oleic acid is a very good collector for both the minerals in the alkaline and acid circuits.

Dextrin — With 1 and 2 mg./100 cc. dextrin concentration both the minerals were found floating in the entire pH range studied. A 3 mg. dextrin/100 cc. limited the lower pH value to 3.6 and 3.7 for hematite and pyrolusite with 5 and 7.5 mg. dextrin/100 cc., lower limit being the same, the higher limit was found to be 9.0 for pyrolusite and 8.2 for hematite. But tests with 1:1 synthetic mixture of pyrolusite and hematite at pH 8.8 failed to give a clean separation.

Tannic Acid — Experiments with tannic acid concentration of 1, 2, 3, 5 and 10 mg./100 cc. were completed. From these experiments, it was observed that pyrolusite floated in the slightly acidic to highly alkaline range and hematite in the acid range only. With 1, 2 and 3 mg. tannic acid/100 cc. concentration, the pH limit being 6.8, the lower pH shifted from 3.2 to 5.2. With 5 and 10 mg. tannic acid/100 cc. concentration, the range of flotation of hematite was found to be 4.8-6.0 pH. In case of pyrolusite, the optimum ranges for good flotation were found to be pH 5.0-10.9, 3.9-10.1, 3.9-10.1, 4.7-10.2 and 6.0-9.6 for 1, 2, 3, 5 and 10 mg. tannic acid/100 cc. concentration respectively. Tests with 1:1 synthetic mixture of pyrolusite and hematite in the pH range of 7.5-10.0 with different concentrations of tannic acid failed to give a clean separation of the two minerals. The causes for this flotation behaviour are being investigated.

12.0 Studies on Electrostatic Separation of Garnetiferous Manganese Ores

Under the Council of Scientific & Industrial Research Fellowship Scheme, studies were initiated on the electrostatic separation of garnetiferous manganese ores.

In the garnetiferous manganese ores of India, garnet is the main gangue mineral which possesses almost the same specific

gravity and magnetic susceptibility as rich manganese-bearing minerals. Hence gravity concentration and magnetic separation method cannot separate the gangue satisfactorily. On the other hand, electrostatic separation method can effect a good separation between the manganese ore minerals and garnets as the former have better electric conductivity than the latter. Quartz, if present, can also be eliminated from the manganese ore minerals as a non-conducting fraction along with garnets.

The scheme involves the following steps:

- (i) Collection of literature on electrostatic separation, and
- (ii) Standardization of the electrostatic separation unit with rutile and zircon,
- (iii) Preparation of pure samples of garnet and manganese minerals, and
- (iv) Experimentation with the pure samples and application of results to natural ore samples.

Pure samples of rutile and zircon of different mesh sizes were prepared. A number of experiments were conducted to standardize the 'Kipp-Kelly' electrostatic separation unit with rutile and zircon. During the course of experiments, it was found that small amounts of rutile collected at the conducting side of the box by the conductive electric field. This is contrary to the accepted notion that rutile is a good conductor of electricity. Further studies are needed to throw more light on this observation.

While standardizing the Kipp-Kelly sutton type electrostatic separator and employing rutile and zircon of Travancore beach sands, the following conclusions were drawn:

- (i) Higher voltage was necessary for coarser particles than the finer ones to acquire better electrical charge,
- (ii) The material acquired better charge when the distance between the needles and the roll was less,
- (iii) At low speeds of the roll (i.e. retention time was high), the material was

pinned to the roll and passed to the non-conducting side,

- (iv) Rutile picked up reversal charge at speeds below 250 r.p.m. in convective electrical field, and
- (v) Temperature had irreversible effect on electrostatic separation. Further work is in progress.

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

The pilot submerged arc furnace was installed for developing the technical know-how for the production of diverse range of ferro-alloys and for the formulation of the electro-smelting technique for the indigenous ores. The furnace has a production capacity of 1-3 tons of ferro-alloys per day depending on the type of alloy produced. Raw materials will be accepted on contract for researches on their amenability to electro-thermal reduction techniques. A full report giving recommendations on smelting practice, power consumption and size of installation required for a given output will be made based on these smelting trials.

During the period under review, two campaigns to study the production of high-carbon ferro-chrome from Orissa chromite ores were taken up.

In the first campaign, high-carbon ferro-chrome, conforming to Indian Standards Specifications (No. 1170 of 1957) was successfully produced. A typical burden consisted of:

Chromite ore . . .	100 kg.
Hard coke . . .	36 kg.
Quartzite . . .	6 kg.

The chromite ore analysed Cr_2O_3 , 53; FeO , 11; and Fe_2O_3 , 5.8 per cent. Hard coke analysed 75 per cent fixed carbon and 21 per cent ash. Quartzite contained 97.8 per cent SiO_2 .

All the raw materials were crushed to a size range of $\frac{1}{4}$ to $\frac{3}{4}$ in. With the above

charge, the furnace operation was smooth and the slag was fluid. The charge level was built up to about half the furnace crucible height and the arc was submerged. The furnace was oscillated through 60° to break the crust formation and to connect the metal pools beneath the electrodes. The furnace was operated on 75 volts at 350 kW. on an average, on automatic controls. Tappings were made at regular intervals of 4-5 hr. and the metal and slag were cast in a cast iron ladle. Metal was separated after cooling and crushed and graded after analysis.

In the first campaign, it was found that silicon could not be effectively controlled and foundry grade high-carbon ferro-chrome was mostly produced. In order to produce low-silicon ferro-chrome, excess of reducing agent over the stoichiometric quantity required was decreased in stages. Also, the ore fines produced during crushing ($-\frac{1}{4}$ in.) were added to the extent of 10 per cent of the ore and no appreciable difficulty in operation was noticed. About 20 tonnes of high-carbon ferro-chrome were produced during this campaign.

Towards the end of the campaign, smelting trials with briquettes of beneficiated Salem magnetite were undertaken. About 4 tonnes of briquettes were charged producing about 2 tonnes of pig iron. It was observed during this short trial that the briquetted Salem magnetite was amenable to electric furnace smelting. For this trial, 'Kolsit', a low-temperature carbonization product, developed by the Regional Research Laboratory, Hyderabad, was used successfully as the reducing agent.

The second campaign was mainly directed to study the relation between silicon pick-up of high-carbon ferro-chrome and (i) total reductant in the charge, (ii) operating voltage, (iii) charging rate. The silicon control was much better in this campaign. A total of 7.6 tonnes of ferro-chrome was produced in a short time of 8 days.

13.1 Electric Smelting of Salem Magnetite in Pilot Submerged Arc Smelting Furnace

The object of this project was to investigate the behaviour of Salem magnetite concentrate briquettes and Neyveli lignite high-temperature carbonized coke. Smelting trials were conducted in the submerged arc electric furnace pilot plant of the National Metallurgical Laboratory. The beneficiation of Salem magnetite had already been investigated at the National Metallurgical Laboratory on pilot plant scales. The briquetting of beneficiated Salem magnetite concentrate was done as a mix comprising 88 per cent concentrate, 6 per cent molasses, 4 per cent lime and 2 per cent moisture. The briquettes were dried before smelting. High-temperature carbonized lignite coke was supplied as 2 in. size briquettes by the Department of Industries, Labour and Cooperation, Government of Madras.

The burden employed was made up of 100 kg. briquettes, 35 kg. lignite coke and 24 kg. limestone. The furnace was operated at a secondary voltage of 50 volts and power input was in the range of 150-250 kW. The slag basicity $(\text{CaO} + \text{MgO})/\text{SiO}_2 + \text{Al}_2\text{O}_3$ was maintained at about 1.075. The slag was free flowing with good fluidity. Good slag-metal separation was obtained. The pig iron produced was of excellent grade and analysed as follows:

	<i>Per cent</i>
C . . .	3.2-3.6
Si . . .	1.03-1.59
Mn . . .	0.06
S . . .	0.06-0.09
P . . .	0.09

The slag analysed as follows:

	<i>Per cent</i>
CaO . . .	43.53
SiO ₂ . . .	39.16
Al ₂ O ₃ . . .	6.5
FeO . . .	2.8
S . . .	0.51

It would be observed that the pig iron was exceptionally low in phosphorus and equally so in sulphur which would fetch high premium for use in foundry industry particularly for the production of malleable iron by virtue of its exceptionally low-phosphorus content which normally ranges in Indian foundry pig iron at 0.3-0.4 per cent. The high purity of the pig iron smelted from Salem magnetite concentrate would eliminate the necessity of pre-refining Indian pig irons for use as base iron for malleable iron production.

The magnetite briquettes did not show any tendency either towards crumbling or sintering in the furnace. The furnace operation was exceedingly smooth. The lignite carbonized coke briquettes were added almost directly beneath the electrodes.

14.0 Smelting of Ilmenite for Removal of Iron

This work, which was earlier taken up with the idea of producing a pig iron and titania-rich slag by smelting ilmenite with a carbonaceous reducing agent and suitable fluxes, was continued using the 50 lb. direct arc furnace lined with magnesite. The pig iron so produced will contain most of the chromium and vanadium present in the ilmenite and the slag will be practically free from iron and other harmful impurities. A total of 19 heats were conducted during the period under review. The ilmenite analysed TiO_2 , 63.1; FeO , 11.24; Fe_2O_3 , 23.21; Cr_2O_3 , 0.28; V_2O_5 , 0.25; MnO , 0.44; and SiO_2 , 0.64 per cent. The coke used in the experiments analysed fixed carbon, 74.76; V.M., 2.1; moisture, 1; and ash, 21.64 per cent; coke breeze analysed 61.3 per cent fixed carbon and 33.3 per cent ash. The magnesite used as flux contained MgO , 47.37; loss on ignition, 49.82; moisture, 0.30; SiO_2 , 1.6; Al_2O_3 , 0.5; Fe_2O_3 , 0.32; and CaO , 1.01 per cent. Dead-burnt magnesite was used as the lining material

for most of the heats; magnesite brick-lined shell was also used for some heats.

The main variables in the smelting trials were the amount of coke or coke breeze and magnesite. Some fluxless smelting trials were also conducted. In order to arrive at a proper evaluation of the material balance, the same proportion of ilmenite, coke breeze and magnesite were used in successive heats. Smelting of pre-reduced material was also carried out. The mixture of ilmenite and fluxes was melted and the reductant was added in stages. The reduction was carried out for at least 1 hr. after addition of all the reductant. Tapping of metal and slag was done in a ladle after the slag became fluid and good slag-metal separation took place. The slag and metal were cast in a sand mold. It was observed that the TiO_2 content of the slag got diluted as a result of attack of the lining material by the corrosive slag. The size of the furnace and the uneven distribution of heat consequent on the improper positioning of the electrodes with respect to the furnace shell also contributed to the excessive attack on the lining. Further work is in progress.

Results of some of the heats are indicated in Table 3.

15.0 Extraction of Magnesium Metal Electrolytically from Magnesium Chloride

The work was taken up initially at the instance of Messrs Tata Chemicals Ltd. for utilization of byproduct bittern from their salt works by extracting the magnesium metal. Nearly 1.1 million tons of magnesium salt are being produced annually from Indian salt industry as byproduct with a very limited market. This bittern can, however, be utilized for production of magnesium metal by electrolytic process.

During the year under review, two types of electrolytic cells with diaphragms of various compositions were tried. Of the

Table 3 — Results of Some of the Heats

Charge	Lining	Weight of metal obtained	Weight of slag obtained	Metal analysis %	Slag analysis %	Remarks
15 lb. Ilmenite	Magnesite	3 lb.	3 lb.	Fe 95.35	TiO ₂ 63.66	Lining was not affected at all
25 lb. Coke			9 oz.	Ti 0.04	FeO 2.68	
3 lb. Magnesite				Cr 0.17	MgO 23.13	
				V 0.13	Cr ₂ O ₃ 0.14	
				C 2.82	V ₂ O ₅ 0.14	
15 lb. Ilmenite	Magnesite	4 lb.	16 lb.	Fe 95.32	TiO ₂ 60.20	Lining was affected at the sides
2.5 lb. Coke				Ti 0.27	FeO 4.55	
3 lb. Magnesite				Cr 0.08	MgO 24.79	
				V 0.04	CaO 1.13	
				C 2.09	Cr ₂ O ₃ 0.13	
					V ₂ O ₅ 0.17	
15 lb. Ilmenite	Magnesite	1 lb.	2.5 lb.	Not analysed	Not analysed	Excess oke affected the yield of slag and metal
3.5 lb. Coke						
3 lb. Magnesite						
15 lb. Ilmenite	Magnesite	4.3 lb.	9.7 lb.	Fe 93.44	TiO ₂ 70.99	Some material from previous heats accounted for the good yield
				Ti 0.35	FeO 1.30	
1.75 lb. Coke				Cr 0.24	MgO 15.3	
				V 0.31	CaO 2.1	
3 lb. Magnesite				C 2.66	Cr ₂ O ₃ 0.06	
					V ₂ O ₅ 0.08	

five compositions of ceramic materials for diaphragm, only one composition was found to withstand the corrosive action of the molten electrolyte tolerably well during small-scale trials. This material will be used for larger electrolytic cells for longer durations.

Current efficiencies for production of magnesium metal in the two types of cells were around 50-55 per cent. This low-current efficiency was due to the recombination of cathodically liberated metal with anodic chlorine and burning of the metal in finely dispersed state. Effect of these two factors was further aggravated by the violent agitation of the electrolyte in the vicinity of the electrodes. In a cell now in fabrication stage, effort was made to eliminate this disturbance by some change in design and enlargement of cathode-anode spacing. The current load of this new cell was provisionally set at 400 amps., in consideration of space and power facilities, though it was felt that a still larger

cell may suffer much less from such disturbances and may in fact improve the current efficiency.

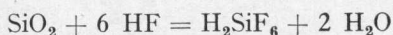
16.0 Electro-refining of Lead

The work was taken up at the instance of Metal & Steel Factory, Ichapore, Ministry of Defence, with a view to defining the exact conditions of electrolysis in relation to the equipment needed for refining secondary lead obtained from spent bullet cores and scrap to a purity of 99.9 per cent lead. This recovery of pure lead will reduce the import of the pure metal used by the Defence Department for making bullet cores.

The composition of the lead bullion received varied from pig to pig with a total impurity content of 2-3 per cent. To homogenize the material, the pig blocks were blended by remelting and recast into pig. The blended mixtures of lead blocks were successively subjected to drossing with

sulphur and softening operation by air to reduce mainly copper and tin. Effective removal of the impurities were possible; copper was finally brought down to 0.043-0.042 per cent and tin to 0.036-0.040 per cent in the metal which did not cause any trouble during the electrolysis. It was found that further removal of tin was associated with heavy loss of lead in the scum as oxides and the period of softening had to be prolonged. With improved drossing technique, the total lead loss as oxides was effectively reduced from 55 to 35 per cent. The blocks after drossing and softening treatment were melted and cast into slabs of $4\frac{1}{2} \times 3\frac{1}{2} \times \frac{3}{8}$ in. for use as anode after mechanically cleaning the surface.

The electrolyte was prepared batchwise. Pure ground quartz was digested first with calculated amount of 38 per cent hydrofluoric acid maintaining a slight excess of sand over the calculated amount as given by the equation



To the clear supernatant liquid (decanted), a calculated amount of basic lead carbonate was added to form lead fluosilicate. After the final addition, the solution was allowed to stand for some time at room temperature. The final acid composition was adjusted suitably for use as electrolyte. Various factors affecting the overall performance, particularly the electrolyte composition, the current density and efficiency aiming at higher purity of the metal were studied for bench scale experiments in a perspex vat, as cell, of $8 \times 5 \times 6$ in.

Various compositions of the electrolyte were prepared and its effect on duration and current efficiency were studied, with current density of 16 amps./sq. ft. over the anode, starting voltage of 0.28 V. and temperature $32^\circ \pm 1^\circ \text{C}$. Glue, 0.1 per cent, calculated on the theoretical amount of deposit was added. Highest current efficiency was

obtained with 65/75 gm. of lead per litre and 90/100 gm. of free acid per litre of electrolyte in the longer duration of electrolysis. The effect of variation of current density and period on the current efficiency was also studied using the electrolyte composition of lead, 70 gm./litre; free acid, 96 gm./litre; and total acid, 145 gm./litre. It was found that in long-drawn out experiments, a current density in the range of 16 amps./sq. ft. gave the highest current efficiency. With somewhat bigger laboratory units (cell) in alkathene welded vats of $18 \times 12 \times 12$ in., anode of $8\frac{3}{4} \times 6\frac{7}{8} \times 1\frac{7}{16}$ in. and cathode of refined lead sheet $9\frac{1}{4} \times 7\frac{3}{8} \times \frac{1}{16}$ in. were used for continuous operation. Anodes and cathodes were changed at the end of every 4 and 2 days respectively. Due to a limited period of anode life about 25-30 per cent of the lead being retained and returned as scrap anode after each dissolution period.

Purity of the metal during preliminary studies varied from 99.94 to 99.9 per cent. The average purity of the metal was 99.97 per cent and current efficiency of 91-92 per cent was obtained during laboratory scale continuous work. The purity of the metal obtained was as follows:

	<i>Per cent</i>
Sb	... 0.005-0.008
Sn	... 0.005-0.007
Cu	... 0.003-0.005
Bi	... 0.005-0.007
Fe	... 0.0022-0.0028
Ag, Cd Zn, As }	Not detected

No attempt was made to treat the anode slime consisting mainly of lead mixed with Sb, Bi, Cu, Fe and Sn which were present as intermetallic complex. The optimum conditions of drossing and softening treatment were evaluated. The lead obtained was subjected to electrolytic refining and average purity of 99.97 per cent with current efficiency of 91-92 per cent was achieved. The process was economically

feasible only when original lead contained appreciable bismuth.

17.0 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 ignition of the reaction mass, powdered aluminium combines with the oxygen content of the metallic oxide until it is consumed and the slag consisting mostly of aluminium oxide and a metal regulus is 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 to the Indian conditions as the present demand of special ferro-alloys such as those of vanadium, titanium, tungsten, carbon-free ferro-chrome, chromium metal, etc., may not warrant setting up individual units. Further systematic studies were undertaken to produce the different grades of ferro-alloys.

During the year under review, carbon-free ferro-tungsten from wolframite concentrates analysing W, 53.9; Fe, 11.34; Mn, 5.6; and SiO_2 , 1.4 per cent was successfully produced. Various factors studied include the effect of fluxes and various amounts of reducing agents. After a series of experiments, the alloy analysing W, 75-80; Si, 0.4-1; and Mn, 0.2-0.6 per cent was produced on a pilot scale and one tonne was supplied to the Ministry of Defence.

18.0 Utilization of Amjhore Pyrite

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 in Mysore. The reserves of

Amjhore pyrites have been estimated to be about 100 million tons with sulphur content up to 40 per cent. The Pyrites Chemical & 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 retain strength without crumbling under load till they reach the softening temperatures in the furnace. The decomposition studies conducted earlier on Amjhore pyrites had indicated that sulphur was evolved at a much faster rate resulting in crumbling.

Petrographic studies of the Amjhore pyrites were carried out to find out the reason for their very fast decomposition. It was seen that the samples contained number of micro-cracks and that the grain size of the pyrites were very small. The micro-cracks resulted in disintegration in the shaft furnace and even briquettes will not stand the burden load at high temperature.

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

Vanadium-bearing titaniferous magnetite occurs in Bihar and Orissa. These ores analyse on the average 55-65 per cent Fe, 8-12 per cent TiO_2 and 0.8-1.8 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 magnetites containing 0.75 per cent vanadium collected from Rairangpur (Orissa) was crushed to -30 mesh B.S.S. Systematic studies on roasting conditions were completed. Sodium chloride and sodium carbonate mixtures were tried in various proportions and at various temperatures for roasting the ore. Sodium

chloride did not prove to be particularly favourable. It was observed that 85 per cent of the vanadium was recovered by roasting the ore at 900-950°C. with 12 per cent soda ash and retaining the mixture for 1 hr. at the high temperature. The roasting studies were conducted in a rotary kiln 12 ft. long and 9 in. inside diameter and rotating at a speed of 0.8 r.p.m. The kiln is lined with sillimanite bricks in the high-temperature zone and high-heat duty fireclay bricks in the remaining zones. The kiln is capable of retaining the charge for only half an hour in the high-temperature zone. To increase the retention period and the flexibility of the operating condition of the kiln, a new drive capable of a minimum speed of 0.1 r.p.m. was designed and installed.

The leaching of the roasted product was carried out on a multi-stage counter-current system with six agitators and steam-heating coils. Studies for evaluating the number of stages required for the maximum recovery and the solid to liquid ratio for the required concentration of the final leach liquor were completed on bench scale. It was found that five-stage leaching was required for the recovery of 95 per cent of the soluble vanadium from the roasted product, with a solid to liquid ratio of 1:2 for a concentration of 15 gm. of vanadium per litre of the final leach liquor and a duration of 2 hr. at each stage at a temperature of 80°C. A centrifuge is being installed for separating and recovering sodium sulphate from the concentrated liquor after precipitation of V_2O_5 . A hot air shelf-drier is also being installed for the final drying of vanadium pentoxide.

Studies are also being conducted to obtain a high-purity vanadium pentoxide by the use of ammonium chloride for precipitation of vanadium from the leach liquor as ammonium vanadate. Vanadium pentoxide made during the trials were sent to National Chemical Laboratory, Poona, for testing purposes.

20.0 Reconditioning of Magnesium Powder

Magnesium powder during storage for long periods deteriorates due to the formation of oxide coating on the surface of the metallic particles and cannot be used for pyro-techniques. The investigation was taken up at the instance of the Defence Establishment with a view to improving the metallic content from about 80 per cent to over 99 per cent. Both mechanical and chemical methods were investigated for the preferential removal of the oxide coating leaving behind a powder with high-metallic contents. A method was established for the preferential elimination of the oxide coating. Effect of various impurities in the water used for making the solution from the inorganic compound was studied. It was observed that $NaCl$, $CaCl_2$, bleaching powder and chlorine affect the purity of the magnesium powder. Systematic studies were carried out to determine the effect of reaction temperature, temperature of drying, concentration of the inorganic solvent. With increase in the reaction temperature from 15° to 80°C., the magnesium content of the reconditioned powder gradually decreased from 99.61 to 89.39 per cent. Attempts were made to regenerate the spent liquor by addition of oxidizing agents and electrolysis. Utilization of the spent liquor itself by addition of partly reconditioned powder was tried. Further work is under progress for counter-current pickling to attain a higher efficiency in the utilization of the inorganic solvent.

On the basis of the preliminary laboratory scale studies, optimum concentration, temperature, reaction period and drying temperature were arrived at to treat bulk quantities of magnesium powder being received from the Defence Stores. Large quantities of different grades such as O, IV, V and III were reconditioned to contain over 99 per cent metallic magnesium from their original metallic contents ranging

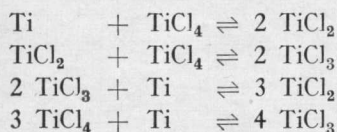
Table 4 — Effect of Time on the Reaction between Ti and TiCl₄ at 750°C.

Expt. No.	Reaction time hr.	Amount of Ti reacted gm.	Amount of Ti with dichloride gm.	Amount of Ti with trichloride gm.	Titanium metal formed by disproportionation of TiCl ₂ gm.
1	Two	0.1808	0.261	0.064	0.03
2	Three	0.290	0.432	0.106	0.05
3	Four	0.412	0.614	0.164	0.08

from 80 to 90 per cent. During the year about 2.33 tonnes of the magnesium powder were reconditioned and despatched to the Defence Establishments in Jabalpur and Kirkee for their use.

21.0 Catalytic Distillation of Titanium from Titanium Bearing Alloys

Aluminium-titanium alloys containing 50-60 per cent titanium can be made by aluminothermic reactions in presence of suitable energizers. Work was taken up to study the recovery of titanium from the aluminium-titanium alloys by treatment with TiCl₄ or AlCl₃. The reaction of TiCl₄ with titanium and disproportionation of subhalides produced may be represented as follows:



An assembly for the reaction of gas mixture containing TiCl₄ at various partial pressures and titanium was set up. Equilibrium vapour pressures of TiCl₄ at various temperatures were determined by bubbling argon gas through TiCl₄ liquid and determining the amount of TiCl₄ in the gas stream, by absorbing TiCl₄ in hydrochloric acid solutions. Table 4 gives the results obtained by carrying out the reactions between Ti and TiCl₄ for different periods

at 750°C., the tetrachloride partial pressures in the gas stream being maintained at 16 mm. of mercury.

Table 5 gives the results obtained at different temperatures by reacting titanium turnings with titanium tetrachloride vapours at a pressure of 16 mm. mercury. The duration of the experiment in each case was 4 hr.

Table 5 — Effect of Temperature on the Reaction between Ti and TiCl₄

Temperature of reaction °C.	Amount of Ti reacted gm.	Amount of Ti with dichloride gm.	Amount of Ti with trichloride gm.	Amount of titanium metal formed by disproportionation of TiCl ₂ gm.
850	0.502	0.71	0.22	0.112
750	0.412	0.614	0.16	0.07
650	0.308	0.41	0.13	0.06

22.0 Studies on the Recovery of Alumina from Indian Bauxite

Systematic studies were taken up to determine the suitability of Bayer's process for the recovery of alumina from Indian bauxites. The properties being studied include mineralogical and chemical composition and various factors which affect the alumina extraction, viz. particle size,

concentration of alkali, molar ratio of caustic to alumina, time and effect of calcination.

Samples were collected from fifteen different deposits in India.

Bauxite Samples from Kutch, Gujarat

The bauxite sample received from Naradi, Kutch district, Gujarat, analysed as follows:

	Per cent
Al ₂ O ₃ . . .	54.10
SiO ₂ . . .	5.7
Fe ₂ O ₃ . . .	24.4
TiO ₂ . . .	4.3
CaO . . .	1.7
MgO . . .	0.28
Moisture . . .	0.52
L.O.I. . . .	28.26

Petrographic examination of the sample revealed that majority of the alumina was present as clachite, Al₂O₃ (H₂O)_x. The sample was found to contain 41.0 per cent alumina as gibbsite, 8.3 per cent alumina as bohemite, 2.8 per cent alumina as kaolinite and 1.8 per cent alumina as diaspor. Systematic studies on the effect of the various factors such as particle size, concentration of alkali, molar ratio of caustic to alumina and effect of calcination of bauxite were carried out. It was observed that 92 per cent of the Al₂O₃ could be extracted after crushing the ore to -10 mesh simply by digesting for 45 min. with a caustic soda solution containing 265 gm. per litre (Na₂CO₃ basis) and Na₂O: Al₂O₃ as 2:1. The effect of calcining bauxite at various temperatures on its solubility in caustic solutions showed Al₂O₃ recovery to decrease from 92.0 per cent at 100°C. to 42.52 per cent at 500°C. which was due to conversion of the readily soluble trihydrate into a less readily soluble monohydrate. The detailed report is being prepared.

Bauxite Sample from Jamnagar

The bauxite sample received from Ran Mines, Jamnagar district, Gujarat, analysed as follows:

	Per cent
Al ₂ O ₃ . . .	61.8
SiO ₂ . . .	1.3
Fe ₂ O ₃ . . .	1.8
TiO ₂ . . .	2.7
CaO . . .	0.32
MgO . . .	0.18
Moisture . . .	0.42
L.O.I. . . .	30.86

Petrographic examination of the sample revealed that majority of the alumina was present as clachite, Al₂O₃ (H₂O)_x. This sample was found to contain 50.11 per cent Al₂O₃ as gibbsite, 8.8 per cent Al₂O₃ as bohemite, 1.8 per cent kaolinite and 1.09 per cent diaspor.

It was observed that 92-93 per cent of the Al₂O₃ could be extracted by digesting the ore crushed to 100 mesh for 45 min. with a caustic soda solution containing 265 gm. per litre (carbonate basis) and a ratio of Na₂O: Al₂O₃ as 2:1.

Bauxite Sample from Bankodi (Gujarat)

The ore sample had the following chemical composition:

	Per cent
Al ₂ O ₃ . . .	57.62
SiO ₂ . . .	1.48
Fe ₂ O ₃ . . .	2.56
TiO ₂ . . .	3.40
CaO . . .	1.2
MgO . . .	1.23
L.O.I. . . .	32.65

Following results were obtained for the quantitative determination of alumina

minerals both by Masletinskii's and plant methods:

Al ₂ O ₃ mineral %	Masletinskii's method %	Plant method %
Gibbsite	52.0	53.18
Bohemite	3.8	—
Kaolinite	1.32	—
Diaspore	0.50	—
Total Al ₂ O ₃ in ore sample	57.62	

Optimum conditions of digestion were as follows: particle size —100 mesh; caustic concentration 265 gm./litre (carbonate basis); molar ratio of Na₂O : Al₂O₃ 2 : 1, leaching time 30 min. and pressure 1 atmosphere.

Bauxite Sample from Bankodi (Gujarat)

This ore sample was somewhat more reddish in colour than sample No. 3 and had the following chemical composition:

	<i>Per cent</i>
Al ₂ O ₃ . . .	56.44
SiO ₂ . . .	1.28
Fe ₂ O ₃ . . .	4.9
TiO ₂ . . .	3.4
CaO . . .	0.56
MgO . . .	1.17
L.O.I. . . .	32.26

The following are the results obtained for the quantitative estimation of alumina minerals:

Al ₂ O ₃ mineral %	Masletinskii's method %	Plant method %
Gibbsite	43.0	50.85
Bohemite	8.25	—
Kaolinite	3.49	—
Diaspore	1.69	—
Total Al ₂ O ₃ in ore sample	56.43	

Optimum digestion conditions were: particle size —100 mesh; caustic concentration 265 gm./litre (carbonate basis); molar ratio 2 : 1; leaching time 60 min.; pressure 1 atmosphere. This ore sample was also suitable for Bayer's process since it was found highly gibbsitic and went into solution easily.

Bauxite Sample from Virpur Mines (Gujarat)

This sample received from Virpur Mines in Gujarat analysed as follows:

	<i>Per cent</i>
Al ₂ O ₃ . . .	55.38
SiO ₂ . . .	1.82
Fe ₂ O ₃ . . .	4.02
TiO ₂ . . .	3.2
CaO . . .	2.2
MgO . . .	1.66
L.O.I. . . .	31.92

Mineral composition is as follows:

Al ₂ O ₃ mineral %	Masletinskii's method %	Plant method %
Gibbsite	39.9	50.47
Bohemite	7.75	—
Kaolinite	4.79	—
Diaspore	2.94	—
Total Al ₂ O ₃ in ore	55.38	

Optimum digestion conditions were reached with particle size —60 mesh; caustic concentration 265 gm./litre (carbonate basis); molar ratio 2 : 1 and 30 min. leaching period; pressure 1 atmosphere. This ore sample has been found suitable for Bayer's process and 92 per cent of the Al₂O₃ can be extracted within a short period.

Detailed reports are being prepared for all the samples investigated.

23.0 Chlorination of Vanadium Bearing Titaniferous Magnetite Ore

The removal of vanadium from vanadium-bearing titaniferous magnetites by chlorination offered the advantage that most of the vanadium can be removed at comparatively low 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 hydrochloric acid with strong H_2SO_4 was passed at a rate of 280 cc. per minute over the vanadium ore contained inside a horizontal tubular furnace. Further work was continued on the rate of volatilization of vanadium at various temperatures which is given in Table 6.

No appreciable difference in the chlorination rate was observed during the first two hours which was attributed to the simultaneous formation of comparatively less volatile iron-chlorides that formed a paste-like mass, inhibiting the progressive chlorination of vanadium. To suppress the chlorination of the iron values, the partial pressure of HCl was decreased by passing

Table 6 — Effect of Temperature on the Rate of Volatilization of Vanadium from Magnetite Ore

Temperature °C.	Percentage vanadium volatilized in			
	60 min.	120 min.	180 min.	240 min.
190	30.38	46	77.2	80.80
200	38	64	65	—
250	36	42	65	—
300	26.5	39	69	—
350	26.28	38	65	—
400	27.24	34.78	36.34	—

the gas along with nitrogen and this correspondingly affected the chlorination of iron and vanadium. The chlorination apparatus is being modified so as to pass the mixture of HCl gas and water vapour over the

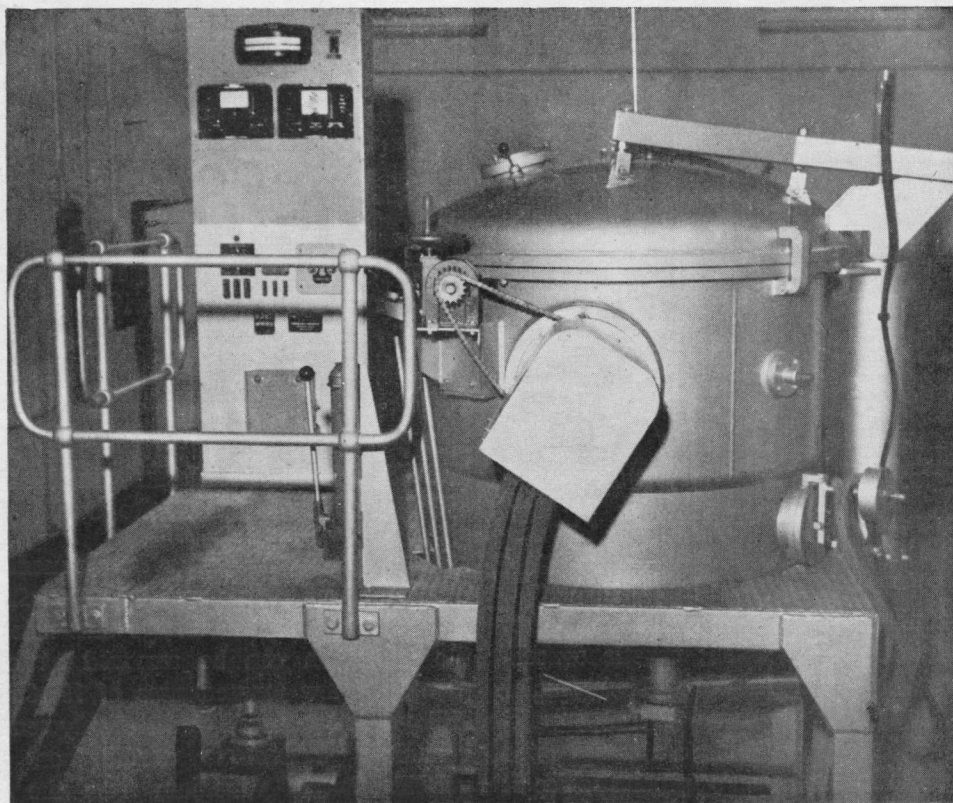


FIG. 12 — A VIEW OF THE VACUUM FURNACE INSTALLED AT THE NATIONAL METALLURGICAL LABORATORY

heated ore, thus preventing the chlorination of iron.

24.0 Extraction of Nickel from Low-grade Siliceous Nickel Ore

A low-grade siliceous nickel ore from Assam was received from the Geological Survey of India. This serpentine type of ore contained 0.65 per cent NiO with high silica and magnesia. Nickel was present in the ore as a complex of hydrated magnesium silicate. Systematic studies on the recovery of nickel by roast reduction followed by ammoniacal leaching were taken up.

The low-grade siliceous nickel ore from Assam analysed SiO_2 , 38.34; Fe_2O_3 , 11.23; Al_2O_3 , 3.15; NiO, 0.65; and MgO, 33.14 per cent. It was not found possible to enrich the nickel content by conventional ore-dressing techniques. A method comprising roast reduction of the ore followed by subsequent ammoniacal leaching was attempted to extract the nickel value in the form of a soluble nickel-ammonium complex. The ore was finely crushed and reduced with hydrogen gas. The reduced ore was cooled, digested in the form of a thick slurry with concentrated ammoniacal liquor for preferential oxidation of iron and subsequently leached with dilute ammoniacal liquor consisting of ammonium hydroxide and an ammonium salt in presence of oxygen for dissolution of nickel in the form of nickel-ammonium complex. Various factors studied so far included effect of temperature, particle size of ore, rate of flow of hydrogen and humidification of hydrogen on reduction, kinetics of reduction at different temperature, effect of briquetting, mechanism of reduction, digestion characteristics of the reduced ore, effect of different ammonium salts, and composition of the ammoniacal liquor on the leaching characteristics.

It was observed that a good degree of reduction could be achieved by reducing -200 mesh (B.S.S.) size of the ore with

dry hydrogen at 750°C. for a period of 90 min. Higher temperatures of reduction were detrimental as they resulted in the formation of an alloy of iron and nickel that could not be readily leached with ammoniacal solution. The reduced ore was digested for 1 hr. and leached at room temperature with an ammoniacal liquor containing 7.5 per cent NH_4OH and 7.5 per cent ammonium carbonate with constant flow of oxygen gas.

The solution containing nickel-ammonium complex was boiled to precipitate basic nickel carbonate. It was observed that almost complete nickel recovery was achieved during precipitation of basic nickel carbonate from the solution. It was also found possible to cycle back the regenerated ammonia during precipitation for leaching further quantities of reduced ore.

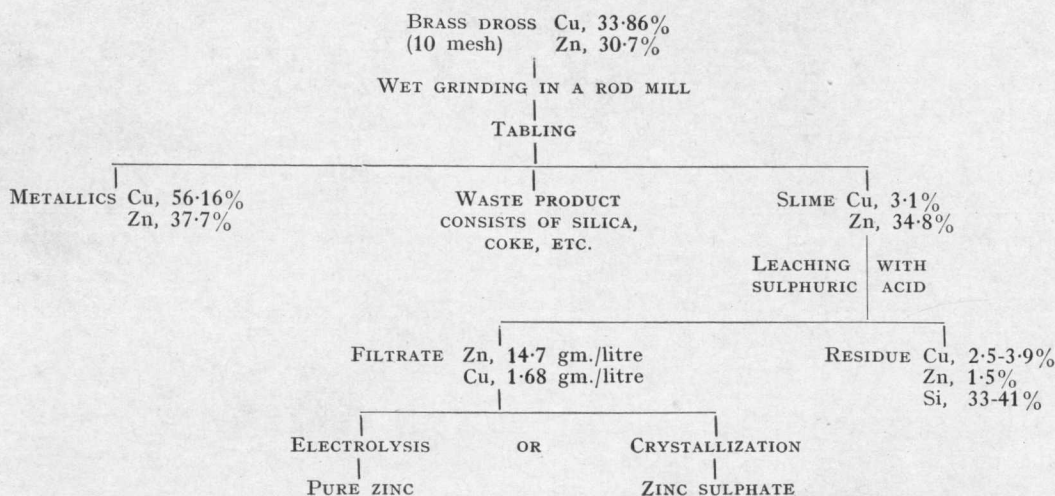
25.0 Recovery of Metallic Values from Industrial Brass Dross

Studies were undertaken for the recovery of metallic values from a typical industrial brass dross containing 34.93 per cent total copper, 34.46 per cent total zinc, 7.49 per cent silica, 1.63 per cent lead and 1.32 per cent iron. Preliminary investigations to dissolve the entire sample with varying strength of sulphuric acid showed that 60 per cent of the total zinc and 1 per cent of the total copper were dissolved in the acid leaving the rest of copper and zinc in the form of metallics and that there was no improvement on increasing the acid strength to more than 10 per cent. Experiments were carried out to separate the metallics by physical methods. The sample was wet ground and washed with water and the metallics were separated. The metallics amounted to 55 per cent of the total weight of the sample taken and analysed 56.16 per cent copper and 37.7 per cent zinc corresponding roughly to 60/40 brass. The slime analysing 3.09 per cent total copper

and 34.5 per cent total zinc was leached with dilute sulphuric acid. 70 per cent of the total zinc and 49 per cent of the total copper was dissolved in the acid. The final residue analysed 2.68 per cent total copper, 10.43 per cent total zinc and 40.1 per cent SiO_2 . An overall recovery of 95.4 per cent of zinc and 94.1 per cent copper was obtained. Thus a simple process of wet grinding to liberate the adhering oxides from metallic and washing with water enabled the separation of metallics which could be used as part of the charge in brass melting. The slime could be treated for recovering zinc and copper by leaching with sulphuric acid. A flow-sheet of the process is given below:

indicated the presence of the gangue in all the fractions. Microscopic examination of the metallic particles revealed oxide coating on the particles and inclusions of the gangue materials. Exhaustive studies on the recovery of metallic values from the dross were undertaken and a method evolved for the same. The final product analysed as:

	<i>Per cent</i>
Metallic aluminium . . .	84.8
Al_2O_3 . . .	2.98
SiO_2 . . .	5.20
Fe . . .	2.47
Si . . .	0.94
Cu . . .	2.40
C . . .	0.34



26.0 Recovery of Aluminium from Aluminium Dross

Aluminium dross containing about 40-50 per cent metallic aluminium in the form of powder is a waste product in the foundries and finds no commercial use as such. Investigation was, therefore, taken up with a view to upgrading the dross sample to a useful product. The dross samples supplied in the powder form around 14 mesh size contained aluminium with a blackish appearance. Screening of the product

The gangue materials were found to be embedded on the metallic particles in the form of inclusions.

27.0 Utilization of Ferro-manganese Slag

The investigation was taken up with a view to recovering manganese from high-carbon ferro-manganese slag. The slag sample received in lumps varied in size. The material was properly sampled, crushed

and analysed. The sample analysed as follows:

MnO, 21.95; FeO, 0.50; Al₂O₃, 9.40%
SiO₂, 29.90; CaO, 32.80; MgO, 4.20%

Investigation was carried out employing ferro-silicon (89 per cent Si) for silico-thermal reduction of the slag producing silico-manganese. It was observed that a considerable part of ferro-silicon oxidized during melting and as such further experiments were carried out to minimize the oxidation of ferro-silicon by adopting suitable methods. Studies are also under progress for the production of carbon-free silico-manganese by employing aluminium as a reductant. Systematic studies were also undertaken to investigate the possibility of recovering manganese values from the slag by leaching with sulphuric acid.

28.0 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 scale on beneficiation of fluorspar was conducted in the laboratory.

For preparing synthetic cryolite, beneficiated metallurgical grade fluorspar was used as the starting material. Preparation of cryolite at an overall efficiency of more than 90 per cent can be done provided

only first leaching and precipitation are taken into consideration. But the spent liquor after precipitation of cryolite should be recycled into the process to make the best use of boric acid for leaching fluorspar. Cyclic experiments conducted earlier as shown in Tables 7 and 8 showed an overall efficiency of 64.01 per cent up to the eighth cycle and loss of 3.7 gm. of boric acid for 100 gm. of cryolite prepared. One cycle consisted of one leaching, removal of residual sulphuric acid and precipitation of cryolite as given in the flowsheet.

Two more cyclic experiments were carried out with the metallurgical grade fluorspar of the following composition:

	Per cent
CaF ₂	84.37
CaCO ₃	9.2
SiO ₂	3.8
Fe ₂ O ₃	0.34
Al ₂ O ₃	1.28
MgO	0.36
S	Trace
Moisture	0.04
Loss on ignition	6.36

For leaching, theoretical amount of sulphuric acid was used. Residual sulphuric acid after leaching was removed with CaCO₃. The data of these cyclic experiments are given in Tables 9 and 10.

Average chemical analyses of NML product and the cryolite specified for aluminium cells are given below:

Radicals	NML cryolite average analysis %	Cryolite specifica- tion for aluminium reduction cell %
P ₂ O ₅	0.05-0.08	Not more than 0.1
SiO ₂	0.1-0.2	Not more than 0.6
Fe ₂ O ₃	0.03-0.06	Not more than 0.25
SO ₃	1.4-3.0	Not more than 5.0
Ca	0.5-0.6	—
Na	22-28	—
Al	17-19	—
F	49-55	—
Na ₃ AlF ₆		Not less than 85

Table 7 — Cyclic Experiment 1

Soln. volume = 500 cc.
Sod. sulphate = 25 gm.
Temp. of leaching = 95°C.
Time = 2 hr.

No. of cycle	Fluorspar gm.	Sulphuric acid	Boric acid gm.	Residual H ₂ SO ₄ gm.	Sodium carbonate gm.	Hydrated alumina gm.	Cryolite obtained gm.
1	100	Theoretical	55	3.42	34	34	60
2	100	do	—	14.37	37.8	37.8	66
3	100	do	—	28.40	32.5	32.5	48
4	100	do	—	46.00	30.5	30.5	45
(purified)							
5	100	do	8	26.45	22.5	22.5	45
6	100	do	—	30.20	30.0	30.0	48
7	100	do	—	40.29	26.0	26.0	46
8	100	do	—	37.4	28.0	28.0	47
9	100	do	—	47.0	30.0	30.0	20

Total cryolite removed up to 8th cycle = 405 gm.
 Boric acid in spent liquor = 48 gm.
 Loss of boric acid = 15 gm.
 Boric acid loss/100 gm. of cryolite = 3.7 gm.
 Total fluorine in 800 gm. of fluorspar = 329 gm.
 Up to 8 cycles fluorine recovered = 210.6 gm.
 Overall recovery = 64.01%

Table 8 — Cyclic Experiment 2

Soln. volume = 500 cc.
Sod. sulphate = 25 gm.
Temp. of leaching = 95°C.
Time = 2 hr.

No. of cycle	Fluorspar gm.	Sulphuric acid	Boric acid gm.	Residual H ₂ SO ₄ gm.	Sodium carbonate gm.	Hydrated alumina gm.	Cryolite obtained gm.
1	100	Theoretical	55	1.9	33	33	64
2	100	do	—	2.33	33	33	60
3	100	do	—	11.10	34	34	58
4	100	do	—	19.30	34	34	53
5	100	do	—	33.80	34	34	33
6	100	do	20	19.85	28	28	28
(purified)							
7	100	do	—	6.40	34	34	63
8	100	do	—	42.00	34	34	49
9	100	do	—	49.00	23	23	25

Up to 8th cycle total cryolite recovered = 408 gm.
 Boric acid in spent liquor = 64 gm.
 Loss of boric acid = 11 gm.
 Boric acid loss/100 gm. of cryolite = 2.7 gm.
 Total fluorine in 800 gm. of fluorspar = 329 gm.
 Up to 8 cycles fluorine recovered = 212.2 gm.
 Overall recovery = 64.01%

Table 9 — Cyclic Experiment 3

Soln. volume = 500 cc.
Sod. sulphate = 25 gm.
Temp. of leaching = 95°C.
Time = 2 hr.

No. of cycles	Fluorspar gm.	Sulphuric acid	Boric acid gm.	Residual H ₂ SO ₄ gm.	Sodium carbonate gm.	Hydrated alumina gm.	Cryolite obtained gm.
1	100	Theoretical	60	1.0	38	38	72
2	100	do	—	5.84	35.6	35.6	68
3	100	do	—	7.40	34.7	34.7	62
4	100	do	—	11.68	35.3	35.3	64
5	100	do	—	12.26	36.0	36.0	60
6	100	do	—	23.36	32.0	32.0	48
(purified)							
7	100	do	—	12.26	31.5	31.5	42
8	100	do	8	36.80	30	30	49
9	100	do	—	46.00	28	28	30
Total cryolite obtained				=	465 gm.		
Boric acid in spent liquor				=	59 gm.		
Loss of boric acid				=	9 gm.		
Boric acid loss/100 gm. of cryolite				=	1.93 gm.		
Total fluorine in 800 gm. of fluorspar				=	329 gm.		
Fluorine recovered				=	241.8 gm.		
Overall recovery				=	73.5%		

Table 10 — Cyclic Experiment 4

Soln. volume = 500 cc.
Boric acid added = 55 gm.
Sod. sulphate added = 25 gm.
Time of leaching = 2 hr.
Temp. of leaching = 95°±2°C.

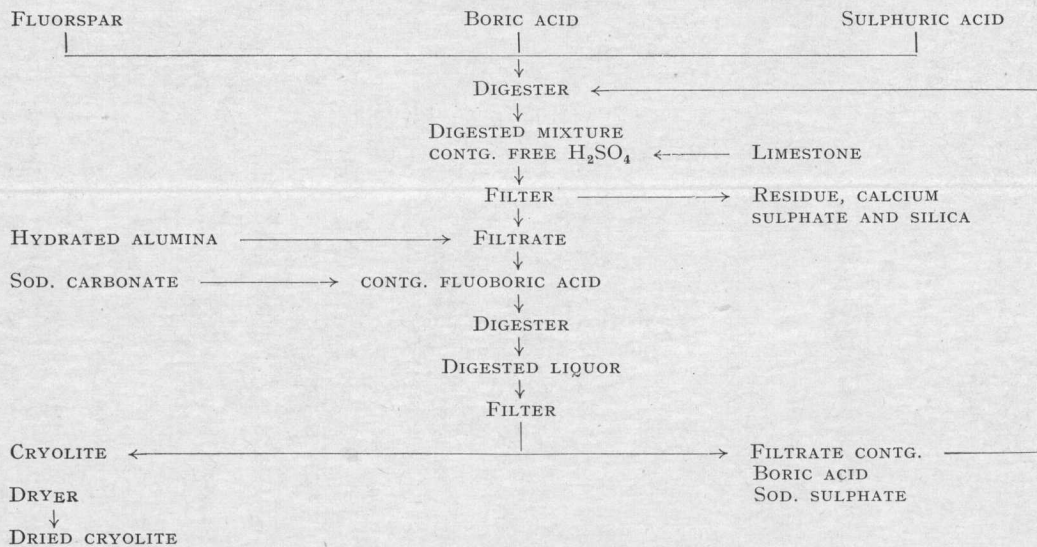
Sl. No.	Amount of fluorspar gm.	Amount of H ₂ SO ₄	Impurity added	Amount of impurity added gm.	Fluorine in fluorspar added gm.	Fluorine leached gm.	Fluorine leached %
1	90	Theoretical	—	—	43	42.14	98
	(acid grade)						
2	90	do	AlF ₃	6.3	43	38.9	90.4
3	90	do	AlF ₃	12.7	43	32.5	75.5
4	90	do	AlF ₃	19.0	43	30.75	71.5
5	90	do	Fe ₂ (SO ₄) ₃	18.0	43	40.9	95.1
6	100	do	FeSO ₄	13.6	41.1	38.9	94.6
	(met. grade)						
7	100	do	Na ₂ SO ₄	75	41.1	36.8	89.6

One kilogram of cryolite made by this process was sent to an aluminium-producing firm for determining its suitability in the aluminium reduction cell. Further work is in progress.

29.0 Preparation of Anhydrous Magnesium Chloride

Hindustan Aircraft Ltd. required approximately 50-100 lb. of anhydrous magnesium

Flow Sheet of Fluoboric Acid Process for Cryolite



chloride per month to be used as flux material for melting, casting and refining of magnesium base alloys. This work was taken up with a view to examining the possibilities of preparing anhydrous magnesium chloride to be used as fluxing material.

It was found that in dehydration of dihydrate magnesium chloride, the presence of NH_4Cl checks the hydrolysis and can be used in place of HCl . Some trial experiments were conducted with mixtures of various proportions of magnesium chloride and ammonium chloride. The mixed salt with various molar ratio of NH_4Cl and $MgCl_2$ was not free from moisture and NH_4Cl in the dried state. The oxide percentage was below 3 and moisture was about 18 per cent up to 1.8 molar ratio of the salts. To remove the moisture and NH_4Cl from the product, the use of a rotary kiln type drier was decided for maintaining a higher drying temperature of about $350^\circ C.$ at the discharge end and about $180^\circ C.$ at the feed end. Shock heating and high-temperature drying tests at different temperature ranges were carried out, using

the already dried mixture of various ratios of NH_4Cl and $MgCl_2$. Results are furnished in Table 11.

For drying at high temperature, the apparatus consisted of a circular muffle furnace open at the top and closed at the bottom by loosely set insulation bricks with controller for regulating the temperature. A glass vessel was used for holding the salt mixture. The furnace was adjusted to $180^\circ C.$, the storing temperature of the salt after previous drying. Each charge contained nearly 100-125 gm. of salt. The temperature was raised stage by stage slowly at the rate of 3° per minute holding for about 2-4 hr. at particular stages till a temperature of $400^\circ C.$ was reached. Before raising the temperature to the next stage, a sample was taken to determine the composition. An optimum programme for drying was developed after a study of the data obtained. The average analyses of dried samples were 97.98 per cent $MgCl_2$, 0.2-0.5 per cent NH_4Cl , oxide below 2 per cent, with traces of other impurities. A rotary drier was designed on the basis of the results obtained.

Table 11 — Data on Drying Tests of Ammonium and Magnesium Chlorides

Salt mixtures ratio NH ₄ Cl: MgCl ₂	Temp. °C.	Period of holding hr.	MgO %	MgCl ₂ %	NH ₄ Cl %	H ₂ O by diff. %
1: 2	250	1 hr.	(a) 3.26	69.72	10.22	16.8
			(b) 4.62	71.33	9.36	14.6
	300	1 hr.	(a) 4.17	70.22	9.34	16.20
			(b) 4.77	72.90	8.66	13.5
	350	1 hr.	(a) 5.11	72.21	8.90	13.78
			(b) 4.93	75.69	7.24	12.14
1: 4	250	1 hr.	(a) 3.35	75.35	6.88	14.42
			(b) 4.16	76.27	7.11	12.46
	300	1 hr.	(a) 4.11	75.89	6.08	13.92
			(b) 5.39	76.73	5.60	12.28
	350	1 hr.	(a) 5.59	76.76	5.13	12.52
			(b) 7.27	77.14	4.08	10.71
1: 6	250	1 hr.	(a) 10.79	79.61	6.09	13.51

The hydrated magnesium chloride alone and with NH₄Cl in ratio of 1:1, 1:1.5 and 1:2 were tried for drying at 165°C. under a vacuum of 28.2 in. Hg. The product did not improve beyond MgCl₂, 1.25 H₂O and MgCl₂, NH₄Cl, 1.5 H₂O. To improve the quality of the final product, a bench scale fluidized bed drying unit was designed and is now being fabricated. A prototype design for chlorination of magnesia-carbon mixture was also made.

30.0 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 on the determination of physical,

mechanical and creep properties of the new steels 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 tensile strength and ageing characteristics of the steel.

During the period under review, weldability, oxidation resistance and low-temperature tensile properties of different compositions of chromium-manganese-nitrogen stainless steels were determined. Chemical composition of the steels used in the investigations are given in Table 12.

Table 12 — Chemical Composition of Cr-Mn-N Stainless Steels

Heat No.	Cr %	Mn %	N %	C %	Si %
B ₂	20.9	13.8	0.68	0.12	0.70
B ₄	22.07	16.63	1.01	0.04	0.40
B ₅	17.9	16.93	0.69	0.05	0.28
B ₆	17.59	16.93	0.66	0.04	0.31

Table 13 — Tensile Properties of the Welded Specimens

Heat No.	Filler metal	Tensile strength kg./mm. ²	Elongation % on 25 mm. G.L.	Location of fracture
B ₂	Parent metal 18/8 type	95.4	50.0	Base metal
		84.4	31.0	At the weld
B ₄	Parent metal 18/8 type	100.3	21.8	At the weld
		108.6	25.0	At the weld

Table 14 — Tensile Properties of Unwelded Base Metal

Heat No.	Tensile strength kg./mm. ²	Elongation % on 25 mm. G.L.
B ₂	94.2	45.3
B ₄	102	50.0

Weldability Tests

Welded specimens were prepared by argon arc welding. Strips cut from the annealed sheets of 0.035-0.040 in. thickness were welded using 18/8 type stainless steel welding rods and thin strips cut from the original sheets as filler metal. Single layer butt joints were produced. The welded strips were ground smooth at the weld and tensile specimens made from the strips were tested at room temperature. Results of the tests are given in Table 13. Tensile properties of unwelded base metal are also given in Table 14 for the purpose of comparison.

Oxidation Resistance Tests

Oxidation resistance tests at 900°C. were carried out in a thermal balance on specimens of heat Nos. B₂, B₄ and B₅ in an atmosphere of dry oxygen. Test was also conducted on a specimen of 18/8 stainless steel for the purpose of comparison. Results of the tests were plotted in Fig. 13, which showed that the steels B₂ and B₄ had slightly higher oxidation rate at 900°C.

than 18/8 stainless steel. Steel No. B₅ containing 17.9 per cent chromium showed much higher oxidation rate. Specimens of heat B₂ and B₄ showed non-uniform scaling.

Sub-zero Tensile Tests

A special fixture was made for sub-zero tensile testing. Specimens of 1 in. G.L. were tested immersed in petroleum ether cooled to the desired temperature by liquid air. Results of the tests are given in Table 15.

Table 15 — Results of Sub-zero Tensile Tests

Heat No.	Temperature	Maximum stress kg./mm. ²	Elongation % on 25 mm. G.L.
B ₂	Room temperature	97.01	45.3
	—10°C.	114.8	46.8
	—20°C.	99.90	43.7
	—30°C.	114.2	43.7
B ₄	Room temperature	104.1	48.4
	—10°C.	122.3	53.0
	—20°C.	116.1	50.0
	—30°C.	122.6	46.8
B ₆	Room temperature	94.82	50.0
	—10°C.	102.8	Broken outside G.L.
	—20°C.	104.7	50
	—30°C.	109.7	Broken outside G.L.

Tests carried out up to —20°C. indicated that at low temperatures, the strength of Cr : Mn : N steels increased slightly with no loss of ductility.

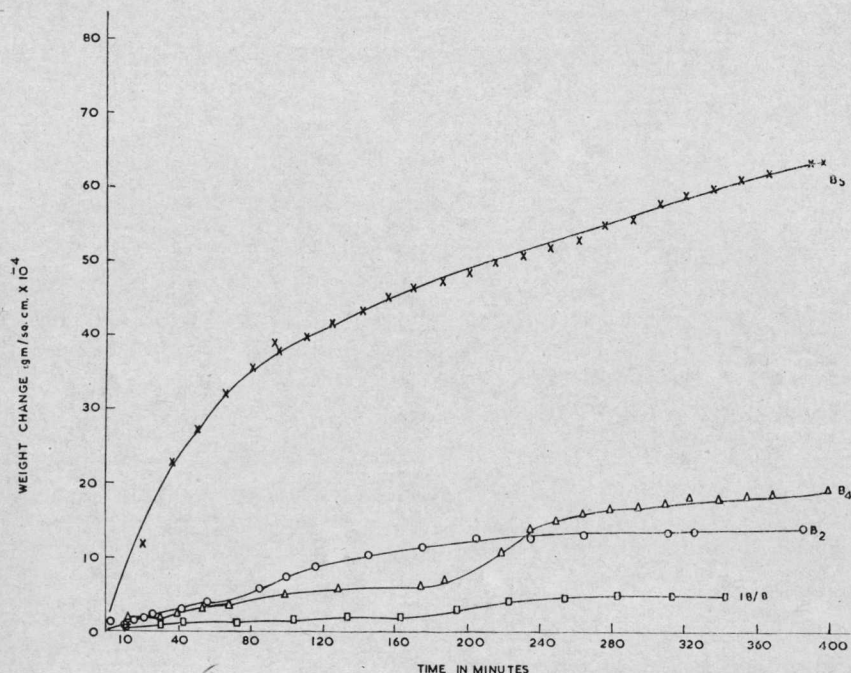


FIG. 13 — OXIDATION CURVES FOR CHROMIUM-MANGANESE-NITROGEN STAINLESS STEEL AND 18/8 STAINLESS STEEL AT 900°C.

Low-chromium Austenitic Stainless Steels

Low-chromium semi-austenitic stainless steels possessing high-corrosion resistance and good formability were prepared at 12 per cent chromium level. In these steels, manganese and nitrogen were used as austenitizing agents and small addition of phosphorus and copper were made to improve the corrosion resistance characteristics of the steel. Results of boiling

nitric acid (65 per cent) tests carried out on a few steels are given in Table 16.

The steels, however, did not show high-corrosion resistance. To improve the corrosion resistance, further small additions of molybdenum and nickel were made in the following heats. The first series of heats in which 2 per cent copper was added showed poor hot-working property. Thereafter, a few heats were made containing 1 per cent copper which were successfully

Table 16 — Results of Boiling Nitric Acid Tests

Heat No.	Percentage composition						Loss in wt. in gm./dm. ² /day
	Cr	Mn	N	C	Cu	P	
A 198	12.43	10.01	0.15	0.04	—	—	1.4090
A 203	12.43	12.23	0.19	0.02	0.99	0.039	1.183
							1.164
A 307	12.6	11.9	0.21	0.02	1.88	0.11	1.1802
							1.0770

Table 17 — Composition of Semi-austenitic Stainless Steel with Ni and Mo

Heat No.	Percentage composition							
	Cr	Mn	N	C	Ni	Mo	Cu	P
A 235	12.14	11.7	0.27	0.07	1.03	0.70	2.0	0.20
A 241	11.47	11.92	0.23	0.04	1.05	0.63	1.01	0.24

forged into bars and subsequently rolled into sheets. Corrosion resistance and mechanical properties of these steels are under study. Typical compositions of the steels made are given in Table 17.

Austenitic Cr : Mn : N Stainless Steels Containing High Carbon

Heats of Cr : Mn : N stainless steels containing carbon in the range of 0.1-0.3 per cent were prepared at 21 per cent chromium level. Chemical composition of two heats in this series are given in Table 18. The objective of this work was to improve the hot-working property of Cr : Mn : N steels by a carbon-nitrogen balance.

Table 18 — Chemical Composition of Austenitic Cr-Mn-C-N Stainless Steels

Heat No.	Percentage composition				
	Cr	Mn	N	C	Si
A 231	21.0	14.0	0.50	0.24	0.40
A 232	21.1	13.8	0.43	0.31	0.26

Tensile properties of the steels determined on the solution-treated sheets are given in Table 19.

Table 19 — Tensile Properties of Austenitic Cr : Mn : C : N Stainless Steels

Heat No.	Proof stress 0.2% off set kg./mm. ²	Tensile strength kg./mm. ²	Elongation % on 25 mm. G.L.	Hardness VPN
A 231	47.3	96.9	56.3	242
A 232	46.5	96.6	62.5	250

Results of constant immersion tests carried out on the steels in vinegar and citric acid plus brine solution are given in Table 20.

Table 20 — Results of Constant Immersion Tests

Heat No.	Vinegar (Loss in weight gm./dm. ² /day, time 72 hr.)	Citric acid plus 1% brine solution. Loss in weight in gm./dm. ² /day, time 72 hr.
A 231	Negligible do	0.0930 0.0040
A 232	0.0012 0.0012	0.1170 0.0730

The steels showed promising properties and are under active investigation.

30.1 Ageing Characteristics of Nickel-free Cr-Mn-N Austenitic Stainless Steel at Elevated Temperature

To study the ageing characteristics at high temperature of nickel-free chromium-manganese-nitrogen austenitic stainless steels developed at the National Metallurgical Laboratory, further experiments, in addition to the work done earlier, were carried out with the steels of heat Nos. A 160, A 171, A 175 and A 180, the chemical compositions of which are given in Table 21. Before subjecting the test specimens for ageing treatment, the test pieces were given a solution treatment (W/Q, 1050°C.). After ageing the specimens for a definite length of time at a given temperature, the test

Table 21 — Chemical Composition and Ageing Treatments of Nichel-free Cr-Mn-N Austenitic Stainless Steel

Heat No.	Composition, %					Ageing temp. °C.	Ageing period hr.	Hardness value VPN	Microstructure
	Cr	Mn	N	C	Si				
1. A 160	17.17	9.8	0.4	0.02	0.53	600	1000	249	Precipitation occurred along the austenite grain boundaries
2. A 160	—	—	—	—	—	700	1000	254	Globular patches consisting of lamellar and granular precipitates developed along grain boundaries by nucleation and growth process
3. A 171	16.65	10.7	0.20	0.03	0.38	600	1000	245	Duplex structure showing depleted austenite and elongated white patches along grain boundaries
4. A 171	—	—	—	—	—	700	1000	251	Duplex structure as A 171/600°C./1000 hr., but here precipitate was more
5. A 175	17.91	5.93	0.32	0.05	0.43	600	1000	230	Small nodules showing lamellar structure developed from grain boundaries
6. A 175	—	—	—	—	—	700	1000	230	Nodules along grain boundaries grew in size and consisted of granular type precipitates
7. A 180	17.2	8.05	0.26	0.26	0.48	600	1000	286	Dark etching nodules having lamellar form grew along the grain boundaries
8. A 180	—	—	—	—	—	700	1000	380	Nodules along grain boundaries showed lamellar and granular type precipitates

pieces were immediately quenched in water at room temperature. In case of heat Nos. A 160, A 171, A 175 and A 180, since the quantity of materials was not sufficiently available, it was not possible to carry out the corrosion tests after ageing of these specimens in different media of nitric acid, citric acid, vinegar, limejuice and salt spray, as was carried out for earlier specimens; only their microscopic examination and hardness measurements were determined. Micro-examination of the steels was carried out with the help of optical microscopes as well as with the electron microscope. Transmission electron microscopic examination of the different heat-treated specimens was carried out with the plastic replicas prepared from them by using $\frac{1}{2}$ per cent Formvar solution and follow-

ing the extraction technique of replica making.

Table 21 shows the experiments completed during the period under review and the results of their micro-examination and hardness measurements.

30.2 X-ray Diffraction Studies on the High-temperature Ageing Characteristics of Ni-free Stainless Steel

During the period under review, precipitation reactions in the low nitrogen, Cr-Mn-C-N steels were investigated at ageing temperatures of 500°, 600° and 700°C. The compositions of the steels used are given in Table 22. These steels can broadly be divided into two categories:

Table 22 — Chemical Composition of the Ni-free Stainless Steels

Steel No.	Cr %	Mn %	N %	C %	Ni %	Si %	Cu %	S %
B ₂	20.9	13.8	0.68	0.12	0.18	0.70	0.12	0.017
B ₃	21.79	18.25	0.75	0.065	0.55	0.55	—	0.012
B ₄	21.56	18.47	0.80	0.06	0.42	0.37	—	0.016
B ₅	17.71	17.55	0.526	0.08	0.45	0.28	—	0.019
A 231	21.0	14.0	0.50	0.24	0.40	—	—	0.015
A 232	21.0	13.8	0.43	0.31	0.26	—	—	—
A 233	22.1	14.2	0.54	0.32	0.42	—	—	—

Table 23 — Phases Present in Steels after Ageing for 1000 hr.

	1050°C./WQ	800°C.	700°C.	600°C.	500°C.	C/N
B	—	Cr ₂ N	Cr ₂ N	Cr ₂ N	Cr ₂ N Cr ₂₃ C ₆	0.176
B	—	do	do	do	Cr ₂ N	0.086
B	—	do	do	do	do	0.015
B	—	do	do	do	do	0.152
A 231	—	Cr ₂ N, Cr ₂₃ C ₆	do	Cr ₂₃ C ₆	Cr ₂₃ C ₆	0.48
A 232	—	do	do	do	do	0.72
A 233	—	do	do	do	do	0.57

(i) Those containing carbon in the range of 0.06-0.12 per cent.

(ii) Those containing carbon in the range of 0.2-0.3 per cent.

The precipitated phases were electrolytically extracted using an electrolyte of citric acid and ammonium chloride and graphite cathode at a current density of 0.025 amp./cm.². The extracted phases were analysed in the diffractometer, using the powder technique. The CrK_α radiation was used. The solution treated and aged samples were also examined in the X-ray diffractometer with the same radiation. The solution treatment was carried at 1050°C. and experimental observations confirmed that this treatment had taken all the carbides and the nitrides into solution.

The results of the investigation can briefly be summarized as follows:

(a) The solution-treated samples always showed austenitic phase.

(b) The nitride and carbide phases detected after ageing at various temperatures for 1000 hr. are given in Table 23.

It will be seen from Table 23 that:

(i) Cr₂N was present in all the samples excepting in higher carbon steels aged at 500°C.;

(ii) Cr₂₃C₆ was not present in the aged steels containing low carbon except in one steel at 500°C.;

(iii) There seems to be some relationship between the C/N ratio and the appearance of the nitride and carbide phases.

31.0 Development of Indigenous Alloy Steels

The objective of this project is based on the basic theme to develop 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

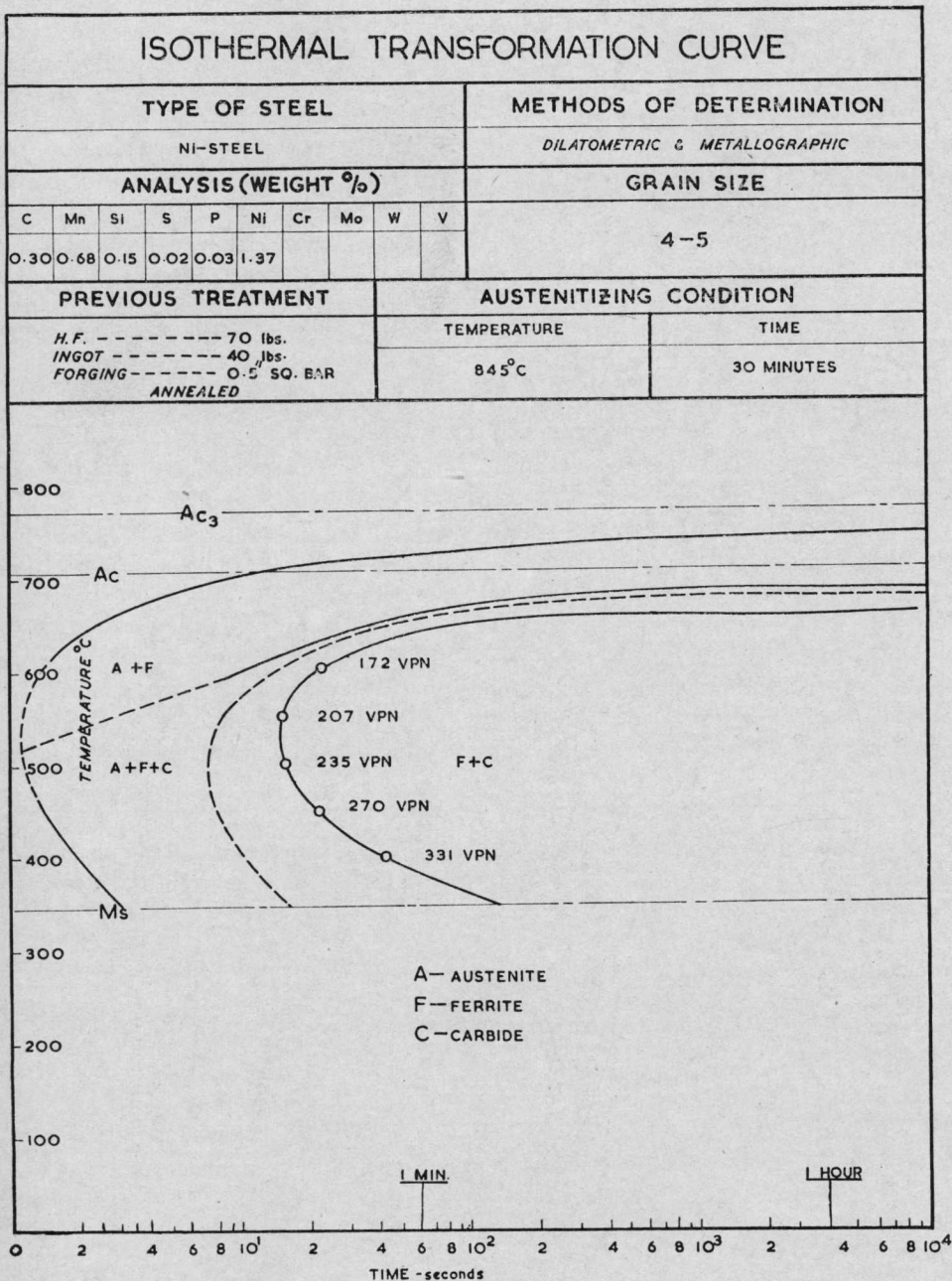


FIG. 14

performance characteristics of standard alloy steels. The project covers the important fields of formulating time-temperature-transformation and continuous cooling transformation curves, hardenability studies *vis-à-vis* ruling sections and optimum heat-treatment cycles and fabrication techniques.

(i) *Tool Steels* — During the period under review, several heats of tool steels were made with the base composition: C, 0.6-0.7; Mn, 6-7; Si, 0.2-0.3; W, 6-7; Cr, 4-5; and V, 1.0-1.5 per cent. The amount of Mn was increased to 6.8 per cent in one series, while in the other series boron was also added. Due precautions were taken while making boron addition in the melt. The amount of Mn was increased to 6.8 per cent with a view to getting higher nitrogen content in the melt. All the Mn was added in the form of nitrided electrolytic manganese. The composition and hardness of these ingots after casting, annealing as well as after forging are given in Table 24.

Further heats of tool steels were made containing 2.6 per cent Mn and 1 per cent Al, with same base composition. Alumi-

nium was added with a view to getting higher nitrogen in the melt. The hardness of the ingots in annealed conditions are given in Table 25.

The specimens from the forged bars of the previous heats made were cut and are being heat-treated to find out the correct hardening and tempering temperature as well as the hardness developed after heat treatment. During hardening treatment, specimens of TS 24 were water quenched, oil quenched, brine quenched, as well as air cooled, after austenitizing at 1250°C. The hardness developed after quenching and tempering are shown in Table 26. The hardening and tempering temperature for other steels are under investigation.

(ii) *Die Steels* — A number of experimental melts of die steels was made with the nominal compositions given in Table 27. Chromium and titanium were added with a view to developing suitable ranges of composition which are expected to yield optimum properties for use as general purpose die steels. Further work is in progress.

(iii) *Iron-Aluminium Alloys* — Additions of silicon and carbon to iron containing

Table 24 — Hardness of Tool Steel in as Cast, Annealed and Forged Conditions

Heat No.	C %	W %	Cr %	V %	Mn %	N %	B %	VPN hardness of the ingots		
								As cast	Annealed	Forged
TS 22	0.7/0.8	6	4	1.2	6	0.032	—	402	308	606
TS 23	0.7/0.8	6	4	1.2	8	0.046	—	427	327	561
TS 24	0.7/0.8	6	4	1.2	6	0.013	0.002	493	626	746
TS 25	0.7/0.8	6	4	1.2	8	0.01	0.002	436	391	412

Table 25 — Hardness of Tool Steel Containing Aluminium in as Cast and Annealed Conditions

Heat No.	C %	W %	Cr %	V %	Mn %	Al %	VPN hardness of the ingots	
							As cast	Annealed
TS 26	0.6/0.7	6/7	4/5	1/1.5	—	1.0	586	251
TS 27	0.6/0.7	6/7	4/5	1/1.5	2	1.0	622	287
TS 28	0.6/0.7	6/7	4/5	1/1.5	4	1.0	543	602
TS 29	0.6/0.7	6/7	4/5	1/1.5	6	1.0	481	550

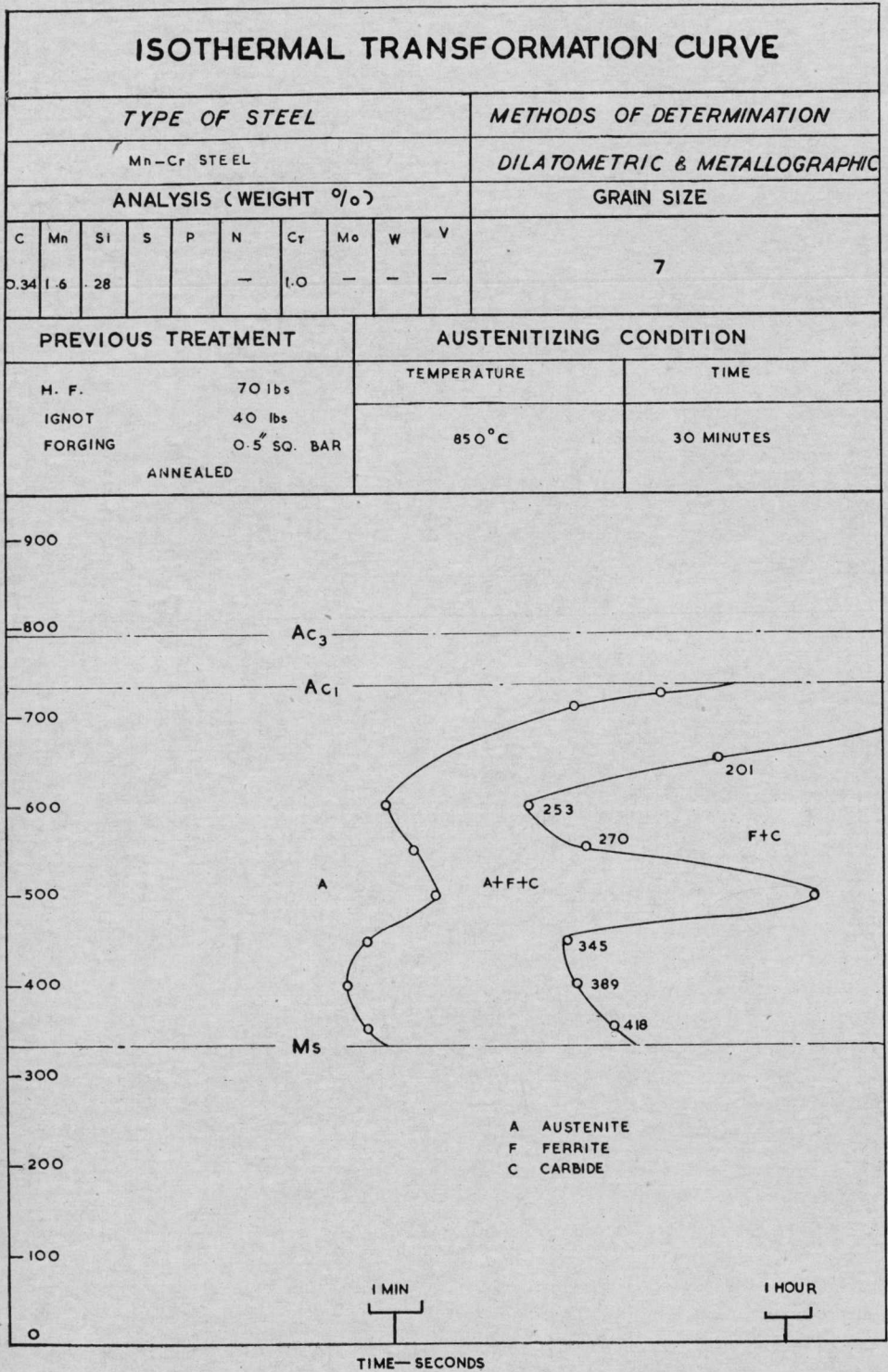


FIG. 15

Table 26 — Hardness of TS 24 after Heat Treatment

Heat No.	Hardening				Tempering in salt bath			
	Temp. °C.	Time min.	Quenching media	VPN hardness	1 hr.		2 hr.	
					Temp. °C.	VPN hardness	Temp. °C.	VPN hardness
TS	1250	6	Air	358	600	415	600	358
	1250	6	Oil	353	600	398	600	383
	1250	6	Water	344	600	400	600	362
	1250	6	Liquid air	348	600	449	600	358

Table 27 — Chemical Composition of Die Steels

Heat No.	C %	Si %	Mn %	Cr %	V %	Ti %
DS 1	0.8/0.9	0.30	0.60	1.0	—	—
DS 2	0.8/0.9	0.30	0.60	2.0	—	—
DS 3	0.8/0.9	0.30	0.60	—	—	0.50
DS 4	0.8/0.9	0.30	0.60	—	0.25	—
DS 5	0.8/0.9	0.30	0.60	—	0.59	—

Table 28 — Mechanical Properties of Some Iron-Aluminium Alloys

Heat No.	% Composition			Ingot As cast hardness VPN	Hot-rolled 0.1" thick sheet	
	Al	Si	C		Max. stress t.s.i.	% Elongation
1S	7.36	1.65	0.2	364	54-64	1.5
2S	8.85	0.78	0.8	327	59.55	Nil
3S	7.20	1.1	1.0	425	Not workable	

7.9 per cent aluminium alloys were tried with a view to increasing the fluidity of melt as the binary alloys of iron and aluminium are viscous. Heats were deoxidized with calcium. Cast ingots were annealed at 800°C. Forging and rolling were carried out from 1150°C. down to 800°C. Results obtained are given in Table 28.

Due to lack of workability and mechanical properties, it was clear that these alloys could not be used in wrought condition.

Additions of vanadium to fix the carbon as vanadium carbide (V.C.) were made with the results as given in Table 29. Composition 1V appeared to have good strength and ductility.

Effect of niobium additions to iron-aluminium (8 per cent) alloys was also studied. Heats were carried out in the 6 kW. H.F. furnace. Results obtained are given in Table 30.

It was found that niobium additions gave good results. Thus, Fe-Al (7.8 per cent)-Nb (2.3 per cent) alloys have a tensile strength of 55-60 t.s.i. and elongation of 12.5 per cent. Some work was initiated on Fe-Al-Mn alloys to prepare cheap stainless steels. A composition Fe-Al (6.8 per cent)-Mn (20-25 per cent) was developed which had a tensile strength of 45 t.s.i. and elongation 11.7 per cent, with good corrosion resistance. Further work is in progress to develop iron-aluminium alloys

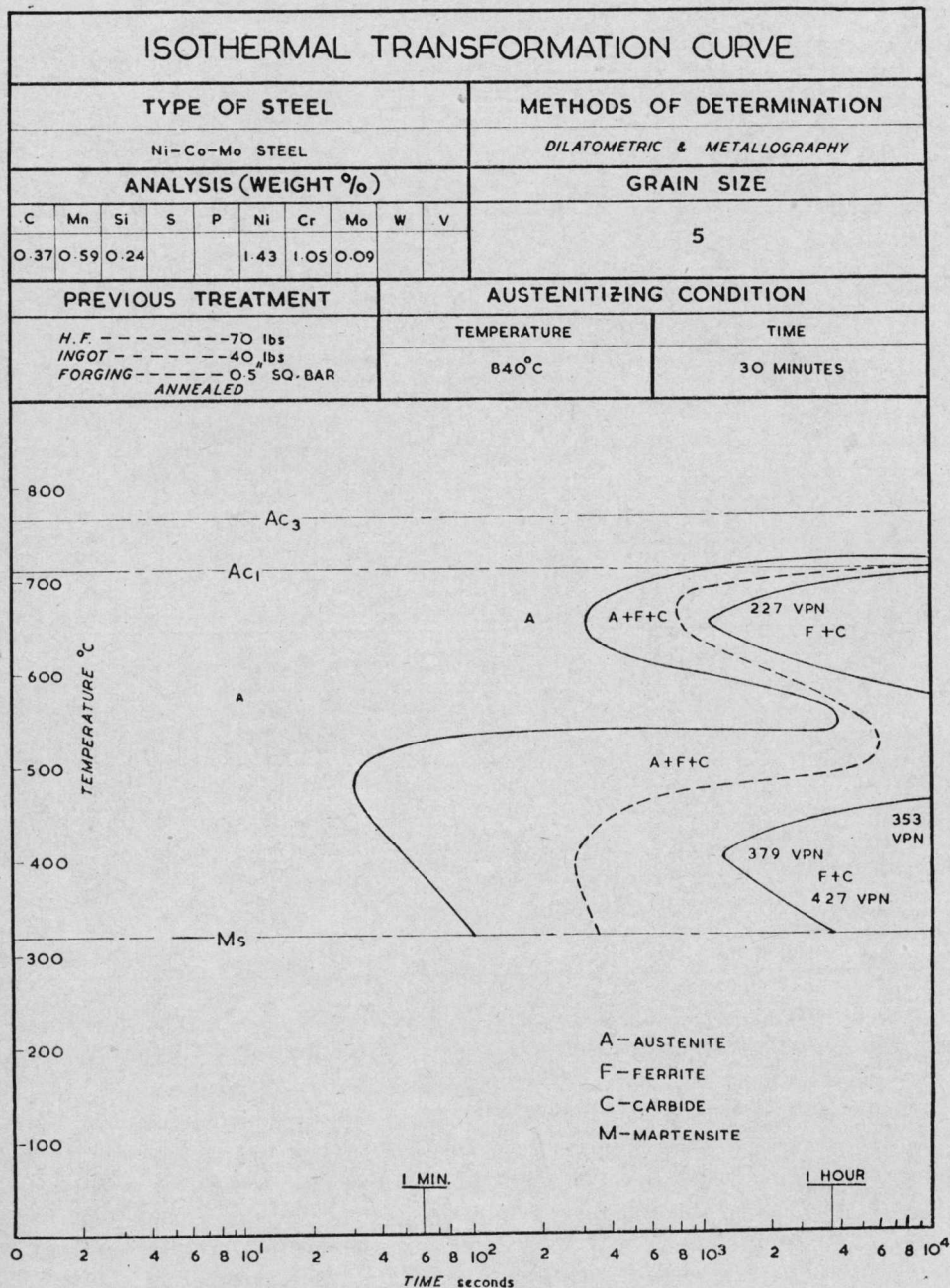


FIG. 16

Table 29 — Effect of Vanadium Addition to Iron-Aluminium Alloys

Heat No.	% Composition				Ingot As cast hardness	Hot-rolled 0.1" thick sheet	
	Al	Si	C	V		Max. stress t.s.i.	% Elongation
1V	13.0	0.17	0.2	0.91	314	46.93	12.5
2V	10.97	2.06	0.37	0.15	468	Brittle. Test piece broke within the grip	
3V	6.2	1.8	0.2	1.94	272	Could not be rolled	

Table 30 — Effect of Niobium Addition on Fe-8 per cent Al Alloys

Heat No.	% Composition		Average hardness		Mechanical properties of 0.1" thick hot-rolled sheet		Salt spray corrosion results; loss in weight mg./dm. ² in 42 hr.
	Al	Nb	As cast	As rolled	Max. stress t.s.i.	% Elongation	
NB 1	8.07	0.79	254	299	58.95	3.1	354, 329
NB 2	8.13	1.61	277	287	50.81	6.3	300, 300
NB 3	7.82	2.15	335	312	56.63	12.5	298, 289
NB 4	7.54	2.64	269	300	58.56	12.5	Not tested

Table 31 — Composition of Steels Studied

Steel	C %	Si %	Mn %	Ni %	Cr %	Mo %
1. 1-½% Mn steel	0.36	0.23	1.48	—	—	—
2. Nickel steel	0.30	0.15	0.68	1.37	—	—
3. Manganese-chromium steel	0.34	0.28	0.60	—	1.0	—
4. Nickel-chromium-molybdenum steel LA 12.3	0.37	0.24	0.54	1.43	1.05	0.09
5. Chromium-molybdenum steel	0.37	0.26	0.8	—	1.3	0.21

comparable in heat and corrosion resistance to some categories of stainless steel and possessing superior nuclear properties.

(iv) *Isothermal Transformation Characteristics of Alloy Steels*—This project was taken up with a view to preparing an atlas of the isothermal transformation diagrams of Indian alloy steels.

For the study of transformation characteristics, number of heats were made in the high-frequency furnaces. Chemical composition of most of the heats were selected from the draft schedule IV of Indian Standard Institution for alloy and special steels. Ingots of steel studied were forged into

$\frac{5}{8}$ in. sq. bar, $1\frac{1}{4}$ in. sq. bar and $1\frac{1}{2} \times \frac{1}{2}$ in. flat respectively. The forged pieces were homogenized for 24 hr. at 950°C.

The isothermal transformation study of the steels completed are given in Table 31.

(v) *Continuous Cooling Transformation of Alloys*—The project was undertaken with a view to studying the continuous cooling transformation characteristics of Indian alloy steels employing the dilatometric technique. The dilatometer was designed and fabricated at the laboratory for the study of the continuous cooling transformation characteristics of alloy steels, during oil cooling of bars up to 152.40 mm. dia. in which the

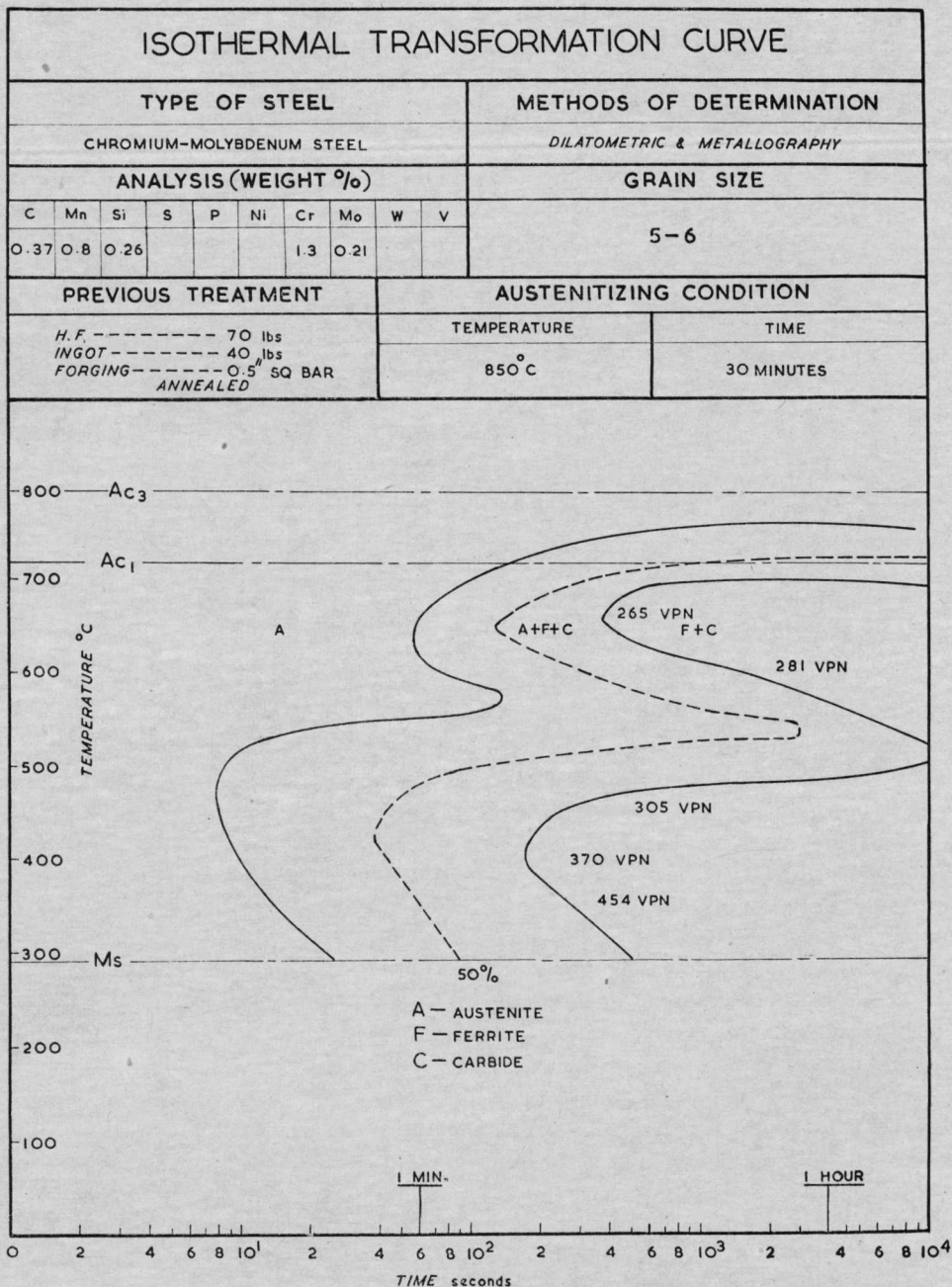


FIG. 17

austenitizing furnace was moved up and down by a pulley arrangement. After austenitizing, the furnace was instantly lowered and the specimen was allowed to cool in air. The drop in dilatation was recorded simultaneously. Continuous cooling transformation curves were plotted from such data. Microstructure and hardness of the dilatometric specimens were also studied. Continuous cooling transformation diagrams of the steels shown in Table 32 were completed.

Table 32 — Composition of Steels Studied

Steel	C %	Si %	Mn %	Cr %	Mo %
Mn-Cr	0.34	0.28	1.6	1.0	—
Cr-Mo	0.37	0.26	0.80	1.30	0.21
Mn-steel	0.38	0.22	1.5	—	—

32.0 Development of Low-alloy High-tensile Structural Steel

This broad-based investigation was taken up with the objective of developing suitable low-alloy structural steel possessing adequate mechanical properties, hot and cold working characteristics, optimum corrosion resistance and favourable welding characteristics based essentially on indigenous alloying elements.

Earlier, a large number of experimental heats was made with slight modifications in the percentages of alloying elements of the existing low-alloy high-tensile steel. Main alloying elements in these heats were manganese, chromium, silicon and copper. The effect of additions of small percentages of vanadium and titanium, which harden the ferrite as well as improve the grain structure, was studied on a few selected compositions wherein improvement in mechanical properties were observed. Simultaneous increase of manganese and chromium in a series of heats produced air-hardening type of steels. In another series, the effects of the additions of silicon on a

few selected compositions were studied. It was found that silicon showed the tendency to improve the tensile properties, especially the yield ratio in presence of copper. The presence of silicon is also not harmful for both arc and gas welding. During the period under review, study was therefore continued in this particular line. Two basic compositions were selected for this purpose. One was selected for making high-tensile corrosion-resistance steel to give a tensile strength above 50 t.s.i. and elongation of not less than 25 per cent; the other composition was selected for making weldable corrosion-resistance steel to give a tensile strength between 40 and 50 t.s.i. The compositions studied are given in Table 33.

Table 33 — Compositions of Steels Studied

(i)	C	0.15-0.25%	Mn	1.2-1.8%
	Cu	0.3-0.4%	P	< 0.09%
	Ti or V	0.1-0.2%		
(ii)	C	0.09-0.12%	Mn	0.9-1.4%
	Cu	0.2-0.4%	P	< 0.08%
	V	0.1-0.15%		

Chemical compositions of the heats made in this connection are given in Table 34. In the case of weldable steels, silicon content was varied from 0.1 to 0.8 per cent and in the case of high-tensile steel, variation of silicon content was made from 0.5 to 1.1 per cent. The charge was calculated for making 20 lb. heats and two ingots were made out of the total charge. All the heats were made in the high-frequency furnace. Hot working of the ingots was carried out at 1150°C. Suitable sections were made for making test pieces for carrying out different tests. Tensile tests were carried out on specimens having 0.282 in. dia. and 1 in. gauge length. For bend test, sheets of the order of 0.1 in. thick were used. Weldability tests of all these steels were made by Reeves Fillet test method (miniature). All the test results are given in the Table 34.

Table 34 — Chemical Composition and Mechanical Properties of Low-alloy High-tensile Structural Steels Studied

Heat No.	Analysed chemical composition (%)										Tensile test results				Results of weldability tests	Hardness values after				Bend test results	
	C	Mn	Cr	Si	P	Cu	V	Ti	Al	Max. stress t.s.i.	Yield stress t.s.i.	El. %	R.A. %	Ann.		Nor.	O.Q.	W.Q.	R: 5mm	Remarks on 180° flat bend	
37/4R	0.25	1.20	0.04	1.10	0.09	0.41	—	0.13	—	51.11	44.68	28.0	58.3	N.W.	185	219	445	476	Nil	Failed	
40/1	0.17	1.76	—	0.51	0.067	0.37	—	0.20	—	55.80	—	25.0	43.6	N.W.	159	235	346	404			
40/2	0.19	1.35	—	0.50	0.063	0.39	—	0.18	—	41.46	27.90	25.0	48.7	W	158	181	278	422	1.2	Withstood	
41/1	0.21	1.42	—	0.61	0.012	0.38	0.20	—	—	56.18	46.80	25.0	56.3	N.W.	168	243	334	473	3.2,3	do	
45	0.18	1.38	—	0.96	0.09	0.36	0.18	—	—	52.01	43.60	23.4	54.8	N.W.	174	204	273	473			
37/5	0.32	1.50	0.02	0.56	0.07	0.41	—	0.126	—	77.73	—	18.7	40.6	—	197	227	523	—	1	Failed	
41/2	0.09	1.11	—	0.26	<0.04	0.45	0.10	—	—	43.20	36.72	29.7	65.5	W	147	222	371	376	5.4	Withstood	
41/3	0.09	1.12	—	0.46	<0.04	0.43	0.11	—	—	48.60	37.44	28.0	64.7	W	157	243	409	420		do	
41/4	0.10	1.10	—	0.31	<0.04	0.42	0.10	—	—	40.67	30.58	31.0	63.4	W	130	214	325	329	4.5		
40/3	0.07	0.63	—	0.20	<0.04	0.39	—	0.011	—	28.44	19.44	37.5	72.6	—	109	140	180	—			
43	0.10	0.81	—	0.37	0.08	0.44	0.18	—	—	35.30	30.38	34.4	71.3	W	133	143	—	191			
44	0.90	0.96	—	0.73	0.08	0.39	0.13	—	—	39.68	31.81	34.4	64.9	W	147	164	—	257			
49	0.12	1.20	—	0.13	0.05	0.19	—	—	Trace	35.39	23.59	34.0	60.0	N.W.	135	164	391	436			
50	0.10	1.40	—	0.27	0.08	0.427	—	—	do	40.04	19.87	33.0	61.0	W	148	186	349	353			
37/4	0.44	0.96	0.12	1.35	0.07	0.47	—	Trace	—	59.12	46.08	25.0	59.3	N.W.	206	309	520	675	Nil	Failed	
47	0.104	1.50	0.53	0.12	0.05	0.176	—	—	Trace	46.82	22.45	23.0	45.0	W	153	239	381	402			
48	0.24	1.40	1.57	0.54	0.06	0.188	—	—	do	66.77	—	1.5	—	N.W.	203	346	419	455			
46	0.17	1.12	1.35	0.18	0.03	0.30	—	—	—	57.73	31.81	17.2	30.0	N.W.	172	276	387	405			

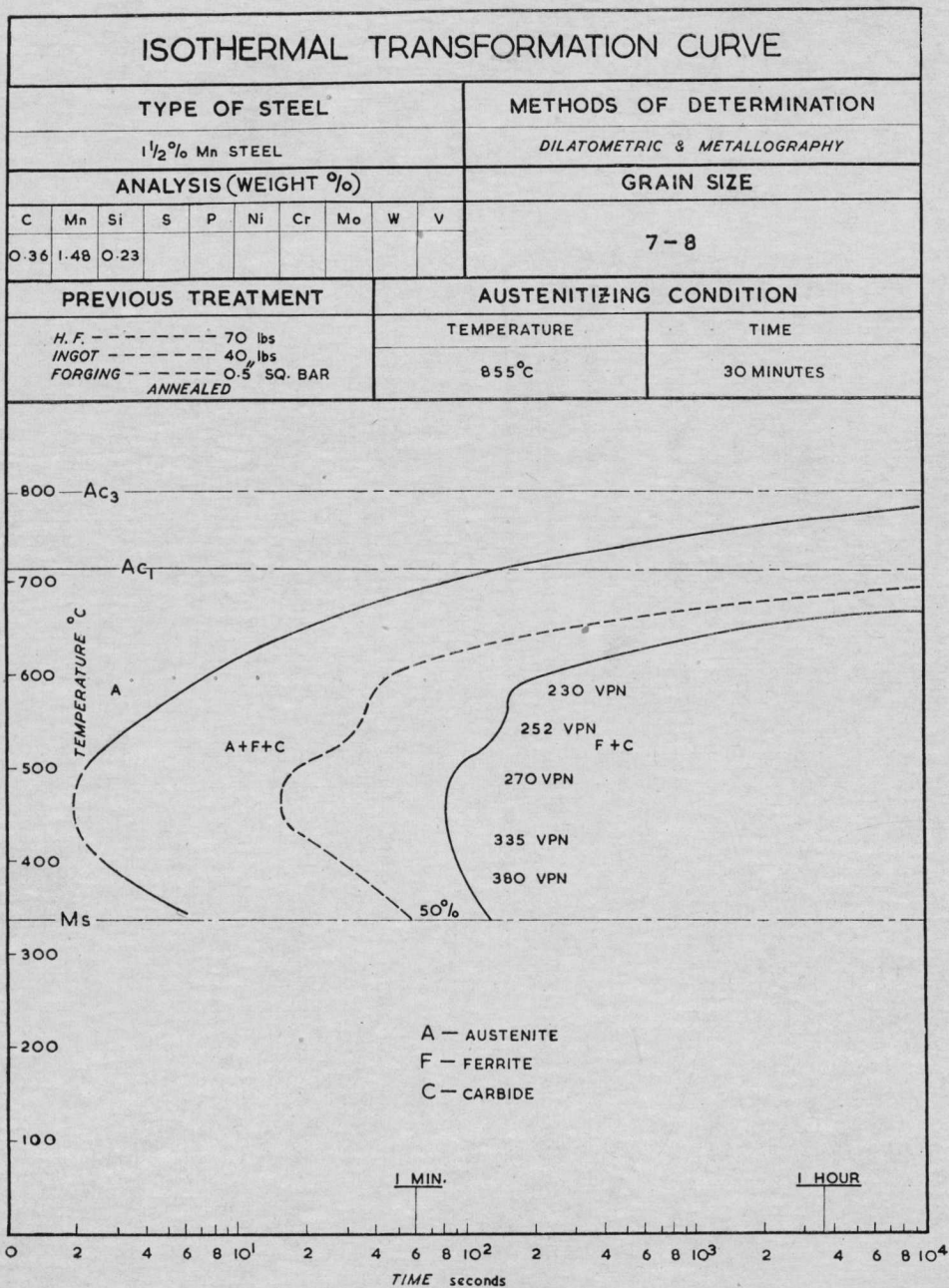


FIG. 18

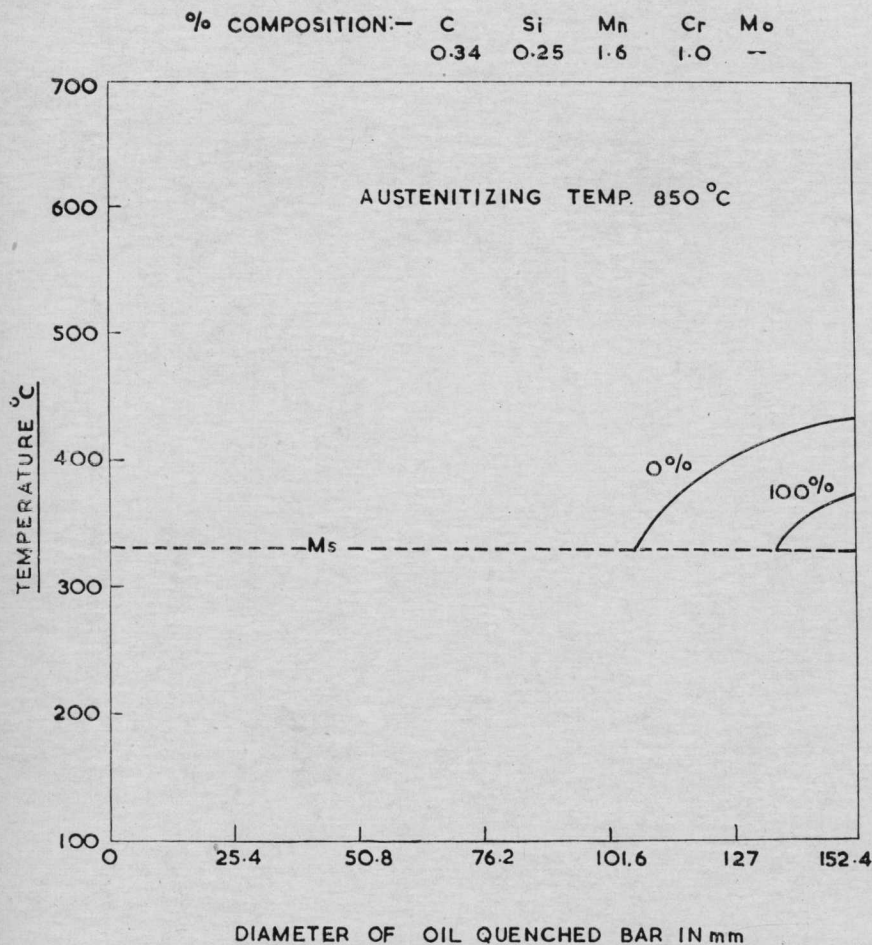


FIG. 19 — CONTINUOUS COOLING TRANSFORMATION DIAGRAM OF Mn-Cr STEEL

First six heats in Table 34 were made to produce high-tensile steels according to the first specification mentioned earlier. It was seen that except heat No. 40/2 all of them gave tensile strength over 50 t.s.i. and yield strength was remarkably high. Almost all of them had an elongation of the order of 25 per cent. Heat No. 37/5 could not give the required elongation probably due to higher carbon content than specified. Heat No. 40/2 which gave lower tensile strength had, however, been found weldable. Next eight heats in the same table were made to pro-

duce weldable grade. All of them were found to be weldable according to the results obtained from Reeves Fillet test method (miniature). Tensile test results of heat No. 41/3 among these heats are noteworthy. It gave tensile strength of 48.6 t.s.i., yield strength 37.44 t.s.i. and elongation percentage 28.

Sub-zero impact tests, accelerated corrosion tests as well as atmospheric corrosion tests of these steels are in progress. Heat No. 37/4, whose carbon and silicon contents were abnormally high for the particular type of steel aimed at, gave very

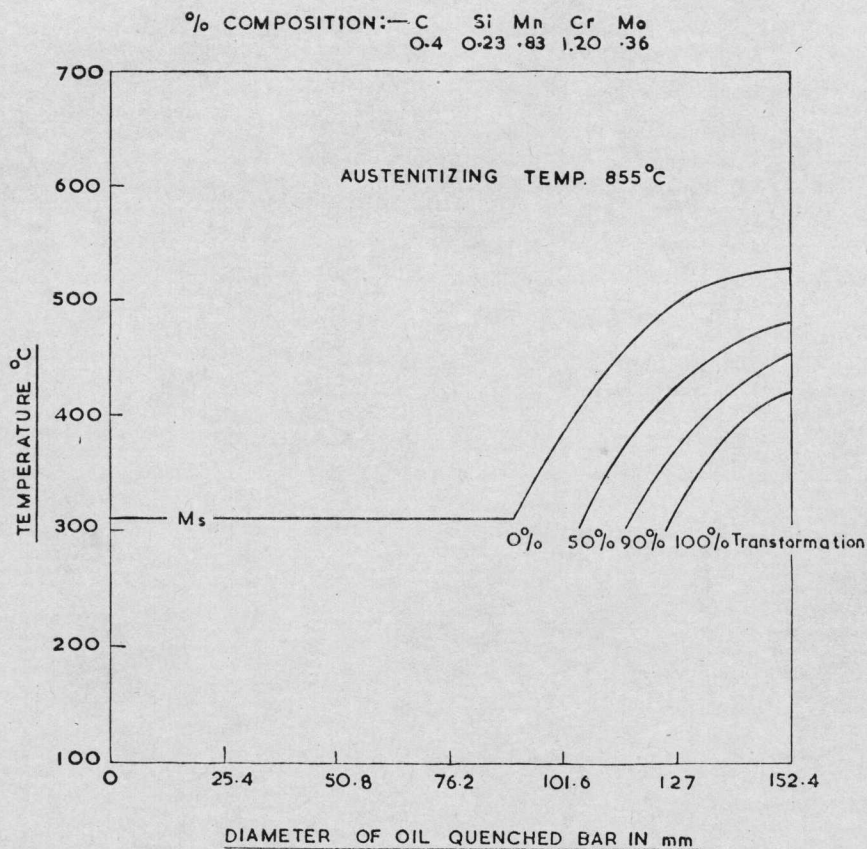


FIG. 20 — CONTINUOUS COOLING TRANSFORMATION DIAGRAM OF Cr-Mo STEEL

interesting tensile properties. It gave a tensile strength of 59.12 t.s.i. with 25 per cent elongation. Yield stress was 46.08 t.s.i. and the yield ratio was found to be 0.796. Bend test results and weldability test results of this steel were of course not satisfactory. The last three heats also contained chromium. The addition of chromium was made using a master alloy of Fe-Cr-Al. Attempts were made to keep the alloy content within the following limits: Mn, 1-1.5; Si, 0.2-0.5; Cu, 0.2-0.3 per cent; carbon and chromium were variables. The hardness results showed that when carbon content was more than 0.15 per cent and chromium more than 1 per cent, the steel produced showed the tendency of air-hardening.

After making a critical study of the test results including weldability and corrosion resistance of the steel, tonnage heats would be made. Work in connection with further improvements in chemical composition from the point of economy of alloying elements and cost of production will also be taken up.

33.0 Development of Electrical Resistance Alloys for Heating Elements

Conventional types of heating elements used for domestic or industrial heating appliances contain high percentages of nickel and some cobalt. Resources of nickel and cobalt do not exist in India. With

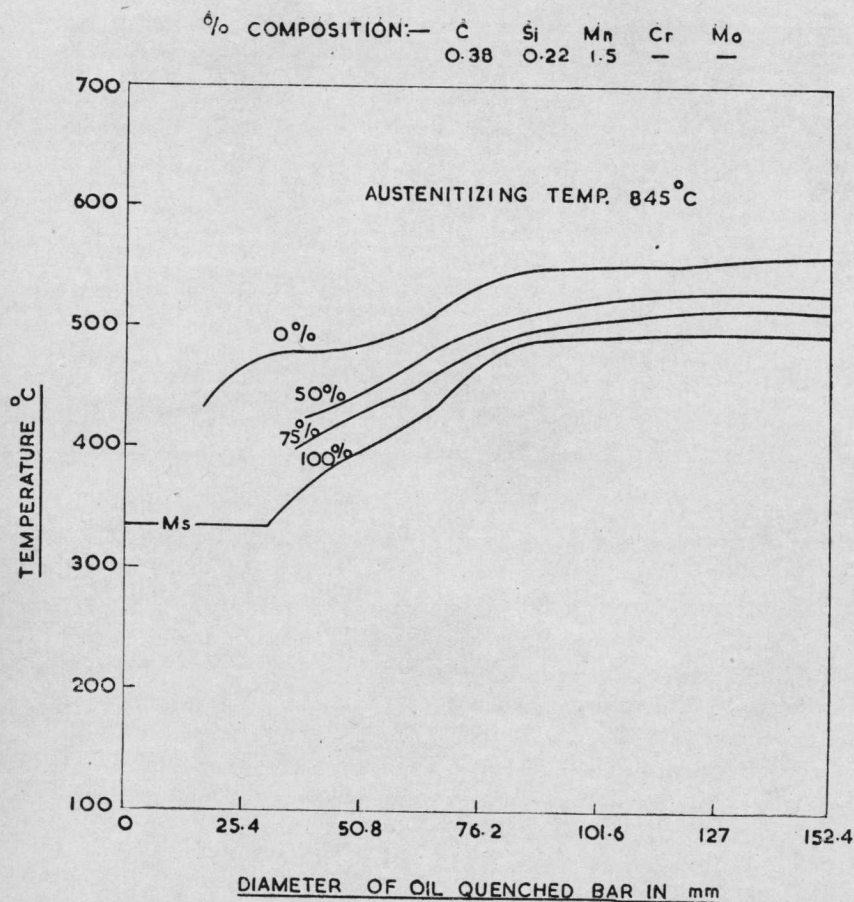


FIG. 21 — CONTINUOUS COOLING TRANSFORMATION DIAGRAM OF Mn STEEL

increase in availability of electric power and industrial growth, demand for such heat-resistance electrical elements for domestic and industrial heating applications will substantially increase. With a view to developing electrical heat-resistance elements containing no nickel or cobalt, intensive research and development work was taken up which lead to the development of indigenous based electrical heating elements. The work was centred round Fe-Cr-Al system based on various considerations including availability of raw materials from indigenous resources and the requirements of physical and mechanical properties for

suitable heating element. As reported earlier, several heats were made using Mn, Si, Zr, V, misch metal, etc., in high-frequency furnace. The hot and cold working behaviour of these alloys were studied and their physical and mechanical properties were also determined. It was seen that the heats which contain relatively higher percentage of aluminium and low percentage of chromium showed better life.

During the year, work was carried out in the range of the basic analysis of heat No. 4E31 which had shown considerably higher life as compared to other heats. In most of the cases, 20 lb. heat was prepared in

Table 35 — Chemical Composition and Hardness of Steels Studied

Heat No.	Chemical composition, %								Hardness (B.H.N.)	
	Cr	Al	Si	Mn	Zr	C	Ce	Ti	Cast	Forged
1A	12.15	6.04	0.64	0.31	0.05	0.05	Trace	—	200	215
1B	13.63	6.23	0.66	0.34	0.05	0.04	do	—	201	224
2	14.00	7.45	0.58	0.17	0.52	0.03	do	—	220	256
3	13.90	7.81	0.92	0.14	0.45	0.05	do	—	233	—
4	13.70	6.74	0.88	0.19	0.51	0.07	do	—	229	222
18E31A	12.63	4.90	0.72	0.70	0.33	0.10	0.18	—	229	—
B	12.57	6.00	0.70	0.73	0.39	0.006	0.18	—	225	217
19E31A	12.13	4.70	0.51	0.10	0.49	0.06	0.06	—	202	214
B	12.64	7.80	0.16	0.15	0.02	0.05	0.11	—	212	222
20E31A	—	—	—	—	—	—	—	—	211	—
B	12.68	5.30	0.13	0.13	0.04	0.05	0.12	—	207	231

the high-frequency furnace using low-carbon ferro-chrome, aluminium, ferro-zirconium, misch metal and low-residue iron made in NML as substitute for Armco iron, as charge. The general procedure was to cast the 10 lb. alloy ingots, machine the surface to remove the scale, cracks, etc., and hot forge to about 1 in. sq. slab at 1200°C., taking care of finishing temperature and forging reduction. These were again machined to remove the surface defects and the slabs after soaking at about 1050°C. finally were hot-rolled to $\frac{3}{8}$ in. rods. The finishing temperature of rolling had to be controlled to have a fine grain size product. The rods were pickled to remove surface defects and cold-drawn using suitable lubricants. Annealing was done at 750°C. after 2-3 passes in successive tungsten carbide dies. The chemical compositions of seven heats with their hardness, in both cast and forged conditions are given in Table 35.

It can be seen from the analysis that the composition is controllable by the use of low-residue iron made in NML. One of the ingots from the last three series in Table 35 was slowly cooled in gas-fired furnace (1050°C.). The other ingot was cooled in air as usual, with a view to observing the effect of controlled cooling on the workability of the ingots. The forgeability of

all the heats was good excepting heat No. 3 with higher pouring temperature which showed cracking along the centre connecting the central pipe in the ingot. The heats 2ET3, 17E31, 18E31, 19E31 showed good drawability. The heat No. 1 (A, B) failed during initial stage of drawing due to heavy coarse grain size of the rod. The other heats are being drawn.

Accelerated Life Test

Five specimens were tested at 1150°C. Comparatively shorter life of the heat No. 18E31 is perhaps due to high-carbon and manganese content. The life test results are given in Table 36.

Table 36 — Data on Life Test

Material	Temperature °C.	Accelerated life hr.
2ET3	1150	75
6E31	1150	132
17E31	1150	71
4E31	1150	250
18E31	1150	32

Weldability Test

Some wires of $\frac{1}{8}$ in. dia. were welded by butt welding under different settings of

pressure and currents. In many cases, the fracture during tensile loading was found to be of brittle nature mainly along the welded zone in which coarse grains were noticed. Experiments are being conducted so as to get a ductile weld for wire-drawing purpose.

High-temperature Oxidation and Grain Growth Study

High-temperature grain growth study of heat Nos. E37 and 4E31 was made at 1200°C. to assess the effect of grain growth inhibiting alloying elements. Both the wires were subjected to prolonged heating up to 200 hr. Microstructure examination results showed that the sample E31 exhibited much greater grain growth inhibiting tendency than 4E31 which appeared to be due to higher aluminium content of the latter. The rate of oxidation of these wires was also measured simultaneously with the grain growth study. The die of both the wires was 0.028 in. The wires were weighed and the increase in weight per unit area of the surface was calculated. Results are given in Table 37.

Table 37 — Rate of Oxidation of the Wires Studied

Sample No.	Increase in weight/area after various periods, gm./cm. ²			
	After 19.5 hr.	After 47 hr.	After 95.5 hr.	After 143.5 hr.
E31	0.1476	0.2148	0.2804	—
4E31	0.1998	0.2789	0.3447	0.3356

Service Test with Domestic Heater

The service trial of the domestic heater (1000 watts capacity) with NML heating element was continued. The heater had already completed more than two years of service at the rate of intermittent heating with actual heat on period of about 42 hr. a week, and is still working.

More heats are being made conforming to the basic compositions of heat No. 4E31 in the 20 lb. high-frequency furnace using low-residue iron made at NML. Studies are also under way to standardize the condition of working behaviour of the alloys as also the grain growth study of two similar alloys, one with addition of Zr and misch metal and the other with the addition of zirconium only. Arrangement is also being made to study the effect of prolonged heating of 4E37 or 6E31 and a similar alloy with misch metal at 1150°C. in nitrogen atmosphere.

34.0 Development of Nickel-free Coinage Alloy

Although nickel is eminently suitable for coinage alloys, its non-availability in India poses a real problem. This project was taken up to develop alloys containing manganese as one of the constituents and having metallurgical and physical properties identical with the present cupro-nickel coinage alloys.

Blanks cut from alloys made at the India Government Mint, Bombay, showed red streaks after annealing and polishing. Further tests were carried out at the Bombay Mint by varying the temperature of soaking of ingots, mode of rolling in the cogging mills, etc. The samples were brought from the mint and thoroughly examined for the various defects. On the basis of observations made during different trials, it was concluded that streaks were formed on the rolled sheets due to the development of fine cracks on the surface of the ingots during the initial stages of hot rolling. These cracks once formed do not penetrate deeper, but elongate during subsequent rolling to give rise to fine streaks. By increasing the rolling temperature from 750° to 825°C., a marked improvement in the appearance of the surface was observed. Decrease in the amount of reduction during each stage was studied by increasing the

number of passes. This was also found to have a marked influence on reducing the number of surface cracks.

35.0 Development of Manganese Bearing Brasses

Brass is extensively used in India for utensil making. Due to 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.

In continuation of the previous studies, corrosion-resistance properties of binary copper-manganese alloys with manganese varying between 0 and 40 per cent was evaluated by salt spray test. Manganese was found to decrease the corrosion-resistance properties of copper. Studies on effect of addition of zinc or nickel to copper-manganese alloys on their resistance to corrosion is in progress.

A detailed report on the properties of various copper-manganese alloys is under preparation. The report will include effect of annealing and different degree of cold working on physical properties, effect of manganese on specific gravity of the alloys, etc.

Studies on electrical and thermal conductivity of binary copper-manganese alloys were continued. Experimental set-up is being made to study in detail the changes in electrical resistivity in the temperature range of -160° to -700°C .

36.0 Development of Magnetic Materials

The demand for magnetic materials is multiplying in India following the rapid growth of telephone, electronics and other industries. At present, the quantity needed is mostly met by import. This project was 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:

- (i) Tempering characteristics of some Alnico type permanent magnets.
- (ii) Study of magnetic and electrical properties of ceramic permanent magnets.
- (iii) Study of binary and ternary alloys of manganese-aluminium iron.

(i) *Tempering Characteristics of Some Alnico Type Magnets*—The effect of tempering between 600° and 900°C . on the magnetic properties of some Alnico magnets was reported earlier. Work was taken up to study the structural changes taking place during tempering by electron microscopy and X-ray diffraction. The electron photographs showed the presence of only one phase in the specimen heat-treated for best magnetic properties, but a second phase was distinctly observed in the specimen heated for 200 hr. at 600°C .; at higher temperatures, however, it appeared in the early stages of tempering, preferentially along certain crystallographic planes. Most of the X-ray diffraction was carried on the tempered Alnico specimens with the diffractometer. A few powder X-ray photographs of the specimen were also taken in the 5.7 cm. camera. The results of the X-ray observations could be summarized as follows:

- (a) The specimen in the optimum heat-treated condition showed (i) B.C.C. structure of lattice parameter approximately equal to that of iron and (ii) an additional intense line.
- (b) On tempering at 600° and 700°C ., this additional line persisted in the early stages of tempering but disappeared on prolonged heating. The presence of this line coincided with the development of high-coercive force.
- (c) On tempering at 800° and 900°C ., lines corresponding to a F.C.C. structure

appeared in addition. No relationship between coercive force and the appearance of this structure could be seen.

Work on this project has since been completed.

(ii) *Study of Magnetic and Electrical Properties of Ceramic Permanent Magnets of Hexagonal Structure* — As was reported earlier, the main problem in the production of ferrites is to prevent grain coarsening to obtain consistent magnetic properties. The study of the effects of ternary oxide addition on (i) sintering temperature and time, (ii) grain growth, and (iii) magnetic properties of ferrites was continued.

The sintering of five mixtures was carried at 1080°, 1176°, 1226° and 1280°C. for durations of 30 min., 2 hr., 4 hr. and 8 hr. The sintering results showed that (i) shrinkage of pure barium ferrite compacts increased with increasing time at any particular temperature; (ii) rapid shrinkage in the early stages of sintering; (iii) maximum shrinkage at 1280°C. after

4 hr. of sintering when the shrinkage value was about 22 per cent; (iv) shrinkage was not materially changed in specimens where the addition of ternary oxide had caused an increase in the coercive force; and (v) increased shrinkage to 30-35 per cent for increased additions of ternary oxide, but the coercive force correspondingly decreased.

Maximum coercive force and remanence for barium ferrite sintered compacts was about 1750 oersteds and 1400 gauss respectively after sintering at 1176°C. for 30 min. The coercive force, however, decreased to 1020 oersted and remanence increased to 1750 gauss when sintered at 1280°C. for 8 hr. The marked change in properties was attributed to greater growth of grains at 1280°C. Small additions of ternary oxide increased the coercive force and remanence values. It also inhibited grain growth with the result that coercive force and remanence did not appreciably change over a range of sintering temperatures and time. The maximum coercive

Table 38 — Coercive Force and Remanence Values for Various Mixtures after Sintering at Temperatures between 1080° and 1280°C. for Various Duration of Time up to 8 hr.

Temp. °C.	Time of sintering	Mixture numbers									
		C-0		C-1		C-2		C-3		C-4	
		H _C	B _R	H _C	B _R	H _C	B _R	H _C	B _R	H _C	B _R
		(Oersted)	(Gauss)	(Oersted)	(Gauss)	(Oersted)	(Gauss)	(Oersted)	(Gauss)	(Oersted)	(Gauss)
1080°C.	30 min.	1181	1285	1776	1587	2227	1636	1907	1528	1750	1422
	2 hr.	1946	1357	1804	1730	1946	1692	1941	1392	1881	1424
	4 hr.	1632	1431	2085	1656	1897	1834	1512	1395	1830	1428
	8 hr.	1791	1612	2197	1703	1992	1770	—	—	—	—
1176°C.	30 min.	1750	1426	2160	1930	1897	1934	1750	1666	1980	1589
	2 hr.	1677	1698	2187	1907	2012	1903	1909	1383	1806	1615
	4 hr.	1677	1473	2162	1920	1897	1824	2067	1664	1590	1458
	8 hr.	1560	1829	2190	1812	2068	1993	—	—	—	—
1235°C.	30 min.	1587	1521	2050	1800	1942	1714	1629	1798	1322	1307
	2 hr.	1750	1511	2033	1834	1817	1797	1557	1639	1326	1309
	4 hr.	1512	1958	1970	1771	1760	1365	1427	1784	907	1248
	8 hr.	1591	1511	2040	1802	1307	1215	—	—	—	—
1280°C.	30 min.	1591	1750	1905	1804	1341	1656	1752	1735	—	—
	2 hr.	1454	1830	1970	1860	1166	1400	1326	1556	—	—
	4 hr.	1342	1741	1772	1763	1136	921	1193	1553	—	—
	8 hr.	1020	1500	1712	1777	1061	811	—	—	—	—

force observed on addition of oxide was 2150 oersted and remanence 1900 gauss. The beneficial effect of the oxide additions was, however, limited to small percentage only. The electrical resistivity of the sintered ferrites was also measured and was of the order of 10^5 micro-ohm-cm.

(iii) *Study of Binary and Ternary Manganese-Aluminium-Iron Alloys* — It has recently been reported that interesting permanent magnetic properties have been found in manganese-aluminium alloy at composition $Mn_{1.1}Al_{0.89}$ having tetragonal structure. It was, therefore, decided to study the magnetic properties of the binary alloys of manganese and aluminium in the composition around $Mn_{1.1}Al_{0.89}$. Attempts to make the alloys using indirect arc and gas-fired furnaces proved abortive due to considerable pick-up of carbon and excessive oxidation. Powder metallurgy technique was, therefore, adopted to prepare the alloys from electrolytic manganese and pure aluminium each of 200 mesh. The powders were weighed, mixed thoroughly, and pressed into compacts at a pressure of 47.25 kg./mm.². The compacts were then sintered at 1050°C. Preliminary work so far carried out showed that considerable volume expansion was associated with the alloy formation. The alloy was weak and brittle. The alloy powder was subsequently repressed into compacts under the same load and resintered at 1050°C. The resintered compacts did not exhibit volume expansion. The alloys were made ferromagnetic after cooling from 1150° to 600°C. at a rate of 30°C./second. Detailed study is in progress.

37.0 Tinless Bearing Alloys

With a view to replacing tin by lead in aluminium-base bearing alloys, experiments were carried out to obtain uniform distribution of lead in aluminium. Injection of lead dioxide in molten aluminium using nitrogen as a carrier gas presented a lot of

difficulties such as dross formation and excessive oxidation of aluminium at temperatures of 760°-820°C. The alloys so produced had a lead content of about 1.2-2 per cent. The microstructure showed oxide inclusions and segregation of lead. Experiments with lead powder obtained by atomization technique added to molten aluminium gave a better microstructure than the technique using lead oxide injection. However, on analysis the percentage of lead in the alloy was found to be only between 1.24 and 1.95. It is known that it is difficult to get good bearing properties in the alloy with such low-lead contents. The lead content retained in some typical heats is shown in Table 39 (the lead powder used for the experiments assayed 97.41 per cent total lead).

Table 39 — Lead Retained by Atomization Technique

Heat No.	Lead content %
A ₁	1.24
A ₂	1.76
B ₁	1.86
B ₂	1.95
C ₁	1.76
C ₂	1.50

38.0 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.

Studies were carried out on the ageing behaviour of Al-Mg (7-10 per cent) alloys with and without misch metal addition. The alloys were given different quenching treatments after solution treatment, to study the effect of varying quenching temperature between 50° and -90°C. on

Table 40 — Composition of Some Typical Heats

Alloy No.	Zn %	Mg %	Si %	Fe %	Mn %	Tensile strength t.s.i.
S1	5.99	2.8	0.13	0.50	0.06	—
S2	6.04	3.47	0.19	0.52	0.03	6.8
S3	5.8	3.16	0.11	0.46	0.07	8.4
S4	5.95	3.01	0.16	0.99	0.06	11.4
S5	5.24	3.33	0.13	0.39	0.06	9.2
S6	6.1	3.15	0.13	0.48	0.05	9.6

age-hardening of these alloys. A comparison was also made between the ageing behaviour of the Al-Mg alloys made from super-pure and commercially pure aluminium. Changes in the microstructure were studied continuously over a period of several hours after quenching at $-90^{\circ}\text{C}.$ and polishing the specimens under ice cold water. Quick appearance of precipitates was observed, but the nature of the precipitates could not be definitely established. Occasionally solution heat-treated specimens developed grain boundary cracks (Fig. 24) on quenching in frozen petroleum ether.

During solution heat treatment, the specimen surface was found covered with a thick oxide layer and to avoid such a formation of oxide layer some experiments were carried out for heat treatment in vacuum. In these experiments, it was observed that Mg metal oozed out from the surface and was deposited on the walls of the vacuum container. It was, therefore, concluded that the specimens could not be solution treated in vacuum under the experimental conditions used. Al-Mg alloys, especially those with high Mg content, are known to be susceptible to stress-corrosion cracking. To study the improvements imparted by the addition of misch metal to Al-(7-10 per cent) Mg alloys, which made these alloys workable; investigation on the stress-corrosion characteristics of the alloys were taken up.

A large number of heats was made of alloys with the nominal composition of

Al-6 per cent Zn-3 per cent Mg, which are well known for their hardenability to high strength under controlled conditions. Table 40 gives the compositions of some of the typical heats made during these experiments.

Ageing characteristics of the above alloys were studied at room temperature, 50° , 100° , 150° and $200^{\circ}\text{C}.$ It was observed that at $50^{\circ}\text{C}.$, the samples showed two hardness peaks with increase in time as shown in Figs. 22 and 23. Further work is in progress.

39.0 Study of the Properties of Indigenous Foundry Moulding Materials

This project was taken up with a view to assessing 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, investigations were completed on six sand samples, five bentonite and two cellulose binders. Besides three indigenous bentonites were studied at elevated temperature for their compressive strength characteristics. A summary of the work done is given below.

I. Moulding Sands

(i) *Batala Black and White Sands*
(Received from Messrs Batala Engineering Company (P) Ltd., Batala, Punjab):

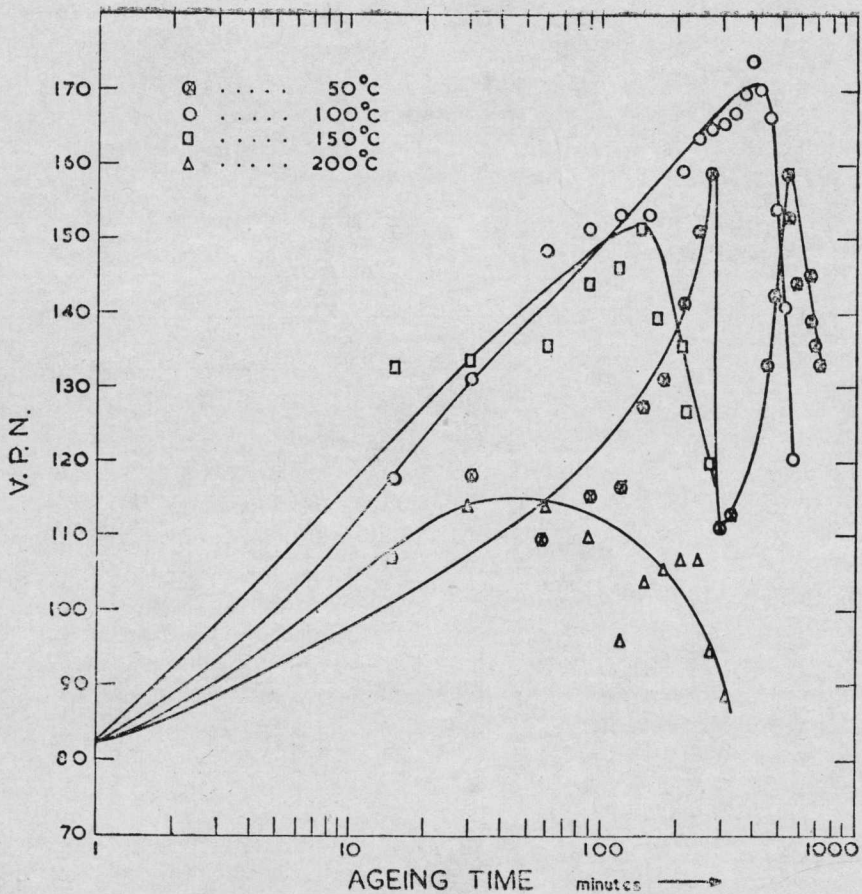


FIG. 22 — AGEING OF Al-6 PER CENT Zn-3.5 PER CENT Mg ALLOY AT DIFFERENT TEMPERATURES AFTER SOLUTION TREATMENT AT $465^{\circ} \pm 5^{\circ}\text{C}$.

(a) *Black Sand* — This was a crude silica sand sample, having grains sub-angular to angular in shape with A.F.S. grain fineness number 43 and containing 1.5 per cent clay. It assayed 81.6 per cent silica with about 5 per cent alkali oxides. The sample consisted primarily of quartz (55-57 per cent) followed by felspar (35-37 per cent) and other minor impurities. The sintering range of the sand was found to be between 1200° and 1250°C . Study of the moulding and casting characteristics showed that the sand was suitable for iron castings. With an addition of 5 per cent bentonite, a moisture content in the range of 3-6 per cent was found optimum for green-moulding purposes,

but for dry-moulding practice 5-8 per cent moisture addition is recommended.

(b) *White Sand* — The sample had an A.F.S. grain fineness number of 53.56 and contained 0.6 per cent A.F.S. clay matter. The shape of the sand grains was sub-angular to angular and the size varied from coarse to medium. The major constituents of the sand was quartz (92-95 per cent) and the total SiO_2 content amounted to about 98 per cent. The sintering temperature of the sample was above 1450°C . The study of its moulding and casting characteristics showed the sand to be suitable for steel castings. When bonded with 5 per cent Hathi Dhani

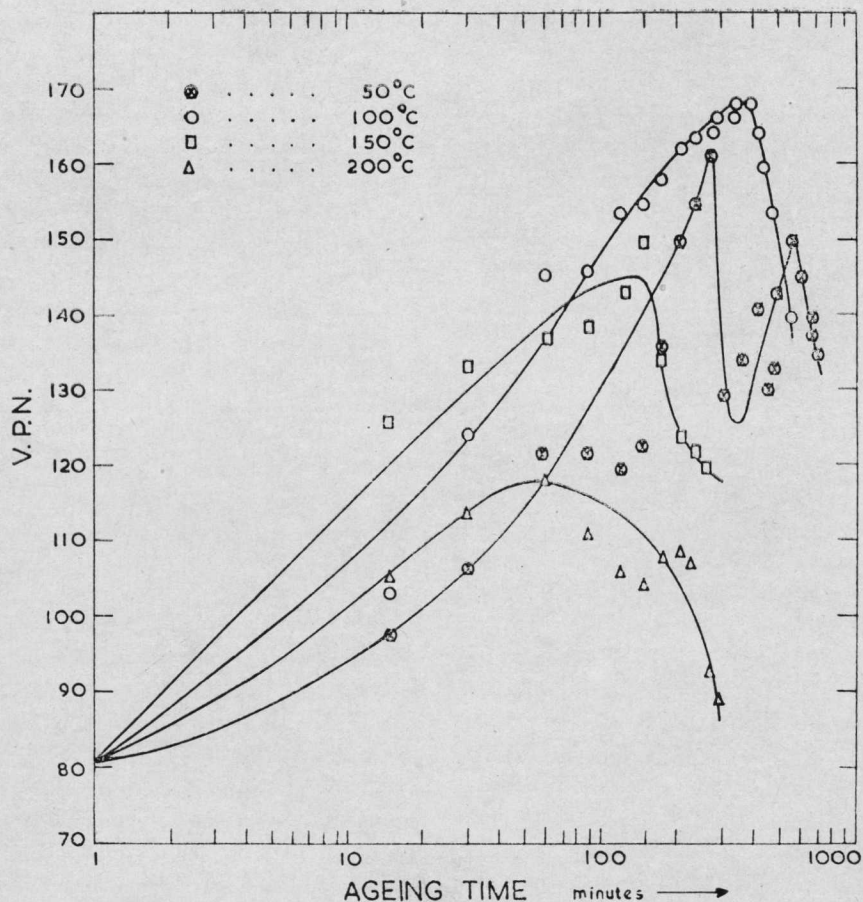


FIG. 23 — AGEING OF Al-6 PER CENT Zn-3 PER CENT Mg ALLOY AT DIFFERENT TEMPERATURES AFTER SOLUTION TREATMENT AT $465^{\circ} \pm 5^{\circ}\text{C}$.

bentonite, it was found satisfactory for green-moulding practice with the addition of 3 per cent moisture content and for dry moulding, above 4.5 per cent moisture is recommended.

(ii) *Madras Silica Sand No. 1* (Received from P.W.D., Ennore, Madras) — This was a clay-free, coarse to medium grained silica sand having 95-98 per cent SiO_2 , mostly in the form of quartz. The sand had an A.F.S. grain fineness number of 37.6. Its sintering temperature was above 1450°C . With the addition of finer silica sand, for better grain size distribution, this might be successfully used for steel castings. The sand seemed to be suitable

for core making also, in which case coatings should be applied to prevent metal penetration.

(iii) *K.S. Sand* (Received from Messrs Rajmahal Quartz Sand & Kaolin Company, Calcutta) — It was a high-silica sand sample (97.7 per cent SiO_2), having A.F.S. grain fineness number 64 and clay content 4.0 per cent. It contained medium to fine grains of sub-angular to angular shape. Mineralogically, it was composed essentially of quartz with traces of other impurities. It had sintering range of 1400 – 1450°C . This sand could be successfully used for light and medium steel castings.

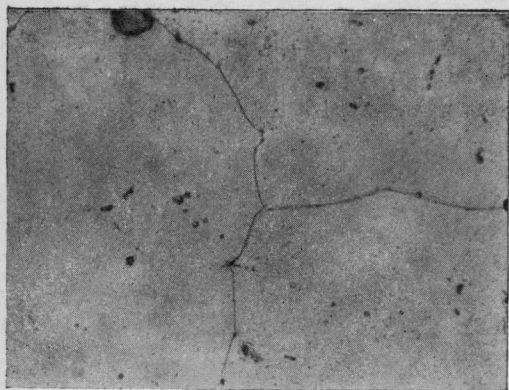


FIG. 24 — GRAIN BOUNDARY CRACKS DEVELOPED ON QUENCHING THE HEAT-TREATED (AT $435^{\circ} \pm 5^{\circ}\text{C.}$) SAMPLES IN FROZEN PETROLEUM ETHER (AT ABOUT -65°C.). $\times 450$.

(iv) *Rajmahal Sand* (Received from Messrs Rajmahal Quartz Sand & Kaolin Company, Calcutta) — The sand was medium-coarse grained and the grains were angular to sub-angular in shape with A.F.S. grain finess number of 51 and clay content 3 per cent. The major mineral constituent of the sand was 98 per cent. The sand sintered in the temperature range of 1500–1550°C. This sand can be successfully used for steel castings.

(v) *Madras Silica Sand No. 2* (Received from P.W.D., Ennore, Madras) — It was a coarse variety of silica sand containing about 97 per cent SiO_2 mostly in the form of quartz. Its clay content was about 3.82 per cent. The grain shape of the sand grains was mostly sub-angular. The sand grains sintered above 1450°C. This sand could be suitable for cores in steel foundries.

II. Bonding Clays

(i) *Cutch Bentonite* (Received from Messrs Hargovindas Shivilal & Company (P) Ltd., Bombay) — This bentonite, occurring in village Wandh, near Mandvi Bunder (Port), Dist. Cutch, was dull yellow in colour and had 11.52 per cent initial moisture. Its swelling index, gel value and pH values

were found to be 18.18, 35 and 7.7 respectively. 64.5 per cent of the dry sample passed through a 200 mesh sieve of the U.S. sieve series, while 87.5 per cent of the sample attained a size of less than 44 microns in wet sieving. Predominance of the montmorillonite mineral was indicated in the petrological study of the sample. Differential thermogram of the sample showed clearly the characteristic features of the mineral montmorillonite. Its total base exchange capacity was 90.62 milliequivalents per 100 gm. and had a sodium to calcium ratio of 2.571 photometrically. Standard Rajmahal sand bonded with 5 per cent of this bentonite yielded high-green strength, permeability and good dry strength.

(ii) *Mahua Bentonite* (Supplied by Messrs Calcutta Mineral Supply Co. (P) Ltd., 31 Jackson Lane, Calcutta 1) — The sample, occurring near Mahua in Bhavnagar, Gujarat, was yellowish-brown in colour and contained 10.32 per cent moisture initially. Its swelling index was 16.67, gel value 20 and pH value 9.4. About 81 per cent of the dry sample passed through 200 mesh sieve of the U.S. sieve series. Petrological examinations and D.T.A. showed montmorillonite and beidellite — the aluminium rich end-member of the isomorphous series of dioctahedral montmorillonites. A Na/Ca ratio of 0.7555 showed that Na was not the predominant exchangeable cation. Total base exchange capacity was 76.47 milliequivalents/100 gm. Rajmahal sand bonded with 5 per cent of this bentonite developed high-green strength, fairly good permeability and low-dry strength values.

(iii) *Bakudi Bentonite* (Supplied by Messrs Calcutta Mineral Supply Co. (P) Ltd., 31 Jackson Lane, Calcutta 1) — The sample, occurring near Bakudi and Tinpahar in Santhal Paraganas (Bihar), was brownish-grey in colour and contained 9.71 per cent moisture. It had high alumina, titania and lime contents. Swelling index, gel value and pH values of the sample were 2.5, 10 and 8.1 respectively. About 89.6

per cent of the dry sample passed through a 200 mesh sieve of the U.S. sieve series. Mineralogically the sample was an admixture of montmorillonites. Endothermic and exothermic reaction characteristics of montmorillonites were recorded in the differential thermogram obtained for the sample. Na/Ca ratio of 0.162 would mean that the sample carried only very low amounts of Na as exchangeable cations. Total base exchange capacity of the sample was only 49.405 m. equivalents per 100 gm. However, Rajmahal sand bonded with 5 per cent of this bentonite developed fairly satisfactory green strength, permeability and dry strength. The sample was found to contain montmorillonites with beidellite in predominance and with very little of Na saturated montmorillonite. Impurities like biotite, calcite, hydromica and some opaque minerals were also present.

The following two foreign bentonites were also investigated under the International Working Group on Bonding Clays as a continuation of the work on English, Spanish and Poland bentonites reported earlier.

(iv) *German Bentonite*—The sample received from Sudchemie AG, Munich, Germany, at the instance of Gei Berei-Institute der Technischen Hochschule, Aachen, was light grey in colour and had an initial moisture content of 14.05 per cent. Swelling index, gel value and pH values of the sample were 2.01, below 10, and 7 respectively. The alkalis were only in trace. The sample appeared to be calcium carrying montmorillonite with minor amounts of saponite (Mg member) and other mineral impurities such as biotite, etc. About 88 per cent of the dry sample passed through 200 mesh sieve of the U.S. sieve series. In wet sieving 63.35 per cent of the sample attained a particle size of less than 44 microns. Presence of montmorillonite in fairly good amounts was indicated by the petrological study on the sample. The sample showed primary and secondary

endothermic reaction characteristics of montmorillonites in the differential thermogram. Total base exchange capacity of the sample was 60.18 milliequivalents per 100 gm., with a Na/Ca ratio of 0.07158. Rajmahal sand bonded with 5 per cent bentonite developed high strength in the green and dry states with fairly good permeability values.

(v) *Australian Bentonite*—The sample had a moisture content of 11.35 per cent. The bentonite could be classed as a 'low swelling' one, as it had a swelling index of only 2.56. The sample had a gel value of 10 which was low for montmorillonites. Results of the dry fineness showed that about 85 per cent of the as received sample had a particle size of 44-53 microns. In wet sieving through a 325 mesh sieve of the U.S. equivalent sieve series, about 70 per cent of it attained a particle size of less than 44 microns. pH value was in agreement with the tentative specifications for Western bentonite of the S.F.S.A. The sample was found to consist of microcrystalline aggregates of montmorillonite group of minerals with montmorillonite in predominance followed by saponite and traces of calcite and hydromica when petrologically studied. From a Na/Ca ratio of 0.220, it was evident that Na was not the predominant exchangeable cation. A total base exchange capacity of 68.255 milliequivalents per 100 gm. was low for montmorillonites.

III. *Cellpro Core Binders [Received from the Imperial Chemical Industries (Private) Limited, Calcutta]*

Evaluation of two samples, Cellpro 'LV' and Cellpro 'HV' of low and high viscosity respectively, was carried out to determine their suitability as core binders. These were white powders containing approximately 50 per cent sodium salt of carboxymethyl cellulose and contained about 7.8

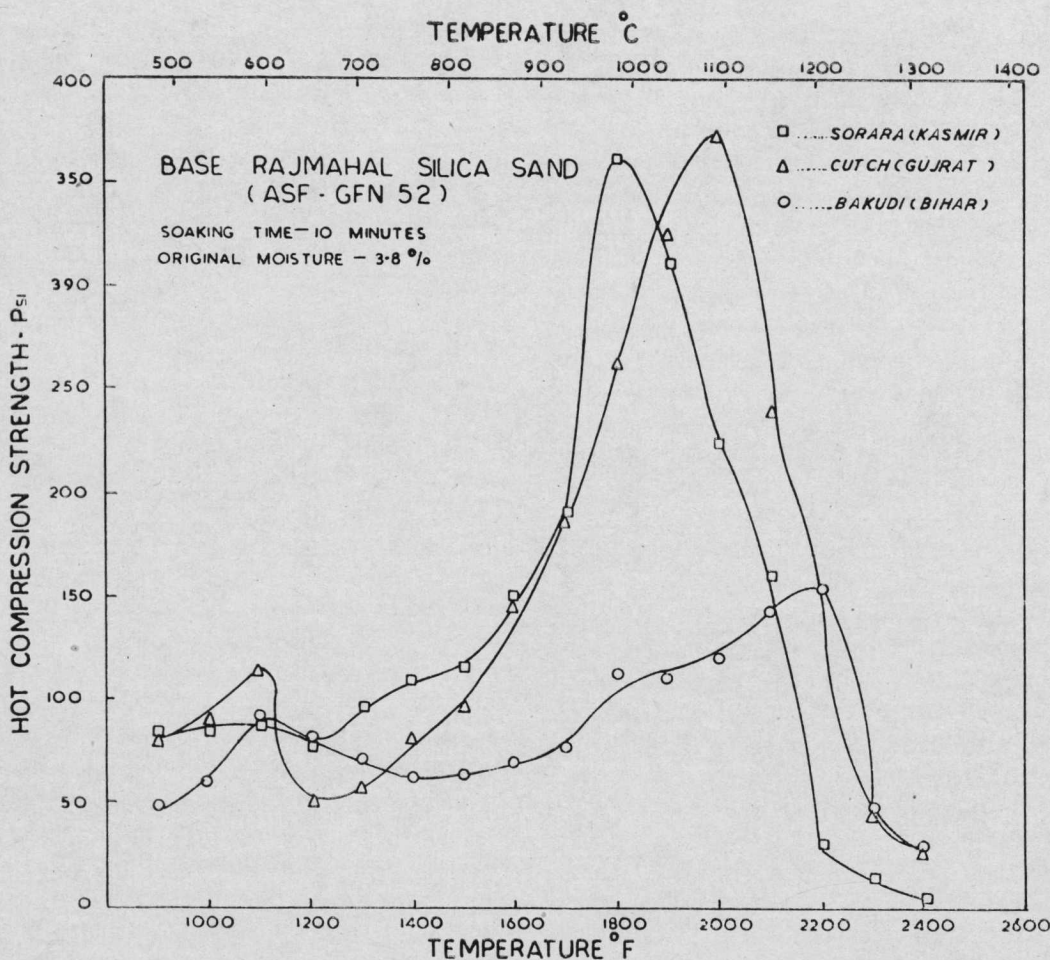


FIG. 25 — HOT COMPRESSION STRENGTH VERSUS FURNACE TEMPERATURE

per cent moisture in the as received state. The core-binding properties of the samples were studied by addition of 5 per cent cellpro to high-silica Rajmahal sand of A.F.S. grain fineness No. 52. The highest green strengths of 4.8 and 4.5 p.s.i. were obtained respectively in 'LV' and 'HV' bonded sand mixtures. Both kinds of cellpro binders behaved similarly in case of baking time and temperature and the optimum properties were obtained by baking for 1 hr. at 200°C. In the case of 'LV' bonded specimens, tensile strength of 103 p.s.i. and permeability number 190 were

observed on baking at 200°C. for 1 hr. and the respective values for 'HV' bonded specimens were 79 p.s.i. and 180. In respect of baked hardness, specimens bonded with either grade of cellpro had a scratch hardness number of 90. The total volume of gas given off measured 34 and 31 cc. per gram of the sand mixture containing 5 per cent cellpro 'HV' and 'LV' respectively when heated at 1000°C. Casting characteristics showed good surface finish of the cast steel and easy shake out of the cores. The investigation is being carried out with various percentages of cellpro to obtain

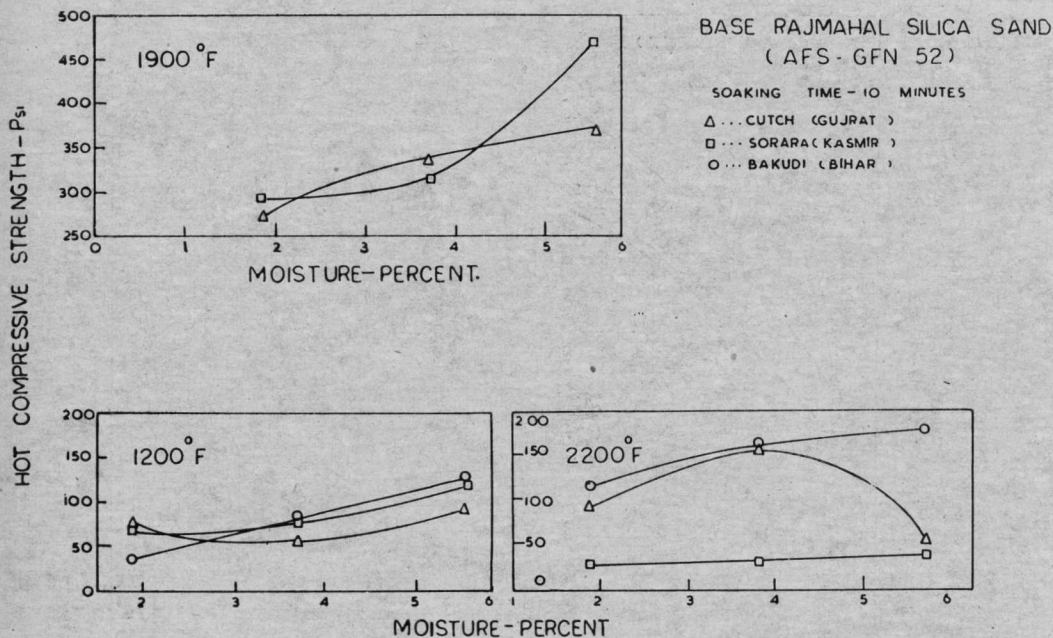


FIG. 26 — HOT COMPRESSIVE STRENGTH VERSUS MOISTURE

the optimum percentage required. In addition the work on the efficacy of wet mixing as compared to dry mixing method is also being carried out.

IV. Elevated Temperature Properties of Foundry Moulding Materials

A study was made of the hot strength characteristics of the three indigenous bentonites, namely Cutch (Gujarat), Bakudi (Bihar) and Sorara (Kashmir). Effect of ramming and moisture on the hot strength was also studied. The temperatures of peak hot strength were also studied. The temperature of peak hot strength values obtained for the three bentonites Sorara, Cutch and Bakudi were at 1800°, 2000° and 2200°F. respectively with Rajmahal silica sand of AFS-GFN.52 as the base sand. The values at the respective peak temperatures were 360, 372 and 154 p.s.i. With the same percentage bentonite additions, rise in moisture content

increased hot strength of the three bentonites; at 1900°F. hot strength of Sorara bentonite increased to the highest value with increase of moisture. Though ramming caused a gradual increase in hot strength of both Cutch and Sorara bentonites, the increase in hot strength of Cutch bentonite was at slightly higher rate than that of Sorara. Under these circumstances, a mixed-bond achieved by employing suitable proportions of Cutch and Bakudi or Sorara and Bakudi appeared favourable.

40.0 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

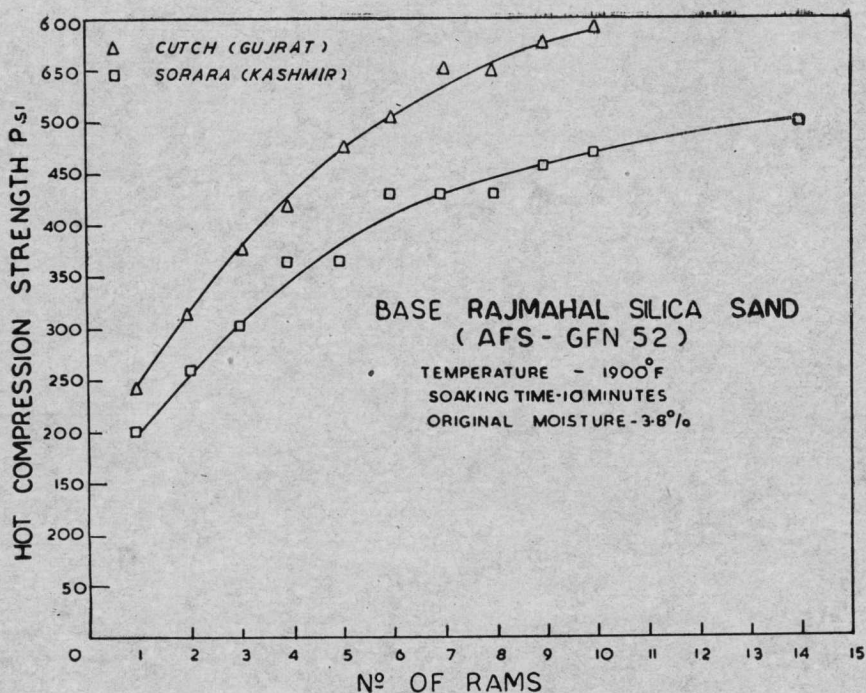


FIG. 27 — HOT COMPRESSION STRENGTH VERSUS RAMMING

been suggested which is effective in both the hypo- and hypereutectic 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.

During the period a comprehensive study was made to evaluate the optimum amount of the modifying agent required to give the maximum refinement in the microstructure and to determine the effect of remelting, holding time and high pouring temperature on the microstructure of the modified alloys.

With the conventional methods of modification, the above points are controversial and a series of experiments were conducted on aluminium-silicon alloys containing 9, 12, 23 and 28 per cent Si with the addition of varying amounts of the modifying agent. The resulting microstructure, mechanical properties and fluidity characteristics were

studied after each addition. It was found that 0.05 per cent of the modifying agent was the optimum amount necessary to retain about 0.01 per cent of the modifying agent to give the best results. Effects of degassing with nitrogen, chlorine and hexachloroethane were separately studied on the modified alloys. Zinc sulphide is believed to be a modifying agent for hypereutectic alloys, but no good results could be obtained with it. Results of all these experiments are being compiled.

41.0 Powder Metallurgy

Work was taken up with a view to (i) producing copper powder continuously by electrodeposition and (ii) to making porous bronze bearings.

It was reported earlier that a successful method had been developed for a

continuous electrodeposition of copper powder using an organic substance. During the period under review, work on the continuous production of copper powder by this method was continued and previous findings on these processes were confirmed, as shown in Table 41. It was possible by this method to shape and alter the characteristics of the powder in the manner required. The use of the reagent is being standardized. Efforts are being made to find the most effective method for the preparation of the organic reagent and for controlling the activity of the reagent in the electrolyte. Work was also done on the post-deposition operations such as obtaining of dried powder from the slurry and annealing of the dried powder. It was observed that drying of the copper powder required protective

action against corrosion, and dichromate solution was tried for this purpose but was not found satisfactory as a corrosion inhibitor. Glycerine was found to be very suitable since it permanently prevents the tarnishing of dried copper powder and also allows rapid drying at high temperature. Annealing of copper powder without loss of the special properties associated with electrodeposited powder was done by the use of linseed oil as coating. It was found that by the use of linseed oil coating, annealing could be done at high temperatures, up to 800°C., and for long duration. It is proposed to study other substances similar to linseed oil for achieving similar results.

It was reported earlier that a procedure for simplifying the manufacture of porous bronze bearings was under study. During

Table 41 — Sieve Analysis of Copper Powder Obtained by Continuous Electrodeposition

Composition of the electrolyte	Duration of continuous electrolysis	Characteristics of powder made		Additive
		Sieve analysis U.S.S.	App. density orifice	
CuSO ₄ , 45 g./l. } H ₂ SO ₄ , 135 g./l. }	5 days	-100 } +140 }	7.8 %	2.2
		-140 } +200 }	3.8 %	
		-200 } +270 }	16.6 %	
		-270 }	45.2 %	
CuSO ₄ , 56 g./l. } H ₂ SO ₄ , 148 g./l. }	5 days	-100 } +140 }	6.5 %	2.3
		-140 } +200 }	16.1 %	
		-200 } +270 }	27.5 %	
		-270 }	49.9 %	
CuSO ₄ , 70 g./l. } H ₂ SO ₄ , 171 g./l. }	2 days	-100 } +140 }	1.2 %	1.9
		-140 } +200 }	15.6 %	
		-200 } +270 }	33.0 %	
		-270 }	50.2 %	
CuSO ₄ , 70 g./l. } H ₂ SO ₄ , 145 g./l. }	2 days	-100 } +140 }	Nil %	1.9
		-140 } +200 }	4.8 %	
		-200 } +270 }	16.7 %	
		-270 }	78.5 %	

the period under review it was observed that the strength of sintered bearings can be improved to the desired extent by a final corrective resintering operation. This resintering could be done either in the presence of a halide gas (which, however, involves risk with the furnace and disadvantage of being highly corrosive at higher temperatures) or a salt bath incorporating halide salt. For the latter purpose a mixture or small amount of Na_3PO_4 was found to be very suitable for the preparation of strong bearings.

42.0 Production of Iron and Steel Using Direct Reduction Technique

An investigation was taken up with a view to producing steel from iron ore directly using direct reduction technique. A sample of iron ore was received from the Orissa Mining Corporation, Bhubaneswar, for this purpose. The Corporation has been granted a lease for mining high-grade iron ore in Keonjhar district, and they are interested in the production of iron and steel at the mine site.

The sample of ore sent was in the form of hard lumps with size varying between 4 and 6 in. The ore was crushed in jaw crusher to $1\frac{1}{2}$ in. size and then to $\frac{1}{4}$ in. in double roll crushers and finally to about 70 mesh by wet-grinding in ball mill. The ore fines had the average analyses of Fe, 66.9; SiO_2 , 0.88; Al_2O_3 , 1.2; P, 0.049; and S, 0.002 per cent. The ore fines were packed in paper tubes of 1 in. in diameter and were embedded in coke breeze and heated in electric muffle furnace for various length, of time at various temperatures. The analyses showed that the reduction at 1000°C . for 8 hr. was sufficient to reduce practically all the iron in the ore to metallic iron. The product analysed: Fe, 96; FeO , 0.52; and carbon, 0.87 per cent. As it was found that the handling of larger quantity of ore fines would be difficult

in the above-mentioned process, it was decided to pelletize the ore fines. Different bonds were used for the purpose. With 0.5 per cent bentonite bond, the pellets had good green strength, but after drying they became friable. The use of 5 per cent molasses gave both good green strength as well as dry strength. The pellets were similarly reduced using coke breeze. The reduced pellets were melted in indirect arc electric furnace under a basic slag. The steel was cast in ingots. The coke was then mixed with 5 per cent lime before being used for reduction of the pellets. These pellets, after reduction, when melted in indirect arc electric furnace, analysed: C, 0.02; Mn, 0.016; Si, 0.02; S, 0.03; and P, 0.014 per cent. This steel was of high grade and could be used for the production of high-grade alloy steels.

Besides reduction with coke breeze, gaseous reduction was also tried. An apparatus was set up for the hydrogen reduction of the pellets. It was noted that hydrogen reduction was much faster and was complete at relatively lower temperature. This product was found to suffer from the defect of being pyrophoric. To avoid this, an arrangement was made to cool the product in an atmosphere of nitrogen after reduction of the pellets with hydrogen. The reduced products were found to be non-pyrophoric when cooled in nitrogen atmosphere.

43.0 Study on Age Hardening: Reversion in Aluminium-Copper Alloys

Reversion in aluminium-3.8 per cent copper alloy had been previously studied after ageing the specimens for different durations. During the period under review, the effect of plastic deformation on reversion was studied. The strip specimens 1.25 mm. thick were solution-treated at 520°C . for 24 hr., quenched in iced water, deformed 10, 20, 40 and 60 per cent by cold rolling and reverted and aged at 170° , 190°

and 250°C. The results showed that reversion behaviour of specimens aged at 170° and 190°C. was nearly identical and reversion hardness value increased with increasing deformation, i.e. drop in hardness caused by reversion was decreased. Specimens deformed by 60 per cent did not show any appreciable reversion. Reversion test at 250°C. for specimen deformed by 10 per cent and 60 per cent showed that general softening of the specimen started after 10 min. of ageing. These tests indicated that zones formed during deformation were more stable and easily transformed to a metastable or stable phase θ' and θ . The stability of the zones to reversion treatment can be attributed to interaction between copper atoms and dislocations.

44.0 Study of Temper Brittleness

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

The wire wrap bend test apparatus, which was fabricated earlier in the laboratory, required certain modifications to give consistent results, and during the period under review, these modifications were carried out. Melts of nickel-chromium steel were made and cast in metal moulds. The ingots were forged and rolled from which the specimen blanks were cut. The specimen blanks were plated with chromium to prevent them from oxidation during heat treatment. The plated specimen blanks were heat-treated suitably to get them in the normal and embrittled conditions. After heat treatment, notches were cut and impact tests carried out to determine the transition temperature. Fractographic and metallographic examinations were also

carried out on the tested samples. One of the ingots of the 30 kg. heat was forged and cold rolled to a thickness of 0.6 mm. Hot rolling was also carried out on a small piece of forged material, but the surface was not good for further studies. From the cold-rolled material, tapered specimens were cut and were stress-relieved at 1000°C. in an atmosphere of oxygen. The annealed specimens were given an extension of 2.5 per cent and were annealed in argon to get single crystals, but without much success. Further experiments are being carried out.

45.0 Curie Temperature of Iron-Chromium Alloys

This project was taken up with a view to determining the electron distribution of iron contributing to ferromagnetism. There is a difference in the ferromagnetic behaviour of iron on the one hand and that of cobalt and nickel on the other. It is felt that Curie temperature which is taken to be an index of ferromagnetism should reflect this difference. The experimental procedure contemplated was to prepare alloys of the required composition and homogenize them.

During the period under review the method for determining Curie temperature was developed and standardized with pure iron and nickel and the method yielded an accuracy of $\pm 3^\circ\text{C}$. A 20 lb. heat of iron-zinc alloy was made. After homogenizing the ingot, it was forged and from the forged rod, specimen for determination of Curie temperature was made and Curie temperature was determined. Metallographic and X-ray examination of the sample was also carried out. As there was difficulty in melting iron-zinc alloys due to volatilization of zinc, iron-aluminium alloys were taken up for the study. Alloys containing 4.5, 9.4, 10, 15, 18 and 24.7 weight per cent of aluminium were made in the high-frequency furnace. The ingots were forged and

subsequently turned to rods of 9.5 mm. diameter. 12.7 cm. of the turned rod was cut and Lowmoor iron rods were welded on both its sides to determine the Curie temperatures of the alloys. As alloys containing 18 and 14.7 weight per cent aluminium were difficult to forge, they were cast into rods of 1.25 cm. diameter. The cast rod was used for experiments subsequently. The Curie temperatures of the alloys are given in Table 42.

Table 42 — Curie Temperatures of the Alloys Studied

No.	Wt. % Al	Curie temperature °C.
Al-6	4.58	737
Al-10	10.00	617
Al-F	14.55	368
Al-E	16.39	139
Al-20	24.7	Not determined

46.0 Preferred Orientation in Cold-rolled Aluminium Steel

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 values 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.

In continuation of earlier work done, commercial aluminium sheet was first annealed at 550°C. before being cold-rolled under the following sequences to develop textures:

- (i) reversing the direction of rolling after each pass
- (ii) reversing and cross-rolling alternately

The textures induced on the interior layers and surface layers of aluminium sheets were determined from the (111) and

(200) pole figures drawn from the intensity data of the (111) and (200) Debye rings of the X-ray transmission photographs. The textures determined from both (111) and (200) pole figures for the interior layers of the aluminium sheet cold-rolled under the rolling sequence (i) were found to be ideal orientations (110) (112), (112) (111) and near (123) (121). The textures determined for the surface layers were the same as found in the case of interior layers, but differed slightly in their scatter. The preferred orientations developed on both the interior and surface layers of aluminium sheet cold-rolled under the second rolling sequence were found to be ideal orientation (110) (112) and near orientations (123) (121) and (100) (001).

47.0 Conductivity of Commercial Aluminium

Silicon and titanium which are ordinarily present in commercial aluminium as dissolved impurities adversely affect its electrical conductivity. It is well established that the conductivity of a solid solution can be increased if the dissolved impurities could be taken out from the parent lattice. The decrease in the solid solubility of both silicon and titanium with falling temperature presents a possibility of their removal from the lattice of aluminium. In order to have a fundamental and rigorous approach to the problem it was decided to study changes in conductivity as precipitation progresses in binary aluminium-silicon alloy (0.5 per cent).

The alloy was cast from aluminium of 99.5 per cent purity, forged and rolled to sheets of $\frac{1}{8}$ in. thickness. The strip specimens were homogenized at 550°C. for 8 hr., quenched and aged at 200°, 250°, 300°, 350° and 400°C. for periods up to 30 hr. The resistances of the individual specimens were measured using a Kelvin double bridge. The results are shown plotted

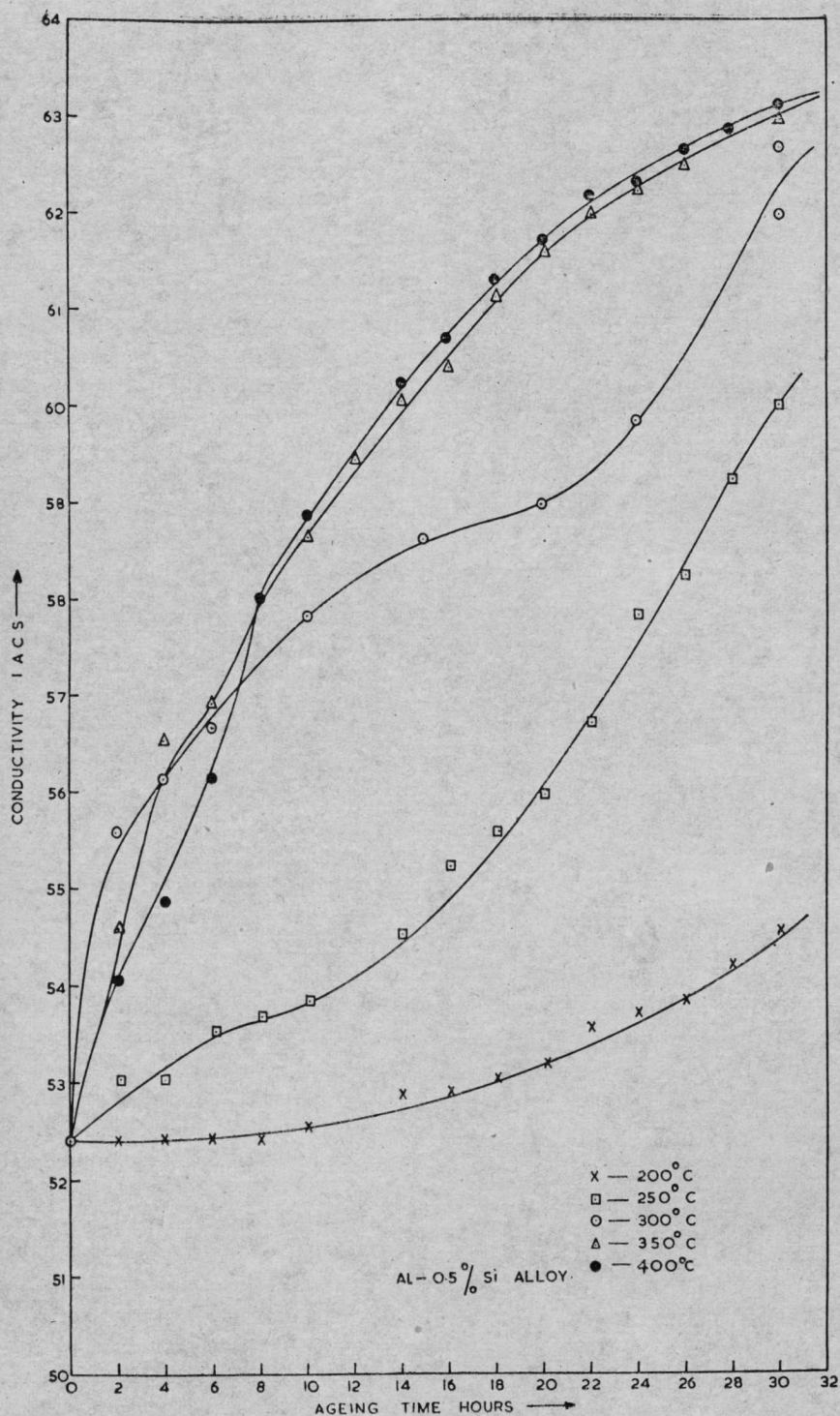


FIG. 28 — VARIATION OF CONDUCTIVITY OF AL-0.5 PER CENT Si ALLOY WITH AGEING TIME

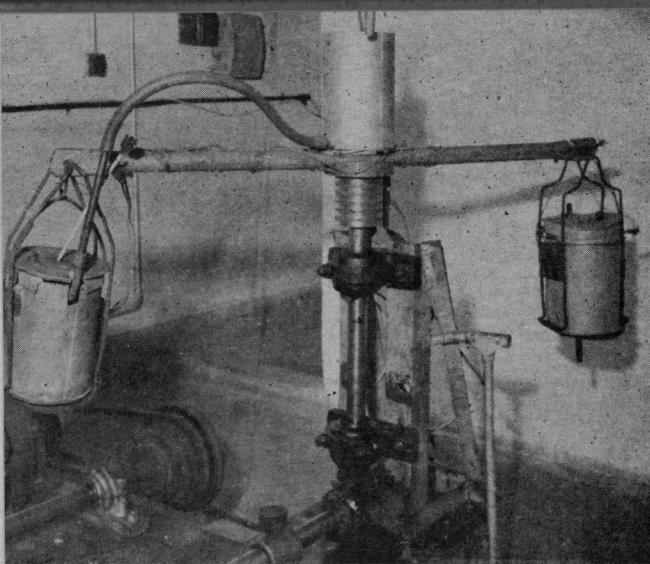


FIG. 29 — A VIEW OF THE LIQUID METAL CENTRIFUGE DESIGNED AND FABRICATED AT THE NATIONAL METALLURGICAL LABORATORY

in Fig. 28 from which the improvement in conductivity after ageing can be readily appreciated. The salient features of the observations were: (i) Marked improvement in conductivity from 53.00 IACS units in the quenched condition to about 62-63.0 IACS in the aged condition for 30 hr. ageing being done between 300° and 400°C. (ii) For isochronal periods of ageing, conductivity increases with increase in ageing temperatures; the increase is most marked after ageing at 350°C.

In order to accelerate the ageing process, the specimens were cold-worked to the extent of 10-12 per cent reduction in thickness after the solution treatment and aged soon after at the above temperatures and for periods extending up to 30 hr. The results so far obtained indicated that such working tends to accelerate the ageing process, although the acceleration was much less pronounced than for the conventional aluminium-copper-magnesium alloys.

48.0 Structure of Liquid Metals

With a view to using 'non-thermodynamic' experimental techniques for studying

the structures of liquid metallic solutions and to obtaining some understanding of the atomic interactions, investigations were taken up on the following lines:

- (a) Study of interactions between Al-Cu, Al-Cu-Mg, Pb-Sn alloys in the liquid state
- (b) Study of the partitioning of magnesium between aluminium and lead
- (c) Enthalpy of liquid alloys

(a) *Study of Interactions between Al-Cu, Al-Cu-Mg and Pb-Sn Alloys* — It is generally accepted that most metallic solutions do not strictly obey the laws of ideal solutions. This implies that in a binary liquid metallic solution there are clusters of either like or unlike atoms. The aim of this investigation is to provide physical evidence of their existence. An apparatus has been built which centrifuges the alloy in the liquid condition whereby the difference in the density of these clusters is magnified several folds, thus causing the lighter clusters to move towards the centre of the centrifuge and the heavier ones to move outwards.

Experimental Technique — A robust centrifuge has been designed and fabricated in the National Metallurgical Laboratory. The molten alloy is kept in a stainless steel crucible concentric with an outer galvanized iron crucible and a furnace. The whole assembly is hung at one end of an arm which is rotated by an electric motor through reduction pulleys. The other end of the arm is counterbalanced by dead weights. A flux of sodium and calcium fluorides is used to prevent surface oxidation in Al-Cu alloys. At the end of the predetermined time water is allowed to rush through the annular space between the stainless and galvanized iron crucibles and this serves to chill the distribution of atoms existing in the liquid state at that instant of centrifuging. The solidified ingot is recovered and analysed. Arrangements have also been made to measure the temperature continuously during centrifuging.

(b) *Study of the Partitioning of Magnesium between Aluminium and Lead* — The distribution coefficient of magnesium between the immiscible liquid lead and aluminium is being studied at 700°, 800° and 900°C. Binary Al-Mg alloy made from superpurity aluminium was melted in a graphite crucible and lead added. After thorough mixing the mixture was allowed to solidify and the ingot chemically analysed.

(c) *Enthalpy of Liquid Alloys* — Apparatus essentially based on the principle of Bunsen's calorimeter was designed and fabricated. It is at present being standardized.

49.0 Effect of Thermal Cycling of Cu-Pb Alloys

This investigation was undertaken with a view to causing separation of Cu from the mixed white metal scrap. As a result of the work done on the liquid metals it seems possible to separate lead from the scrap by physical methods. Gravity segregation due to its density can be enhanced either by centrifuging the metal or by repeated thermal cycling through the solidification range. At present the segregation of lead during thermal cycling in Cu-40 per cent Pb alloy is being studied.

50.0 Effect of Hydrogen in Overheating and Burning of Steel

One of the most serious obstacles facing the makers of high-quality alloy steels is the presence of hydrogen as dissolved impurity. It is established that hydrogen in steel can cause porosity, embrittlement and hairline cracks. It is also generally accepted that the steel made in the electric furnace is more susceptible to overheating and burning than the one made in the open hearth due to the fact that the hydrogen content of electric steel is generally more than that of open-hearth steel. It is, therefore, decided to study the effect of hydrogen

on overheating and burning of steels. Blank specimens and specimens soaked in hydrogen at 1100°C. for 1 hr. are heated for half an hour at various temperatures with a view to getting them overheated. Overheating temperature range was determined metallographically after etching in nitro-sulphuric acid and also after etching electrolytically in saturated aqueous solution of ammonium nitrate. So far, six plain carbon and alloy steels were examined.

51.0 Measurement of Carbon Activity in Iron and Its Alloys

Under the C.S.I.R. Research Fellowship Scheme work was initiated on equilibrium

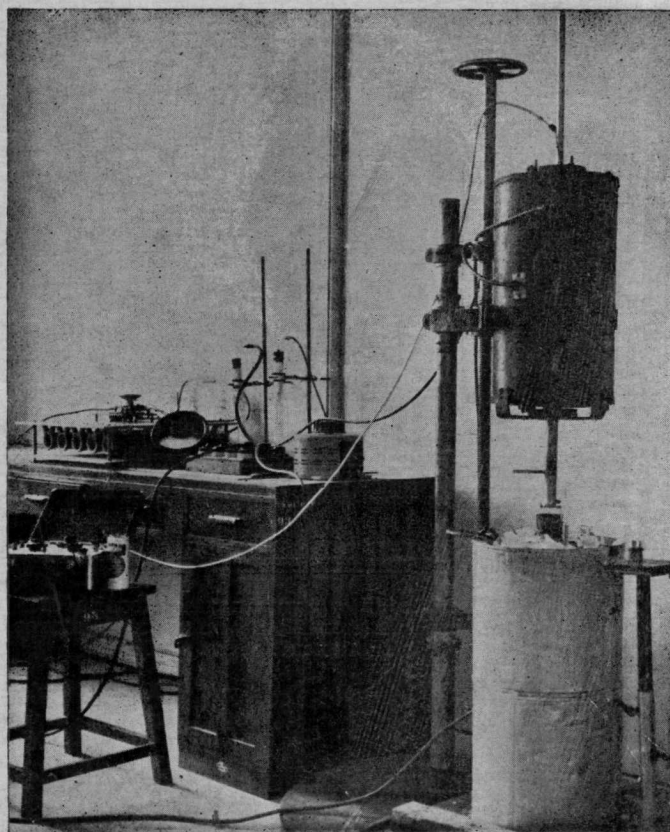


FIG. 30 — A VIEW OF THE CALORIMETER FOR MEASURING THE HEAT CONTENT OF LIQUID METALS, DESIGNED AND FABRICATED AT THE NATIONAL METALLURGICAL LABORATORY

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 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.

Measurements of the activity of carbon in austenitic iron at 1000°C. were completed. Thirty-four experiments, besides those performed in the previous year, were carried out utilizing a glass solenoid pump. The glass solenoid pump was found to be most suitable for circulating gas mixtures of CO and CO₂ over the specimen in a closed all-glass vacuum-tight system. Carbon analysis of the sample corresponding to the values of the partial pressure ratios of the gas mixture under equilibrium was determined. In some of the experiments, the duration of equilibrium was extended to more than 72 hr. and after equilibrium was established, the system was flushed with a stream of purified argon and the sample was quickly cooled in argon atmosphere. Experimental results of equilibrium of CO-CO₂ gaseous mixtures with austenitic iron at 1000°C. are given in Table 43.

52.0 Solubility of Nitrogen in Plain Carbon and Alloy Steel

Under the C.S.I.R. Research Fellowship Scheme, a study was taken up with a view to determining the solubility of nitrogen in nickel-free austenitic Cr-Mn-N-Cu stainless steel in solid and liquid states. Preliminary studies conducted earlier revealed that data based on the measurement of hot-volume and volume of nitrogen absorbed for the determination of solubility of nitrogen on the Sievert's type apparatus were not found reliable as the last traces of oxygen could not be removed from the system. The nitrogen solubility was subsequently determined by chemical analysis and vacuum fusion technique from the samples that were quenched in water after exposing to purified nitrogen gas at one atmospheric pressure. The sample was treated with purified nitrogen gas till the attainment of equilibrium 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. Experiments were carried out with thin strips of 18-8 stainless steel having uniform thickness of 0.01 in. at 1000°C. and one atmospheric pressure of nitrogen for various periods. The results obtained are indicated in Table 44.

Table 43 — Experimental Results of Equilibrium of CO-CO₂ Gaseous Mixtures with Austenitic Iron at 1000°C.

Expt. No.	P _{CO} =vol. % CO	P _{CO₂} =vol. % CO ₂	p ² CO ₂ in atmos.	Carbon in wt. %
31	63.88	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	93.03	6.97	12.42	0.28
29	98.0	1.8	53.34	1.12

Table 44 — Effect of Nitriding Time on Nitrogen Content of the Sample at 1000°C.

Time hr.	Wt. per cent of nitrogen
24	0.93
50	1.2
50	1.24
50	1.12
70	1.31
70	1.29
70	1.42
96	1.61
96	1.54

It was observed that with increased exposure to 96 hr. the nitrogen absorption had increased. Further experiments are in progress to measure the nitrogen absorption on continued exposure for longer period. The possibility of formation of nitrides is also being investigated.

53.0 Phase Transformation Study on Copper-Tin Alloys

Under the Council of Scientific & Industrial Research Fellowship Scheme, systematic studies were taken up on the phase transformation in electrolytic copper-tin alloys at constant cathode potential. An attempt is being made to verify the applicability of postulations made by Banerjee and Allmand, which explain the existence of metastable phases in electrolytic alloys.

Copper-tin alloys of various compositions ranging from 15 to 100 per cent Cu were deposited at constant cathode potential in an electrolytic cell of the propeller-agitation type designed to ensure efficient circulation of the electrolyte round the electrode. The structural states before and after annealing were examined by X-ray powder studies. Some of the results obtained so far are given in Table 45. Electrical resistivity measurements of the alloy are being taken up.

54.0 Development of Thermostatic Bimetals

Thermostatic bimetals are temperature-sensitive elements which are widely used in a great variety of ways for indicating, regulating and controlling temperature, and as thermal relays, circuit-breakers and overload protection devices. The entire requirements of bimetals are met by imports. With the increase of the electrical industry in India the need for bimetal will be increasingly felt. This project was, therefore, undertaken to develop suitable manufacturing techniques for different types of bimetals.

Table 45 — Results of Phase Transformation Study of Deposited Cu-Sn Alloys

Composition of the electrolyte:												
Copper cyanide Sodium cyanide Volume of the electrolyte Temperature				= 0.1656 M. = 0.3378 M. = 4500 M. = 25°C.				Sodium stannate Caustic soda pH		= 0.1888 M. = 0.3775 M. = 12.3		
Expt. No.	π	C/37 cm. ²	c.d. amp./Dm ²	t (min.)	Area	Yield	C.E.	% Cu in the deposit	π_0	Colour of the deposit	Equilibrium phase	Structure
												without annealing Annealing
B ₁	-1.3276	1.85	5.0	35	55.04	0.1302	12.48	15.1332	-0.7916	Feeble black	$\theta + \eta$	$\theta + \eta$
B ₂	=1.3776	2.1	5.6	55	119.07	0.2648	7.76	67.16	-0.8406	Greyish white	$\alpha + \epsilon$	$\gamma(\delta) + \epsilon$
B ₃	-1.4276	3.9	10.54	50	134.425	0.05	1.216	71.11	-0.7896	Greyish white	$\alpha + \epsilon$	$\alpha + \epsilon$
B ₄	-1.4526	4.85	13.1	50	168.95	0.0458	0.686	100	-0.7336	Coppery	α	α
π = Constant cathode potential.												
π_0 = Static cathode potential.												

π = Constant cathode potential.
 π_0 = Static cathode potential.

Earlier, physical and mechanical properties of some of the alloys chosen for the production of thermo-bimetal were studied and suitable melting and casting practices and hot-working techniques for each of these alloys were developed. A number of thermo-bimetals were prepared and their physical and mechanical properties were reported. During the year under review, stress was laid on the development of overall technology for the production of all-ferrous bimetal and bimetal with copper base alloys as high extrusion component.

(i) *Production of All-ferrous Bimetal*—Extensive work was carried out to standardize the method of production of bimetal with ferrous alloy as both the components. NML-45 was chosen as a representative type. Difficulties encountered in each stage of production were successfully tackled. Use of ingot mould of different size and shape than those previously used, deoxidation of the melt by CaSi and use of hot top were found necessary for obtaining sound ingots. For successful hot working of these alloys, especially the low expansion ones, it was found necessary to employ controlled heating schedules and proper forging temperature and to avoid exposure to high temperature for longer time. Method of bonding was also standardized. It was concluded on the basis of several trials that a reduction of at least 15-20 per cent in the first pass was necessary for obtaining sound bonding at a temperature of 1150°/1200°C. With the rolling mill available, it was possible to produce a bonding of composite of maximum thickness of 0.5 in. by rolling only. For composites of higher thickness, compression at high temperature of at least 5 tons/in.² followed by hot-rolling was necessary for successful bonding. Composite of maximum thickness which had been bonded successfully was 1.1 in. thick, weighing about 6 kg. Heating in controlled manner was also found necessary in this case for obtaining better surfaces.

As proper surface finish is also important, appropriate pickling and cleaning technique was evolved. A schedule of cold-rolling and annealing under controlled atmosphere was worked out for obtaining good surface finish, appropriate grain size and enhanced mechanical properties.

(ii) *Bimetal with Copper Base Alloys as High Expansion Component*—Alpha-brass was initially chosen for the purpose. Previous attempts to bond brass in solid state by hot-rolling technique had not succeeded. Brass was, however, successfully bonded by fusion technique. Brass was melted over invar under reducing atmosphere. The composite was then cold-rolled to appropriate thickness with intermediate annealing. Work is under progress for bonding Al-bronze and silicon-bronze with invar by this technique; a little modification of the technique is, of course, necessary to suit individual cases.

(iii) *Bimetal with High Expansion Mn-Cu-Ni Alloys*—Attempts to bond Mn base high expansion alloys with invar by hot-rolling technique were not quite successful. Only partial bonding was obtained in some cases. The chief factors contributing to failure so far were due to (i) comparatively low working temperature of Mn base alloys (900°C. maximum), (ii) low capacity of the rolling mill, and (iii) inappropriate welding of the periphery of the composite. At such a low temperature, the maximum thickness of the composite that can be given sufficient reduction (about 25 per cent) for obtaining bond was about 0.2 in. (This limitation to thickness is imposed by the capacity of the mill.) While welding the periphery of such a thin composite, the bonding surface got badly oxidized and this hindered bonding. Work is under progress to try argon arc welding technique for welding the periphery of the composite. A few trials were also made to bond this alloy with invar by fusion technique. Perfect bonding was obtained in each of the trials, but excess diffusion of iron from invar and absorption

of carbon from the surrounding atmosphere led to the failure of the Mn alloy during hot and cold-working of the composite. Work is under progress to modify the technique so as to overcome these defects.

55.0 Development of Clad Metals

The investigation was taken up with a view to developing a suitable technique to bond dissimilar metals by rolling so as to have a combination product which will give the desired properties not obtained by a single metal and also to utilize the same technique for the production of different engineering components.

The process of cladding aluminium on mild steel and the determination of optimum conditions required for good bond strength were conducted earlier. After standardizing the technique for bonding, the work was done on utilizing the same technique for cladding aluminium alloys on mild steel. Obvious choice was for aluminium base bearing alloy cladded on mild steel so as to work as

a bimetallic bearing. Attempts were made in the first instance to make a suitable aluminium base bearing metal of improved bearing properties and roll-cladding it on hard backing material such as mild steel.

Heats were made for standardizing a suitable melting technique of the aluminium base alloy having uniform dispersion of lead and antimony in the matrix of solid solution of aluminium. For all the heats, charges were kept the same to obtain the aluminium base bearing alloy having 2 per cent antimony and 4 per cent lead as alloying elements. Moreover, a few other heats were also made using copper in addition to antimony and lead. Keeping the charge constant, the technique of addition of alloying element and furnace conditions were varied. For one heat, 1 per cent nickel was added to the melt containing lead and antimony to study its effect on dispersion.

The pieces from all the heats were examined under microscope after polishing and etching with sodium hydroxide solution. The alloys showed uniform dispersion of

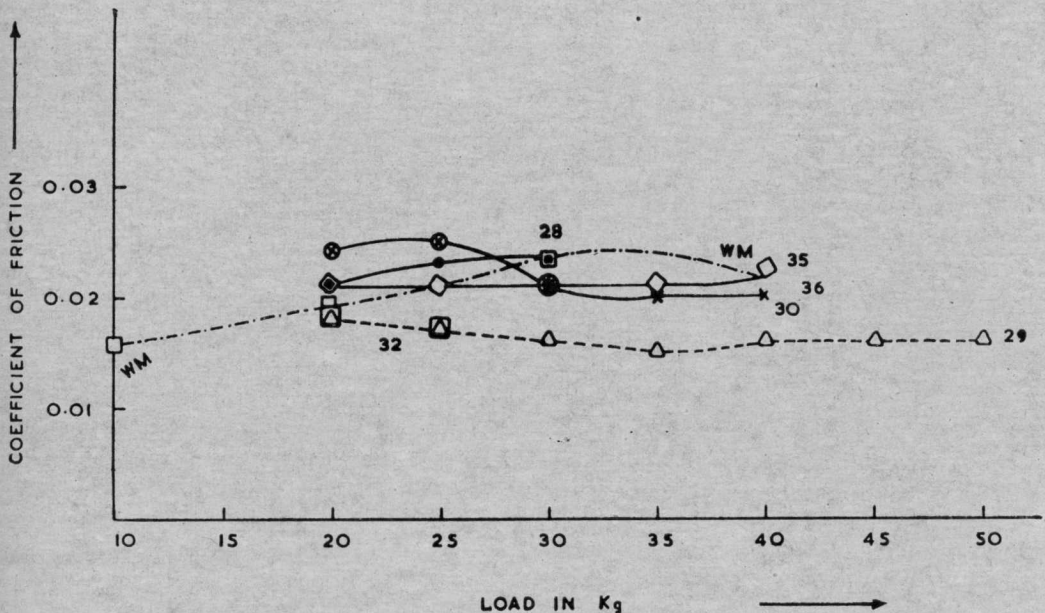


FIG. 31 — VARIATION OF COEFFICIENT OF FRICTION WITH LOAD

lead and antimony in the white etched matrix of aluminium. Much improvement in dispersion of lead and antimony was observed in the alloy AB 29 which contained 1 per cent nickel. Dendritic structure showing as cast condition was observed for the alloys AB 28 and 31 which contained copper. The structure consisted of deep etched black patches over the solid solution of copper in aluminium.

Mechanical properties of all the alloys are given in Table 46. The heats 32 to 36 exhibited the requisite mechanical properties of bearing; moreover, it can be forged and rolled. The frictional properties of the heats mentioned above had been shown in Fig. 31 and it was compared with the standard bearing metal (white metal). From the figure it was observed that the frictional properties of these materials were quite comparable with the standard bearing metal.

Table 46 — Mechanical Properties of the Alloys Studied

Heat	Ultimate tensile strength (tons/sq. in.)	Elongation percentage	Hardness in B.H.N.	Co-efficient of friction
AB 28	14.10	6.25	50.00	0.023
AB 29	8.40	12.50	36.50	0.016
AB 30	6.72	12.50	27.80	0.020
AB 31	13.90	6.25	50.50	0.019
AB 32	4.85	18.5	23	0.018
AB 33	4.8	12.5	27	0.021
AB 34	4.857	12.5	25	0.021
AB 35	5.076	10	28	0.021
AB 36	5.832	25	33	0.021

56.0 Study on Internal Friction of Metals

The aim of the project is to study the phenomenon of strain ageing, temper brittleness and the interactions of interstitial solute atom of nitrogen and carbon in iron through measurement of internal friction. Further friction studies were undertaken on high-purity iron supplied by BISRA and Armco iron in the K type torsion pendulum of 1.2 c.p.s., fabricated in the laboratory.

During strain ageing, the interaction of solute atoms with dislocations gave rise to slow-moving dislocation peak near the Snoek peak in the internal friction/temperature curve. This was studied at various temperatures because at high temperatures the precipitation and at lower temperatures the atmosphere formation were more pronounced. The high-purity iron wire of 19 SWG size was each time annealed at 650°C. for 1 hr. in dry hydrogen, water-quenched and its internal friction measurements taken. Ageing of these samples was done at room temperature for different periods of time as well as after restraining. Fig. 32 shows the results obtained.

The design of the inertia disc of the internal friction apparatus was changed to enable easy changing of frequency by putting additional weights on the disc, thus changing the moment of inertia. Armco iron wire samples were aged at 73°C. for different periods of time and internal friction measured. Fig. 33 shows the corresponding internal friction/temperature curves. The ageing curve is given in Fig. 33a, the curve being of exponential nature. Six other samples of Armco iron of 19 SWG size were annealed in hydrogen atmosphere at 650°C. for 1 hr. and quenched in water. The specimens were given about 4.5 per cent plastic strain in tension. They were then aged at room temperature for different periods and the internal friction measured at room temperature and up to 47°C. Table 47 gives the results of internal friction 10^{-3} at 1.5 c.p.s.

57.0 Photoelastic Stress Analysis

Photoelastic stress analysis covered the study of distribution of stresses in engineering components as such a study is necessary from the point of view of understanding the behaviour of the components as well as for improving the design. Photoelastic evaluation of stress concentration factor due to multiple notches was undertaken to

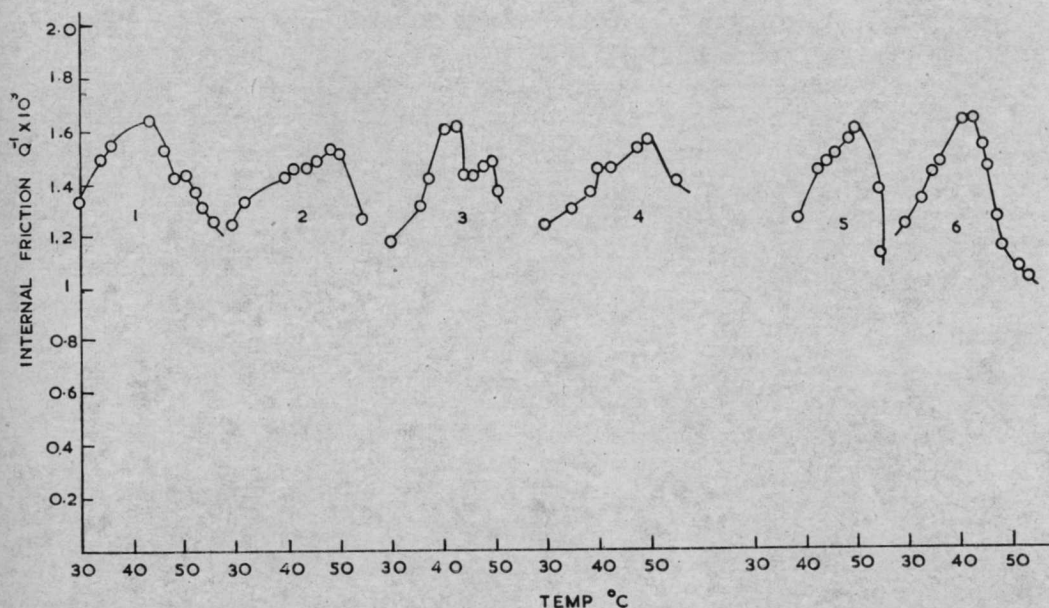


FIG. 32 — INTERNAL FRICTION OF HIGH-PURITY IRON AFTER AGEING FOR VARIOUS PERIODS [1, Initial; 2, 20 hr. ageing at room temperature; 3, 92 hr. ageing at room temperature; 4, 116 hr. ageing at room temperature; 5, restraining (just after); 6, aged for 186 hr.]

Table 47 — Values of Internal Friction at 1.5 c.p.s.

Approx. tempera- ture °C.	tan $\alpha \times 10^{-3}$ within half an hour after quenching	tan $\alpha \times 10^{-3}$ after room temperature ageing for							
		24 hr.	24 hr. & re- strained	48 hr.	72 hr.	72 hr. & re- strained	96 hr.	96 hr. & re- strained	120 hr.
22	—	—	—	3.55	3	—	29	—	—
23	2.73	—	—	—	—	—	—	—	—
25	2.84	—	—	—	—	—	—	—	—
27	3.45	—	—	4.75	3.65	—	—	—	—
30	3.83	—	—	—	—	—	—	—	—
31	4.25	—	3.55	—	—	—	—	3.0	2.84
32	4.4	8.55	4.04	0.08	—	3.6	—	—	—
34	—	4.05	—	—	3.94	3.7	3.2	3.5	3.55
35	4.7	—	—	5.2	—	—	—	—	—
36	5.7	4.3	—	—	—	—	—	—	—
37	5.35	4.3	4.3	5.35	4.3	3.83	3.55	3.75	3.83
38	5.68	5.68	—	4.4	—	—	—	—	—
39	—	4.9	4.5	—	4.7	—	3.83	—	—
40	5.68	—	4.56	5.55	—	—	—	3.85	4.2
41	5.72	—	—	—	—	—	4.1	—	—
42	5.72	5.08	4.4	5.55	4.9	4.2	—	4.2	—
43	—	5.30	4.3	5.65	—	—	4.05	3.93	4.75
45	5.68	—	—	5.55	4.7	3.4	3.83	3.6	4.0
				(44°C.)	(44°C.)	(44°C.)	(44°C.)	(44°C.)	(44°C.)
47	5.07	4.8	4.2	5.3	4.6	3.3	3.75	3.5	3.9

Some other specimens were aged at 120°C. Other experiments would be conducted at various ageing temperatures, viz. 30°, 50°, 75° and 100°C.

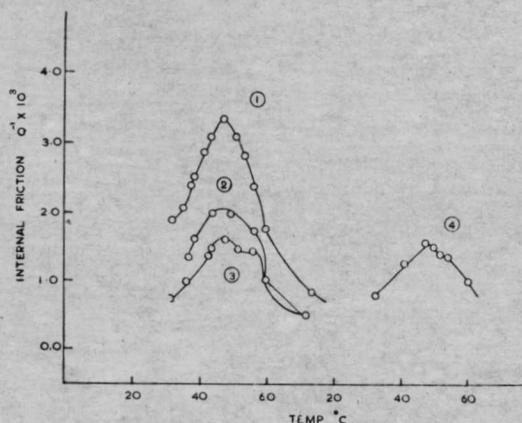


FIG. 33 — INTERNAL FRICTION VERSUS TEMPERATURE CURVES FOR ARMCO IRON ANNEALED AT 650°C. W.Q. [1, Initial; 2, 17 hr. at 73°C.; 3, 37 hr. at 73°C.; 4, 79 hr. at 73°C.]

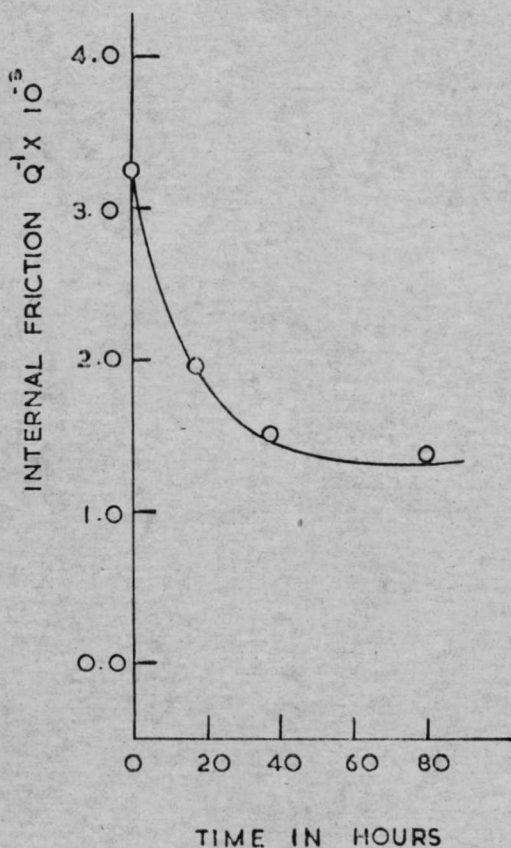


FIG. 33a — AGEING CURVE FOR ARMCO IRON

obtain quantitative measure of the stress-relieving effect of neighbouring notches. A rectangular beam 3 in. wide, 0.2 in. thick and 9 in. long with central notch $\frac{1}{4}$ in. dia., and 1 in. deep was stressed in pure bending. Two additional notches of same diameter were machined symmetrically on either side of the central one. Stress concentration factors (S.C.F.) were measured with different depths and proximity of the additional notches. The results obtained are furnished in Table 48.

Table 48 — Stress Concentration Data at Different Notch Depths

Depth of the extra notches	S.C.F.	Percentage reduction in S.C.F.
(1) Horizontal distance between the root of notches: $\frac{3}{4}$ "		
Single notch	3.23	—
$\frac{1}{8}$ "	3.23	—
$\frac{1}{4}$ "	3.04	6
$\frac{3}{8}$ "	2.99	7
$\frac{1}{2}$ "	2.88	11
$\frac{3}{4}$ "	2.35	27
1"	1.78	45
(2) Horizontal distance between the root of notches: $\frac{3}{4}$ "		
Single notch	2.73	—
$\frac{1}{8}$ "	2.58	5
$\frac{1}{4}$ "	2.51	8
$\frac{3}{8}$ "	2.57	6
$\frac{1}{2}$ "	1.93	29
1"	2.02	26
(3) Horizontal distance between the root of notches: $\frac{3}{8}$ "		
Single notch	3.06	—
$\frac{1}{8}$ "	3.31	—
$\frac{1}{4}$ "	3.30	—
$\frac{3}{8}$ "	3.03	—
$\frac{1}{2}$ "	2.78	8
1"	2.28	22

Further experiments with notches of different stress concentrations are being carried out to arrive at a general conclusion regarding the stress-relieving effect of notches.

58.0 Study on the Hot Extrusion Characteristics of Aluminium Alloys

This work was undertaken in order to make a systematic study on the effect of

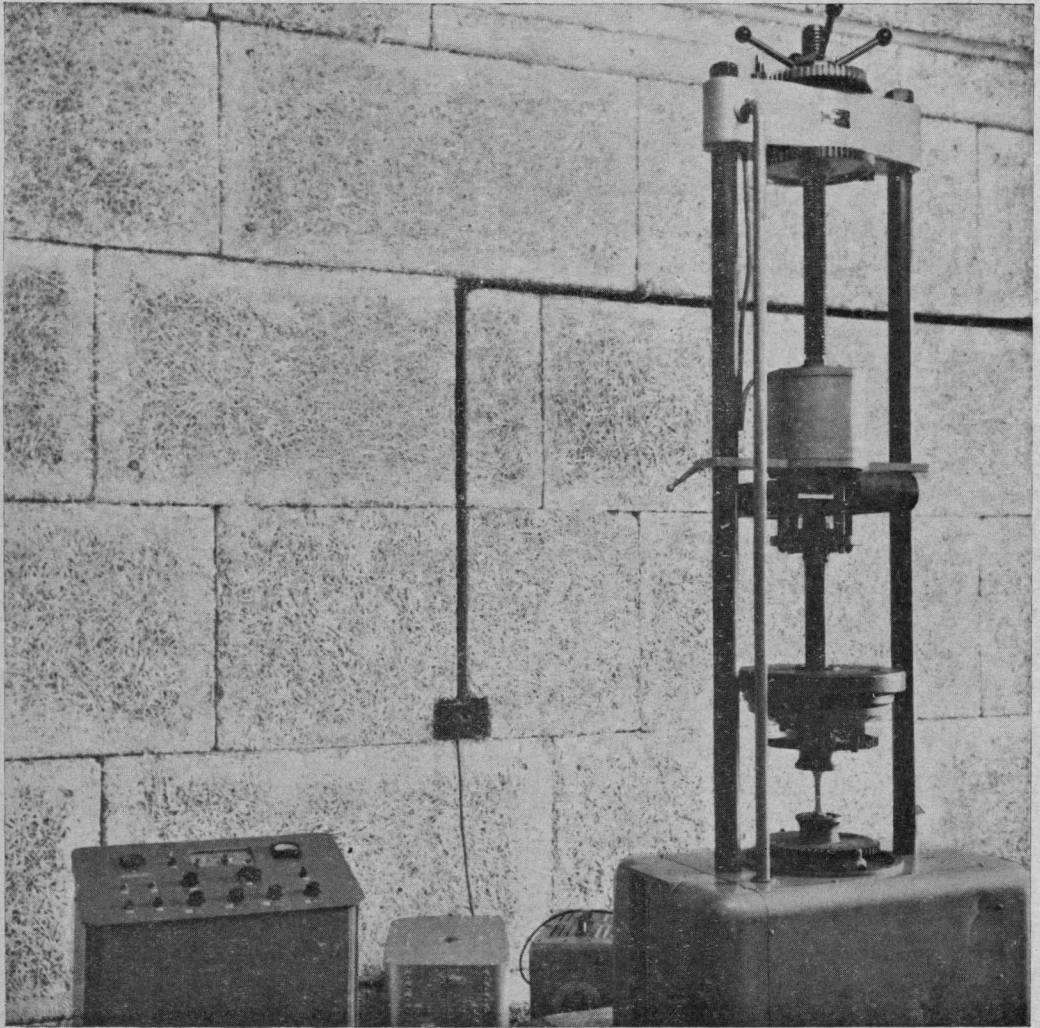


FIG. 34 — A VIEW OF THE HIGH-FREQUENCY VIBROPHORE INSTALLED AT THE NATIONAL METALLURGICAL LABORATORY FOR STUDYING THE FATIGUE PROPERTIES OF METALS AT ROOM AND HIGH TEMPERATURES

various factors such as shape and finish of the die aperture, temperature of billet and tools, speed of extrusion and nature of lubricant on the dimensional variation and metallurgical quality of the extruded duralumin type alloys.

Chill-cast and machined billets of the aluminium alloys of the composition given below were used for this investigation. Extrusions were carried out in the 500-ton

oil hydraulic direct extrusion press installed at the National Metallurgical Laboratory.

Material	Nominal analysis			
	Cu %	Mn %	Si %	Mg %
L 65	3.8-4.8	0.4-1.2	0.06-0.9	0.55-0.65

As the heterogeneity of the alloys of this group had pronounced effects on their extrusion behaviour, the machined billets were given prehomogenization treatment for 30 hr. at 460°C. After the prehomogenizing treatment, the billets were heated in the electric furnace for 4 hr. at temperatures of 400°, 410°, 430° and 460°C. respectively and transferred to the container of the extrusion press which was electrically heated to a temperature of 400°C. Various extrusion speeds were followed starting from the lowest available ram speed of 4 in. per minute to 29 in. per minute. Experiments were confined to the lower speed range because the alloys of this group had low melting constituents which might fuse at high temperatures developed in high-speed extrusion, giving rise to surface cracks of the extruded products. Shape of the die was also varied from plain cylindrical shape to dies with different bearing surface lengths and entry radii. The lubricant was also changed from aluminium front disc lubrication to grease and graphite lubrication on die and in the container. Extrusion pressure was noted in all cases and the surface condition of the extruded bar was carefully observed and dimensions thereof were accurately measured along the entire length of the product. Hardness measurements were made along the length of a few bars to reveal any characteristic changes taking place. Metallographic examination of billets in as-cast and as-homogenized condition was also made for assessing the extent of homogenization.

From the above series of experiments, it was observed that extrusion of round bars of high-strength aluminium alloys through a well-finished die with small entry radius and small bearing length gave good surface finish and extrusion could be effected at low pressure provided lubrication did not fail. The failure of lubrication gave rise to serious trouble such as seizure and consequent scouring at the die, and excessive extrusion ratio can be achieved in direct

extrusion if the billet length is less, since the friction between the container and billet, which has a profound effect on overall extrusion pressure, was noticed to increase almost linearly with the length of the billet. For a given die size, higher speed was noticed to give a smaller reduced diameter of the product presumably due to greater contraction during cooling. Too low a temperature of extrusion results in grain growth during subsequent heat treatment and too high a temperature and speed invariably gave a surface typical of the cracked surface due to melting of low melting constituents.

59.0 Production of Phosphor-Bronze

At the instance of the Railway Design & Standards Organization, investigation on production of phosphor-bronze for springs was undertaken and studies were made to obtain the desired properties of springs. The basic raw materials for the charge were cut pieces of electrolytic copper ingot, pieces of tin and phosphor-copper containing 13 per cent phosphorus for deoxidation and for introducing the specified amount of phosphorus in the bronze. The melts were carried out in a gas-fired frit furnace and ingots were cast in a rectangular mould. Composition of the phosphor-bronze ingots used for investigation is furnished in Table 49.

The ingots, after casting, were shaped to remove any surface defects for studying the effect of hot and cold-rolling properties of ingots made. Hot-working was carried out at 400°, 500° and 600°C. respectively. The optimum temperature for hot-rolling was found to be 500°C. The material could not be rolled at 400° and 600°C. as it failed while rolling. The cold-working properties of the phosphor-bronze ingots were found satisfactory. The ingots were reduced 60 per cent of their thickness by cold-rolling. Intermediate annealing was done at 500°C. after 60 per cent reduction.

Table 49 — Chemical Composition of the Phosphor-bronze Ingots Produced for Investigation

Heat No.	Sn %	P %	Pb %	Bi %	Cu %	Remarks
1	7.22	0.39	Trace	—	Remainder	—
2	7.22	0.39	do	—	do	—
3	7.22	0.39	do	—	do	—
4	7.30	0.36	do	—	do	—
5	7.08	0.31	do	—	do	—
6	6.60	0.28	do	Trace	do	Phosphor-copper was added only on the basis of higher limit, no extra addition was made for deoxidation

To give the requisite spring temper to the material, the cold-rolled and annealed pieces were given different percentages of cold reduction. Mechanical properties of the pieces were studied after each percentage reduction. This was carried out until the optimum reduction per cent was obtained which will give the requisite mechanical properties given in different standards for phosphor-bronze springs (flat products). Data obtained are furnished in Table 50.

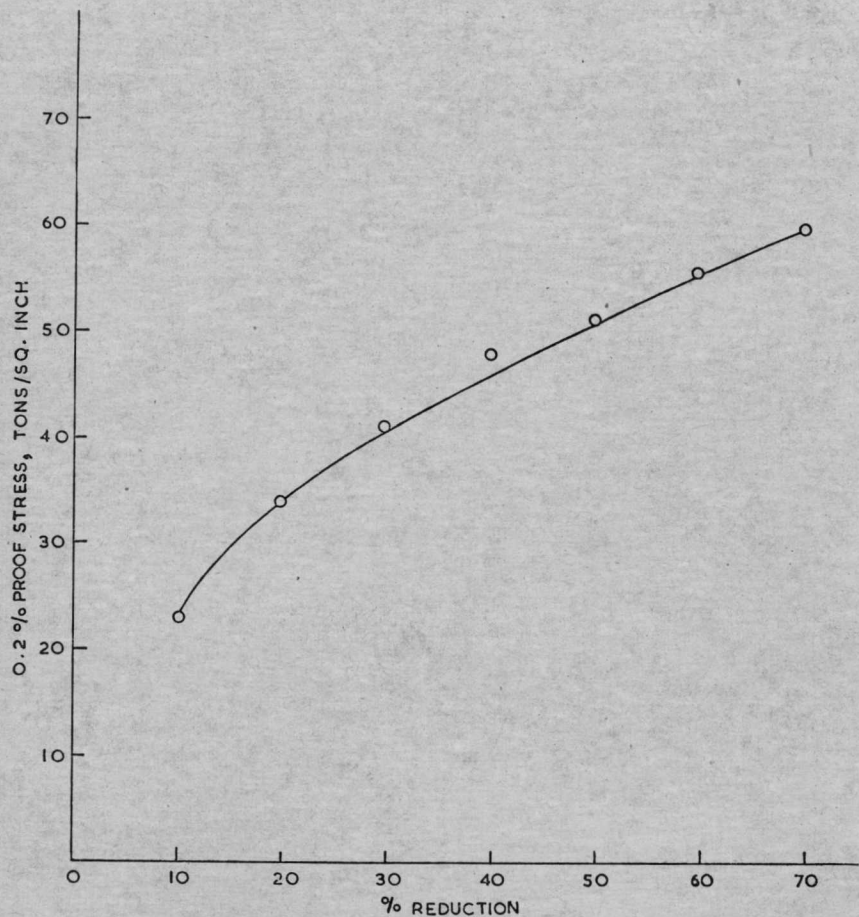


FIG. 35 — VARIATION OF PROOF STRESS WITH PERCENTAGE REDUCTION OF PHOSPHOR-BRONZE

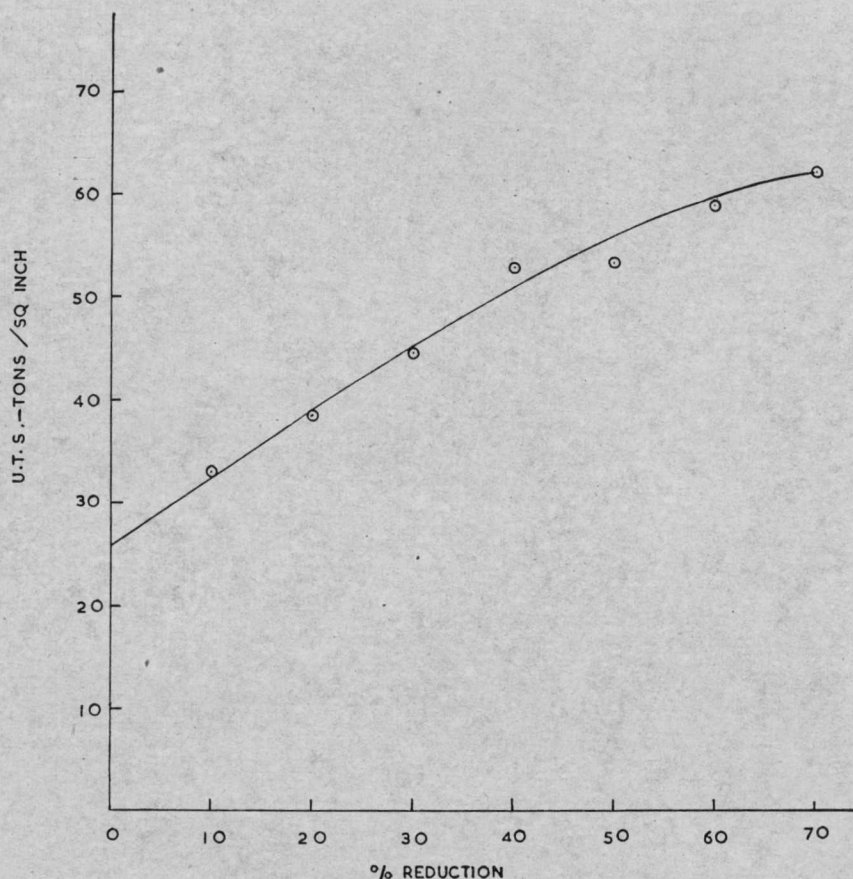


FIG. 36 — VARIATION OF ULTIMATE TENSILE STRENGTH WITH PERCENTAGE REDUCTION OF PHOSPHOR-BRONZE

Table 50 — Mechanical Properties of the Phosphor-bronze Spring Strips

Reduction %	0.2% Proof stress tons/sq. in.	U.T.S. tons/sq. in.	Elongation %
10	23.25	33.21	43.7
20	34.38	38.67	27.3
30	41.55	44.74	14.0
40	48.50	53.01	6.0
50	50.90	53.89	6.0
60	56.40	59.02	1.5
70	60.03	62.50	Nil

the internal stresses of the material without hampering the mechanical properties.

60.0 Study on Welding

Studies on pressure welding of aluminium strips were continued. In order to study the variation of bond strength with the thickness of the material, a series of experiments were carried out on aluminium strips of different thicknesses and of different hardness. For aluminium strips having a hardness of 48 VPN three gauges of sheets were used, i.e. 0.127, 0.067 and 0.039 in. and for strips of hardness 24 VPN two gauges of sheets were used, i.e. 0.125 and 0.067 in. Bond strength was

After giving the spring temper, the materials were subjected to stress-relief annealing at different temperatures and times. The temperature of the above annealing was so chosen as will only reduce

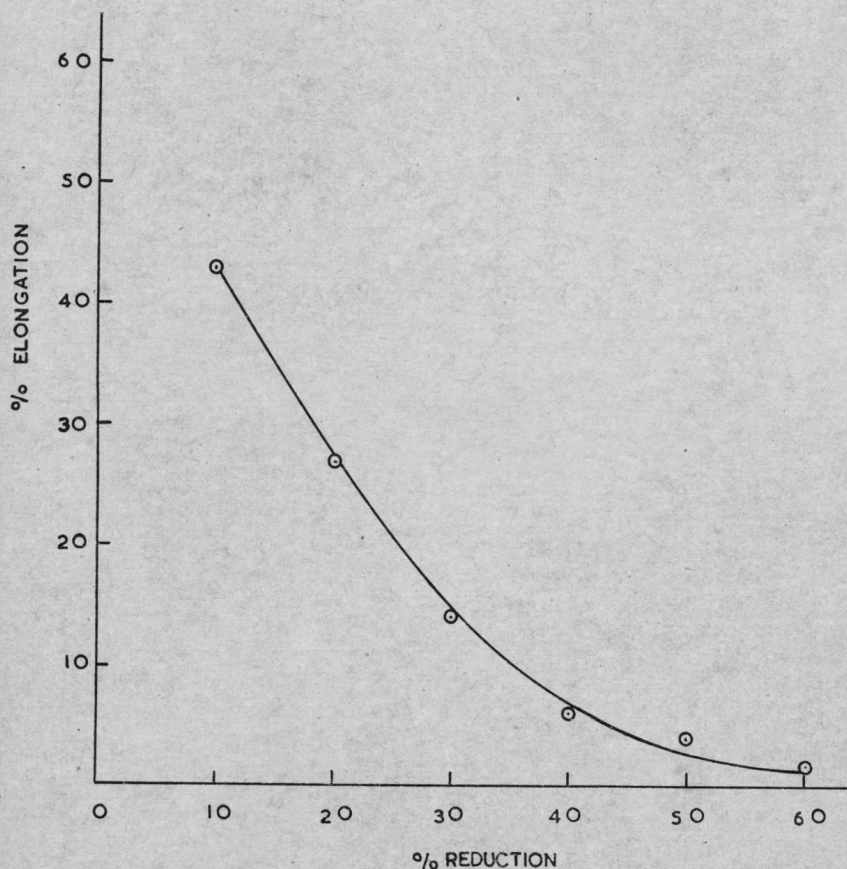


FIG. 37 — VARIATION OF PERCENTAGE ELONGATION WITH PERCENTAGE REDUCTION OF PHOSPHOR-BRONZE

determined in each case and it was plotted against percentage reduction as shown in Fig. 38. The width of the tool was kept the same in all cases, i.e. $\frac{1}{8}$ in. thick.

61.0 Production of Basic Refractories from Indigenous Resources

(i) *Almorah Magnesite* — This investigation was taken up with a view to evaluating the physical and refractory properties of Almorah magnesite and to assess its suitability for the manufacture of refractories.

During the period under review two research reports were prepared. The first

report dealt with the mineralogy of Almorah magnesite. A detailed study of petrographic, D.T.A. and X-ray methods had shown that the magnesite had some ferric oxide in its structure. Lime and silica were present in the form of dolomite and talc. Some chlorite was also detected. The second report dealt with the effect of heat on the constitution, crystal growth and densification of Almorah magnesite. X-ray, electron microscopic and petrographic methods were adopted in the study. The salient features were as follows.

On heating Almorah magnesite, periclase formation took place at temperatures of 600°C. and above. The periclase formed after the dissociation was highly stained

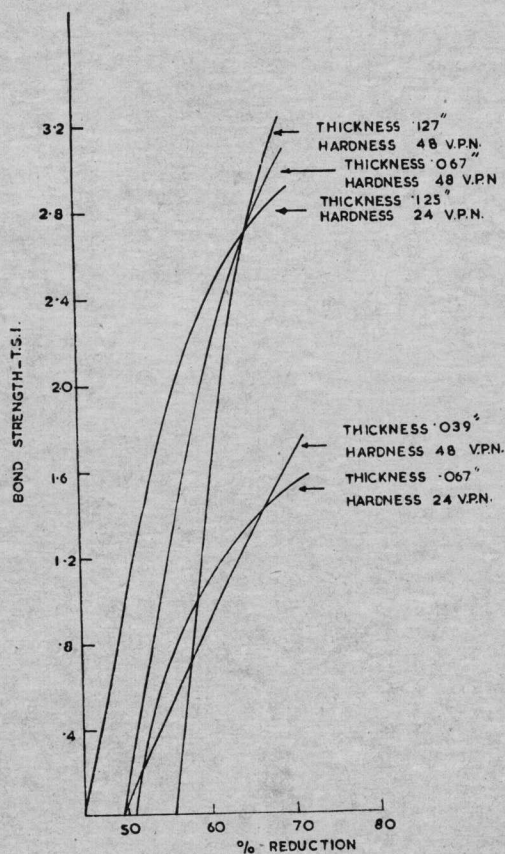


FIG. 38 — VARIATION OF BOND STRENGTH WITH PERCENTAGE REDUCTION OF PHOSPHOR-BRONZE

and recrystallized between 800° and 850°C. (Fig. 39a). Considerable increase in crystal size of periclase took place between 1200° and 1300°C., and at higher temperatures the size of the periclase crystal further increased. The initial grain size of magnesite studied had little effect on the crystal size development of periclase at higher temperatures. The impurities of dolomite and talc broke down between 800° and 900°C. and reacted with magnesia to form monticellite and forsterite at high temperatures. Magnesioferrite formation was detected at 800°C. and above. Significant sintering and densification of Almorah magnesite took place between 1200° and 1300°C. and increased with increasing temperatures up to 1700°C. (Figs. 39b and 39c). Ball-milling

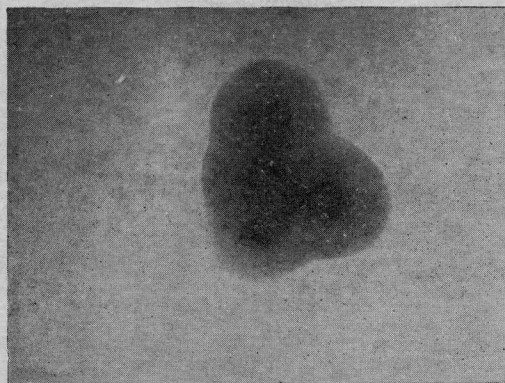


FIG. 39a — ELECTRON MICROGRAPH OF ALMORAH MAGNESITE FIRED TO 800°C. $\times 120,000$

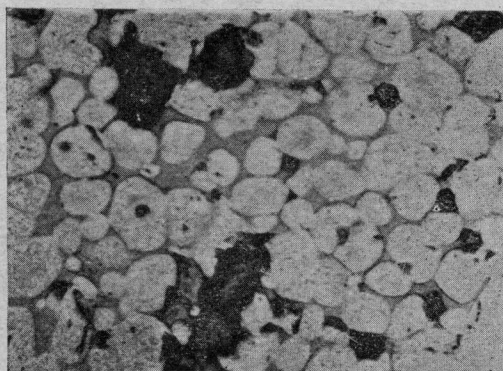


FIG. 39b — MICROPHOTOGRAPH OF ALMORAH MAGNESITE FIRED TO 1400°C. $\times 450$

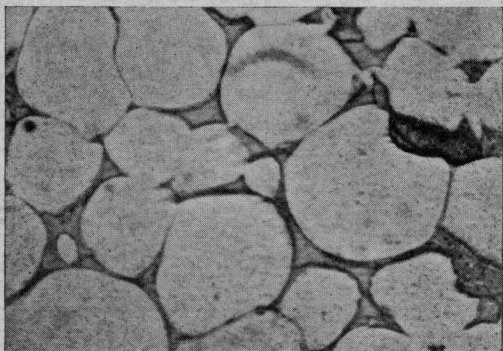


FIG. 39c — PHOTOMICROGRAPH OF ALMORAH MAGNESITE FIRED TO 1000°C. $\times 450$

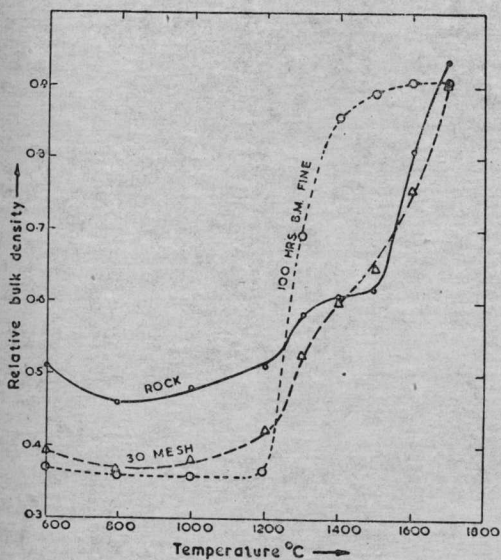


FIG. 40 — DENSIFICATION OF ALMORAH MAGNESITE

improved the extent of sintering and densification at temperatures above 1200°C. (Fig. 40). In order to get a dense sinter for magnesite bricks, it was not necessary to crush the magnesite below $\frac{1}{2}$ in. size.

In continuation of the work conducted on the study of physical and refractory properties of Almorah magnesite without recourse to beneficiation, lime in calculated proportions was added to the mixes and fired to high temperature. The test specimens obtained thereon indicated excellent physical and refractory properties. The refractoriness under-load value significantly improved to 1780°C. (Ta) and above.

(ii) *The Correlation between the Constitution and Physical Properties of Magnesite Refractories* — The investigation was started with a view to determining the effect of variation of chemical compositions, especially lime and silica, on the constitution and physical properties of magnesite. The results obtained would be helpful in forming standard specifications for basic refractories.

The magnesite studied had the following analysis after dead burning: SiO_2 , 5.25; Al_2O_3 , 0.5; Fe_2O_3 , 5.24; CaO , 1.15; and

MgO , 89.86 per cent, with lime/silica molar ratio of 0.378 per cent. Four compositions were made adding lime progressively, such that lime/silica molar ratio increased to 1.908. Another four compositions were made such that the ratio decreased to 0.1206. All the fired samples were examined by X-ray diffraction studies. With progressing lime addition, the amount of forsterite and monticellite decreased or even disappeared with a subsequent increase of merwinite and dicalcium silicate. With progressive silica addition, the amount of forsterite considerably increased. With lime addition, the porosity was decreased with a slight increase in bulk density. There was no significant change in crushing strength. With silica addition there was no significant change in the porosity, but the crushing strength and refractoriness under-load were improved. Further tests are being conducted.

62.0 Development of High-alumina Refractories

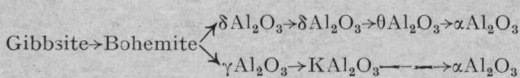
High-alumina refractories are required to meet a wide variety of service requirements in metallurgical furnaces and cement kilns. The presence of high titania in many bauxite deposits made them unsuitable for manufacture of refractory bricks containing above 60 per cent alumina. Investigation work was, 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* — In continuation of the investigation conducted earlier, about one ton representative sample of Kashmir bauxite was collected. Part of it was calcined in lump form at different temperatures and another part was crushed and ground to -80 mesh and mixed with 15 per cent plastic clay, pressed into briquettes

at 5000 lb./sq. in. and fired at different temperatures. The grog obtained by both the methods was crushed and graded and on the basis of packing density studies, coarse, medium and fines were mixed in requisite proportions to give maximum bulk density, and 2 in. diameter buttons, quarter size briquettes as well as full-size bricks were pressed at pressures indicated in the table using 4 per cent clay as the binder. The specimens were fired at different temperatures given in the table. The physical properties of the different specimens are given in Table 51.

(ii) *Development of Foamed Alumina Bricks* — In pursuance of the work on the development of foam alumina bricks, the composition of the calcined alumina to make a dense sintered mass was standardized with suitable addition of sintering agents like TiO_2 , Cr_2O_3 , etc. The procedure for producing a suitable foam and stabilizing the foam was standardized. Foam bricks were cast in plaster mould as well as wooden moulds. It was found that bulk density of the foamed product was about 1 gm./cc. To decrease the bulk density of the product, a few trials were made by adding combustible material in the batch which gave encouraging results. Further experiments were planned to study the effect of fineness of the sintered alumina and addition of different combustibles.

The thermogravimetric study and the X-ray studies revealed that Bayer's alumina, which has the structure of gibbsite, breaks down on heating in the following manner:



63.0 Development of Calcium-Aluminate Cement

High-alumina cements offer wide application in refractory castables and concretes that can withstand up to 1300°C. and above. These cements, which are hydraulic setting, are composed essentially of calcium-aluminates and varying percentages of other

Table 51 — Physical Properties of High-alumina Refractories from Kashmir Bauxite and 4 per cent Clay as Bond

Composition	Calcining temperature	Forming pressure of fired specimens	Firing temperature of fired specimens	Apparent porosity %	Bulk density	Specific gravity	Firing shrinkage (Linear) %	Cold crushing strength lb./sq. in.	Refractoriness under load °C.
Bauxite lumps	1600°C. for 3 hr.	5000 lb./sq. in.	1550°C. for 3 hr.	30-31	2.3	3.2-3.3	2.5	4000	N.D.
do	1600°C. for 5 hr.	10,000 lb. by 500-ton hydraulic press	1500°C. for 5 hr.	26	2.5	3.385	1.0	1700	Ta 1580 Te 1800
do	1650°C. for 3 hr.	10,000 lb./sq. in.	1600°C. for 3 hr.	20.5	2.7	3.46	5.50-6.0	14,000	N.D.
Bauxite — 80 mesh	1600°C. for 5 hr.	10,000 lb./sq. in.	1500°C. for 5 hr.	30.0	2.3-2.32	3.28	0.5	5500	Ta 1550 Te 1700
15% plastic clay									
do	1600°C. for 5 hr.	10,000 lb.	1600°C.	22-24	2.60	3.36	3.5-4.5	16,000	Ta 1540 Te 1700

compounds. They are now used in large quantities in India for various applications in the iron and steel industry as also in some high-temperature furnaces and the country's requirements of these cements are met by import. It is expected that demand will increase in future owing to the growing trend towards the use of monolithic linings and refractory concretes, particularly in locations where special and complicated shapes made by conventional refractory-making practice, involving the employment of skilled labour and high cost, are used. In view of this and the availability of raw materials for their manufacture in our country, a two-pronged study was taken up on the development of high-alumina cements, namely (i) studies on pure calcium aluminates for high-temperature applications and (ii) development of molten aluminous cements of the 'cement fondu' type from indigenous raw materials.

(i) *Pure Calcium Aluminate* — The physical properties of calcium monoaluminate mixed with different percentages of graded aggregates like calcined bauxite, sillimanite and kyanite were studied. With increased percentage of cement in the aggregate, the porosity was lowered and the crushing strength was increased. The refractoriness under-load tests showed variable results with different types of aggregates. The spalling resistance of these aggregates was excellent. The effect of firing temperature

on the modulus of rupture is being studied.

(ii) '*Cement Fondu*' — The principal raw materials, e.g. bauxite and limestone, for the manufacture of molten aluminous cements of the cement fondu type contain, in addition to Al_2O_3 and CaO , other oxides such as FeO , Fe_2O_3 , SiO_2 and MgO which play a significant role in the preparation of the cements as well as in the refractory and cementing properties of the product. Indian bauxites and limestones containing these oxides in varying proportions pose special problems for the formation of suitable mixes from them for cement making. Experiments were, therefore, planned on the systematic study of the optimum conditions for making these cements, utilizing bauxites and limestones from different well-known localities. Several series of melts of cement were made using bauxite from Kashmir, Lohardaga and Katni and limestones from Madras and Bisra, with the $\text{Al}_2\text{O}_3/\text{CaO}$ ratio of the mixes varying from 0.6 to 1.4. Optimum range of $\text{Al}_2\text{O}_3/\text{CaO}$ ratio for the different series was fixed and a number of compositions giving good cements were arrived at. Properties of some of the good cements prepared in this study are given in Table 52 along with those of two imported cements. It is seen from the table that cements developed in this study compared well with the imported cements. Extension of the studies using some more bauxites and limestones from other sources is under progress.

Table 52 — Comparison of Physical Properties of Imported Cements with Those Developed at NML

Property	Cements developed in the study			Imported cements	
	M-4	L-1	KM-5	1	2
Setting time initial (mt.)	140	120	183	20	35
Final (mt.)	245	225	337	200	360
Cold compression strength (lb./sq. in.)*	4280	4420	4290	4570	5710
P.C.E. (Orton cone)†	—	14	14	16	—

*As per ASTM specification C-266-58 (Gilmore needle method).

†As per ASTM specification C-109-58.

64.0 Studies on Carbon

The objective of this project is to study carbonaceous materials from the point of view of their ultimate utilization as raw materials for the production of carbon products for diverse applications in metallurgy. Such a study involved in the first instance examination of their pyrolytic characteristics with and without pretreatment as well as additions which will 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 Jharlia and Raniganj area and lignite from Neyveli and petroleum coke from Digboi were initially taken up for studies.

Pyrolyses in the range of 360-450°C. of a coking bituminous coal, two high volatile, high sulphur coking coals of Assam and lignite from Neyveli were studied. Analyses and other properties are given in Table 53. Differential thermal analysis study was also conducted.

Thermogravimetric studies at constant temperature were made with the help of a thermal balance which consisted of a 'Sartorius' analytical balance placed over a vertical kanthal wound tubular furnace. Specific gravity was determined with kerosene oil as the fluid medium. Agglutination value was determined according to the method specified by ASTM.

The differential e.m.f. curves obtained with various coals are given in Fig. 41. It indicates that the bituminous coking coal has a characteristic endothermic peak in the range 415-490°C. which other material did not exhibit. In thermogravimetric analyses study, the experimental data were graphically represented as plots of percentage weight loss (based on the total weight of the sample) versus time and it was found that the curve conformed to the equation

$$Y = \frac{X}{a+bX} + c, \text{ where } X \text{ is any given time,}$$

Y the corresponding cumulative percentage

Table 53 — Proximate Analyses, Ultimate Analyses, Agglutinating Values and Densities of the Materials

Coal location	Proximate analyses					Ultimate analyses (DMM-free bases)			
	Giridih	Assam-1 5' seam Tikak mine	Assam-2 20' seam Tikak mine	Neyveli lignite		Giridih	Assam-1	Assam-2	Lignite
Moisture (%)	0.50	2.35	2.05	As received	Dried at 110°C.	90.07	80.58	80.34	71.99
Ash (%)	5.05	1.85	2.45	54.13	—	5.00	5.77	5.69	4.98
Volatilities (%)	27.70	43.15	43.69	4.83	10.53	0.49	3.61	4.94	0.99
Fixed C. (%)	66.75	52.65	51.81	23.22	50.6	1.85	1.54	1.49	0.72
				17.82	38.87	2.59	2.50	7.54	21.32
Nature of sample			Specific gravity			Agglutinating value			
						Giridih	Assam-1	Assam-2	Assam-2
As received	1.317	1.268		Assam-2	Lignite	1.8	Nil	Nil	Nil
Dried at 110°C.	N.D.	1.28		1.283	1.162	9.25	Nil	Nil	Nil
				1.289	1.413	28.7	Nil	Nil	Button of no strength
						53.0	Button of no strength	3.6	
						122.0	11.7		25.7

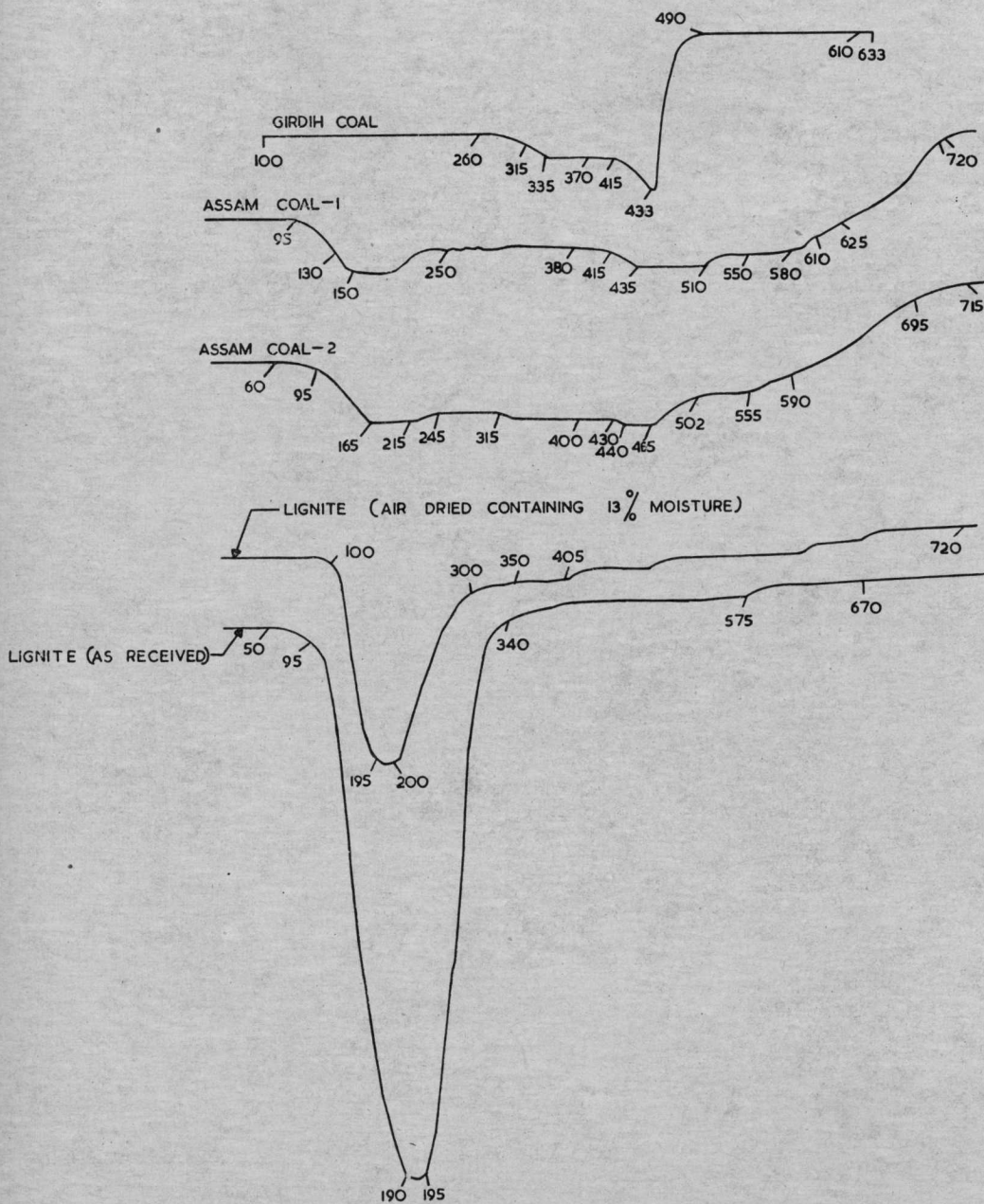


FIG. 41 — D.T.A. OF VARIOUS COALS

loss and a , b and c are constants — c being the value of the intercept on the time axis at experimental zero times. From this equation, the experimental data were extrapolated to determine the theoretical zero time and

the corrected cumulative percentage loss versus time curves were plotted. Some of the data obtained are given in Fig. 42.

In order to obtain the rate constants and other kinetic data from these curves, two

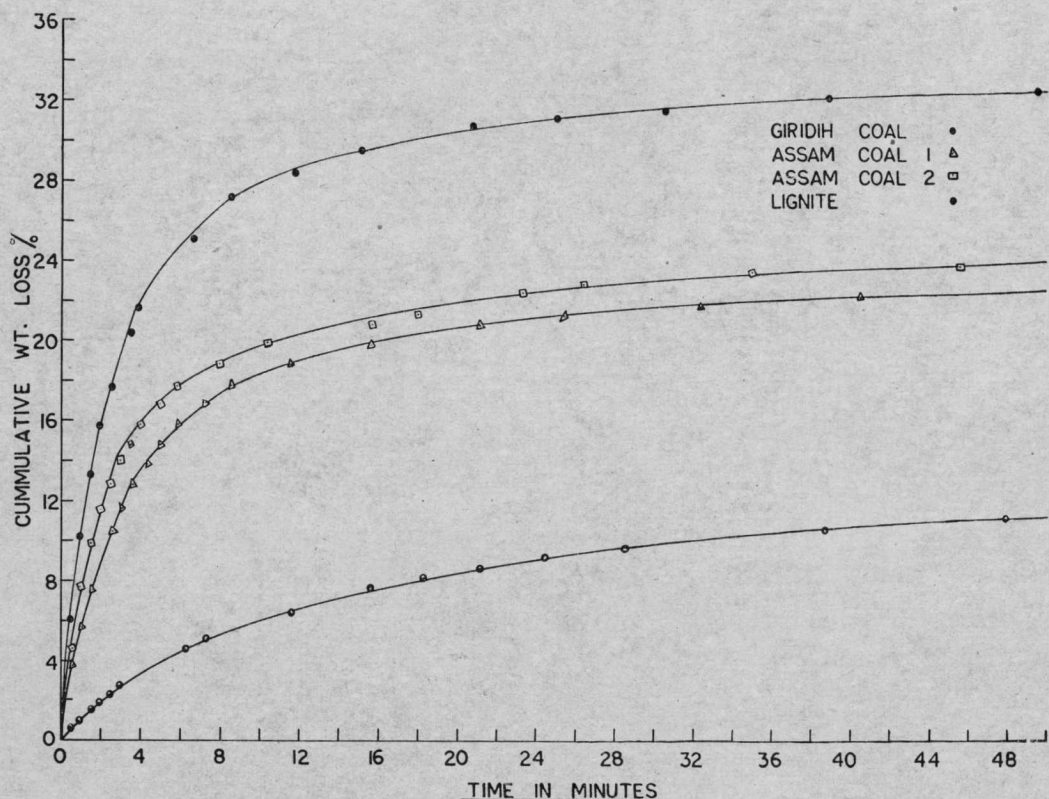


FIG. 42 — CORRECTED INTEGRAL CURVES OF THERMAL DECOMPOSITION OF VARIOUS COALS EXTRAPOLATED TO ZERO LOSS AT 425°C.

methods were adopted. In one, according to the procedure followed by Madorsky *et al.*, the rate per cent loss per minute was plotted against the cumulative percentage loss based on the original samples. In the other case, the procedure followed by Murry and White was adopted. According to this procedure, an approximate half time was obtained from the corresponding curve assuming the

limiting loss $\left(y = \frac{1}{b} + c\right)$ as L_{∞} and a

multiple of this value was assumed as ΔT . $L - L$ at various time intervals T , T_1 , T_2 , etc., was calculated and $\log_{10} \Delta L$ was plotted against T . The plots of rate per cent per minute against cumulative percentage loss were straight lines up to approximately 50 per cent of the limiting loss in

the case of Giridih coal, 50-70 per cent in the case of Assam coals and 50-65 per cent in the case of lignite.

Activation energy was calculated from the initial rates, assuming that the reacting part of coal was constant at all temperatures under study and that the limiting loss obtained for the various experimental curves merely indicated the point at which the reaction slowed down so much that these could not be followed further. The Arrhenius plots based on this assumption indicated that the pyrolysis of all the materials under study followed two simultaneous first-order reactions in the temperature range under study. In the case of Giridih coal, the point of inflexion was observed at 406°C., in Assam coals at 380°C. and in lignite at 400°C. In the second

method, i.e. according to Murry and White, $\log_{10} \Delta L$ versus T were plotted. Such plots indicated two reactions with differing velocity constants taking place simultaneously at all temperatures. At high reaction times, the slopes should correspond to the K value of the slow reaction. On this assumption, at all temperatures under study, two or more reactions must be taking place in these coals. The slope at lower reaction time under such circumstances corresponds to an average value of the K values of all the reactions taking place simultaneously. The Arrhenius plot obtained indicated that activation energies of the reactions that are rate controlling at high temperature were of the order of 45 k.cals. per mole in the case of coking coals while with lignite the activation energy was only about 16 k.cals. per mole. From the data obtained it is possible to assume that the reacting part of coal is constant in the temperature range 415-450°C.

65.0 Study on Refractory Raw Materials from Kerala

Under the Council of Scientific & Industrial Research Fellowship Scheme, work on this project was continued.

(i) *Study of Sillimanite Sand* — Previous experiments had shown that addition of rare earth chloride would help to sinter sillimanite. So further work was done for finding out the optimum condition under which sillimanite brick could be made using rare earth chloride as a sintering agent. It was found that if sillimanite sand and powder (—200 mesh) were mixed in proportions varying from 50 to 80 in the case of raw sand and 50 to 20 in the case of fine respectively and rare earth chloride solutions to the extent of 2-3 per cent of the mix were added, good sillimanite brick could be obtained by firing at 1600°C. and soaking for 3 hrs. X-ray analysis of the fired product showed that mullitization had taken place to a considerable extent.

(ii) *Study of Zircon* — The investigation of the application of rare earth chloride solutions in sintering of zircon was also continued. Zircon powder mixed with raw zircon sand in the ratio 50 : 50 and heated with 2-3 per cent of rare earth chloride solutions formed a good mix to produce zircon bricks at 1400°C. It was found that the composition containing 50 per cent grog, 25 per cent fines and 25 per cent raw sand gave a good mix with the above-mentioned quality of rare earth chloride. The above composition was also found to be useful for the preparation of crucibles. Investigations are being continued to see whether any further improvement in refractory properties could be effected.

(iii) *Separation of Rare Earth* — Previous experiments on the action of a mixture of calcium oxide and calcium chloride on rare earths like cerium oxide, lanthanum oxide, praseodymium oxide and samarium oxide had shown that lanthanum oxide formed some complexes with the above better than the others. Assuming that ingredients of rare earth combination which will form complexes with the above flux may exhibit difference in properties and taking advantage of this difference in properties for effecting the separation, the heated mixture was chlorinated at 1300°C. The volatile products were analysed and found to contain heavy rare earths. The residue left behind was acidified with dilute hydrochloric acid and it was found that concentrates of neodymium oxide and lutecium oxide could be separated from the acid leaching at different pH . It was also found that the ultimate residue left after the acid leachings resembled praseodymium oxide.

66.0 Thermal Conductivity of Insulation Refractories

Thermal conductivity and physical properties of some more samples of insulating bricks collected from different sources were determined. In addition to physical

properties like reheat change (linear) at 1250°C. (24 hr.), refractoriness under-load with a load of 10 lb. per sq. in., permeability and modulus of rupture were determined with a view to correlating these physical properties with each other and the ultimate insulating characteristics of the bricks. The physical properties studied during the year under review are furnished in Table 54.

Table 54—Physical Properties of the Insulation Bricks Studied

Sl. No.	M.O.R. in lb./sq. in.	Permeability through one skin C.G.S. unit	Reheat change at 1250°C. (24 hr.) (linear)	Refractoriness under-load °C. at 10 lb./sq. in.	
				Ta	Te
Foreign					
1	183	1.48	0.22	1435	1630
Indian					
2	263	0.96	0.65	1145	1430
3	223	0.85	3.9	1075	1315
4	N.D.	0.14	5.4	N.D.	N.D.
5	159	0.55	N.D.	940	1150
6	193	0.61	0.2	1360	1490
7	273	1.07	0.7	1100	1340
8	307	0.46	0.048	1580	1650
9	229	0.76	0.12	1250	1570
10	339	0.92	0.2	1260	1290
11	132	0.56	N.D.	980	1145

67.0 Plasticizing of Non-plastic Refractory Materials

An important and major advance in ceramic technology is that of producing artefacts from high-temperature refractory raw materials which do not have binding properties in the raw state without the addition of binders which alter chemical composition and more or less adversely affect their high-temperature properties. Pure oxides like alumina, magnesia and thoria are examples of such refractories which are 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.

A full report on the first stage of this project was prepared and the salient conclusions arrived at were as follows:

- (i) In pot-milling of sillimanite sand, the production of material finer than 2.5 microns was directly proportional to the duration of grinding.
- (ii) The ratio of pebble weight to weight of sillimanite had a distinct influence on the amount of fines produced up to a size of 4 microns. Up to this size, the relationship between the pebble/sillimanite ratio and the weight of material finer than specified size was parabolic and conformed to the equation $y = a + bx + cx^2$, where y was percentage finer than a specified particle size, x was the pebble/material ratio and a , b and c were constants. This relationship did not appear to hold good in finer sizes.
- (iii) There was also an optimum pebble/sillimanite ratio at which maximum amount of grinding took place. Under the present experimental conditions, this ratio was 2.4.
- (iv) The pH of the fluid medium appeared to have a profound influence on grinding. Under the present experimental conditions, a starting pH of 5 had maximum effect. It was felt that more experiments would be necessary to establish quantitative relationships.
- (v) Base exchange capacities of ground sillimanites showed a definite trend and had a linear relationship with the amount of material finer than 1 micron that the grinds contained. The quantitative relationship conformed to the equation $y = 0.02x + 0.18$, where y was the base exchange capacity in m.e. and x the weight percentage of material finer than 1 micron.
- (vi) Grinding sillimanite finer than 1 micron released considerable quantities

of alumina which could be leached out with hydrochloric acid. The nature of such a break-up and its influence on casting and binding properties of the sillimanite grinds are expected to form interesting aspects for further study.

In continuation of the grindability experiments, batches of sillimanite, pebbles and water were charged in 10½ in. dia. pot mills and run on the rack for 72 hr. The ratio of sillimanite to pebbles was constant at 2.4. Ratios of sillimanite to water charged were 1:0, 1:0.5, 1:1, 1:1.5 and 1:2. Table 55 gives the particle size distribution of sillimanite so ground.

Table 55 — Particle Size Distribution of Sillimanite					
Particle size microns	Sillimanite/water ratio				
	1:0	1:0.5	1:1	1:1.5	1:2
	Cumulative percentages				
26	92	100	100	100	100
8	66	92	100	100	98
6	56	79	95	91	85
4	47	64	81	78	72
3.4	43	53	72	67	62
2.6	37	44	59	56	50
1.1	20	23	31	24	19
0.8	13	15	20	15	17
0.6	12	11	14	13	13

After determining the grinding characteristics of sillimanite and base exchange characteristics of sillimanite samples of different finenesses, larger batches of sillimanite were ground in a ball mill with 2.4 times the weight of pebbles and same weight of water. The mill was run for 72, 120, 168 and 216 hr. The sillimanite slip was then dried and sampled. The particle size distribution of the sillimanite is furnished in Table 56.

X-ray photographs of sillimanite samples pot-milled to various durations indicated that only sillimanite phase is present. Action of electrolytes like hydrochloric acid and sodium carbonate on fluidity of 50 per cent solids suspensions of sillimanite ball-

Table 56 — Particle Size Distribution of Ball-milled Sillimanite				
Particle size microns	Ball-milling time (hr.)			
	72	120	168	216
	Cumulative percentages			
26	100	97	100	100
8	80	86	89	95
6	64	75	83	84
4	52	63	68	70
3.4	44	54	60	59
2.6	35	44	50	53
1.1	16	22	28	29
0.8	10	16	21	20
0.6	8.5	10	15	13

milled to the various durations was studied. The results of the preliminary study of relationship between pH and fluidity of these suspensions indicated that there was no well-developed minima on the curve. This minima was obtained in the case of clay slips and other casting slips.

In order to test the extrudability of plastic sillimanite pastes sillimanite samples ball-milled for various durations were taken and extrusion experiments made in the brass extrusion press. Starch paste was used as a temporary binder for extrusion. This paste was made from a mixture of starch of ammonium hydroxide and water which was evaporated to half the mass and later cooled for about 5 hr.

On the basis of the small-scale trials, 10 lb. samples were extruded in the experimental pug mill. One to one and a half in. dia. rods were extruded using 20 per cent by weight of starch paste. Shrinkages and modulus of rupture values of the dried bars were edetermined. Further work is in progress.

68.0 Development of Steel-pouring Pit Refractories

Considerable difficulty is experienced in obtaining the pouring pit refractories of satisfactory quality from indigenous sources. As these refractories form a vital link in steel production, the Durgapur Steel Project authorities desired that investigation to

Table 57—Comparative Study of the Imported and Indigenous Nozzles

Properties	Imported nozzle	Imported insert	Indigenous nozzles			
			K	X	SN	B
Chemical analysis, %						
SiO ₂	55.23	51.14	59.20	58.75	59.28	49.51
Al ₂ O ₃	33.09	40.78	53.80	34.36	35.00	40.91
Fe ₂ O ₃	6.51	3.20	1.53	2.65	2.05	3.13
TiO ₂	1.03	1.11	2.57	2.30	2.34	3.05
CaO	1.78	1.73	0.73	0.51	0.72	0.58
MgO	1.85	1.58	0.25	0.33	0.29	0.18
Na ₂ O	—	—	0.99	0.62	0.65	1.63
K ₂ O	—	—	—	—	1.69	1.20
Apparent porosity, %	17.8	18.5	23.75	25.01	23.51	19.15
Bulk density, g./cc.	2.24	2.32	2.038	1.986	2.071	2.14
Refractoriness (Orton cone)	23-26	21-32	28-29	N.D.	30	28-29
Refractoriness under-load						
Ta (°C.)	1270	N.D.	1440	1370	1405	1435
Te (°C.)	1500	—	1600	1670	1600	1650
Cold crushing strength, kg./cm. ²	118.02	119.07	116.5	58.21	109.78	N.D.

develop the same from indigenous raw materials should be taken up to meet the pressing problem.

A detailed study was made of the nozzle at present imported from foreign countries and the indigenous nozzles that were reported to have given poor performance in service. Table 57 gives the data of tests performed on the imported and indigenous nozzles.

Other tests like erosion by slag, reheat change and spalling resistance are being done. Work on the development of suitable technique for producing satisfactory nozzle composition based on indigenous raw materials is underway.

69.0 Studies on Clays

(a) *South Indian Clays*—The project on 'Studies on South Indian Clays' has been concluded and a report embodying the results has been prepared and sent for publication in the *Transactions of the Indian Ceramic Society*.

(b) *Base Exchange Study of Clays*—Further work on this project showed that cations had no mineralizing action on mullite formation, but the cristoballite phase was very much affected. The cristoballite

was found to be the low form and of the disordered type in view of higher 'd' values. The variation in intensity of the characteristic lines of cristoballite with different cations in X-ray diffractograms was considered to be due to the degree of disorder brought by cations on cristoballite lattice. Even though further work was needed to arrive at positive conclusions on the subject, the possibility of glass formation in the case of Na⁺ and K⁺ cations resulting in the complete disappearance of cristoballite phase cannot be ruled out.

70.0 Studies on Corrosion of Metals and Alloys

Long-term studies on various aspects of corrosion research under Indian tropical weather conditions were undertaken. The setting up of the Marine Corrosion Research Unit at Digha on the sea coast of West Bengal is actively progressing.

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

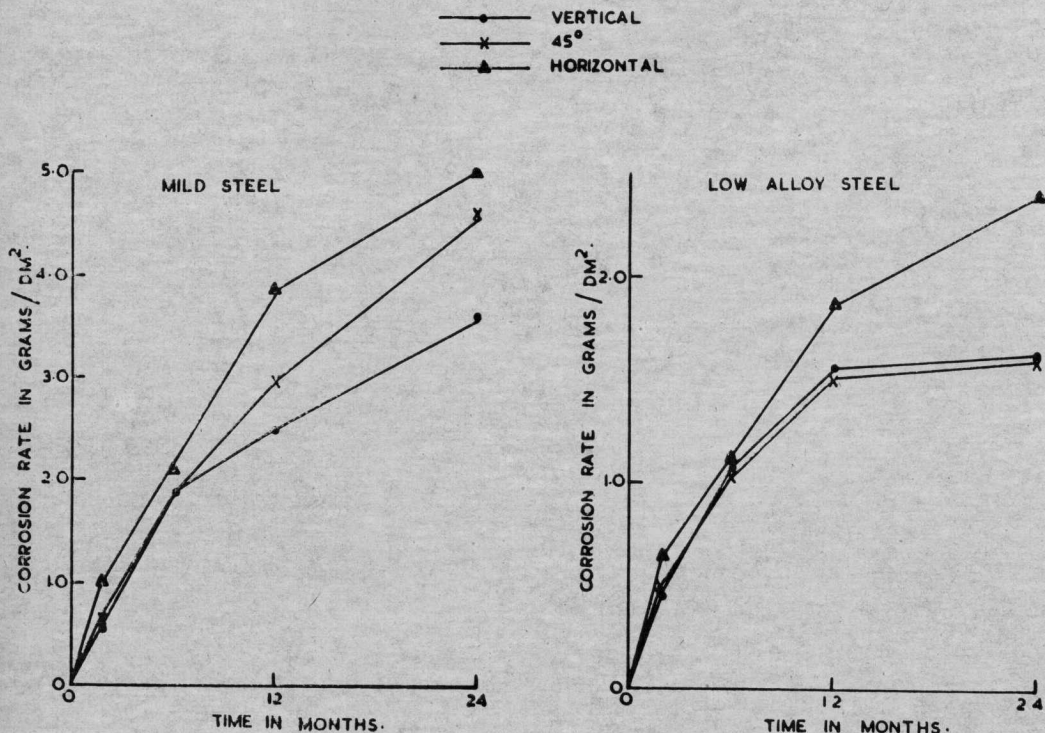


FIG. 43 — CORROSION RATE OF MILD STEEL AND LOW-ALLOY STEEL

on long-term studies on the atmospheric corrosion of metals under industrial atmospheres as well as a number of corrosion problems.

(i) *Studies on Atmospheric Corrosion of Metals and Alloys* — Atmospheric corrosion of metals and alloys is influenced by the nature of atmospheric pollution, relative humidity, state of exposure and composition of materials. The present investigation, which was taken up to evaluate the influence of these various factors, was continued.

(a) Corrosion studies on different ferrous and non-ferrous metals and alloys exposed to atmosphere after 1956 are being continued. Samples were last removed in 1961. The test will be in progress for a period of at least 10 years. The next batch of the samples will be removed after completion of seven years of exposure.

(b) To study the changes in properties of rust on mild steel and low-alloy steel and their effect on resistance to corrosion, further set of samples were removed after the completion of 2, 6, 12 and 24 months' exposure. The loss in weight was determined for different periods of exposure of 2, 6, 12 and 24 months. In addition, rust collected from samples was analysed for total sulphate, ferrous iron, total iron and moisture content. Porosity of rust and anodic polarization of the rusted samples with rust *in situ* were also determined. Marked influence of angle of exposure on corrosion was observed as will be evident from Fig. 43. Further, mild steel was found to corrode at a rate much higher than that of low-alloy steel. Ferrous iron content of the

rust in low-alloy steel was found to be less than that in mild steel. Anodic polarization also showed a marked effect of time of exposure and composition of base metal. Detailed monthly record of sulphur pollution of atmosphere and meteorological data was kept for proper assessment of the effect of climatic pollution factors on corrosion resistance. SO_2 pollution of the atmosphere was determined by 'Lead peroxide candle' method. Monthly variation in atmospheric pollution at Jamshedpur is shown in Fig. 44 for the year 1962.

- (c) Evaluation of performance of organic and inorganic coating in the industrial atmosphere of Jamshedpur. This work was taken up during the period under review. Aluminium-

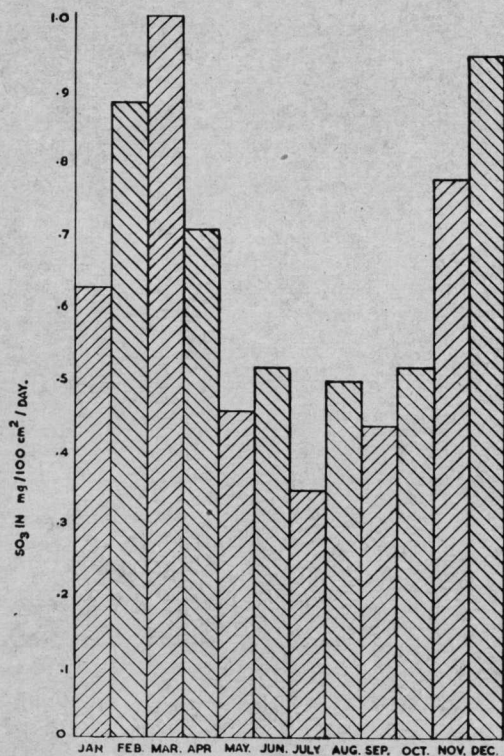


FIG. 44 — SULPHUR POLLUTION DATA FOR 1962

sprayed mild steel panels were exposed with and without paint. The paint system used consisted of one coat of primer paint (red-oxide zinc chrome) and two coats of finishing paint (red-oxide). Some of the painted samples exposed were damaged by scratching and by striking with a round hammer. Mild steel samples were also exposed with and without paint. The samples were first pickled in 10 per cent sulphuric acid. This was followed by a finish using '0' emery paper. The samples were then degreased and painted with one coat of primary paint and two coats of finishing paint. Some paints, as those used for the metallized panels, were also used in the present case. A detailed record of periodic observation of the condition of painted panels is being kept.

(ii) *Impingement Corrosion Studies* — Failure of non-ferrous metals and alloys in moving water system is a serious problem in industrial cooling systems and ship condensers. This type of corrosion is generally caused by impingement of moving water which results in erosion of the protective film on the metal, followed by corrosion of the exposed areas. The severity of attack is increased by the entrained air and is intensified with increasing water speeds. The present investigation was taken up to study the impingement corrosion resistance of various non-ferrous alloys with an idea to develop new alloys which would be resistant to this type of attack. In addition to this, effects of various physical and chemical factors will also be studied so as to have a better understanding of the mechanism of impingement corrosion and also to find out means for its mitigation.

The apparatus fabricated for studies on impingement corrosion resistance of different alloys at the National Metallurgical Laboratory is based on a design used by

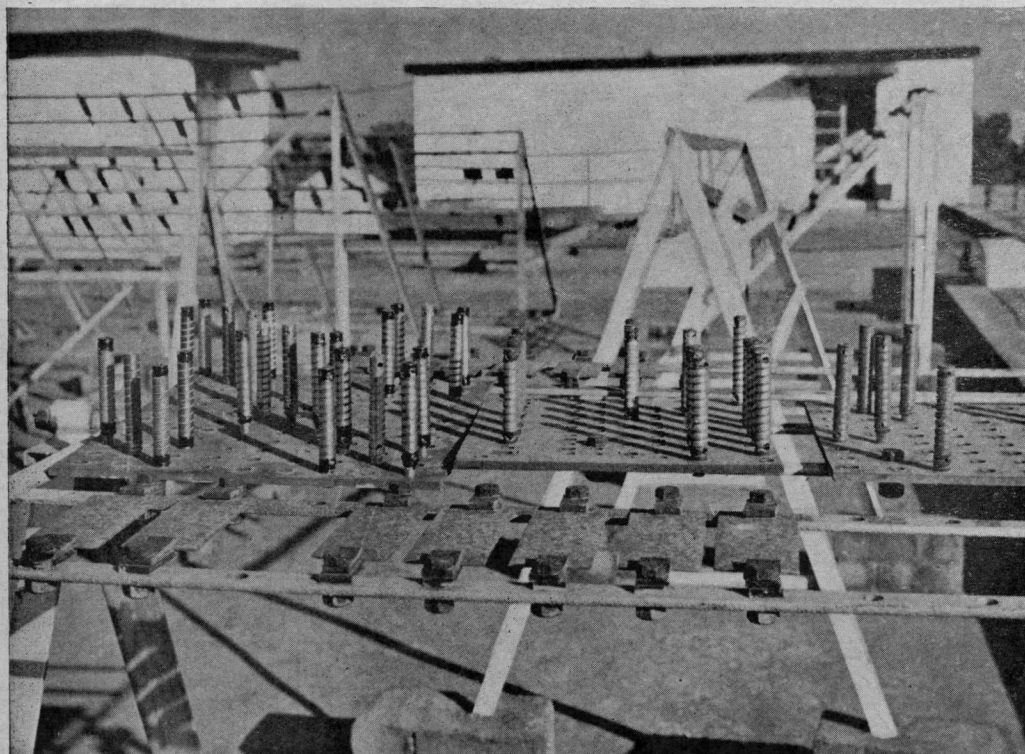


FIG. 45 — A VIEW OF THE SET-UP FOR ATMOSPHERIC CORROSION STUDY

H. W. Brownsdon. For the test, compressed air at a pressure of 20 in. of mercury was first filtered through glass wool and then saturated with moisture by bubbling through water. The air was then allowed to impinge on the sample through a glass jet of 0.035 cm. dia., drawn out from 1 mm. capillary tube. The sample was placed at an angle of 60° to the impinging jet in corroding media contained in a beaker. The apparatus was designed for testing twenty samples at one time under varying conditions of constant air pressure. Tests are in progress so as to fix the conditions of maximum attack by varying physical and chemical factors. For these studies 60/40 brass, which is known to possess poor resistance to impingement attack, is being used. The test solutions used are 3 per cent NaCl and synthetic sea-water

containing the following salts in 1 litre of solution:

NaCl		29 gm.
MgCO ₃ .7H ₂ O		7 gm.
MgCl ₂ .6H ₂ O		5 gm.
CaCl ₂ .6H ₂ O		2.4 gm.
NaHCO ₃		0.2 gm.

(iii) *Investigation on Effect of Variation in Solution Temperature in Preece Test* — Uniformity of coating of zinc on galvanized articles is determined by Preece test based on immersion of the article successively for 1 min. in a copper sulphate solution till an adherent deposit of copper is obtained. The ISI specification (Nos. 429-1954 and 728-1956) states that the test should be carried out at $15.5^\circ \pm 5^\circ\text{C}$. As the ambient temperature in India varies

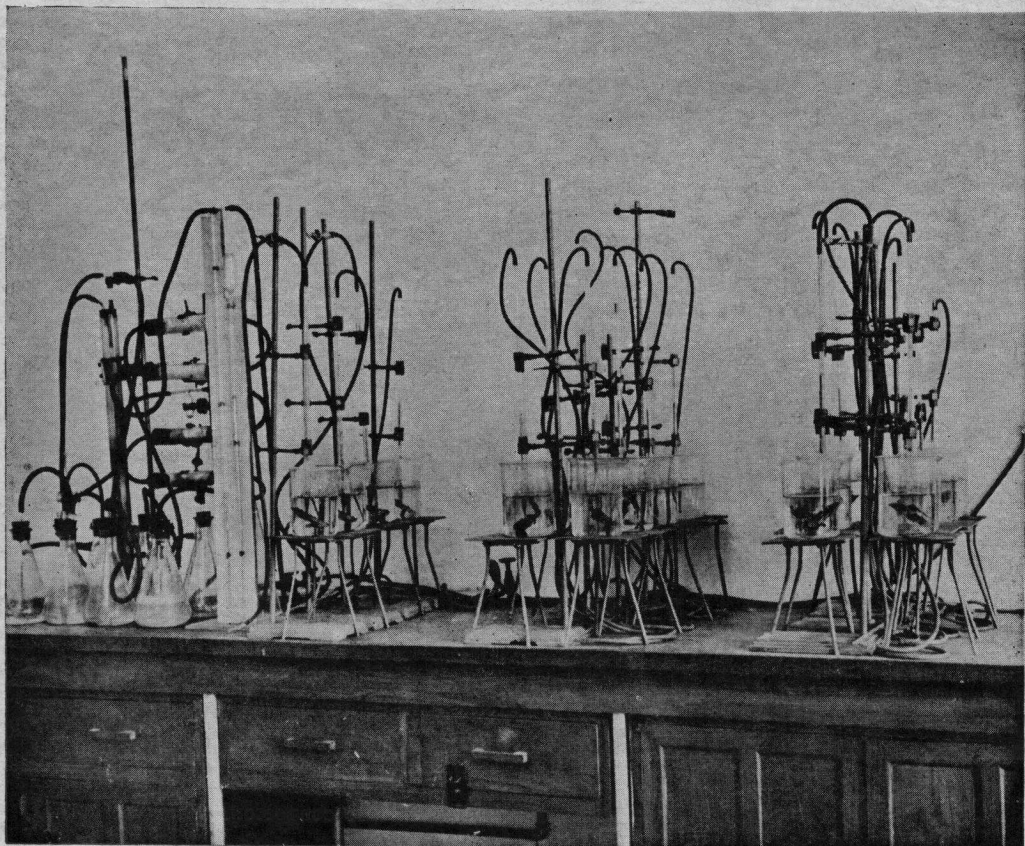


FIG. 46 — A VIEW OF THE SET-UP TO STUDY THE IMPINGEMENT CORROSION

from place to place and from season to season, investigation was taken up to determine the effect of solution temperature on the reliability of Preece test.

The test was carried out at 15.5°C . as per ISI specification and also in the following ranges of temperature: $20-25^{\circ}\text{C}$., $22-27^{\circ}\text{C}$., $25-30^{\circ}\text{C}$., $27-32^{\circ}\text{C}$. The increase in test temperature generally resulted in decrease in number of dips necessary for reaching the end point.

Stripping Test — Weight of zinc coating was first determined for all the materials as per IS: 429-1954 and IS: 728-1956. Zinc coating was dissolved in conc. HCl containing antimony chloride and weight of coating per unit area was calculated in each case. Preece's test to find out the

uniformity of zinc coating and stripping test to find out the weight of zinc coating per unit area were carried out on the following galvanized steel articles:

- | | |
|--------------------------------------|---|
| a) Tisco | Galvanized sheets |
| b) IISCO | Galvanized sheets |
| c) Guest Keen Williams & Co., Howrah | Bolts and nuts (galvanized) |
| d) Indian Tube Co., Jamshedpur | Galvanized tubes of different diameters |
| e) IISCO Ltd., Burnpur | Galvanized wire |
| f) Post & Telegraph | Galvanized wire |

(iv) *Mechanism of Chromate Passivation* —

Resistance to corrosion of zinc, aluminium, magnesium, etc., can be improved considerably by forming chemical conversion coating on the surface. This is generally accomplished by treating the material in

acid chromate solution. Investigation was taken up to study the mechanism of film formation and its relation to improved resistance to corrosion.

After the study of chromate passivation of zinc reported earlier, the study of mechanism of chromate film formation on aluminium and its alloys was taken up. The composition range of the solution studied was as follows:

Chromic acid	2.0-6.0 gm./l.
Sodium fluoride	0.5 gm./l.
Sodium dichromate	3.0-3.5 gm./l.
pH	1.5
Temperature	At room temperature (25-30°C.)

Aluminium samples were cut to a size of 6×4 cm. and were cleaned in hot sodium hydroxide solution prior to chromate treatment. Films were developed in the above solution by varying the amount of chromic acid from 2 to 6 gm./l. and then of sodium fluoride from 0.5 to 1.5 gm./l. To study the effect of time of treatment, immersion period in chromate solution was varied between 1 and 10 min. All the samples were weighed before and after chromate treatment. The film formed after treatment was removed in concentrated nitric acid. Amount of metal attacked and weight of chromate film formed were determined from these weighings in each case to study the properties of the film. Increase of immersion period from 1 to 10 min. had generally resulted in slow increase in weight of the film formed, which decreased with further increase in the acid. Films formed during immersion periods of more than 5 min. appeared to be somewhat loose and non-compact. Increasing additions of sodium fluoride from 0.5 to 1.5 gm./l. also did not change the properties of the film to any noticeable extent. Corrosion tests were also carried out to find out the protective properties of the treatment in salt spray and humidity

chamber. Untreated samples developed white spots after 2 or 3 days both in salt spray and humidity chamber maintained at 90 per cent R.H. and 30°C. temperature. Treated samples showed no corrosion after 280 hr. of test. In salt spray chamber, chromate-treated samples showed no tarnishing, except that in some cases chromate film was washed away at the completion of the test. Salt spray test was carried out for 170 hr. Protective properties of the films on ageing, and on drying the samples at temperatures ranging from 50° to 200°C. are under study.

(v) *Studies on the Mechanism of Stress-Corrosion Cracking* — Under specific conditions of stress and corrosion, most of the alloys are susceptible to stress-corrosion cracking. The mechanism of stress-corrosion cracking, in spite of considerable research, is not yet fully understood to explain all the observations. The investigation was taken up to study different metallurgical and chemical factors which affect the susceptibility of both homogeneous and heat-treatable ferrous and non-ferrous alloys to stress corrosion.

During the period under review, studies of different binary and ternary copper base alloys were taken up. Two methods of testing were adopted in the present studies for sheet and wire specimens. The sheet specimens were tested by loading to constant strain and wire specimens by direct tensile loading based on constant load device. For the direct tensile loading, apparatus based on that of Hoar and Hines was designed and fabricated. The testing solution was kept in a glass apparatus which was provided with arrangements to measure corrosion potential and polarization of the specimen tested. The sheet specimens were tested in ammonia atmosphere while the wire specimens in ammoniacal copper sulphate solution. The tests carried out till now showed profound influence of annealing temperature, degree of stress, composition and type of alloying

element of stress-corrosion cracking susceptibility of copper base alloys.

(vi) *Studies on Phosphatizing of Steel* — Amongst various chemical conversion coating processes, phosphate treatment of steel has received much industrial importance, specially for its good absorbent characteristics to form a good base for painting or to aid lubrication to the rubbing and wearing surfaces and also for its own rust-proofing characteristics. There are various proprietary processes developed, but very little information could be obtained in the literature regarding the solution composition and other details of the processes. Proper operational control such as selection of the bath composition, adjustment of acidity in the bath, working temperature, time of treatment, preparation of the samples, etc., deserve more attention for producing a good degree of coating. Investigation was, therefore, taken up to determine the optimum conditions to get a good coating by controlling different chemical and physical factors. On the basis of these studies, it was decided to compare the various characteristics of the coating obtained on their protective and other properties.

During the period under review, attempts were made to develop a non-sludge-forming bath. For these studies solutions containing zinc phosphate and phosphoric acid with the addition of certain other chemicals were tried. Effect of pH of the solution on the nature of the deposit was studied. At lower pH , the coating obtained was found to be thin and non-adherent, though the solution remained free from precipitation after successive treatments. With the adjustment of pH to a certain level the coating was found to become denser and more adherent, but treating of even 2-5 samples gave profuse turbidity. Addition of certain organic acid in the solution could prevent such precipitation in successive treatments of the samples. Analysis of the phosphate coating for its moisture

and PO_4 content was also done. An appreciable difference in PO_4 and moisture content was not observed with the change in time of phosphate treatment. Tests for oil-retaining characteristics of the coating were also performed. The coating was found to retain about 0.05 gm./dm.² of a thin lubricating oil. The corrosion resistance properties of the coatings were evaluated by exposing the phosphate oil or lanolin solution under the condition of constant condensation. The samples treated in oil or lanolin solution showed no development of rust spot in two months.

(vii) *Prevention of Discolouration of Copper Wires during Transit* — It is frequently observed that copper wires get discoloured during transit. This is undesirable for the manufacture of certain articles, e.g. cables. The investigation was taken up to study the effectiveness of different treatments in reducing this type of attack.

The scope for the application of the following preventive measures were studied:

- (a) Storing under low humidity
- (b) Chromate passivation
- (c) Lacquer coating

Storage under low humidity (60-70 per cent) was found to cause no discolouration during a test period of three months under fluctuation of room temperature which varied between 15° and 30°C. Chromate passivation of copper wires was carried out in a solution containing 10-30 gm./l. of sodium dichromate. To this, various amounts of chromic acid were added to give a pH value of 2.4-2.6. The bright pickled wires were treated in these solutions at 90°C. for 3 min. For lacquer treatment, shellac lacquer diluted with benzene in the ratio of 10:0, 10:1, 10:2 and 10:3 was used. The protective power of the treatment was determined by dipping the treated and untreated wires in 2 per cent sodium sulphide solution in water. This solution was found to discolour untreated copper wires within 5 sec., but the treated ones remained bright for 5-10 min. Based on these,

rapid-evaluation long-term test was carried out by placing small wires of copper in a desiccator under constant condensation. The untreated wires were found to be discoloured within 3-4 days, but chromate-treated ones did not tarnish even after 6 months. Lacquer-coated samples were found to remain without change for about one month.

71.0 Preparation of Plating Salts

The electroplating industry in India has so far been using imported plating salts, polishing compositions and plating equipment. Due to strict import restrictions, these proprietary plating salts are not available in the market and the electroplating industries in India are experiencing serious difficulties in procuring plating materials. Some of the Indian firms 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.

The formulation of dull nickel salt was standardized and its performance was experimented and tested in continuous production plating practice in a light engineering firm to assess its suitability in commercial plating practice. Dull nickel-plating salt had established its superiority in service performance compared to imported salt.

71.1 Dull Nickel-plating Salt

In the formulation of dull nickel-plating salt, the main ingredients that were used

were nickel sulphate, nickel chloride and boric acid together with some conducting salts. Investigations were carried out to assess the possibility of replacing NiCl_2 by NaCl in the dull nickel-plating salt without affecting the plating characteristics for economic reasons. In this connection anode and cathode efficiencies were measured during the course of electrolysis from the electrolyte containing NaCl in place of NiCl_2 in the range of current densities from 6 to 12 amps./ft.². The results indicated the possibility of substituting NiCl_2 by NaCl in the proposed formulation.

71.2 Bright Nickel Plating

A suitable organic brightener was formulated which when added to Ni-plating bath produced bright nickel plates without buffing. The brightener was exhaustively tested in bench experiments in the laboratory and it has given satisfactory results. To assess the suitability of NML-formulated brightener in commercial production plating practices, large-scale experimental trials are in progress with the organic brightener in a light engineering firm in Calcutta.

71.3 Hard Chromium Plating on Aluminium

Hard chromium plating on aluminium had been practised for some years, principally on such components as cylinders, pistons and bearings and brake drums. The method of producing hard, wear-resistant and, in some cases, oil-retaining surfaces had considerably advanced the use of aluminium alloys for many engineering applications. However, no component should be hard chromium-plated which is subjected to impact or shock in service as the substrate is soft aluminium. In the investigation on plating of hard chromium on aluminium as reported earlier, use had been made of interlayers of nickel and iron separately after etching the aluminium surface through a series of operations.

This etching made the aluminium surface free from the oxide layer. Subsequent plating from chromic acid bath between 200 and 300 amps./ft.² current density after removal of the interlayer in acid dips has resulted in dense, white, adherent, uniform deposits of chromium having hardness between 950 and 1242 VPN. Similar encouraging results have been observed while using an interlayer of zinc by immersion in zincate solutions of etched aluminium surfaces before plating with chromium in the usual chromic acid bath. Hardness of chromium plating using zinc interlayer has been found to exceed 1000 VPN in all cases. The technique of plating hard chromium directly on the aluminium without any interlayer but by etching only is currently on trial. Etching aluminium surface with chromic acid and HCl acid mixture of proper concentration to remove the oxide layer was found to produce an adherent, dense and hard chromium surface on aluminium after plating.

72.0 Gases in Metals

Different samples of metals and alloys in connection with different investigations along with samples received from other laboratories and institutes have been successfully analysed for oxygen, nitrogen and hydrogen by vacuum fusion method.

Accurate determination of hydrogen in metals and alloys of high-vapour pressure and low-melting point was difficult by the conventional hot vacuum extraction method, as the vapour condenses on the wall of the furnace tube absorbing hydrogen and lowers the hydrogen value obtained.

These difficulties have been overcome by introducing carrier gas technique for the determination of hydrogen. A few samples of high-vapour pressure and low-melting point metals and alloys have been analysed by the carrier gas method and have been compared with those obtained by the conventional vacuum heating method. The

results obtained by the carrier gas method are found to be better than those of vacuum heating method as shown in Table 58.

Table 58 — Determination of Hydrogen in Metals and Alloys of Low-melting Point and High-vapour Pressure

Sample	No. of detn.	Operational temperature °C.	cc. of H ₂ per 100 gm. by vacuum heating method	cc. of H ₂ per 100 gm. by carrier gas method
Magnesium powder	1	500	90.0	95.0
	2	500	81.0	96.0
	3	500	78.0	96.0
Electrolytic zinc	1	350	41.5	56.0
	2	350	50.0	54.0
	3	350	37.3	54.5
Copper-cadmium alloy (electrolytic)	1	350	36.3	41.1
	2	350	32.9	43.3
	3	350	28.2	42.9

Hydrogen pick up by steel during pickling with some common industrial inhibitors has also been studied and results obtained are shown in Table 59. Criteria of the efficiency of the inhibitors was determined by the ratio of the hydrogen pick up in cc. per 100 gm. of the sample to the percentage loss of the sample by dissolution. Lesser the value of the ratio, better is the inhibitive action of the inhibitors in respect to the hydrogen pick up.

73.0 Rapid Method for the Determination of Manganese in Nickel-free Austenitic Stainless Steel

Determination of manganese in steels containing high percentages of chromium necessitates prior separation of chromium. This investigation was taken up for developing a rapid method for determining manganese in the presence of chromium. It was possible by oxidizing both chromium

Table 59 — Behaviour of Some Inhibitors during Pickling in 4NH₂SO₄ Bath for 4 hr. Time

Sample	Inhibitors	Quantity of inhibitors/ 100 cc. of bath soln. gm.	cc. hydrogen pick up/ 100 gm.	Weight percentage loss of the sample	Ratio of hydrogen pick up to % loss
Mild steel	Starch	0.025	6.57	11.77	0.56
do	Dextrine	0.050	5.95	10.57	0.56
do	do	0.025	8.0	13.71	0.58
do	Dextrose	0.025	23.11	38.39	0.60
do	Potassium iodide	0.025	10.2	22.47	0.45
do	Arsenious oxide	0.05	0.48	1.59	0.30

and manganese by ammonium persulphate in the presence of 3-4 per cent (V/V) silver nitrate as catalyst and determining both the total permanganate equivalent for chromium and manganese and that of chromium alone. The amount of manganese present was then calculated from the difference between the two titres. It was found that both the constituents can be determined in about 40 min. This method has since been applied for the determination of chromium and manganese in nickel-free austenitic stainless steels and other ferrous materials containing high percentage of chromium and manganese.

74.0 Spectrographic Analysis of Antimony in Solders

The object of this study is to develop a method to estimate antimony, both high and low amount, in lead-tin solder spectrographically.

Estimation of antimony in lead-tin solders by chemical methods is cumbersome and time-consuming, and the methods, occasionally, under certain circumstances lead to erroneous results. Results obtained by spectrographic methods have been found to be more consistent and reliable. Applicability of the methods depends, however, upon the antimony content of a solder sample. There are generally two kinds of tin-lead solder in common use, namely antimonial and non-antimonial (containing trace antimony) solders. For estimation of high

amount of antimony (0.5-10.0 per cent) in antimonial solders, porous cup technique and for low amount of antimony (0.001-0.2 per cent) in non-antimonial solders, oxide arc technique were developed. In both the methods 'Hilger' medium quartz spectrograph was used. The work is completed.

75.0 Spectrographic Analysis of Phosphorus in Steel

This work was taken up to develop a suitable method for analysis of phosphorus in steel. In continuation of previous year's work, few more experiments under different excitation conditions were carried out. More encouraging results were, however, obtained when intermittent A.C. arc of 15 kV., current 6 amps. was employed for excitation using rotating electrode with spec. pure nickel rod tipped at 120°C. cone angle as counter-electrode.

Further work in regard to reproducibility and accuracy of the method is in progress.

76.0 Preparation of Slag Cement from Low-shaft Furnace Granulated Slag Containing more than 5 per cent MgO

It was earlier reported that low-shaft furnace granulated slags have good hydraulic properties and that slag cements

produced from them possessed adequate compressive and tensile strengths and were resistant to salt water attack. The slags used in these investigations contained less than 5 per cent MgO to satisfy the requirements set up by IS and BS specifications. Many of the iron blast furnaces in India now produce slags containing more than 5 per cent MgO due to dolomite addition in the furnace burden. Keeping these facts in view, investigation was taken up to examine the scope for utilization of slags containing more than 5 per cent MgO in cement making. The samples of slag cement were prepared by mixing powdered granulated slag and portland cement as activator in different proportions.

The following samples were prepared:

No.	Amount of slag (granulated and powdered)		Amount of portland cement
1	30 parts	+	70 parts
2	40 parts	+	60 parts
3	50 parts	+	50 parts
4	60 parts	+	40 parts
5	70 parts	+	30 parts

Each sample was mixed in the ratio of 1 part of slag cement sample with 3 parts of standard sand (—14, +18 mesh).

The following tests were carried out:

(a) Determination of water of consistency, initial setting time and final setting time by Vicat's needle test (samples without sand — *vide* Table 60):

Room temperature for all the experiments Dry 81°F.
Wet 75°F.

Humidity 65-70 per cent

(b) Determination of tensile strength of the mixture of 1 part of slag cement and 3 parts of standard sand (*vide* Table 61).

(c) Determination of compressive strength of the mixture of 1 part of slag cement + 3 parts of standard sand (*vide* Table 62).

Tests for sulphate water resistance of the samples were also carried out. Bars 5×1×1 in. were prepared with 1 part of the sample +5 parts of standard sand. After ageing for 24 hr. in moist atmosphere they were immersed in a 0.15 molar solution of sodium

Table 60 — Determination of Water Consistency, Initial and Final Setting Time

No.	Composition of sample	Water of standard consistency per 100 gm. of sample	Initial setting time		Final setting time	
			hr.	min.	hr.	min.
1	70 slag 30 portland cement	23 cc.	2	45	6	5
2	60 slag 40 portland cement	23 cc.	2	3	5	38
3	50 slag 50 portland cement	23 cc.	3	25	5	20
4	40 slag 60 portland cement	22.5	3	0	5	5
5	30 slag 70 portland cement	22.0	2	0	3	5
6	100 parts portland cement (standard)	22	1	30	2	45

Table 61 — Tensile Strength of the Mixture of 1 Part of Slag Cement and 3 Parts of Standard Sand

No.	Composition of sample	Strength and period of tests (strength in lb./sq. in.)		
		3 days	7 days	28 days
1	70 slag + 30 portland cement	60	79	199
2	60 slag + 40 portland cement	75	112	213
3	50 slag + 50 portland cement	86	121	225
4	40 slag + 60 portland cement	93	130	300
5	30 slag + 70 portland cement	105	142	347
6	100 portland cement	370	388	488

Table 62 — Compressive Strength of the Mixture of 1 Part of Slag Cement and 3 Parts of Standard Sand

No.	Composition of sample	Compressive strength in lb./sq. in.		
		3 days	7 days	28 days
1	70 slag + 30 portland cement	476	590	743
2	60 slag + 40 portland cement	511	707	1267
3	50 slag + 50 portland cement	595	711	1302
4	40 slag + 60 portland cement	621	774	1729
5	30 slag + 70 portland cement	686	798	1890
6	100 portland cement	1340	2840	3245

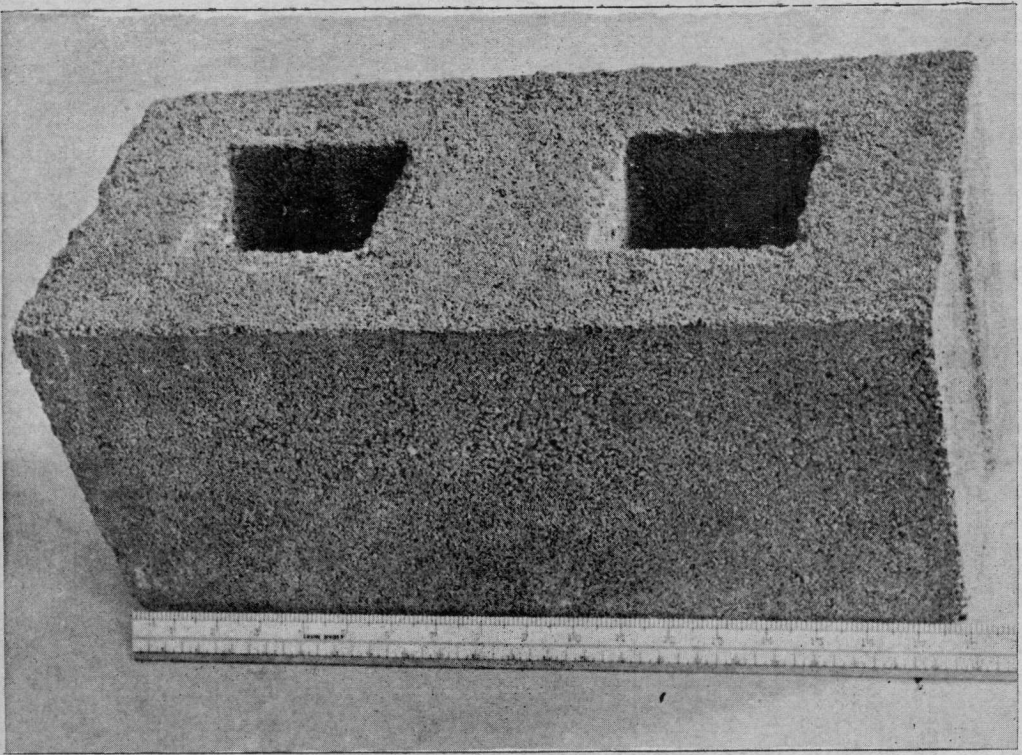


FIG. 47 — HOLLOW BLOCKS MADE OUT OF LOW-SHAFT FURNACE SLAG

sulphate in stoppered bottles. The change in shape, size or any crackings, etc., in the bars were examined at intervals of one week. Similar bars of portland cement with 5 parts of sand were also prepared and stored in the same way. The slag cement bars were not affected even after storing for about 8 months, whereas the bars of portland cement were totally disintegrated in less than that period.

77.0 Utilization of Low-shaft Furnace Slag for Light-weight Aggregate

The concrete made by mixing the light-weight aggregate from expanded slag, cement and water on volume basis was found to possess low-compressive strength while thermal conductivity values were within the range of specifications. This was probably due to the incorrect propor-

tioning, causing undersanded concrete and giving a honeycombed structure to the concrete. The approximate water-cement ratio also affected the compressive strength of the concrete. Trials were, therefore, made to determine the proper concrete mix on weight basis according to the tables provided by the National Slag Association of U.S.A. These mixes were on the basis of proportion of different ingredients by weight per cubic yard of concrete with particular cement-water ratios intended for desired compressive strength. Seven different mixes were cast into concrete blocks and their physical and mechanical properties were determined. Hollow blocks of $18 \times 8 \times 8$ in. and $18 \times 8 \times 6$ in. were also cast (Fig. 47). Further trials were made on expansion of low-shaft furnace slag by pit method to get an expanded slag with still lower unit weight and more strength.

PILOT PLANTS

78.0 Low-shaft Furnace Pilot Plant Project

Based on the comprehensive investigations conducted with the regional raw materials received from the State Governments of Maharashtra, Andhra Pradesh and Punjab, comprehensive Investigation Project Reports indicating feasibility of smelting technique with non-metallurgical fuels including preliminary cost calculations and capital investment have been issued to the respective State Governments.

During the period under review, extensive smelting trials were undertaken at the request of the Government of Rajasthan with the iron ore and limestone occurring in the State employing nut coke as fuel. Due to the non-occurrence of any type of coal in Rajasthan, it was recommended that it would be necessary to use either nut coke procured from the nearest integrated iron and steel plant or carbonized Palana lignite briquettes, if possible, for smelting the iron ore eventually. The possibilities of electrothermal smelting with consequent reduction of fuel can be considered as and when the requisite power becomes available. An Investigation Project Report detailing the results of the investigation has been issued to the State Government of Rajasthan.

Smelting trials were also conducted at the request of the Government of Nepal with iron ore and limestone occurring there. In view of the total absence of coal in Nepal, smelting trials were conducted with nut coke as fuel and reductant. A detailed Investigation Project Report has since been issued indicating the feasibility of installa-

tion of a small iron-smelting unit in Nepal based on either nut coke imported from India or utilization of charcoal available in Nepal.

Programme of Work

The long-term programme of investigations based on the following phases were continued:

Phase I

- (1) Iron ores from Orissa and Bihar with non-coking coals from Raniganj and Dishergarh coalfields either in bedded charge or by briquetting iron ore, limestone and non-coking coal.
- (2) Iron ores from Chanda district and non-coking coals from Ballarpur, Kamptee and Wardha Valley, Maharashtra State.
- (3) Iron ores from Chapra, Antribeharipur in Mahindergarh, Punjab, with nut coke or non-coking coals.
- (4) Iron ores from Anantpur, Warangal, etc., with non-coking coals (and low-temperature carbonized coke made thereof) from Kothagudem, Yellandu, etc., in Andhra Pradesh.
- (5) Iron ore from Katni in Madhya Pradesh with non-coking coals from adjacent coalfields (Kanhan and Pench Valleys).
- (6) Iron ores from Nathara-ki-Pal near Udaipur and Morinja (Chomu-Samod) near Jaipur, Rajasthan, initially with nut coke and then with high-temperature carbonized Palana lignite (as and when it becomes available) [*vide* Phase III (1)].

Phase II

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

Phase III

- (1) Utilization of lignites after its high-temperature carbonization for iron smelting.
- (2) Utilization of Salem magnetite after its beneficiation and briquetting in conjunction with carbonized coke.

Phase IV

Research and development investigation on oxygen enrichment of air blast, injection of naphtha oil and gas including low-shaft furnace clean gas through auxiliary tuyeres and study of overall economics of production of such operations.

Phase V

Utilization of agglomerated fine-grained soft iron ores, blue dust in the form of sinter, self-fluxing sinter, pelletization and briquetting and study of fuel requirements, etc.

Phase VI

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

Based on the raw materials employed for smelting and the objectives in view, the smelting trials during the period under review can be grouped into the following campaigns.

Campaign XV — Smelting characteristics with non-coking coals from Singareni and Kharkenda collieries. During this campaign, exclusive use of non-coking coals

from Singareni and Khaskenda collieries were attempted, but serious furnace operational difficulties like hanging were experienced with 25 per cent Singareni coal in fuel burden, while exclusive utilization of non-coking coal from Khaskenda colliery was possible.

Campaign XVI — Smelting characteristics of fine-grained iron ore (—12 mm.) from Orissa with a view to achieving optimum fuel efficiency.

Campaign XVII — Smelting trials with iron ore and limestone from Rajasthan employing nut coke as fuel and reductant.

Campaign XVIII — Smelting trials with iron ore and limestone from Nepal employing nut coke as fuel and reductant.

Campaign XIX — Smelting characteristics of fine-grained iron ore (—5 mm.) from Orissa (Orissa Minerals) with low-temperature carbonized coke to achieve the optimum fuel rate.

Sixteenth Furnace Campaign

In this campaign, smelting trials were conducted with fine-grained iron ore from Orissa, low-temperature carbonized coke (C.F.R.I.) made from non-coking coals, using limestone from Orissa and dolomitic limestone from Andhra. The object of this campaign was to study the smelting behaviour of fine-grained ore with a view to obtaining the optimum fuel rate without disturbing the regularity of smelting operation and sacrificing the quality of pig iron. The chemical analysis of raw materials employed is given in Table 63. The screen analyses of the raw materials are recorded in Table 64.

The furnace was blown under the following conditions.

Tuyere diameter	75 mm.
Wind volume	1850-2200 Nm ³ /hr.
Wind pressure	1450-2000 mm. WG.
Hot blast temperature	580-590°C.
Top gas temperature	350-400°C.

Table 63 — Chemical Analysis of Raw Materials

1. Proximate analysis of low-temperature carbonized coke (C.F.R.I.)

H ₂ O %	Ash %	V.M. %	F.C. %
8.5	25.9	4.1	61.5

2. Analysis of ash of L.T.C.

SiO ₂ %	Fe %	Al ₂ O ₃ %	CaO %	MgO %	P %	Total sulphur
53.8	4.62	28.0	5.68	1.88	1.8	0.23

3. Analysis of iron ore

Fe %	SiO ₂ %	Al ₂ O ₃ %	CaO %	MgO %	Mn %	S %	P %
63.21	2.88	5.6	Nil	Nil	—	Trace	Trace

4. Analyses of fluxes

Location	CaO %	MgO %	SiO ₂ %	Al ₂ O ₃ %
Birmitrapur (Orissa)	44.8	3.27	6.96	1.6
Andhra Pradesh	32.2	25.0	0.3	0.56

Table 64 — Screen Analysis of Raw Materials Employed

Raw materials	In percent					
	Above 2"	2"-1"	1"-½"	½"-¼"	¼"-⅛"	Below ⅛"
L.T.C.	25.72	37.16	18.50	5.36	4.83	8.43
Iron ore	—	—	—	58.43	40.54	1.03
Limestone (Andhra)	—	27.78	56.24	7.80	8.18	—
Limestone (Birmitrapur)	—	—	—	—	40.35	59.63

During the initial stages, the furnace was operated exclusively with nut coke and fine-grained iron ore, but after obtaining regularity in smelting, the nut coke was progressively replaced by low-temperature carbonized coke. Although the ore consisted of fines, the fuel consumption was 2.32 tonnes/tonne of pig iron associated with slag volume of 1.97-2.11 tonnes/tonne of pig iron. Despite several attempts, the fuel could not be reduced as the furnace showed evidence of chilling. The regularity of burden descent was affected by occasional small bridging which, however, broke at intervals but affected the preparation of the burden and the fuel rate. Under these

conditions, about 7.5 tonnes of raw materials were consumed for the production of a tonne of pig iron, the ore rate and flux rate being 1.46 and 1.80 tonnes per tonne of pig iron respectively. A production figure of 6.4 tonnes per day was achieved under blowing in condition of 2400 Nm³/hr. at a pressure of 2000 mm. WG. The compositions of pig iron and slag during this campaign are given in Table 65.

The high-fuel consumption was attributed to the large particle size of the low-temperature carbonized coke as compared to the iron ore, and also due to the poor porosity and reducibility of the iron ore. Flue dust loss amounted to 8-10 per cent of the

Table 65 — Composition of Pig Iron and Slag

	Per cent
(a) Composition of pig iron:	
Total carbon	2.50-3.00
Silicon	3.00-4.45
Sulphur	0.04-0.09
Phosphorus	0.40-0.60
Manganese	0.30-0.40
(b) Composition of slag:	
CaO	33.0-38.0
SiO ₂	34.0-36.0
FeO	0.5-3.0
Al ₂ O ₃	22.0
MgO	5.4

raw materials, which was found to contain high percentage of carbonaceous material. The investigation was not particularly successful in achieving acceptable fuel rate.

Seventeenth Furnace Campaign

Smelting Trials with Iron Ore and Limestone from Rajasthan

Due to the non-occurrence of any type of coal in Rajasthan, preliminary pilot plant trials were undertaken with Rajasthan iron ore and limestone employing nut coke as fuel and reductant.

In Rajasthan, one of the two major occurrences of iron ores is at Nathara-ki-Pal hills in Daralmata and Ansivali areas with an estimated reserve of about 10 million tonnes. It is situated at a distance of about 64 km. south-east of Udaipur. The other major occurrence is in Jaipur division, situated at Moriia at a distance of about 9.6 km. from Chomu-Samod railway station. It is estimated that over 10 million tonnes of iron ore will be available from this region.

The full assessment of these deposits, both as regards their physical and chemical characteristics, and reserves needs detailed investigations, but even the proved deposits assure adequate ore supplies for the installation of a pig iron plant with an annual output of 100,000 tonnes.

The Nathara-ki-Pal ore consists mostly of specular hematite with minor amounts of magnetite. The iron mineral is associated with quartzite. The Chomu-Samod Moriia ore consists mainly of hematite associated with quartzite. Both these deposits are substantially free from phosphorus and will enable production of standard low-phosphorus foundry pig iron required for special purposes like rolls, etc. The Nathara-ki-Pal ore is, however, highly siliceous and its exclusive use will necessitate appropriate prior beneficiation and agglomeration treatments before smelting. Besides these two major deposits, several minor iron ore deposits occur in Rajasthan.

Limestone occurs in many parts of Rajasthan State. In Jodhpur division, limestone occurs at Sojat, Gotan and other places and these are generally suitable for iron smelting. There are also many other deposits.

Rajasthan does not possess any coal deposits, but lignite occurs at Palana, Khari, Channeri, etc. Deposits at Palana, 14 km. south of Bikaner, is most extensive, the estimated reserves being of the order of 30 million tonnes. The water content of raw lignite is about 40-45 per cent.

Lignite can be carbonized at low temperature to produce soft coke. The chemical and physical characteristics, reactivity of carbonized lignite coke briquettes for iron smelting have to be thoroughly investigated. It may be mentioned that due to the non-availability of lignite coke, no tests could be conducted for iron smelting. Most of the sulphur present in lignite remains in the ash, which will prevent its use for iron smelting due to high-sulphur input in the metal. In any case, the possibilities of obtaining carbonized lignite coke briquettes and their availability for iron smelting is outside the scope of this report. Further, the economics of production and operation of lignite-coking plant have to be separately considered including the byproduct recovery plant.

Table 67 — Analysis of Coal, Coke, Coal Ash, Coke Ash, Limestone, Dolomite and Manganese Ore

1. Proximate analyses of coke and coal

Fuel	H ₂ O %	V.M. %	F.C. %	Ash %	Total S%
Nut coke (from Tisco)	3.00	1.80	75.10	20.10	0.50
Lower Kenda coal	2.80	30.20	49.86	17.14	0.50

2. Ash analyses of coal and coke

Fuel	SiO ₂ %	Al ₂ O ₃ %	CaO %	MgO %	FeO %	P %
Nut coke	52.08	33.00	3.80	2.12	7.71	0.68
Lower Kenda coal	49.04	24.93	2.44	1.35	4.37	0.59

3. Analyses of limestone and dolomite

Location	CaO %	MgO %	SiO ₂ %	Al ₂ O ₃ %
Rajasthan	51.60	3.30	1.20	1.70
Andhra Pradesh	32.20	25.00	0.30	0.56

4. Analyses of manganese ore

Location	Mn %	Fe %	SiO ₂ %	Al ₂ O ₃ %
Dharmadihi, Bihar	50.70	5.60	11.20	11.30

In view of the non-availability of suitable coal in Rajasthan for iron smelting, establishment of a regional iron-smelting plant at Rajasthan will be based on the use of surplus nut coke available from existing steel plants; in this particular case nut coke from Bhilai (M.P.) Steel Plant will be required, being the nearest to Rajasthan. In the event of using nut coke, a small low-shaft blast furnace of 150-200 tonnes/day capacity can be employed. The other alternative is to adopt electrothermal smelting of pig iron using carbonized lignite coke briquettes or preferably nut coke as the reductant. However, the electrothermal process of iron smelting is outside the scope of investigation.

Analysis of Raw Materials

Analysis of raw materials employed in the smelting trials are given in Tables 66 and 67.

Table 66 — Analysis of Iron Ore

Location	Fe %	SiO ₂ %	Al ₂ O ₃ %	P %	S %
Rajasthan	61.6	9.9	1.2	0.5	0.2

Physical Characteristics of Rajasthan Raw Materials

The Rajasthan ore as received was in big lumps unsuitable for dried smelting. The ore was crushed and screened through 25.4 mm. mesh screen. A large volume of fine-grained product was generated on jaw crushing of the lumpy ore. Limestone also received in big lumps was crushed similarly and screened through 19.05 mm. mesh screen. Although prior preparation of raw materials is not specifically necessary for smelting, the little reduction time afforded during the descent of the burden

Table 68 — Screen Analysis of Raw Materials Employed for Smelting Trial

Raw material	Sieve fraction percentage				
	—50.8 mm. +25.4 mm.	—25.4 mm. +12.7 mm.	—12.7 mm. +6.35 mm.	—6.35 mm. +3.175 mm.	—3.175 mm.
Iron ore (Rajasthan)	2.20	67.20	18.90	4.20	7.50
Limestone (Rajasthan)	8.80	40.70	20.70	14.10	15.70
Nut coke (Tisco)	29.10	46.10	3.40	3.20	18.20
	(38.1 mm. + 25.4 mm.)				
Lower Kenda	50.60	6.10	1.40	0.80	1.30
Non-coking coal	39.80	—	—	—	—
	(Above 50.8 mm.)				
Limestone (Andhra)	14.10	62.40	10.60	5.20	7.70
Manganese ore	4.30	74.10	5.20	11.30	5.10

in low-shaft furnace necessitates proper sizing and grading of the raw materials, since fuel consumption is influenced by the particle size of the raw materials employed. For operation with nut coke, the size of iron ore and limestone should preferably be —19.05 mm. +6.36 mm., although a small proportion of larger and finer size can be tolerated. The screen analyses of raw materials employed in the trial are recorded in Table 68.

Appraisal of Rajasthan Iron Ore

The average apparent porosity of the iron ore was 6.05 per cent which is extremely low compared to iron ores available in Bihar-Orissa area and the other ores previously smelted in the low-shaft furnace pilot plant. The lack of porosity was expected to affect the reducibility of the ore. Another factor affecting the smelting behaviour is the fusion point of the ore. Under oxidizing conditions, the fusion point varied from 1435° to 1475°C. which is comparable to other Indian ores.

The as received ore sample was massive, hard and compact, medium to fine crystalline, granular with a metallic lustre and cherry-red streak. Fine quartz grains could be distinguished with hand lens and at times with naked eye.

Petrological examination of polished ore sections under incident illumination and

crushed ore under transmitted light showed that the sample consisted mostly of crystalline hematite with quartz as the main gangue mineral followed by small quantities of feldspars. The hematite commonly showed lamellar twinning. Hypidiomorphic and tabular grains were also seen interlocked with coarse to medium-grained quartz. A few grains of magnetite with exsolved hematite along the octahedral cleavage planes were also observed. Hematite crystals with twin lamellae forming octahedral network were also quite common. This pattern could be due to metamorphism which had resulted in the alteration of original magnetite to hematite, at the same time leaving the exsolved hematite (along octahedral cleavage directions) lamellae undisturbed.

Examination of the sieve fractions of the crushed sample revealed that the total gangue present in the sample was about 11 per cent by weight, and was well liberated at about 100 mesh size.

Although the reducing intensity of the gas and the thermo-mechanical stresses to which the ore is subjected during its descent in the low-shaft furnace smelting cannot be simulated in the laboratory, a determination and comparison of physical characteristics, chemical composition, reducibility of various ores may throw some light on their behaviour during smelting. The suitability of an ore for smelting depends on its

composition particularly in relation to the nature of gangue, physical strength to enable mechanical handling and its reducibility.

The handling strength of the ore was determined by shatter and abrasion tests. For shatter test, 2000 gm. of ore was allowed to fall freely from a height of 3.658 m. and the particles were subjected to screen analysis. For the abrasion test, 2000 gm. of the material was tumbled in a 30.48 cm. diameter and 35.56 cm. long drum with a baffle plate inside, which revolves at 75 r.p.m. for 18 min.

The crushing strength and abrasion resistance of the ore and limestone were satisfactory for smelting in low-shaft furnace.

Reducibility Characteristics of Rajasthan Iron Ore

The reducibility of the ore was determined employing hydrogen gas as the reductant at different temperatures of reaction, gas velocities and particle size of the ore. At a reaction temperature of 900°C., at hydrogen flow of 600 cc./min., Orissa Minerals ore was 90 per cent reduced in 30 min., while in the same time Rajasthan ore was only 66 per cent reduced. It was observed that reducibility of Rajasthan ore was inferior to the other ore. Besides, visual examination of the particles of the ore after reduction disclosed the presence of lumps of quartz SiO_2 embedded in the reduced mass. The reduction treatment made the ore friable and similar behaviour at appropriate height of the shaft may promote reducibility of the ore.

Details of Smelting Operation

At the initial stage, a trial was conducted primarily to ascertain the smelting behaviour of the ore and the fluxing characteristics of the limestone, when optimum FeO content of the slag demonstrated adequate reduction of the ore. Following the observation that the ore was completely

reduced in the low-shaft furnace, different burden schedules with variations in operational factors such as pressure and volume of the air blast, basicity ratio of the slag aiming at low-slag volumes were employed to attain optimum smelting rate coupled with reasonable fuel consumption. On the basis of previous experience gained dolomite was added in the burden to result in 8-10 per cent MgO content in the slag which improved the fluidity of the high-alumina slag. It was found that for the production of desirable grade pig iron, a burden charge consisting of 26 kg. nut coke, 20 kg. Rajasthan iron ore, 4 kg. Andhra limestone, 7 kg. Rajasthan limestone and 5 kg. of non-coking coal with a calculated slag composition of CaO , 35.85; SiO_2 , 30.73; Al_2O_3 , 16.69; and MgO , 11.16 per cent, with a calculated slag basicity degree $\text{CaO}/\text{SiO}_2 = P_1$ of 1.17 and fuel consumption at the rate of 1.6 tonnes of fixed carbon per tonne of iron, worked most satisfactorily. As the shaft height of the furnace was inadequate for the transfer of sensible heat of the ascending reducing gases to the descending burden during smelting with nut coke, 5 kg. of non-coking coal were added to bring down the furnace top and gas temperatures. Most of this coal disintegrated at the top of the stack and was carried away in the flue dust. In order to introduce manganese in the pig iron, manganese ore assaying Mn, 50; Fe, 5.6; SiO_2 , 11.2; and Al_2O_3 , 10 per cent was added to each charge. The recovery of manganese in the pig iron was about 50 per cent.

Several factors are known to affect the fuel rate. A change in the basicity degree of slag was made to study its effect on the fuel rate and the sulphur partition between the pig iron and the slag when it was found that on lowering the basicity degree CaO/SiO_2 from 1.0 to 0.9, sulphur in the pig iron increased to 0.12 per cent, whilst a basicity of 1.2 was adequate in maintaining the sulphur contents in the pig iron at 0.04 per cent.

As indicated earlier, the tuyere openings can be altered from 40, 60, 75 and 100 mm. which influence the driving rate of the furnace. To assure smooth working of the furnace, velocity of the air blast has to be suitably adjusted in relation to particular raw materials smelted. Based on early experience, for these trials the furnace was blown with 75 mm. dia. opening when the daily production was assessed 7-8 tonnes. An increase in blast volume with an identical velocity will increase daily iron output. The optimum burden schedule obtained during the smelting trials, which resulted in satisfactory results both from analyses and operation angle, is given in Table 69. The operational characteristics of the furnace with these raw materials were quite satisfactory. The descent of the raw materials in the furnace was smooth and regular and no hanging or other irregularities were met with.

Table 69 — Operational Data of the Smelting Process

Data (average)	Basicity degree $\text{CaO/SiO}_2 = \text{P}_1 - 1.2$
Fuel consumption:	
F.C. in tonnes/tonne of pig iron	1.60
Volume of blast, Nm ³ /hr.	2600-2900
Pressure of blast, mm. WG.	1700-2000
Blast temperature, °C.	500-550
Top gas temperature, °C.	400-500
Flux rate per tonne of pig iron	0.80
Ore rate per tonne of pig iron	1.49
Slag volume per tonne of pig iron	1.00
Production tonne/day	7-8
Composition of slag:	
CaO, %	35.00-39.00
SiO ₂ , %	27.00-30.00
FeO, %	0.50-1.30
Al ₂ O ₃ , %	16.00-18.00
MgO, %	7.00-9.00
Composition of pig iron:	
C, %	2.50-3.00
Si, %	2.50-3.50
S, %	0.05-0.08
Mn, %	0.20-0.90
P, %	0.08-1.00
Gas analysis (by volume):	
CO, %	21.00-23.00
CO ₂ , %	3.00-4.00
CH ₄ , %	4.00-6.00
CO/CO ₂ ratio	6.00-6.70

Although pig iron of standard analysis was obtained with optimum metallic yield and slag analyses, the fuel consumption was rather high. It is well known that due to the short height of the shaft and limitation imposed on indirect reduction, the fuel rate in a low-shaft furnace is generally higher than a conventional blast furnace. For operation with nut coke, the height of the shaft can well be increased with improvements in heat transfer and indirect reduction. The burden was calculated with a lime basicity degree of 1.17. Due to the contamination of limestone/ore with clay, the basicity often was lower than this value, resulting in high sulphur in metal. The presence of 8-10 per cent MgO in the slag assured satisfactory fluidity despite variations in basicity degree. However, to economize the flux rate, reduce the slag volume and thereby reduce the fuel rate, the basicity degree should be maintained at its minimum needed for adequate desulphurization. In industrial plant, adapting proper grading and matching of particle size of raw materials with high-hot blast temperature and constancy of operation over long periods at optimum driving rate, the fuel rate is expected to be of the order of 1.3-1.4 tonnes of nut coke per tonne of pig iron. For industrial cost calculations, a fuel rate of 1.3-1.4 tonnes per tonne iron can well be assumed in the present case.

Even after the addition of non-coking coal, the temperature of the furnace top gas remained high varying between 350° and 400°C. The gas analyses showed CO/CO₂ ratio of about 6.5, which showed inadequate indirect reduction presumably due to the lower reducibility of the ore. The high-fuel rate was directly related to the poor utilization of chemical heat of the gas and can be improved by using ore of finer sizes. The amount of dust generated was reasonable at 4-5 per cent of the burden. In view of non-availability of any solid fuel in Rajasthan, the preliminary smelting trials were conducted with nut coke.

Eighteenth Furnace Campaign

Furnace Campaign Smelting Trials with Iron Ore and Limestone from Nepal

Nepal has no iron and steel industry and the limited requirement of these materials is met with by imports. In view of the availability of iron ore, limestone and timber from the extensive forests, the possibilities of winning iron was explored employing nut coke as fuel and reductant. Charcoal can be employed as and when it becomes available.

Distribution of Raw Materials in Nepal

The iron ore occurs at Phul Choki at a distance of 161 km. by road from the proposed site of the plant at Hitaura. In the absence of any railway connecting these places, the ore will have to be transported by road. The installation of ropeway will reduce the distance to 64 km. and reduce the cost of transport accordingly. Besides,

the ropeway will assure steady supply of the ore which will not be affected by changes of weather and conditions of the road. The occurrence of limestone at Bhainse Dhuban is adjacent to the proposed site and can be transported by road in trucks.

In the absence of any coal deposits in Nepal, iron smelting of ore can be conducted with nut coke from India or charcoal made at the plant site.

The chemical analyses of raw materials employed for smelting trials are given in Tables 70-73.

Screen Analysis of Raw Materials Employed for Smelting

As the particle size of the raw materials plays vital role on the smelting characteristics, their screen analysis was conducted and the results are given in Table 74.

No large-scale wood-carbonization plant is in operation in Nepal. The recovery of

Table 70 — Proximate Analyses of Nut Cokes

Location	Moisture %	Volatile matter %	Ash %	Fixed carbon %
Tata Iron & Steel Co. Ltd.	3.00	1.80	20.10	75.10
Durgapur	2.30	2.30	24.70	70.70

Table 73 — Analyses of Fluxes

Material	CaO %	SiO ₂ %	Al ₂ O ₃ %	MgO %
1. Limestone of Bhainse Dhuban (Nepal)	42.20	16.60	5.03	2.40
2. Limestone (M)	54.30	0.88	1.23	1.01
3. Limestone (A)	32.20	0.03	0.56	25.00
4. Dolomite (B)	32.60	3.90	1.60	20.40

Table 71 — Ash Analyses of the Nut Coke

Location	Fe %	SiO ₂ %	Al ₂ O ₃ %	CaO %	MgO %	P %
Tata Iron & Steel Co. Ltd.	6.00	52.08	33.0	3.80	2.12	0.69
Durgapur	9.70	50.90	25.60	3.10	1.90	0.27

Table 72 — Analysis of Iron Ore (Nepal)

Location	Fe %	SiO ₂ %	Al ₂ O ₃ %	CaO %	MgO %	Mn %	S %	P %
Phul Choki, Godavari (Nepal)	60.00	9.00	2.80	0.70	1.0	Trace	0.038	0.0105

Table 74 — Screen Analysis of Raw Materials

Material	+50.8 mm.	50.8 mm.- 25.4 mm.	25.4 mm.- 12.7 mm.	12.7 mm.- 6.35 mm.	6.35 mm.- 3.175 mm.	Below 3.175 mm.
1. Nut coke (T)						
(A)	Nil	25.3	29.5	7.8	8.3	29.1
(B)	4.6	52.2	39.4	2.8	0.2	0.8
2. Nut coke (D)	4.9	60.2	24.4	3.1	2.3	5.1
3. Lower Kenda coal	26.5	50.0	9.2	1.5	2.9	9.5
4. Nepal iron ore (A)	Nil	6.5	72.5	11.9	4.4	4.7
5. Nepal limestone	Nil	3.8	80.2	11.2	1.7	3.2
6. Limestone (M)	Nil	8.2	26.9	19.2	19.5	26.5
7. Limestone (A)	Nil	16.2	72.4	5.3	1.7	3.5
8. Dolomite (B)	Nil	16.2	25.6	17.0	13.6	27.6
9. Manganese ore	Nil	4.4	76.7	3.0	13.2	2.7

(A) As received.

(B) After screening through 12.7 mm. screen.

byproducts from destructive distillation of wood would improve the overall economics of carbonization. A small amount of electric power will be required for ancillary uses during iron smelting, the cost of availability of the power and water will have to be assessed at site.

Appraisal of Raw Materials

From the analyses of iron ore and flux, it was apparent that despite moderately high-iron contents in ore, its high-silica contents coupled with high-silica contents of the limestone would result in high-slag volume which in turn will substantially increase the fuel rate and decrease the iron output. Individual pieces of ore particles were noticed to contain high silica.

The apparent average porosity of the ore was 3.27 per cent which is very low compared to iron ores of India. Such low porosity will not afford adequate penetration of reducing gases into the ore body. The density in gm./cc. of the ore was 4.04, the low value being due to its high-silica content. The fusion point of the ore under oxidizing condition was found to be 1435-1465°C. which was similar to Indian iron ores.

Petrological Examination of Limestone and Iron Ore

Limestone — The limestone consisted of white, buff and grey-coloured pieces. Examination of the thin sections of limestone showed uniformity of texture, consisting of crystalline calcite with multiple twinning in association with quartz and muscovite.

The petrological structure exhibited the crystalline nature of the limestone with quartz and mica.

Iron Ore — Examination of the iron ore revealed it to be of dense and massive nature as well as of laminated character. In between the hematite laminae, presence of jasper and other gangue was noticed.

Examination of ore-polished sections and of crushed material indicated the ore to be essentially hematite. Presence of quartz and chlorite was noticed. Hematite was seen replacing jasper bands as well as other silicate gangue which showed signs of brecciation.

Physical Characteristics of Iron Ore

In order to find out the degradation of the ore during its handling, transportation,

storage in hoppers at the plant site and also during charging and its behaviour during its descent in the furnaces, the ore was subjected to shatter and tumbler tests. In shatter test, 2000 gm. of the ore was allowed to fall freely from a height of 3.6576 metres. For the tumbler test, 2000 gm. of the ore was placed in a cylindrical drum, 355.6 mm. long and 304.8 mm. diameter, having a baffle plate at its middle and was revolved at 70 r.p.m. for 28 min.

The results of shatter and tumbler tests confirmed that the ore possessed adequate crushing strength and abrasion resistance.

Reducibility Characteristics of Iron Ore

Reducibility characteristics of Nepal iron ore containing Fe, 54 per cent and SiO_2 , 16.3 per cent were investigated. This confirmed the view expressed earlier that pockets of the ore contained high silica. It may, therefore, be necessary to separate the ore containing high silica by suitable treatment. These tests were conducted with hydrogen gas as the reductant with variations in reaction temperature, gas velocity and particle size of the ore. It was observed that the reducibility of the Nepal iron ore was extremely poor. It was observed that for the same period, at 900°C ., reduction in the case of Orissa Minerals ore had progressed to 91.7 per cent, while in the case of Nepal ore only 73 per cent was reduced. At a lower temperature of 800°C ., for the identical time of reduction, the Orissa Minerals ore attained 79 per cent reduction compared to 47 per cent of Nepal ore. It was, therefore, evident that Nepal iron ore possessed poor reducibility. On reducing the size of Nepal ore to $-6.35 \text{ mm.} + 3.175 \text{ mm.}$, the reducibility was somewhat improved. Decrepitation of the ore did not occur on heating in a reducing atmosphere as it generally happens with other ores. From these observations it was evident that the ore had to be crushed to a smaller size to effect

its reduction in the limited time afforded in the low-shaft furnace.

Details of Smelting Trials

As the use of raw materials of unknown smelting characteristics may lead to operational difficulties, raw materials from Nepal were introduced in the burden in progressive stages. Based on the fluxes employed, the extensive trials with the Nepal Government raw materials were subdivided into three groups as follows:

Investigation Series No. A

Nut coke (T & D)
Nepal iron ore
Nepal limestone
Dolomite (B)

Investigation Series No. B

Nut coke (T & D)
Nepal iron ore
Nepal limestone
Limestone (M)
Limestone (A)
Dolomite (B)
Manganese ore

Investigation Series No. C

Nut coke (T & D)
Nepal iron ore
Limestone (M)
Limestone (A)
Manganese ore

Investigation Series No. A

After the operational characteristics of the low-shaft furnace became normal, extensive smelting trials were conducted with Nepal iron ore and limestone employing Indian nut coke as the fuel and reductant, with some dolomite additions to maintain optimum MgO content of 6-8 per

cent in the slag. During these trials, operational variables such as the volume, pressure of the preheated blast and burden schedules were suitably adjusted to yield smooth furnace operation and attain desired analysis of pig iron. The furnace was operated under the following operational conditions:

Operational Data of the Smelting Trial

Effective dia. of the tuyeres	75 mm.
Hot blast volume (Nm ³ /hr.)	2600
Hot blast pressure (mm. WG.)	1700
Hot blast temperature (°C.)	500
Top gas temperature (°C.)	400
Slag volume per tonne of pig iron	1.9

The composition of slag, pig iron and top gas were as follows:

Composition of Slag

CaO, %	32-38
SiO ₂ , %	36-39
FeO, %	3-4
Al ₂ O ₃ , %	15-20
MgO, %	6-8

Composition of Pig Iron

T.C., %	2.75
Si, %	1.0-3.50
S, %	0.08-0.20
Mn, %	0.3-0.5
P, %	0.3-0.5

Top Gas Analysis (by volume)

CO, %	24-28
CO ₂ , %	3-4
CH ₄ , %	5-7
CO/CO ₂	6-8

A small amount of non-coking coal was primarily added to keep the temperature of the furnace top and the gas at the desired level. As most of it disintegrated and was carried away in the flue dust, it did not

function as a fuel. The observed fuel consumption of 3 tonnes of coke/tonne of pig iron was rather high. The comparatively larger particle size of the iron ore together with high-silica content coupled with very high-silica contents of limestone resulted in abnormal slag volume, which in turn increased the thermal load on the furnace and thereby adversely affected the fuel rate. It has been observed that fuel rate depended on several factors such as reducibility, porosity and chemical analyses of the ore, the indices of direct and indirect reduction in the smelting and chemistry of the pig iron and slag made. Due to larger particle size and low reducibility of the iron ore, the amount of indirect reduction was exceedingly poor thereby increasing the fuel rate and the CO/CO₂ ratio in the furnace top gas. However, the exclusive utilization of raw materials of the Nepal Government enabled the establishment of optimum fuel rate—employing nut coke as the fuel and reductant. On the whole, furnace operation was normal. Some hanging of burden in the furnace was observed which fed inadequately prepared and preheated burden to the smelting zone and affected the fuel rate, composition of pig iron, etc., as a result of which the analyses of pig iron, slag and the top gas varied widely during this investigation and the slag sometimes contained high amount of sulphur and FeO. The retention time of the slag and the metal inside the furnace was extremely low for which the partition of sulphur between the metal and slag was poor. Due to the higher silica content of the limestone, the furnace had to be operated at a low-slag basicity.

Investigation Series No. B

In order to study the effect of better grades of limestone on the smelting characteristics of the Nepal ore using nut coke as the fuel, the Nepal limestone was progressively replaced in this investigation by

suitable Indian limestones. The MgO content of the slag was, however, maintained in the range of 9-12 per cent by incorporating some dolomite or dolomitic limestone in the burden. Requisite quality of high-grade manganese ore assaying Mn, 50.7; Fe, 5.6; SiO₂, 11.2; and Al₂O₃, 10.3 per cent was incorporated in the burden to yield the necessary manganese content.

Burden calculations for smelting with varying amounts of Nepal limestone along with superior grades of limestone (M) and limestone (A) and composition of individual charge were also worked out. The operational data are summarized in Table 75.

Operating data clearly indicated that the basicity of the slag CaO/SiO₂ could be increased from 1.2 to 1.32 without increasing the slag volume whilst ensuring improved desulphurization but causing high-fuel rates.

Investigation Series No. C

In this investigation the Nepal ore was smelted with nut coke as fuel, with exclusive use of limestones of low SiO₂ content with the object of reducing the slag volume and the fuel rate.

The composition of the metal, slag and the top gas during this investigation were observed to be as follows:

Composition of the Metal

C, %	2.4-2.75
Si, %	2.30-2.50
S, %	0.07-0.08
P, %	0.47
Mn, %	0.19

Composition of Slag

CaO, %	33.6
SiO ₂ , %	34.2
FeO, %	4.2
Al ₂ O ₃ , %	17.05
MgO, %	6.85

Table 75 — Operational Data on Progressive Replacement of Nepal Limestone with a Better Grade Limestone

Data on	A 40% Nepal limestone 60% other fluxes	B 30% Nepal limestone 70% other fluxes	C 23% Nepal limestone 77% other fluxes
Fuel rate F.C./tonne of pig iron	2.43	2.54	2.66
Ore rate in tonnes/tonne of pig iron	1.53	1.53	1.53
Flux rate in tonnes/tonne of pig iron	1.76	1.76	1.69
Slag volume in tonnes/tonne of pig iron	1.95	1.99	1.94
CaO/SiO ₂ -P ₁ (calculated)	1.24	1.34	1.31
<i>Average metal analysis</i>			
C, %	2.03	2.40	2.43
Si, %	3.55	3.10	2.40
S, %	0.32	0.21	0.16
P, %	0.46	0.49	0.26
Mn, %	0.26	0.46	0.14
<i>Average analysis of slag</i>			
CaO, %	32.58	38.80	38.05
SiO ₂ , %	39.20	39.00	36.20
FeO, %	1.40	2.50	0.70
Al ₂ O ₃ , %	17.05	19.23	14.60
MgO, %	6.93	7.00	8.00
<i>Average analysis of top gas (by volume)</i>			
CO, %	27.20	27.90	27.70
CO ₂ , %	3.20	3.45	3.20
CH ₄ , %	3.80	4.21	3.70
CO/CO ₂	7.10	6.60	7.40
Hot blast volume, Nm ³ /hr.	2600	2700	2600
Hot blast pressure, mm. WG.	1800	1700	1750
Hot blast temp., °C.	500	570	510
Average top gas temp., °C.	400	430	410

Composition of the Top Gas by (volume)

CO, %	30.61
CO ₂ , %	3.45
CH ₄ , %	2.12
CO/CO ₂	8.80

The use of limestone containing higher amount of CaO in it improved the smelting characteristics. It was observed that

standard foundry grade pig iron could be produced with the Nepal iron ore with high-lime base limestone. To raise the Mn content in the pig iron to the required range, manganese ore was incorporated in the burden. The addition of dolomite or dolomitic limestone to maintain 6-8 per cent MgO in the final slag was useful. In view of poor desulphurization in the earlier trials, the lime basicity of the slag was simultaneously increased to 1.4 with the use of better grade limestone. Equating for the higher basicity of the slag and its volume with earlier trials, the calculated fuel rate will obviously be lower with limestone containing lesser amount of SiO_2 . It will be, therefore, necessary to employ in industrial scale smelting limestones containing as little as SiO_2 , which will reduce the fuel rate and improve the quality of iron produced.

Discussion of Results — Recovery of Byproducts

The chemical analysis of iron ore revealed the presence of high and varying amounts of silica in it. The chemical analysis of limestone showed the presence of silica with the result that effective CaO for fluxing was as low as 26 per cent at CaO/ SiO_2 ratio of 1.2. High-silica content of the iron ore coupled with high silica of the limestone yielded a highly siliceous slag which in turn required heavy additions of limestone. It was, therefore, difficult to attain the desired lime basicity of the slag CaO/ SiO_2 of 1.2-1.3 so essential for desulphurizing the metal. Alternatively smelting can be conducted with an acid slag and the metal can be ladle desulphurized outside the low-shaft furnace. A study of physical characteristics of the ore revealed that ore possessed optimum crushing strength and abrasion resistance. An exhaustive study of reducibility characteristics in the laboratory with hydrogen gas demonstrated the ore possessed very

poor reducibility which added to the difficulties of smelting and required a high-fuel rate. The ore did not decrepitate during heating in a reducing atmosphere and therefore prior crushing of the ore to a suitable size before charging in the furnace will be necessary.

Nineteenth Furnace Campaign

In this campaign, object was to study the fuel requirements for smelting fine-grained iron ore (0.5 mm.) and limestone with either low-temperature carbonized coke obtained from Central Fuel Research Institute or from Regional Research Laboratory. The chemical analysis of raw materials employed in this campaign are given in Table 76.

Screen analysis of raw materials used in this campaign has been furnished in Table 77.

The furnace was blown in under the following operational conditions:

Tuyere diameter	75 mm.
Wind volume	2100-2500 Nm^3/hr .
Wind pressure	1700-2100 mm.WG.
Hot blast temperature	490-510°C.
Top gas temperature	350-400°C.

During initial stages, the furnace was operated with low-temperature carbonized coke obtained from Central Fuel Research Institute, when due to large particle size of fuel and its wide variation in size, high-ash and low fixed carbon content, the fuel requirement varied from 2 to 2.3 tonnes of fixed carbon. The replacement of this fuel by low-temperature carbonized coke from Regional Research Laboratory, Hyderabad, was immediately reflected on the lower fuel consumption when a fuel rate of 0.95 tonnes F.C./tonne of pig iron with slag volume of about 0.66 tonnes/tonne of pig iron was achieved. In other words, the fuel rate was 1.44 tonnes of Kolsit/tonne of pig iron. A consolidated data with Kolsit operation is given in Table 78 which shows

Table 76 — Chemical Analysis of Raw Materials Employed for Nineteenth Furnace Campaign

1. Analysis of iron ore

Location	Fe %	SiO ₂ %	Al ₂ O ₃ %	CaO %	MgO %
Messrs Orissa Minerals	64.14	3.28	4.57	Trace	Trace

2. Proximate analysis of low-temperature carbonized cokes

Location	Moisture %	Ash %	V.M. %	F.C. %
C.F.R.I. (Jalgora)	5.80	28.00	4.60	61.60
R.R.L. (Hyderabad)	2.30	21.20	8.90	65.67

3. Ash analysis of carbonized fuels

Location	SiO ₂ %	Fe %	Al ₂ O ₃ %	CaO %	MgO %	P %	S %
C.F.R.I.	56.31	7.61	22.00	3.45	2.20	1.83	0.27
R.R.L.	62.07	7.60	22.72	1.59	2.18	0.11	(Total S) 0.26

4. Analysis of fluxes

Location	CaO %	MgO %	SiO ₂ %	Al ₂ O ₃ %
Birmitrapur (Orissa)	44.80	3.57	6.96	1.60
Madras	54.31	1.01	0.88	1.23
Andhra Pradesh	32.20	25.00	0.30	0.56

Table 77 — Screen Analysis of Raw Materials

	Wt., %					
	+ 50.8 mm.	50.8 mm.- 25.4 mm.	25.4 mm.- 12.7 mm.	12.7 mm.- 6.4 mm.	6.4 mm.- 3.2 mm.	< 3.2 mm.
Kolsit	Nil	52.2	38.3	4.6	1.5	3.4
Orissa Minerals ore	Nil	Nil	6.4	35.7	30.1	27.8
Madras limestone	Nil	15.3	36.7	16.5	12.3	19.2
Andhra limestone	Nil	27.3	57.5	9.6	2.9	2.7
Bisra limestone	Nil	Nil	2.4	10.8	46.1	40.7

progressive decrease in fuel rate with continuity of smelting.

It is considered that further reduction of fuel rate can be possible in industrial furnace. Smelting during this campaign was exceedingly regular and no irregularity in the descent of the burden was noticed which primarily contributed towards the optimum fuel efficiency. The gas-cleaning plant was continuously in operation during this period.

In the second series of investigation, extensive smelting trials were conducted

with fine-grained iron ore from Orissa, Rajasthan and Andhra limestone employing either nut coke or low-temperature carbonized coke (F.R.I.) as fuel and reductant. The MgO content in the slag was increased in stages from 5 to 15 per cent specifically to find out the effect of increasing amounts of MgO in the slag on smelting behaviour of the furnace, the composition of the pig iron produced, partition of sulphur, the fuel and flux rate, etc.

The operational data with different MgO content in the slag are given in Table 79.

Table 78 — Consolidated Data on Operation with Kolsit and Fine-grained Iron Ore Showing Decrease of Fuel Rate with Progress of Smelting

Fuel rate F.C./tonnes/T.P.I.	2.00	1.82	1.61	1.49	1.28	1.19	0.99
Slag vol.	1.20	1.66	1.08	0.99	0.88	0.82	0.69
Av. analysis of metal							
C, %	3.36	3.40	2.80	2.90	3.60	3.40	2.90
Si, %	1.86	2.64	1.60	2.30	5.14	3.74	2.60
S, %	0.13	0.155	0.10	0.076	0.014	0.067	0.073
P, %	0.40	0.11	0.09	0.097	0.094	0.09	0.063
Mn, %	0.62	0.69	0.77	0.42	0.75	0.25	0.34
Av. analysis of slag							
CaO, %	35.10	36.10	38.50	41.80	—	39.70	—
SiO ₂ , %	36.50	35.90	34.90	32.40	—	33.60	—
FeO, %	2.00	2.50	1.50	1.20	—	2.00	—
Al ₂ O ₃ , %	18.00	17.20	16.40	16.00	—	16.70	—
MgO, %	7.50	7.80	7.90	7.90	—	7.70	—
S, %	0.33	0.48	0.49	0.52	—	0.47	—
Av. analysis of gas (by vol.)							
CO, %	24.60	24.10	24.60	N.D.	22.00	22.20	22.40
CO ₂ , %	5.20	5.20	5.80	—	5.10	5.40	5.60
CH ₄ , %	4.80	5.20	5.00	—	6.30	6.00	5.80
CO/CO ₂	4.80	4.60	4.30	—	4.20	4.10	4.00

N.D. = Not determined.

Table 79 — Effect of Increasing MgO Contents in Slag on Smelting Characteristics

Data on	5% MgO	10% MgO	15% MgO
Fuel rate	2.10	2.54	2.58
Slag basicity (CaO/SiO ₂)	1.03	1.16	1.19
Wind volume, Nm ₃ /hr.	2600	2600	2700
Wind pressure, mm. WG.	2000	2000	1900
Blast temperature, °C.	500	500	500
Flux rate tonne/tonne of pig iron	0.78	1.35	1.56
Production tonne/day	8.14	6.00	4.10
Slag volume	0.90	1.80	2.03
Raw materials consumed/tonne of pig iron	5.20	7.00	7.24
Av. analysis of metal			
C, %	3.20	3.02	2.80
Si, %	2.65	1.87	1.54
S, %	0.11	0.10	0.08
Mn, %	0.48	0.53	0.24
P, %	0.39	0.72	0.14
Av. analysis of slag			
CaO, %	35.50	35.20	37.20
SiO ₂ , %	32.30	32.40	36.92
FeO, %	2.40	0.90	1.00
Al ₂ O ₃ , %	21.60	17.50	18.40
MgO, %	7.00	7.50	8.20
Av. analysis of top gas (by vol.)			
CO, %	23.90	23.50	22.20
CO ₂ , %	3.70	3.40	3.10
CH ₄ , %	3.30	5.03	5.03
CO/CO ₂ ratio	6.40	6.90	7.16

A critical review of the data revealed that MgO contents in slag were not compatible with the MgO additions of dolomite in the burden and the results are, therefore, not considered conclusive. It was, however, observed that the productivity of the furnace decreased from 8.14 tonnes/day to 4.1 tonnes/day particularly because of the fact that the slag volume increased from 0.9 to 2.03 tonnes/tonne of pig iron. Similarly the fuel consumption increased from 2.7 tonnes F.C. to 2.58 tonnes F.C./tonne of pig iron. The effect of MgO on desulphurization index could not be ascertained because of identical MgO contents in the slag. Further experiments with different MgO content in the slag are being planned.

78.1 Investigations on Briquetting

Briquetting of Beneficiated Limestone for Steelmaking Purposes — In the as-received condition, the limestone assayed: 45.5 per cent CaO, 10.5 per cent total insolubles.

After beneficiation the concentrate assayed: 50 per cent CaO, 3.0 per cent total insolubles.

Briquetting Process

As the average particle size of the beneficiated product was —200 mesh and cannot, therefore, be employed directly for steel-making, it has to be agglomerated either by briquetting or by pelletizing. The beneficiated concentrate was thoroughly mixed with either 6 per cent molasses or 6 per cent sulphite lye as binder and briquetted in a roller-briquetting press. The moisture content of the mix prior to briquetting was kept around 4.5 per cent. The green briquettes were allowed to age for about 24 hr. and were then suitably heat-hardened to develop adequate physical strength. The percentage of moisture in these briquettes was found to vary between 1.5 and 3 per cent.

Physical Properties of Briquettes after Heat-hardening — In order to assess the degradation of briquettes during handling, transportation and charging in basic open-hearth steelmaking furnace, the briquettes were subjected to shatter test. In shatter test, the briquettes were allowed to fall freely from a height of 3.66 mm. (12 ft.) and the screen analysis of the product is recorded in Table 80.

As the main source of breakage of briquettes is during handling, a test based on tumbling resistance of briquettes was conducted to ascertain this behaviour. For this, briquettes were tumbled in a 30.5 cm. dia. and 35.6 cm. long drum with a baffle plate inside. The drum was revolved at

75 r.p.m. for 30 min., +3.2 mm. fraction was found to be 33.8 per cent. The room crushing load of the briquettes was found to vary between 400 and 450 kg. It was, therefore, considered that the briquettes possess an adequate physical strength to sustain handling and charging in the open-hearth furnace for steelmaking.

About 120 tonnes of briquettes were employed in 8 experimental steelmaking heats in 200 tons basic open-hearth furnace of the Tata Iron & Steel Co. Ltd. with efficient removal of phosphorus and considerable reduction in time of steelmaking, which will increase the output.

78.2 Utilization of Fine Size Manganese Ore, Coke and Limestone by Briquetting for Smelting Ferro-manganese in Electric Furnace

With a view to utilizing the fine-grained raw materials obtained during the manufacture of ferro-manganese, manganese ore, limestone and coke were obtained from Meesrs Khandelwal Ferro-alloys Ltd., Kanhan, Maharashtra, for conducting investigations on the production of briquettes suitable for smelting in electric furnace. Different types of binders were employed for investigation. In order to assess the physical characteristics, shatter index, crushing strength and under-load tests at elevated temperatures were conducted. Briquettes with bentonite and burnt lime were found unsatisfactory. Due to poor bonding of inorganic binders, the organic binders such as molasses and sulphite lye were employed

Table 80 — Screen Analysis after Shatter Test of Briquettes

Condition of briquette	50.8 mm.- 25.4 mm.	25.4 mm.- 12.7 mm.	12.7 mm.- 6.25 mm.	6.35 mm.- 3.2 mm.	Below 3.2 mm.
Air dried, wt. %	77.2	12.3	6.0	1.1	3.4
Heat-hardened, wt. %	88.3	7.3	2.6	0.4	1.4

in amounts of 4-6 per cent. Although these binders considerably improved the physical properties particularly after heat treatment around 200°C., the strength at elevated temperature was rather poor. Amongst these two, sulphite lye was found to yield slightly better physical properties. The physical properties of briquettes with 4 per cent sulphite lye were identical with 6 per cent molasses as binder up to a certain temperature. Treatment of briquettes at progressively higher temperatures showed considerable deterioration. In order to secure adequate high-temperature stability, 5, 10 and 15 per cent coking coal was added and optimum results were obtained with 10 per cent coking coal. With this amount of coal addition, a variation of sulphite lye addition of 4, 6 and 8 per cent did not appreciably affect the physical properties. The properties of briquettes with 4 per cent sulphite lye and 10 per cent coking coal gave almost identical results. However, the availability of coking coal *vis-à-vis* the economy of binder addition will need assessment.

As grain sizes of ingredients affected the quality of briquettes, the grain size of coking coal alone was varied from below 2.41, 1.00 and 0.50 mm. in briquettes with either 4 per cent sulphite or 4 per cent molasses. It was observed that the variations in grain size of coking coal did not cause any outstanding benefit. Similarly the addition of 10 per cent coking coal to briquettes pressed with 4 per cent molasses as binder conferred high-temperature stability. However, those formed with 4 per cent sulphite lye produced more stable briquettes than obtained by 4 per cent molasses. The under-load characteristics of these briquettes were

satisfactory and they are expected to be suitable for smelting. In view of the difficulties of obtaining coking coal in Maharashtra it may be worth while to study smelting characteristics of briquettes with either 6 per cent molasses or 4 per cent sulphite lye.

78.3 Briquetting with Kiriburu Iron Ore for Export Purposes

As desired by the State Trading Corporation, the briquetting of Kiriburu iron ore fines was studied with a view to exporting these briquettes to Japan. The analysis of the ore was as follows:

Fe, %	...	64.2
SiO ₂ , %	...	1.89
Al ₂ O ₃ , %	...	1.10
L.O.I. at 950°C., %	...	4.92

The screen analysis of the ore employed for briquetting is recorded in Table 81.

The crushed ore was thoroughly mixed with 6 per cent molasses as binder and briquetted in a roller-briquetting press. After ageing, briquettes were heat-hardened at a temperature range of 400-600°C. The shatter strength was determined by allowing a sample of briquette to fall once from a height of 3.6 m. (12 ft.) when nearly 100 per cent was above 25 mm. size. The crushing load of the heat-hardened briquettes was found to vary between 250 and 700 kg. These physical properties indicated adequate stability for handling of briquettes.

78.4 Briquetting of Coke Breeze

Coke breeze was obtained from a firm in Calcutta for studying the briquetting

Table 81 — Screen Analysis of Iron Ore

+2.41 mm.	2.41 mm.- 1.00 mm.	1.00 mm.- 0.50 mm.	0.50 mm.- 0.25 mm.	0.25 mm.- 0.15 mm.	0.15 mm.- 0.08 mm.	Below 0.08 mm.
%	%	%	%	%	%	%
38.5	16.3	11.4	4.7	5.8	10.5	12.8

Table 82 — Screen Analysis of Coke Breeze

In per cent								
+ $\frac{1}{4}$ "	$\frac{1}{4}$ "- $\frac{1}{8}$ "	$\frac{1}{8}$ "- 0.0949"	0.0949"- 0.0395"	0.0395"- 0.0197"	0.0197"- 0.0099"	0.0099"- 0.006"	0.006"- 0.003"	+0.003"
14.7	33.12	1.92	19.45	16.20	3.75	6.45	2.11	2.30

characteristics. Screen analysis of the coke breeze in the as-received condition is given in Table 82.

The material in the as-received condition was found to be quite suitable for briquetting without further crushing and screening. Several series of trials were made on suitably briquetting the coke breeze. Briquettes were prepared by thoroughly mixing the material in a mixing worm along with 4-6 per cent molten coal-tar pitch employed as a binder and thoroughly pressing the mixture in roller-briquetting press. The crushing load of the green briquettes was determined after an ageing period of 24 hr. It varied between 115 and 125 kg. on the briquette.

The shatter index of the green briquettes after an ageing period of 24 hr. was determined by allowing the briquettes to freely fall from a height of 12 ft. The screen analysis of the product is given in Table 83.

Table 83 — Screen Analysis of Briquettes after Shatter Test

2"-1"	1"- $\frac{1}{2}$ "	$\frac{1}{2}$ "- $\frac{1}{4}$ "	$\frac{1}{4}$ "- $\frac{1}{8}$ "	$\frac{1}{8}$ "
72.4	13.8	6.8	3.7	3.39

From the crushing load as well as the shatter index of the green briquettes it was observed that suitable coke breeze briquettes can be made for diverse uses. Due to the presence of coal-tar pitch, copious smoke could be expected on burning the briquettes in a domestic oven. However, the binder employed would be the cheapest of those readily available for briquetting coke breeze.

78.5 Malleabilization Characteristics of Low-shaft Furnace Pig Iron

Smelting of pig iron in low-shaft furnace with low-temperature carbonized coke made from totally non-coking coals yielded a pig iron containing exceedingly low-phosphorus contents which is ideally suitable for making malleable castings. A project was initiated with a view to studying the malleabilizing characteristics of such irons with or without the addition of elements which are known to shorten the malleabilization cycle. Pig iron containing C, 2.8; Si, 2.70; Mn, 0.40; P, 0.30; and S, 0.42 per cent was used as base material, to which desired amount of steel scrap was added to obtain white iron suitable for making malleable castings. In a cast sample analysing C, 2.52; Si, 1.12; Mn, 0.56; S, 0.02; and P, 0.22 per cent, the period of first stage of malleabilization was varied from 12, 24 and 36 hr. at 980°C. followed by secondary stage at 730°C. for 12 hr. followed by slow cooling in all cases. It was noticed that a period of 24 hr. in the first stage and 12 hr. for second stage were adequate for graphitization. For studying the effect of prior microstructure on the morphology of graphite nodules, samples previously heat-treated such as by normalizing, oil quenching and water quenching were malleabilized, but the prior treatment did not alter graphitization characteristics.

The effect of addition of boron and aluminium on malleabilization cycle is being investigated. In iron containing C, 2.60; Si, 1.74; Mn, 0.38; S, 0.06; P, 0.40; and B, 0.018 per cent, the time for complete

Table 84—Analytical Survey of Operational Characteristics of Steelmaking by the Basic-lined Side-blown Converter Process

Heat No.	Time of blowing min.	Lime added kg.	Hot metal analysis, %					Blown metal analysis, %					Analysis of slag					Remarks		
			C	Si	S	Mn	P	C	Si	S	Mn	P	SiO ₂	Fe ₃ O ₃ +FeO	Al ₂ O ₃	MgO	CaO		P ₂ O ₅	MnO
3	22	20	3.4	4.6	—	—	0.41	2.88	2.39	—	—	0.45	—	—	—	—	—	—	—	L.S.F. iron Fe-Si added. Inadequate blowing L.S.F. blower developed back pressure
4	25	20	3.1	4.7	0.07	0.26	0.63	2.40	2.40	0.02	0.18	0.48	41.30	14.98	—	10.60	27.10	0.24	—	Addition of ferro-silicon to L.S.F. iron, already high in Si, rendered decarbonization difficult
5	35	30	3.9	1.16	0.035	0.7	0.25	0.6	0.62	0.04	Trace	0.058	17.6	39.93	6.67	11.8	21.9	—	1.30	Basic iron was used instead of L.S.F. pig
7	32	40	3.6	1.16	0.05	—	0.25	1.8	0.04	0.045	—	0.03	11.84	61.10	2.1	5.5	18.19	—	—	Part decarbonization was due to inadequate blowing time
8	30	40	3.5	1.3	0.05	—	0.25	0.36	0.24	—	—	—	14.64	59.5	2.48	16.4	6.8	—	—	Metal slag separation was unsatisfactory due to very viscous slag
9	30	40	3.6	1.4	0.05	—	0.25	0.72	0.23	—	—	—	39.28	33.3	2.3	13.4	11.34	—	—	do
19	15	40	3.19	2.28	—	0.51	0.28	0.60	0.15	—	Trace	0.069	17.6	28.43	3.14	36.1	10.1	3.9	0.50	Two blowers used in parallel were employed
20	15	30	3.82	1.40	—	0.62	0.17	1.77	0.35	—	Trace	0.102	19.8	28.86	2.94	36.2	9.6	2.37	0.50	do
21	15	30	3.74	1.70	—	0.61	0.15	1.74	0.48	—	0.27	0.056	17.64	31.34	3.62	21.9	20.17	2.96	2.20	do
22	18	30	3.87	1.70	—	—	—	0.054	0.85	—	—	—	—	—	—	—	—	—	—	do
23	35	49	3.87	1.29	0.05	0.51	0.29	2.66	0.04	0.03	Trace	0.016	8.9	64.6	—	9.3	11.6	2.0	—	Vigorous slopping in vertical position
24	32	30	4.07	1.03	0.06	0.51	0.27	0.04	0.52/0.15	0.047	Trace	0.11	25.9	32.3	—	13.5	23.2	2.3	—	Slopping minimized by changing the tilt

graphitization was found to be 48 hr. in cast condition. Prior heat treatment shortened it to 24 hr. In a cast sample analysing C, 1.9; Si, 1.36; Mn, 1.02; S, 0.07; P, 0.24; and Al, 0.20 per cent, 48 hr. were necessary for complete malleabilization in as cast state, which was lowered to 24 hr. by prior heat treatment. However, the various prior heat treatments such as normalizing, oil or water quenching did not show any difference on the malleabilizing cycle.

78.6 Pneumatic Method of Steelmaking in Side Air-blown Converter

A 500 kg. magnesite lined side air-blown converter, earlier designed and fabricated at the National Metallurgical Laboratory, was installed. The steelmaking operations of this converter in the earlier experiments were faced with many handicaps and problems. Collecting of the hot metal from oil-fired tilting furnace and a cupola and its transfer by a manually operated crane often caused delay and chilled the hot metal before it could be poured in the converter. To overcome this difficulty, a hot metal receiver was designed, fabricated and installed in conjunction with a cupola, from which 500 kg. of the hot metal could be received at a time. This arrangement, however, did not permit super-heating of molten metal, a factor of considerable importance for intermittent pilot plant test trials.

With low-shaft furnace smelted pig iron containing high percentage of silicon it was observed that removal of silicon or carbon were inadequate. This was due to high percentage of silicon (4.6 per cent) in the hot metal. In view of it, trials were conducted with basic pig iron containing 1.3 per cent silicon. Several trials were conducted. In a few cases, per cent C was found too low, otherwise in most of the cases per cent C was on the higher side. Due to the non-availability of a volume meter, the amount of air blown could not be ascertained. In order to obtain higher blast volume,

two blowers were connected in parallel before the blast was introduced in the converter. With these modifications, considerable improvement during blowing was noticed and encouraging results were obtained. The operational characteristics were suitably altered to convert pig iron into acceptable grades of steel. Results obtained are summarized in Table 84.

78.7 Experimental Shaft Furnace with Provision for Injection of Supplementary Fuels through Auxiliary Tuyeres

A shaft furnace, 8 metres high and 0.51 metre diameter, has been designed and fabricated at the Low-shaft Furnace Project of the National Metallurgical Laboratory for the purposes of assessing the smelting characteristics of substandard grades of raw materials for iron production in cottage industry scale. The shaft of the furnace has been lined with suitable refractory lining of 225 mm. thick up to a height of 7 metres from the bottom. The furnace is provided with facilities for injecting supplementary fuels through a set of auxiliary tuyeres placed at the same height as air blast tuyeres, but inclined to one another so that the gas stream and air blast impinge at an appropriate point inside the furnace. There are 4 tuyeres for supplying air blast and 4 separate tuyeres placed alternatively to air blast benefits for injection of either fuel oil or gaseous fuel. Valves have been inserted at suitable places for regulating the flow of gaseous fuel or air. In order to recover a sensible heat from the top gas and eliminate the necessity of expensive blast-heating equipment, arrangements have been provided for heating the cold blast at the top of the shaft furnace. This arrangement is expected to raise the temperature of the blast to 200-250°C. which will be adequate for smelting iron in small scale. By suitable adjustment of valves, air blast supplied by

Table 85 — Analysis of Iron Ore (Orissa Minerals)

Location	Fe %	SiO ₂ %	Al ₂ O ₃ %	CaO %	MgO %	S %	P %
Orissa	65.46	1.8	1.85	Trace	0.8	0.01	0.02 ₃

the turbo-blower of the low-shaft furnace and heated in its recuperator can also be employed. An automatic skip hoist arrangement has been provided for feeding the furnace with raw materials. Suitably designed water sprays have been provided to cool the outer mild steel shell of the furnace excepting the topmost part where the blast-heating arrangement is located. The furnace is provided with slag and tap holes at 180° angle which are placed at appropriate heights for the removal of slag and metal at desired intervals.

It will not be difficult to obtain indigenously manufactured blowers suitable for supplying the requisite quantity of air blast for smelting purposes. It is presumed that the Pilot Shaft Furnace can be suitably scaled up for producing 20-30 tons of pig iron per day in small commercial scale. The possibilities of utilization of low-temperature carbonized coke (Kolsit), which gave exceedingly promising results in the 12-15 tonnes/day low-shaft furnace plant, were investigated in the shaft furnace. The analysis of raw materials employed are recorded in Tables 85-87.

At the initial stages, the charge consisted of nut coke, limestone and low-shaft furnace slag primarily intended for heating up the furnace. Then a burden consisting of low-temperature carbonized coke, very fine-grained iron ore and fluxes was smelted. The screen analyses of these raw materials are given in Table 88.

The air blast supplied by the turbo-blower of the low-shaft furnace was pre-heated in the recuperator to 350-400°C. The temperature was dropped to 190-220°C. at the hot blast main of the shaft furnace. After initial smelting with a blast pressure of

Table 86 — Analysis of Fluxes

Material and location	CaO %	SiO ₂ %	MgO %	Al ₂ O ₃ %
Limestone (Madras)	54.31	0.88	1.01	1.23
Dolomitic limestone (Andhra)	32.20	0.30	25.00	0.56

80-80 mm. Hg, the pressure was raised to 100-200 mm. Hg to obtain the desired smelting rate. Although the descent of the burden was irregular and the temperature in the combustion zone was inadequate, these improved with progress of smelting. The top gas temperature of 500°C. was rather high. In the absence of stock line recorder, the burden was fed at regular intervals. As volume and pressure of the blast were not recorded, the tapping and slagging were done at regular intervals.

The fuel consumption of 3 tonnes/tonne of pig iron was rather high and can be attributed to several causes such as low temperature of hot blast, high-heat loss by the furnace and slow rate of smelting.

78.8 Smelting of Self-fluxing Briquettes Containing Coke Breeze, Iron Ore Fines and Dust Limestone

In order to utilize the huge amount of coke breeze which accumulates in any integrated iron and steel plant concurrently with fine-grained iron ore and limestone (both of which are too fine for sintering purposes), these ingredients were briquetted together (containing 55 per cent coke breeze, 29 per cent fine-grained ore and 16 per cent

Table 87 — Analysis of Low-temperature Carbonized Coke (Kolsit)

	General range Coal (air dry basis) %	*Kolsit %	Typical individual sample			
			Coal (air dry basis)		Kolsit	
			I	II	I	II
			%	%	%	%
	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	—	11,000	—	—	—	—
(B.t.u./lb.)	9,800-10,500	11,400	10,500		11,400	

*Kolsit is the trade name of low-temperature carbonized coke made out of Singareni non-coking coal.

Kolsit — Ash analyses

	%	%
SiO ₂	63.65	62.07
Al ₂ O ₃ -TiO ₂	20.23	22.72
MgO	1.5-2.5	2.18
CaO	2.5-3.0	1.59
Fe ₂ O ₃	10-15	10.98
SO ₃	0.5-0.7	0.36
Alkali (by difference)	less than 1	0.10
Fusion range under mildly reducing and milding oxidizing atmosphere, Lietz heating microscope method	1500-1550°C.	1555°C.
Sources of coal	Singareni Collieries Co. Ltd., Kothagudem	

Table 88 — Screen Analysis of the Raw Materials Used

Material	+50.8 mm. size wt. %	50.8 mm.- 25.4 mm. wt. %	25.4 mm.- 12.7 mm. %	12.7 mm.- 6.35 mm. %	6.35 mm.- 3.4 mm. %	Below 3.4 mm. %
Kolsit	12.05	32.41	47.74	7.04	0.35	0.41
Orissa Minerals ore	—	—	5.7	23.0	28.1	43.2
Madras limestone	—	9.7	32.5	16.1	15.5	26.2
Andhra limestone	—	16.5	52.6	10.8	6.9	13.2

limestone) with the addition of 5 per cent bentonite as binder. The physical properties like shatter index, room temperature, crushing strength, etc., indicated satisfactory stability for handling and transportation of briquettes, but these properties deteriorated at elevated temperatures and the furnace stability of these briquettes was rather unsatisfactory. However, the descent of the burden composing exclusively of these briquettes was regular, but a large volume of ignited particles was noticed to eject from the top opening of the furnace. The

furnace utilization of the burden ingredients was poor. The analyses of pig iron and slag are given in Tables 89 and 90.

Table 89 — Analysis of Pig Iron

C	Si	Mn	S	P
%	%	%	%	%
2.7	2.7	0.29	0.2	0.54

Table 90 — Analysis of Slag

CaO	SiO ₂	FeO	MgO	Al ₂ O ₃	S
%	%	%	%	%	%
36.7	29.68	7.9	4.4	20.1	0.08

78.9 Investigation on the Use of the Substitute Fuels for Foundry Coke for Melting in the Cupola

Difficulties are experienced by iron foundries in obtaining supplies of foundry coke for melting pig iron in cupola. In order to find out the possibility of utilizing inferior grades of fuel, such as Jhama coal, low-temperature carbonized coke made from totally non-coking coals, an investigation was initiated.

Initially Jhama coal from Jama Doba collieries, Tisco, was tried. The chemical analyses of Jhama coal and its ash are recorded in Tables 91 and 92.

Table 91 — Proximate Analysis of Jhama Coal on Air-dried Basis

Location	Moisture %	V.M. %	F.C. %	Ash %
Digwadih (Jharia)	1.0	14.0	69.8	15.2

Table 92 — Analyses of Ash of Jhama Coal

Colliery	SiO ₂ %	Al ₂ O ₃ %	CaO %	MgO %	Fe %
Digwadih	52.8	34.2	3.4	4.7	4.1

Foundry coke was progressively replaced by 25, 50 and 100 per cent of Jhama coal, but the bed was made from foundry coke. In the second experiment, foundry coke was used for the bed only and melting was conducted exclusively with Jhama coal in the ratio 5 : 1 metal : Jhama coal. The size of Jhama coal was between 50 and 75 mm. The operation was quite smooth and the temperature of hot metal and slag were normal. In the next experiment, bed composed of 50 per cent Jhama coal and 50 per cent coke, but the result was not very satisfactory as the metal temperature was low and the bed prepared from Jhama coal and coke lasted for a very short time. In subsequent

experiments, bed in the cupola was made exclusively of coke and the ratio of Jhama coal to metal was varied in the ratio of 1 : 6 and 1 : 7 (the latter of pig iron), to effect optimum fuel economy. In these experiments 20 per cent steel scrap was also added in the last few charges. Temperature of tapped metal varied from 1200° to 1250°C.

In the second series of these experiments, C.F.R.I. soft coke was substituted for the foundry coke. In this series of experiments, metal/coke ratio was altered from 7 : 1, 6 : 1, 5 : 1. In the last few charges in all the experiments 20 per cent steel scrap was also added. The operation was found to be very normal and the metal temperatures were obtained as follows:

Metal/ lowering tempera- ture of carboniz- ed coke	Temperature of molten metal, °C.		Remarks
	Without steel scrap	With steel scrap	
7 : 1	1280	1200	The examination of the tuyere zone revealed inadequate heat which was presumable due to small size of L.T.C. coke which burnt away before it reached the tuyere region. This experiment was repeated with the size of coke 2-3 in.
6 : 1	1300	1200	
5 : 1	a) 1220	1170	
	b) 1340	1240	

In the third series, low-temperature carbonized coke made from totally non-coking coals of Singareni collieries, Andhra Pradesh (Kolsit), was substituted for the foundry coke ; the bed, however, was made of foundry coke. Melting with metal/coke ratio of 5 : 1 and 6 : 1 was found to be unsatisfactory. In the ratio of 6 : 1 the charge did not descent to the tuyere level and in the 5 : 1 ratio, the metal temperature was low. It was considered that due to the high reactivity of Kolsit, it burnt before it reached the melting zone.

79.0 Appraisal of Raw Materials for Iron Making

The economic exploitation of iron ore for the production of iron depends on its physical, chemical and petrological characteristics. The expansion of iron and steel industry necessitates the exploitation of hitherto unknown ores. The recent trends of higher productivity of the metal demand prior assessment of raw materials. With this in view, a comprehensive appraisal of iron-making raw materials and their correlation with smelting characteristics, particularly the fuel rate and slag chemistry has been initiated. The determination of the following properties are investigated:

- (1) Breaking load or compressive strength at room and elevated temperatures.
- (2) Shatter strength.
- (3) Abrasion strength.
- (4) Decrepitation property of iron ores.
- (5) Porosity of iron ores and other materials.
- (6) Abrasion of iron ores at elevated temperatures under different atmospheres with varying oxygen potentials.
- (7) The apparatus to study the different variables simulating the blast furnace stack conditions.
- (8) Behaviour of ores at elevated temperatures and under atmospheres of different oxygen potentials for their reducing characteristics.
- (9) Study of the decomposition characteristics of limestone.
- (10) Specific heat and thermal conductivity of iron ores for determination of the heat transfer characteristics.
- (11) Distribution characteristics of raw materials in a model low-shaft furnace.
- (12) Petrological examination.

The results are given in Table 93. The decrepitation test simulates the effects of time, temperature and furnace atmosphere on iron ores during its descent in blast

furnace and indicates probable breakage of ore and its loss as flue dust.

The porosity test consists of filling the voids successively with measured volumes of mercury and gases, preferably helium gas. It has been shown that mercury will penetrate all exposed pores greater than 14.1 microns in diameter at atmospheric pressure, helium will enter pores greater than 1.8 Å. in diameter. The difference between the value is a measure of the porosity relative to the fluids employed.

The abrasion of iron ores at elevated temperatures and at varying reducing atmospheres is important as it will give an idea of reducing action of the gas with temperature and time as well as the mechanical stressing of the ore. The apparatus consists mainly of an electrically heated rotating cell and apparatus for gas generation and analysis. The rotation will simulate the conditions and will give the abrasion.

The apparatus to study the variables simulating the blast furnace conditions will show the conditions at various depths below stockline level and in particular the thermal or chemical limitation. This can deal with the following problems:

- (a) Minimum coke rate for a given burden and products under given operating conditions.
- (b) Minimum coke rate for a given burden with different operating conditions.
- (c) The top gas conditions and direct and indirect reductions.
- (d) Heat and mass transfer rates in the stack.
- (e) Comparison of real and idealized furnace conditions.

The apparatus for the study of distribution characteristics of the low-shaft burden will deal with sequence of charging, the type of raw materials and their size fractions. Arrangements have been made to see the actual colour obtained of the distribution by sectioning into exact half by means of perspex sheet.

Table 93 — Chemical Analysis and Physical Properties of Some Iron Ores

Iron ore	Visual examination	Chemical analysis						Crushing strength tons/sq. in.	Shatter strength % + $\frac{1}{8}$ " fraction	Abrasion strength % + $\frac{1}{8}$ " fraction	Reducibility						
		Fe %	SiO ₂ %	Al ₂ O ₃ %	CaO %	MgO %	S %				P %	Size - $\frac{1}{8}$ " + $\frac{1}{4}$ "		Size - $\frac{1}{4}$ " + $\frac{1}{2}$ "			
												At 900°C. and 800 cc./min. of hydrogen flow	% Reduc- tion min.	At 800°C. and 800 cc./min. of hydrogen flow	% Reduc- tion min.	At 900°C. and 80 cc./min. of hydrogen flow	% Reduc- tion min.
Orissa Minerals	Mostly laminated	64.14	3.28	4.57	—	—	0.01	0.02	4.0	89.54	71.00	91.7	32	83.5	42	92.1	32
Punjab Govern- ment iron ore	Massive and hard	62.6	6.65	2.1	—	—	0.03	0.13	4.8	97.36	89.85	80.9	62	70.7	76	75.5	60
Andhra Govern- ment iron ore	Massive but laminated	59.8	4.0	5.92	—	—	0.05	0.12	1.52	96.70	79.30	95.6	74	95.9	82	94.8	68
I Variety																	
Andhra Govern- ment iron ore	Mostly porous but hard	63.74	3.8	2.0	—	—	0.03	0.06	3.7	98.00	89.35	96.9	139	92.8	95	97.1	130
II Variety																	
Nepal Govern- ment iron ore	Massive and hard	60.00	9.00	2.8	0.7	1.0	0.038	0.01	3.08	97.23	93.4	74.7	34	59.4	47	83.2	32
Rajasthan Government iron ore	Massive and hard	61.6	9.9	1.2	0.3	0.9	0.20	0.50	2.8	97.6	94.22	73.5	22	53.4	32	74.6	22

The programme includes study of iron ores:

(a) which have been successfully smelted in low-shaft furnace and those which can be used for the installation of small-scale plants with low-shaft furnace,

(b) which are utilized by Hindustan Steel Ltd.

(i) Rourkela

(ii) Bhilai

(iii) Durgapur

(c) which will be used by Bokaro Project.

80.0 Thermal Properties of Blast Furnace Raw Materials

The blast furnace can be considered as (i) a chemical unit and (ii) as a heat exchanger. In the past, the chemical aspect of the blast furnace operation overshadowed the heat exchange which occurs during the descent of the furnace operation and led to the evaluation of the iron ore on its iron content. With the recognition that the thermal efficiency of the blast furnace essentially depends upon the heat exchanger, the physical and the thermal characteristics of the iron ores also become important for the evaluation of the iron ores.

In order to fully appreciate the heat transfer in the blast furnace, it is necessary to know the heat content of all the raw materials, and the products of reduction, etc. No such information is generally available and requires extensive experimentation. A copper block calorimeter to determine the heat capacity of the iron ore up to about 900°C. was designed. The results obtained are shown in Figs. 48 and 49 and each point

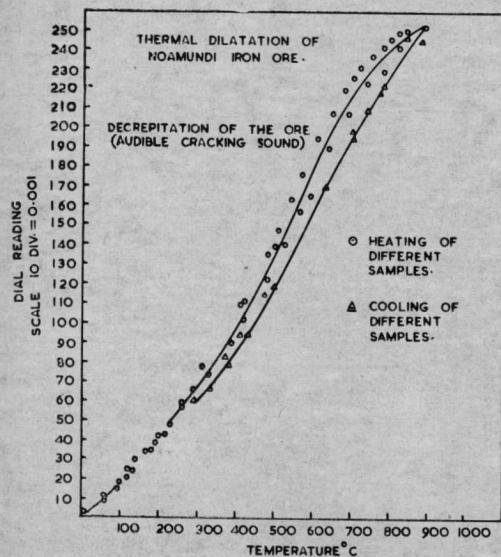


FIG. 48

represents an average of several readings. The thermal data of Kelley on pure Fe_2O_3 are also shown for comparison. During dilatation studies on iron from Noamundi and Barsua, audible decrepitation was observed between 450° and 600°C. The size of the sample was $2\frac{1}{4}$ in. length and $\frac{3}{8}$ in. diameter, and took 3 hr. to heat through this range of temperature. The diameter of the stack of the blast furnace progressively increases as it approaches bosh for smooth running. Although the accompanying increase in volume is much more than necessary to accommodate the thermal expansion of the raw materials, the expansion of iron was studied for its own sake. Similar work is in progress on other types of ores.

81.0 Mineral Beneficiation Pilot Plant

Based on the comprehensive studies made in the National Metallurgical Laboratory on beneficiation of low-grade ores of India, a flexible flowsheet was developed for a pilot plant capable of treating all types of ores by suitable modifications in the flow of materials. The development of flowsheet, drawing up of specifications of equipment, designing of the layout and installation of the pilot plant including water and power supplies were done entirely by the staff of the National Metallurgical Laboratory. The equipment involving foreign exchange was obtained through funds provided by the U.S. Eximbank credit scheme. The plant installed at a capital cost of Rs. 50 lakhs is located in an area of 6 acres of land.

The plant is divided into different sections, namely (a) Crushing; (b) Washing and grinding; (c) Reduction roast and magnetic separation; (d) Heavy media separation; (e) Flotation; (f) Jigging, tabling and Humphreys' spiral treatment; (g) High-intensity and high-tension separations; and (h) Agglomeration.

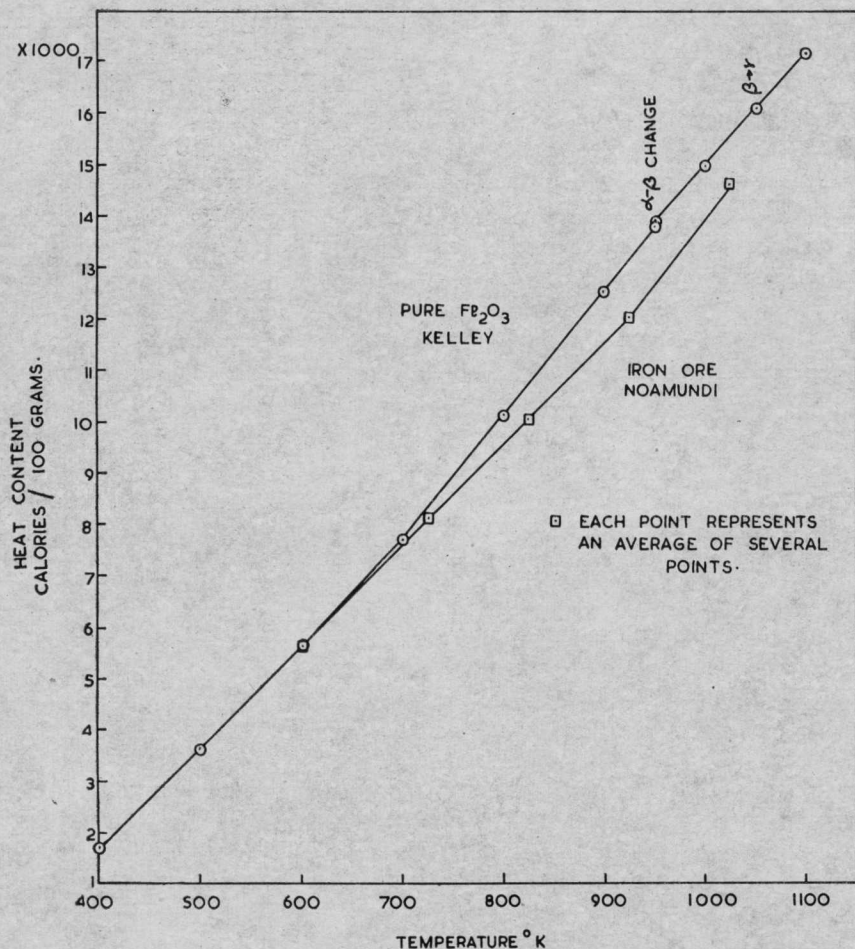


FIG. 49 — THERMAL DATA OF KELLEY ON PURE Fe_2O_3

Section A — Crushing

Ore from the railway siding is transported by two 5-ton tilting trucks, weighed on a weigh bridge and then unloaded into a hopper before feeding to a vibrating grizzly. The oversize is crushed in a jaw crusher, whereas the undersize along with the jaw crushed ore is fed to secondary crushers (gyratory or cone crusher) working in closed circuit with a vibrating screen. Screen undersize is conveyed to a fine ore bin and provision has been made for collecting a representative sample by means of an

automatic sampling system. Secondary crushing may not be necessary when the ore has to be handled in the washing, heavy media or reduction roast sections and arrangements are made to bypass the screen in such cases. This section has a capacity of 8-10 tons of ore per hour

Section B — Washing and Grinding

The crushed ore bin has a constant weight feeder fitted at its discharge opening and a reversible belt conveyor. This conveyor

can feed the ore either to washing section or to grinding mills or direct to an inclined conveyor leading to washed ore bin.

The washing section consists of a scrubber, wet screen and a crossflow classifier. The scrubbed ore is wet screened and the under-size is dewatered and deslimed using the classifier, whereas the oversize is conveyed to the washed ore bin for further treatment in Section C or D. Provision is also made to convey the classifier sand for jigging, if desired.

The reversible belt can feed ore either to a ball mill, rod mill, or a roll crusher through suitably designed chutes. Ball mill works in closed circuit with a rake or cyclone classifier. The overflow is pumped to Section E or flotation, whereas the roll crusher discharge can be sent to Section F for further treatment.

Section C — Reduction Roast and Magnetic Separation

Ore from the washed ore bin, either washed or direct from the crushed ore bin, can be fed through apron feeder to any of the two reduction furnaces, viz. vertical kiln or rotary furnace. Gas from a gas producer and stored in a 15,000 cu. ft. capacity gas holder is used for magnetizing reduction roast of the ore. Reduced ore is crushed in a roll crusher and passed through drum or belt type wet magnetic separators. The non-magnetic fractions after dewatering in a crossflow classifier can be ground finer in a rod mill and passed again through another wet magnetic separator. The magnetic product is sent to Section J for agglomeration.

Section D — Heavy Media Separation

Arrangements are being made to produce a 5-10 tons per hour capacity heavy media separation unit. The unit proposed will be a complete one consisting of separating vessel, drainage screens, pumps, dewatering classifier, medium reclamation equipment, etc.



FIG. 50—PROF. HUMAYUN KABIR, MINISTER FOR SCIENTIFIC RESEARCH & CULTURAL AFFAIRS, ADDRESSING THE DELEGATES AT THE OPENING OF MINERAL BENEFICIATION PILOT PLANT

Section E — Flotation

The classifier overflow obtained in Section B is pumped to hydroclassifier for desliming, if necessary, or to a conditioner where flotation reagents are added through wet reagent feeders. There are 12 big size flotation cells for roughing and scavenging and 12 of smaller size for cleaning operations. The flow of material can be made in any desired manner by suitable placing of launders and pumps. Soft water can also be supplied from a softener, if required.

Flotation concentrate is pumped to a thickener in Section G, and the tailings are pumped to waste dumps outside the building.

Section F — Jigging, Tabling and Humphreys' Spiral Treatment

Roll crusher or rod mill discharge from Section B is the feed for this section. Two Harz type jigs and a duplex mineral jig are installed. Three half-size Wilfley tables

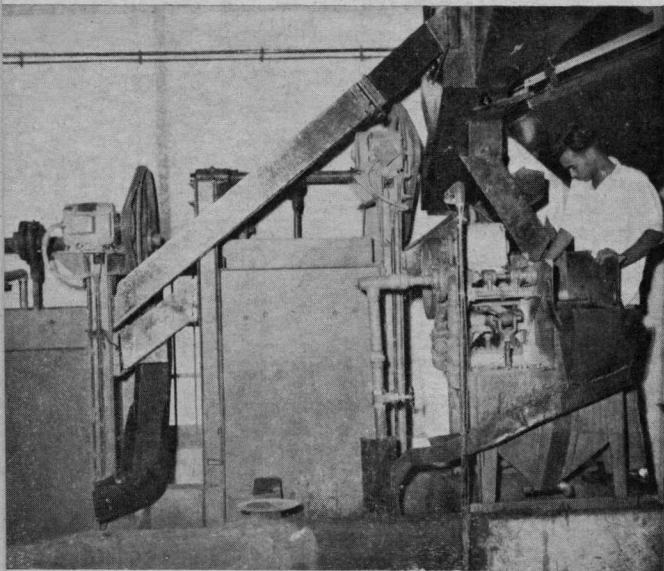


FIG. 51—A VIEW OF TWO HARZ JIGS AND DUPLEX MINERAL JIG IN MINERAL BENEFICIATION PILOT PLANT

and two Humphreys' spirals, pulp distributor, hydrosizer and vibrating screen comprise the remaining equipment in the section.

Section G — Thickening, Filtering and Drying

This section consists of two thickeners, a 4-disc filter, three rotary driers and conveyors. Products from any of the sections mentioned earlier that require filtration are brought to this section and finally stored in bins in Section J.

Section H — Electrostatic and High-intensity Magnetic Separation

A dry grinding ball mill with an air classifier is proposed to be installed to prepare feed for this section. Three high-intensity dry magnetic separators and one double roll electrostatic separator have been installed below the bins fitted with table feeders.

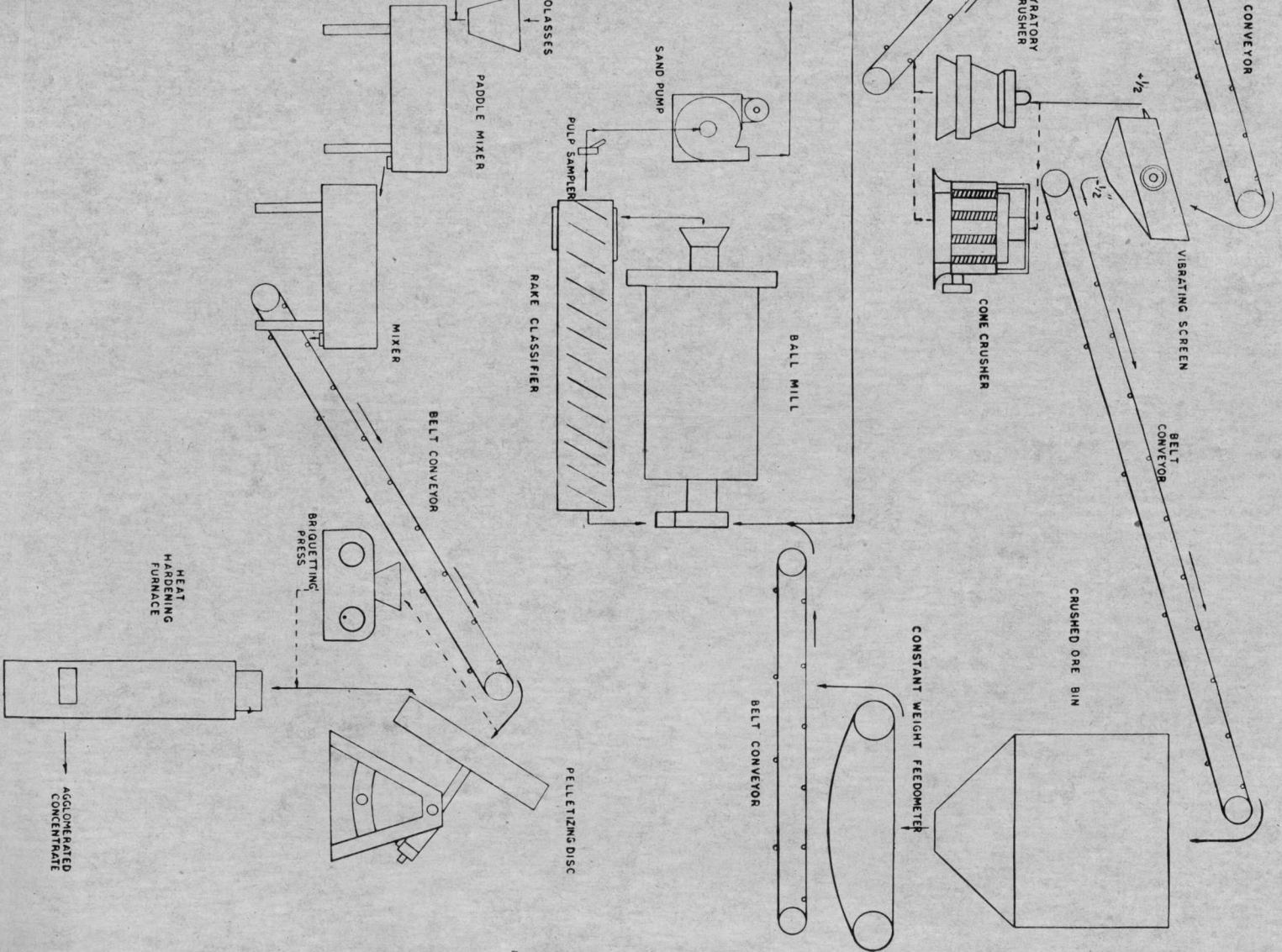
Section J — Agglomeration

Concentrates obtained in fine size can be sintered, pelletized or briquetted. An 18 in. wide and 8 ft. long continuous sintering machine has been ordered and is expected in a few months time. A 6 ft. dia. disc pelletizer and also a drum pelletizer have been installed and the briquetting machine is awaiting installation. For physical and chemical analyses of ore samples, a control laboratory is provided with facilities for petrological and microscopic studies, X-ray fluorescence analysis, thermal analysis, etc. The plant is instrumented for continuous measurement of pH values, pulp density and also has gas and water flow rate indicators, oxygen analyser, CO/CO₂ analyser and recorder, multipoint temperature recorders, etc.

Beneficiation of Limestone

Since November 1962, the plant had been treating low-grade limestone to make it suitable for use in steelmaking. About 800 tons of limestone sample assaying CaO, 46.7; L.O.I., 38.2; and SiO₂, 7.1 per cent was received for upgrading. The concentrate produced was handed over to Tisco after agglomeration for actual trials in open-hearth furnaces.

The sample was crushed to $-\frac{1}{2}$ in. size, using jaw and cone crusher. The crushed sample was ground in a ball mill working in closed circuit with rake classifier. The classifier overflow (80 per cent, -200 mesh) was conditioned with sodium silicate and emulsion of oleic acid and sodium hydroxide. Rougher concentrate collected from eight cells was refloatated in six smaller cells, whereas the tailing was floated in four scavenger cells producing a final tailing and a scavenger float for regrinding. The cleaner tailing was also recirculated in the circuit. The refloat concentrate assaying CaO, 50.3; L.O.I., 41.1; and SiO₂, 2.0 per cent, with a CaO recovery of 94.7 per cent in it was thickened, filtered and dried. The dried



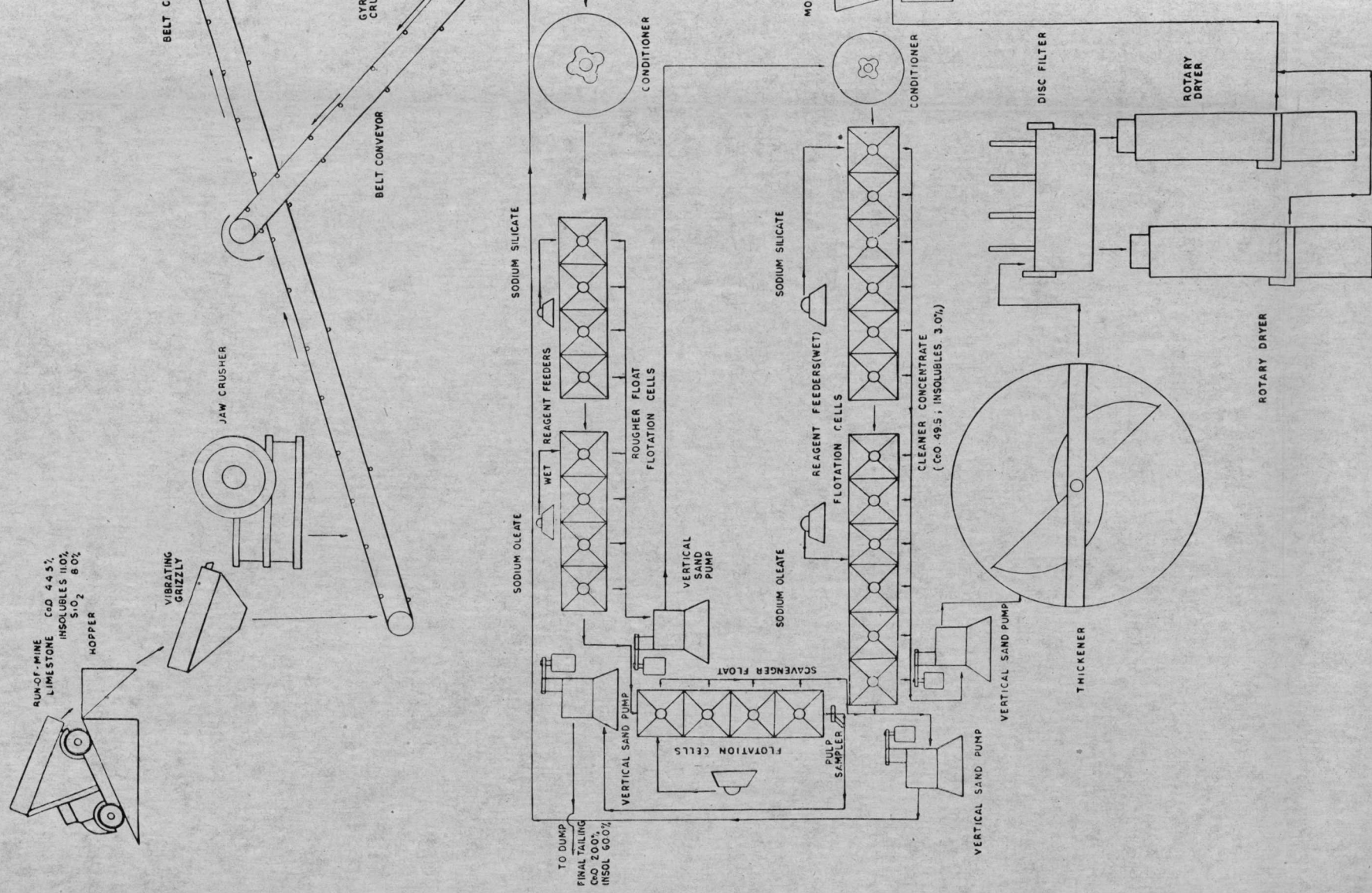


FIG. 52 — LIMESTONE FLO

concentrate was mixed with molasses and made into briquettes as well as pellets and supplied to Tisco. Full-scale melts using limestone concentrate briquettes in Tisco open-hearth furnaces are in progress and the results obtained so far have been quite encouraging, whereas trials using pellets are expected to be started soon.

Flowsheet employed for the pilot plant tests is given in Fig. 52.

82.0 Electrolytic 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 such as development of thermostatic bimetals, development of magnetic materials, nickel-free coinage alloy, manganese brass, etc. The erection and fabrication of all the equipment for the 100 lb. manganese metal plant was completed and the plant was made ready for regular production of metal. A highly ferruginous manganese ore from Orissa containing 30 per cent manganese was used for production of manganese metal. Though the time necessary for iron precipitation and subsequent filtration was found to be relatively high, the recovery of manganese from the ore was satisfactory.

83.0 Electrolytic Manganese Dioxide Pilot Plant

This pilot plant was operated quite successfully during the year. As in the case with manganese metal plant, a highly ferruginous manganese ore containing 30 per cent manganese was used for production of manganese dioxide. The average analysis of electrolytic manganese dioxide obtained on the above anodes gave the following figures:

MnO ₂ , %	...	93.35
Total Mn, %	...	59.02
H ₂ O (at 110°C.), %	...	3.90

Acid insoluble, %	...	0.28
Sulphate, %	...	1.00
Lead, %	...	0.77
Iron, %	...	0.07

The successful operation of this pilot plant confirmed the feasibility of putting the process into large commercial scale with almost the same current efficiency and recovery of manganese as that obtained from laboratory scale experiments. This plant would further study the behaviour of siliceous and other complex ores as well as different anode materials such as graphite. Graphite anodes were found to give a product relatively free from lead.

84.0 Pilot Plant for Hot-dip Aluminizing of Steel

(i) *Field Trials at Visakhapatnam* — During the period under review, a study on the corrosion resistance of aluminized wire in saline atmosphere was completed. Samples, after exposure near the sea shore at Visakhapatnam, were examined. Eighteen months' exposure trials showed the superior behaviour of aluminized wire as compared to the galvanized one. While the zinc, zinc alloy and aluminium layers disappeared, the alloy layer of the aluminium coating resisted corrosion and thus increased the service life of the aluminized wire. The corrosion products of aluminized wire also remained adherent, whereas those of the galvanized wire either dissolved in dew and rain or flaked under the action of wind, thereby exposing new surface for fresh corrosion attack to start. Fig. 53 shows the microstructure of exposed aluminized wire revealing the presence of the protective alloy layer besides the random distribution of metallic aluminium in the outer corrosion product. On the other hand, Fig. 54 shows the microstructure of exposed galvanized wire revealing only rust on the surface of basis metal steel. No metallic zinc could be found in microscopic survey. The electrical

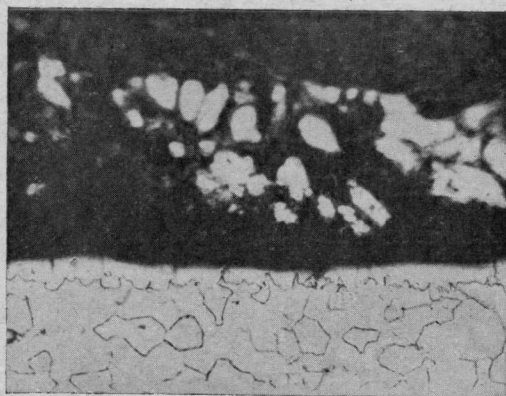


FIG. 53 — MICROSTRUCTURE OF EXPOSED ALUMINIZED M.S. WIRE SHOWING RESIDUAL ALUMINIUM IN THE OUTER LAYER EMBEDDED IN CORROSION PRODUCTS, ALLOY LAYER INTACT AND THE BASIS METAL. ETCHED IN NITAL $\times 450$



FIG. 54 — MICROSTRUCTURE OF EXPOSED GALVANIZED M.S. WIRE SHOWING ONLY CORROSION PRODUCT OVER THE BASIS METAL. ETCHED IN NITAL $\times 450$

resistance measurements made on the two kinds of wire aluminized and galvanized showed a sharp rise in resistance in the case of galvanized wire as compared to only a slight increase in the aluminized wire. Extensive tests on the soundness of aluminized wire such as the nitric acid test showed that there were no pin holes. Welding of aluminized M.S. wire was also investigated and it was found that good weld joints could be obtained.

(ii) *Hot-dip Aluminizing of H.T. Transmission Tower Members* — An investigation

was undertaken to find the feasibility of hot-dip aluminizing of structural members of H.T. transmission line towers, sub-station structures, nuts and bolts and line hardware. It was observed that aluminizing could be done by NML patented processes and that the products thus obtained compared favourably with similar galvanized materials. A comparative macrostructure study (Figs. 55 and 56) showed that though the coating was continuous, an excess of aluminium was deposited in the threads, whereas in the case of galvanized coating, wetting of threads appeared poor and some portions remained uncoated. The aluminium coating showed a passive behaviour towards neutral salts owing to a film of alumina, whereas zinc did not possess this property. Salt spray corrosion test also indicated superior corrosion resistance behaviour of aluminium coating compared to that of galvanized articles. The effect of aluminizing on the mechanical property of high-tensile steel angles was studied and it was observed that a very slight decrease in strength took place. Adhesion, though sound as shown in Fig. 57, was less than in the case of galvanized steel resulting in the peeling off at $36-50^\circ$ bend and also during tensile test earlier than the failure of the basis material. This peeling off may be attributed to the

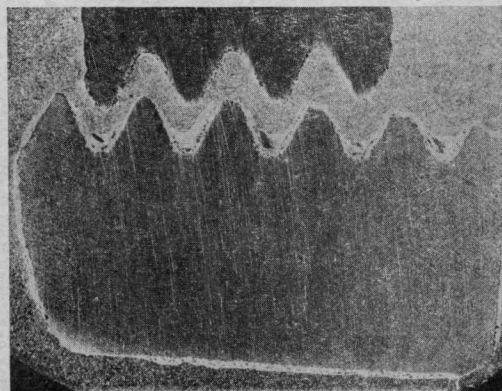


FIG. 55 — MACROSTRUCTURE OF ALUMINIZED THREADS T.S. SHOWING EXCESSIVE DEPOSITS OF ALUMINIUM. $\times 7$ APPROX.

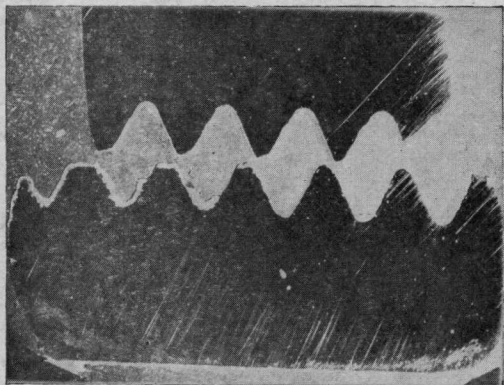


FIG. 56 — MACROSTRUCTURE OF GALVANIZED THREADS T.S. SHOWING LACK OF COATING IN SOME OF THE THREADS. $\times 10$ APPROX.

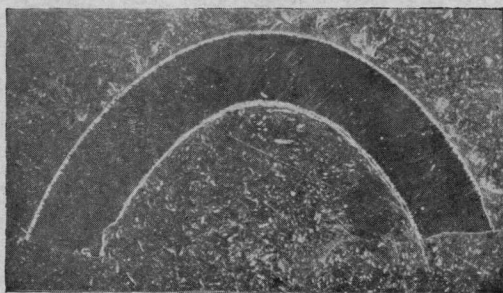


FIG. 57 — BEND TEST SAMPLE OF ALUMINIZED ANGLE SHOWING COATING INTACT ON BENDING THROUGH 180° . $\times 4.5$

composition of the basis material which being high in manganese appeared to diminish the strength of bond between the coating and steel.

(iii) *Training and Demonstration to Licensees* — During the period under review, the working of the process was demonstrated to a number of licensee firms and their representatives were trained in the technique of aluminizing.

(iv) *Field Trials of Mufflers* — Two sets of 3-ton 'Dodge' mufflers were aluminized and sent to Army Base Workshops, Delhi Cantt., for determining the actual service life. The mufflers were fitted and they have so far withstood the test.

Argon arc welding of Fiat 1100 main and auxiliary mufflers was carried out and six each of assembled mufflers were sent to Messrs Premier Automobiles Limited, Bombay, for trials. The results of the tests are awaited.

(v) *Aluminizing of High-tensile Steel Wires* — 4.17 and 1.26 mm. diameter high-tensile steel wires received from Messrs Alind, Kundara, were aluminized and despatched to the party for their perusal and trial. Besides the above, IS SWG 0.6 carbon steel wire received from R. & D.O., Jahapur, for Defence Laboratory (Electronics), Bangalore, was also aluminized.

85.0 Pilot Plant for Refractories

(i) *Pilot Plant Erection* — The following machines were installed in the Refractories Pilot Plant:

1. Sagger-cum-brick Press.
2. Perforated Pan Mill.
3. Jigger Jolley.
4. Binder tanks along with fitting of agitators.
5. Centrifugal pums for pumping binders.
6. Down draught furnace of 7-ton capacity going up to 1000°C . was designed, erected and operational tests were carried out. All the parts including burners, gas and air manifolds were fabricated in NML.
7. Cutting table for pug mill.
8. Dust extraction system designed at the National Metallurgical Laboratory
9. Continuous type ball mill of 2 ft. inside dia., 3 ft. long supplied by Messrs A.C.C. and lined at the National Metallurgical Laboratory was installed and some operational tests were also carried out.

(ii) *Effect of Pressing and the Influence of Bonds on the Compaction of Granular Ceramic Powders* — A report on the work so far done on the subject embodying the results of research investigation on the compaction of granular ceramic powders under pressure

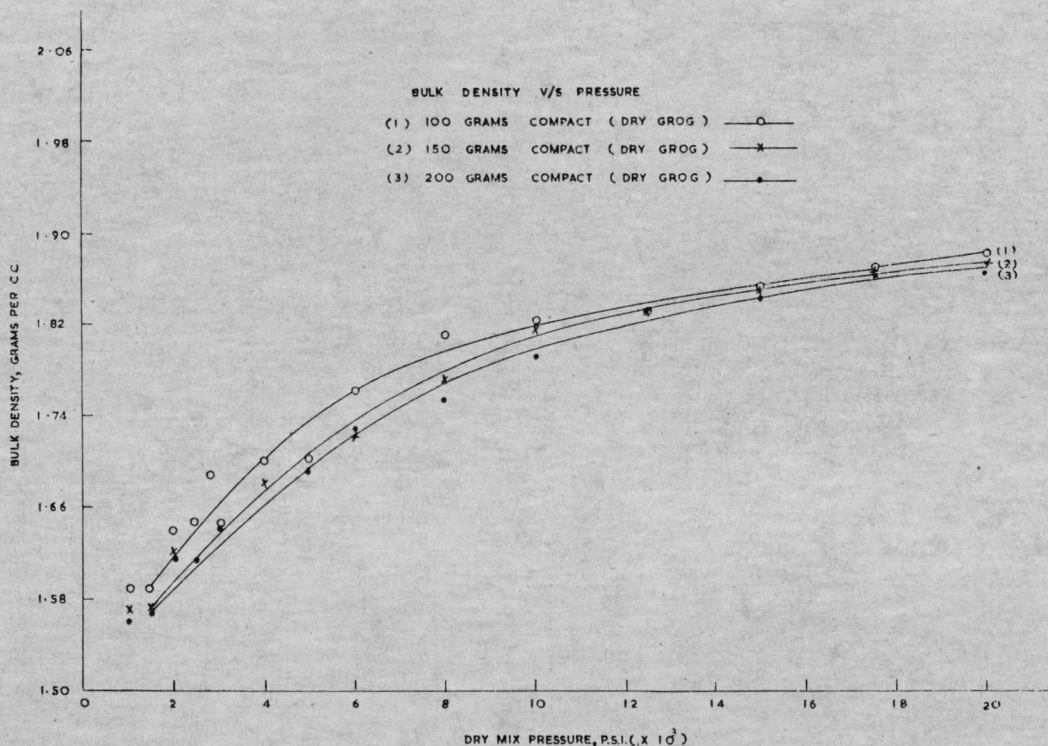


FIG. 58 — COMPACTION OF DRY GROG UNDER HYDRAULIC PRESS.

applied at one end was prepared. Experimental procedure and the properties of the grog were reported earlier. The conclusions drawn were as follows:

- (a) Bulk densities of granular ceramic mixes compacted under pressure applied from one end increased according to the equation $BD = \frac{P}{a+bP} + c$ where BD is bulk density in gm./cc., P = pressure in p.s.i. and a , b and c are constants. This is parabolic relationship as shown in Fig. 58.
- (b) It was possible to arrange the bulk density/pressure relationship in a manner analogous to a first-order chemical reaction, the increase in bulk density being considered analogous to the reaction and P to time and $\log_{10} \Delta BD$ (increment in bulk density) against P (applied pressure in p.s.i.).

Such plots being intersecting straight lines as shown in Fig. 59, it was possible to conclude that compaction in these powders took place by two different mechanisms and the maximum increase in bulk density that can be obtained by the mechanism are shown in Table 94. While it could not be categorically stated as to what the two mechanisms were, it was suggested that one of them was the sliding down of particles under pressure and the other was infiltration of fines subsequently produced into the void spaces.

Bonding agents normally employed at the industry act as lubricants during compaction. Their effect was significant even at low concentration. Fig. 60 shows the effect of addition of bonds sulphite lye, dextrin and water, etc., and the porosity of the powder compacts at 1500 and 20,000 p.s.i. Fig. 61 shows the effect of

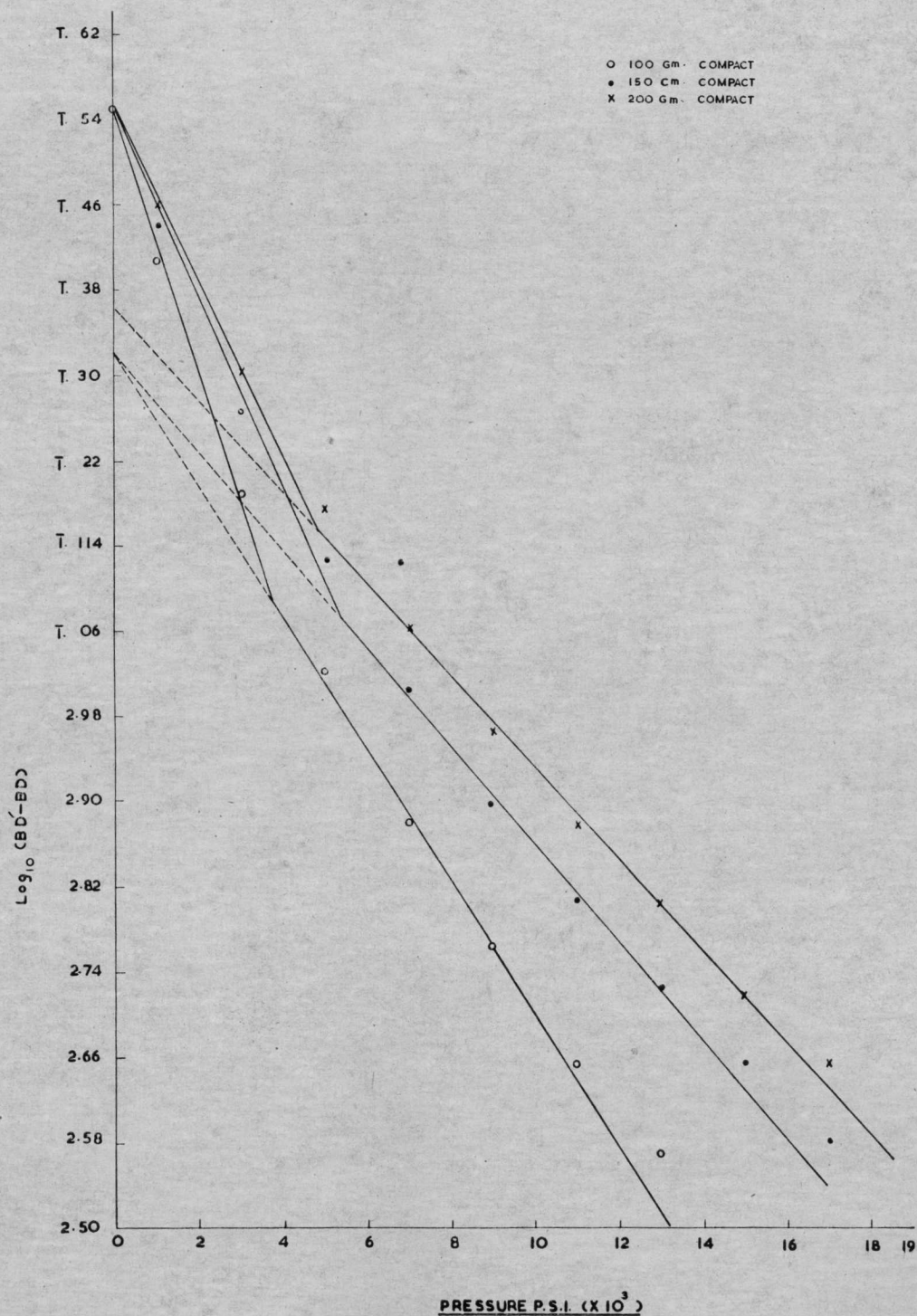


FIG. 59 — INCREMENT IN BULK DENSITY AGAINST APPLIED PRESSURE

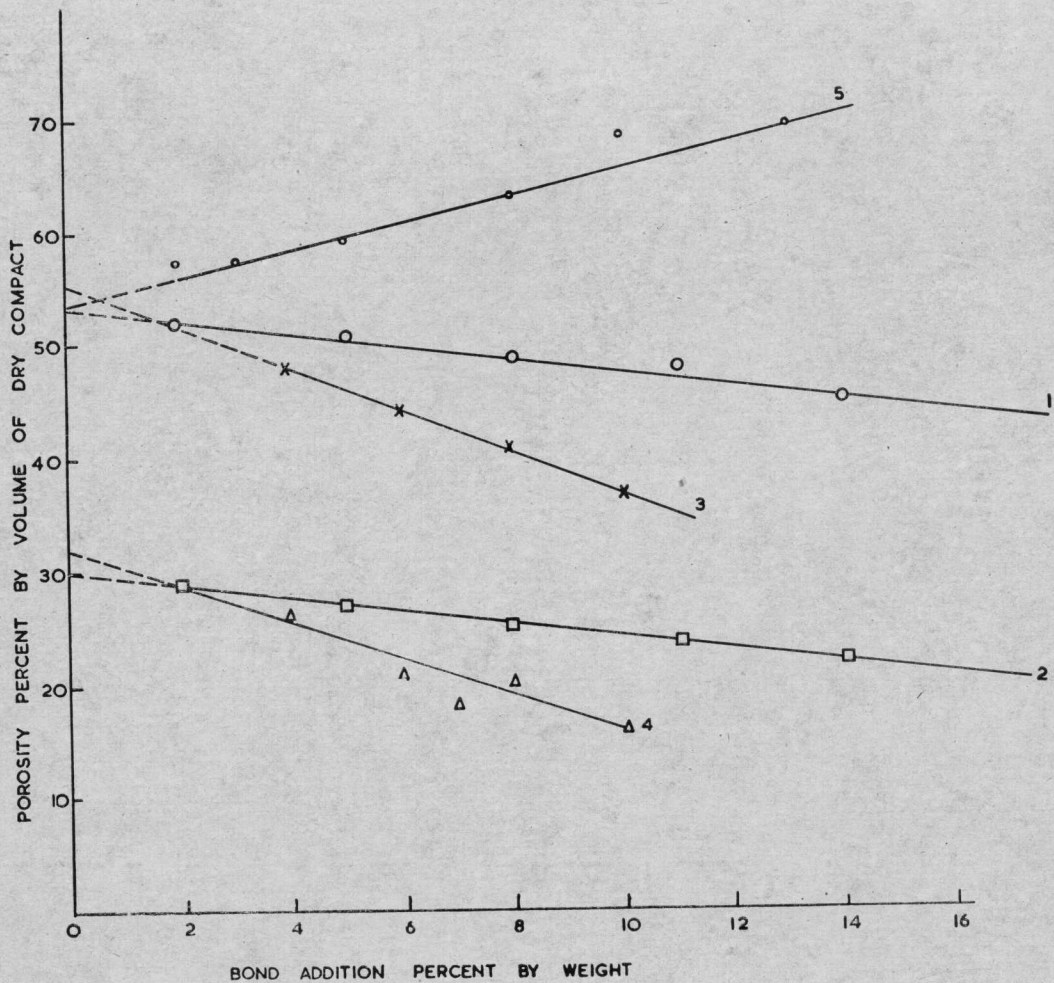


FIG. 60—EFFECT OF ADDITION OF BONDS ON THE POROSITY OF POWDER COMPACTS [1, sulphite lye, 1500 p.s.i.; 2, sulphite lye, 2000 p.s.i.; 3 dextrin, 1500 p.s.i.; 4, dextrin, 20,000 p.s.i.; and 5, water, 15,000 p.s.i.]

Table 94—Characteristics of the Two Compaction Mechanisms

Wt. of compact in gm.	Mechanism A		Mechanism B		Ratio B.D. A/B.D. B	Actual B.D. due to both	B.D. as calculated from asymptote $y = \frac{1}{b} + c$ gm./cc.
	Rate constant $\times 10^{-4}$	B.D. A max. increment in B.D. gm./cc.	Rate constant $\times 10^{-4}$	B.D. B max. increment in B.D. gm./cc.			
100	6.92	0.10	1.41	0.31	0.33	0.41	0.52
150	6.70	0.08	1.07	0.32	0.25	0.40	0.53
200	6.42	0.07	1.00	0.34	0.20	0.41	0.54

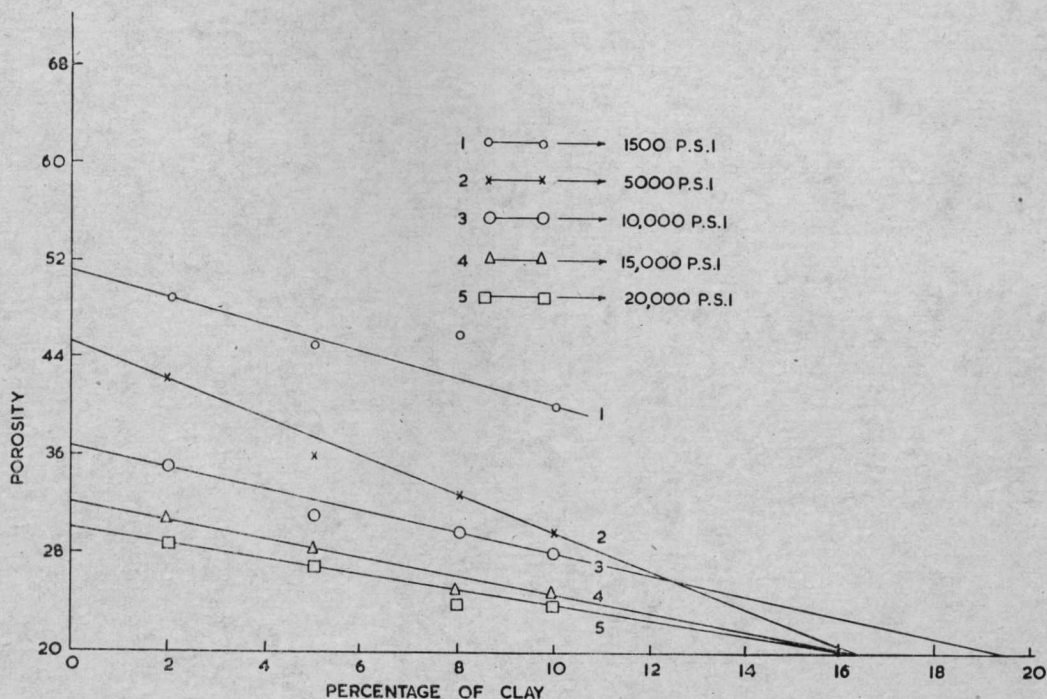


FIG. 61 — EFFECT OF ADDITION OF CLAY IN THE GROG, WATER 8 PER CENT CONSTANT FOR 100 GM. OF DRY MIX

addition of clay in the grog, water 8 per cent constant for 100 gm. of dry mix at 1500 5000, 10,000, 15,000 and 20,000 p.s.i.

The relationship between porosity and clay concentration P is shown in Table 95.

Table 95 — Relationship between Porosity and Clay Concentration at Various Pressures

Compacting pressure p.s.i.	Relationship
1500	$P = -1.125 P + 51.25$
5000	$P = -1.5 P + 45.0$
10000	$P = -0.875 P + 36.75$
15000	$P = -0.7 P + 31.5$
20000	$P = -0.625 P + 30.25$

(iii) *Comparative Study of Some Methods of Compaction of Mixes of Dry Press Consistency* — While one-stage pressing is a widely used method for fabrication of

refractory bricks, blocks and other shapes are usually tamped or jolted. It was, therefore, thought that a comparative study of all these methods would yield interesting information. Experimental mix and its physical properties were same as reported earlier.

Experimental Procedure

Mixing — Weighed quantities of dry grog powder and clay were first introduced in a mixer and later a weighed quantity of water was added. The mix was homogenized.

(a) *Bumping under Maximum Weight* — A weighed quantity of mix was introduced in the sample container of Westman & Hugill apparatus. Bumping was started and readings were taken after frequent intervals up to 60 min.

(b) *Tamping by A.F.M.S. Rammer* — A weighed quantity of mix was introduced

into the die and number of blows were dealt with the rammer. The height of the free space above the compaction was measured with the help of vernier depth gauge.

(c) *Pressing under Hydraulic Press*—Ames hydraulic press with 4 in. dia. ram and 30 tons' capacity was used. A known weight of mix was introduced into the die and compacted under required pressure.

(d) *Tamping under Pneumatic Hammer*—The mould was fixed securely to the anvil. A known quantity of mix was introduced into the die and plunger placed in position. All the tampings were done by adjusting the valve control and locking the disc. A vernier depth gauge was used to measure the free depth of the mould and the height of the compact was obtained by difference.

Bulk densities of the 100, 150, 200, 250 and 300 gm. compacts were obtained by various methods. In all the cases the data followed a parabolic relationship with the compacting force (bumps, blows and pressures respectively), following the equation

$y = \frac{x}{a+bx} + c$, where y is bulk density in gm. per cc., x is pressure and a , b and c are constants. Table 96 shows the value of constants a , b and c obtained by different methods of compaction with the variation of the weight of the mix.

In all these cases, plot of $\log_{10} \Delta BD$ (increment in bulk density) against P (applied pressure) intersecting straight lines were obtained which follow the analogy of first-order chemical reaction.

(e) *Forsterite Refractories*—During the year, experimental work on sampling and crushing studies on serpentine rock; making bricks at various pressures under 500 tons hydraulic press and tamping under pneumatic hammer; four mixes containing various grain sizes of serpentine rock, dead burnt, light calcined and raw magnesite was carried out. The chemical analysis of the serpentine rock used is given in Table 97. The

Table 96 — Values of the Constants and Asymptote in the Equation $y = \frac{x}{a+bx} + c$

Wt. of compacts	Westman & Hugill apparatus				A.F.M.S. rammer				Hydraulic press				Pneumatic hammer			
	a	b	c	Asymp-tote	a	b	c	Asymp-tote	a	b	c	Asymp-tote	a	b	c	Asymp-tote
100	178	3.13	1.36	1.68	27.3	2.43	1.37	1.78	8763	2.434	1.64	2.05	—	—	—	—
150	262	3.03	1.34	1.67	35.7	2.38	1.34	1.76	11460	2.70	1.65	2.02	—	—	—	—
200	317	3.03	1.325	1.66	42.8	2.41	1.34	1.75	12570	2.75	1.65	2.01	10	2.13	1.88	2.35
250	404	3.11	1.33	1.65	39.9	2.46	1.33	1.71	14580	2.71	1.65	2.02	—	—	—	—
300	—	—	—	—	—	—	—	—	14638	3.053	1.645	1.97	—	—	—	—
				× = No. of bumps.				× = No. of blows.				× = lb. per sq. in.				× = No. of blows.
																Asymptote corrected to mix sp. gravity of 2.406 = 2.12.

**Table 97 — Chemical Analysis of Serpentine
Rock Used**

SiO ₂ , %	38.32
Al ₂ O ₃ , %	0.39
Fe ₂ O ₃ , %	9.12
Cr ₂ O ₃ , %	2.26
MgO, %	37.29
CaO, %	Traces
Alkalies	Traces
Loss on ignition, %	12.19

results are under study in order to select the optimum conditions for obtaining end products with most desirable properties.

86.0 Pilot Plant for Production of Magnesium Metal

An oil-fired furnace has been obtained and work on the production of magnesium metal by silico-thermal reduction of dolomite will be taken up after the erection of the plant has been completed. A tablet-making machine and a conical blender for mixing the briquette constituents were received and are under installation.

ELECTRIC FURNACES

THE three cement compositions developed by the Electric Furnace Committee, clay-kyanite, phosphoric acid-kyanite and an aluminous composition with calcium-aluminate formulated earlier, were found highly useful in fabrication of all laboratory furnaces required by the laboratory. None of the composition reacted with kanthal or nickel-chromium resistance wires. The technique of making internally wound heating sheaths developed earlier was systematically utilized for making tubular furnaces required for laboratory work. This practice greatly minimized the demand for new furnace tubes.

A laboratory tubular furnace for operation at 1300-1500°C. was designed and fabricated in connection with work on ferrites. The furnaces had the following characteristics:

- (1) Resistance material — Pt-Rh (10 per cent) wire, 0.5 mm. dia. wound on alumina tube.
- (2) Lagging material — hydraulic aluminous cement prepared in the laboratory.
- (3) Insulation material — sintered alumina powder.

- (4) Design — An auxiliary heating element made of kanthal wire was provided concentrically round the Pt-Rh (10 per cent) tubular element with a view to reducing power load on the noble metal element. Operating data of the two circuits were as follows:

Auxiliary element	Noble metal element
Voltage, 110 V.	200 V.
Current, 3 amps.	4.5 amps.
Temperature °C., 1000	1450 ± 5, over a length of 4.5 in. and controllable with an energy regulator

The furnace gave complete satisfaction during use.

A design for a molybdenum wound furnace for operation at 1800°C. was also completed. The design utilizes a porous recrystallized alumina tube and provides for a continuous flow of hydrogen through the furnace body during operation. There is, however, facility for working under controlled atmosphere in a separate working tube placed concentrically inside the alumina tube. The fabrication of the furnace is underway.

ENGINEERING SECTIONS

Design Engineering

A number of new design projects, in addition to those initiated in the previous year, were undertaken during the year under review. Some of the major design projects executed and underway for different Pilot Plants and for research and development work in general are given below.

(i) *Strip Aluminizing Plant* — The development of suitable designs for a complete plant for continuous hot-dip aluminizing of steel strip was continued. A major portion of the design and drafting work was completed. Among equipment designed and detailed were: (a) the process tanks for fluxing, hot washing and plating complete with pull-over rolls, pinch rolls, scrubber rolls, wiper rolls, etc.; (b) a motorized reel stand for coiling of the aluminized strip; and (c) variable tension looping pit/accumulator for storing of strip required for feeding the plant while the finishing end of one roll and starting end of the other roll are being squared and welded. Considerable follow-up work with regard to equipment being fabricated for this plant in the workshop was also undertaken.

(ii) *Dry Chlorination Furnace* — A furnace for obtaining molten anhydrous magnesium chloride from magnesium oxide was designed and detailed.

(iii) *Magnesium Chloride Dehydrator* — A batch type dehydrating furnace for removing the chemically combined water from magnesium chloride was designed and detailed. The furnace has a capacity of 30 kg. and is electrically heated. The moisture evolved is carried away by a continuous blast of dehydrated air which

is preheated by passing over the heating coils.

(iv) *Rotary Kiln and Cooler* — A 1100 mm. dia. and 7500 mm. long rotary kiln for roasting of vanadium and chrome ores on a pilot plant scale is being designed. Details of a rotary cooler hermetically sealed to the rotary kiln was also worked out. The kiln will be capable of working at temperatures up to 1400°C. and will have an output of 1.5 tonnes per day.

Electronics Engineering

(i) *Phototransistor Temperature Controller* — A phototransistor temperature controller was developed using Mullard OCP 71 phototransistor. This controller can be used with any electrical resistance furnaces and has an accuracy of about $\pm 2^\circ\text{C}$. up to a temperature of 1600°C. The sensing element is a thermocouple. Compensation for variation of ambient temperature is provided for temperatures up to 30°C. Design of compensatory circuits for higher ambient temperatures is under progress. Fig. 62 shows the controller.

(ii) *Electronic Titrimeter* — The prototype using alternating current was fabricated and is under observation in analytical chemistry section. Further work is in progress using stabilized direct current.

(iii) *Wide-band Stabilized Power Supply* — The prototype was designed and fabricated. The stabilized power supply (0-500 volts, 300 ma.) was found to be stable for wide range of input voltage and load variations.

(iv) *Damping Capacity Meter* — Some modifications were made in the design of the circuit and assembly is under progress.

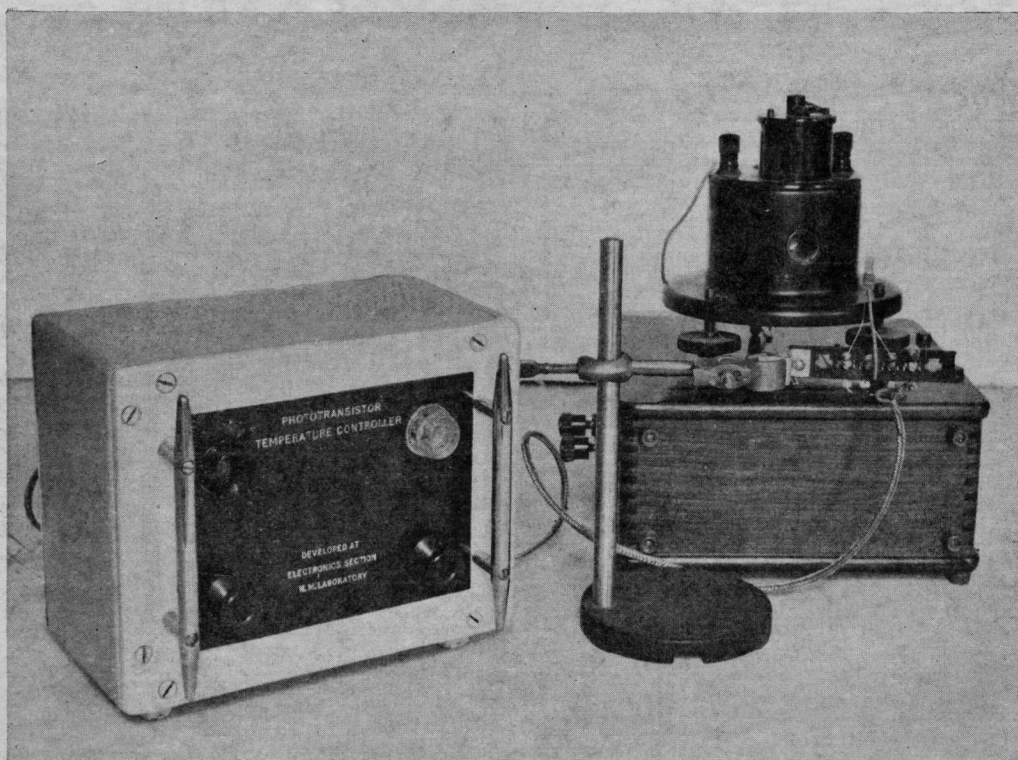


FIG. 62 — PHOTOTRANSISTOR TEMPERATURE CONTROLLER DEVELOPED AT THE NATIONAL METALLURGICAL LABORATORY

The time taken for the amplitude of vibration to fall to half its value during free decay is measured by means of a circuit which transmits two pulses to a standard electronic timer at the appropriate moments.

Several major maintenance, installation and calibration jobs were carried out during the year under review.

Mechanical Engineering

During the year under review, the workshop completed about 1700 different jobs for the various research divisions of the laboratory. These jobs ranged from break-down work to fabrication and installation of research tools and equipments. A few

of the important jobs are detailed below with their salient features.

(i) *Sample-splitter* — A sample-splitter for the sampling of Mn ores was fabricated and the same was provided with a motor drive. A speed-variator was incorporated in the design to adjust the r.p.m. of the output shaft as per requirement.

(ii) *Fabrication of Furnace Stand* — A furnace along with its stand and accessories was designed and fabricated for the determination of the heat content of liquid metals (Al-Cu alloys). The furnace which is placed in a vertical position can be moved up and down through a system of screw and clamps.

(iii) *Magnetostriction Apparatus* — An apparatus for the measurement of changes in dimension of ferro-magnetic materials

placed in a magnetic field was designed and fabricated in the workshop. The changes in dimension of the specimen are transferred to a roller through a system of delicate lever and finally recorded.

(iv) *Vibrating Machine* — A vibrating machine for compacting mortar cubes was fabricated for the determination of the compressive strength of slag cement.

(v) *Fabrication of Centrifuge* — A liquid metal centrifuge was made and installed to study the constitution of the liquid alloys. The essential constructional features are as follows:

A central arm is supported by thrust bearings vertically. This arm can be rotated in vertical position by a horizontal drive-shaft connected by bevel gears. Five different speeds are available due to step-down pulleys driven by a motor. A horizontal beam with two furnaces at the extremities are connected to the central arm. The elements inside the furnaces can be heated up electrically. The cooling arrangement is also provided to cool the furnaces as required.

Electrical Engineering

During the year under review, a number of design, development and investigation work were planned and executed for

various Pilot Plants and laboratory requirements apart from repair and maintenance of electrical equipment of the National Metallurgical Laboratory.

(i) *Installation of Electrical Equipment in Mineral Beneficiation Pilot Plant* — In view of the urgency to commission the Pilot Plant, a temporary substation consisting of 250 kVA. transformer 6600/415 volts, one 6600 volts 30 amperes switch-fuse and one 490/440 volts 200 amperes oil circuit-breaker was installed and commissioned. 415 volts power was taken to flotation shed main distribution board by means of underground cable and therefrom it was distributed to various control panels. Nearly one hundred A.C. motors ranging from fractional h.p. to 30 h.p. along with their control devices were installed, tested and commissioned.

(ii) *500 kVA. Transformer* — One 500 kVA. transformer was received from one firm in damaged condition. It was repaired and tested for its satisfactory performance.

(iii) *Installation of High-frequency Heating Furnace* — One valve generator type high-frequency equipment, manufactured by Wild Barfield Electric Furnace Ltd., was installed, tested and commissioned.

(iv) *High-temperature Furnaces* — Design and fabrication of graphitization furnace and molybdenum furnace are under progress.

INDUSTRIAL LIAISON, OPERATIONAL RESEARCH AND INFORMATION SERVICES

DURING the year under review, close contact was maintained with the industrial organizations, scientific societies, professional associations and institutions so as to ensure coordination in research efforts and fruitful utilization of the research results. Technical information and research results of the processes developed by the National Metallurgical Laboratory were disseminated in the form of technical papers, bulletins, brochures, press-releases, etc. Practical demonstrations were also arranged and industries to whom the processes were released were invited to attend the demonstration so as to impart the technical know-how of the processes for commercial exploitation.

During the period as many as eight practical demonstrations were arranged on the 'Hot-dip Aluminizing of Ferrous Materials' at the NML Pilot Plant for the benefit of licensees of the above process. Practical demonstration on 'Liquid Gold' was also arranged for the benefit of the licensees. A number of industrialists visited the laboratory to discuss their problems and helpful suggestions were furnished to them. A large number of industries showed keen interest in commercial exploitation of some of the NML developed processes and products like production of nickel and cobalt-free heating elements, bimetals, ceramic and permanent type magnets, ferro-alloys, submerged arc welding flux, iron powder for autogenous cutting, etc. Full particulars regarding the techno-economic value of the processes, market demand and other relevant details were furnished.

Patents and Processes Passed on to Industry

As a result of intensive research and pilot plant work done at the National Metallurgical Laboratory, twenty-two new techniques and processes were made ready for commercial exploitation. Out of these twenty-two processes, the following twenty-one have already been passed on to industries and commercial scale production of some of the products are already under way:

- (1) An improved process for electrolytic production of high-purity manganese dioxide.
- (2) An improved method for the production of manganese salts from manganese ores, and its application for the regeneration of the spent electrolytic manganese sulphate baths.
- (3) An improved process for the production of electrolytic manganese metal.
- (4) Hot-dip aluminizing of ferrous materials.
- (5) Improvements in or relating to magnesium silicate refractories and use of the same.
- (6) A process for making completely stabilized dolomite refractories.
- (7) Refractory compositions comprising graphite and silicon carbide (carbon-bonded graphite crucibles).
- (8) Refractory compositions comprising graphite and alumino-silicate materials and glazes to render such compositions resistant to oxidation (clay-bonded graphite crucibles).

- (9) Refractory compositions containing non-refractory chrome ore and refractory products made therefrom.
- (10) Preparation of liquid gold.
- (11) Electroplating of metals on aluminium or its alloys.
- (12) Chemical polishing of aluminium.
- (13) Metallization of non-conductors.
- (14) Brass plating from non-cyanide bath.
- (15) Production of dense carbon aggregate suitable for being used as base material for carbon products in general and Soderberg paste in particular.
- (16) Production of electrical resistance alloys for heating elements.
- (17) Technology of production of thermo-static bimetals.
- (18) Production of Alnico type permanent magnets.
- (19) Production of chemically bonded metal-clad or unclad basic refractory.
- (20) Production of iron powder for auto-genous cutting.
- (21) Production of carbon-free ferro-alloys by alumino-thermic reactions.

Consultancy Work

Consultancy service or assistance has assumed a serious magnitude in the present context of India's development plans and today the NML is being consulted both at the government level as well as by the industries both in public and private sectors on multitudes of problems. Such consultancy services are not necessarily restricted to small industries and medium industries alone and, in fact, this laboratory has been consulted by major industries such as steel plants for finding effective solutions to some of their pressing problems for increasing their productivity. As, for instance, considerable difficulties were experienced by the Hindustan Steel Ltd. in the handling of iron ores in monsoon season due to the blinding of the screens, since the indigenous ores are associated with high

alumina and this high-alumina content has also affected adversely the smelting operations particularly in relation to minimum fuel ratio and maximum productivity in quality. Based on the comprehensive investigations conducted on the iron ore from Noamundi, Joda, Bolani, Barsua and Rajhara mines, it was recommended that scrubbing of the ores followed by wet screening and classification would solve ore handling and screening problems encountered.

State Governments also consult this laboratory on some of their industrial projects such as prospects of establishing an iron and steel plant in South India utilizing Salem magnetite and Neyveli lignite (low-temperature carbonized coke) as also the prospects of establishing a low-shaft furnace plant in Punjab, Maharashtra, Rajasthan and also in Andhra Pradesh utilizing regional raw materials. Such consultancy work obviously involved comprehensive smelting trials on the regional raw materials and a fair assessment of the reserves of raw materials, data relating to the transport of raw materials and finished products, optimum plant size, reasonable evaluation of the cost of production and capital cost of the entire project, utilization of byproducts, etc.

Another important problem of consultancy work undertaken by the NML is the utilization of limestone in steelmaking which at present due to the high-silica content presents considerable operational difficulties. This problem has been successfully solved through beneficiation techniques in the mineral beneficiation pilot plant and the silica content was brought to below 2 per cent. Actual in-plant trials were also made with the limestone briquettes after beneficiation, in the 235-ton open-hearth steel-melting furnace at the Tisco. 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 per day with equipment specification, cost

of beneficiation and briquetting of the limestone. Similar work was also carried out on four different consignments of limestone from Purnapani mines on behalf of Hindustan Steel Ltd.

Another consultancy work referred to this laboratory would refer to the recovery of diamond from diamondiferous tuff and gravel samples sent by the National Mineral Development Corporation, Government of India, in connection with the development of historic Panna Diamond Mines. Suitable ore-dressing techniques for the recovery of diamond were formulated along with the detailed flowsheets for setting up of treatment plants for efficient recovery of diamonds. Similar consultancy work was also undertaken on behalf of the Government of Rajasthan to set up a beneficiation plant for fluorspar in the Dungarpur district of Rajasthan as also on the Khetri copper ore on behalf of the National Mineral Development Corporation. Requests for consultancy work have also been received from the Governments of Nepal and Jammu & Kashmir State on problems relating to the utilization of indigenous ores for metallurgical purposes.

Besides, a number of industries have approached this laboratory to give impartial assessment of the causes of major failures and breakdown in their plants and in such cases, specialized staff members are deputed to carry out on the spot study and follow it up with the appropriate investigation to arrive at the causes of failure, etc.

Project Reports

Preparation of detailed project reports for different metallurgical industries was initiated earlier at the National Metallurgical Laboratory. The Project Reports prepared can be broadly classified into the following three groups:

- (1) Investigation Project Reports on the basis of the investigation carried out on behalf of the sponsoring body.

- (2) Project Reports on the processes developed by the laboratory for commercial exploitation.
- (3) Project Reports based on the available information for setting up of a new industry.

Project Reports under category (1) provide adequate technical data on the projects under investigation to assess the application of a particular process for the raw materials available and also provide a sound basis for evaluating the scope of a particular technique before large-scale investments are made on the project. The following Investigation Project Reports were prepared during the year:

- (i) Report on the operation of low-shaft furnace pilot plant at the NML for the production of foundry grades of pig iron with Andhra Pradesh raw materials.
- (ii) Report on the operation of low-shaft furnace pilot plant at the NML for the production of pig iron with East Punjab raw materials from Mahindergarh.
- (iii) Report on the operation of low-shaft furnace pilot plant at the NML for the production of pig iron with raw materials from Maharashtra State.
- (iv) Report on the operation of low-shaft furnace pilot plant at the NML for the production of pig iron with iron ore and limestone from Nepal.
- (v) The operation of low-shaft furnace pilot plant at the NML for the production of pig iron with raw materials from Rajasthan.

Project Reports falling under the second category provide information on the technology of the processes evolved, properties of the products developed, range of application and market demand, availability of requisite raw materials, plant and machinery and technical personnel required, approximate idea of the capital for a reasonable size of plant, etc. These reports are designed to give an idea about the salient features of the process or products developed

to a prospective entrepreneur. The following is the list of such preliminary Project Reports prepared so far on the projects developed at NML:

- (1) Production of iron powder for auto-genous cutting.
- (2) Electrical resistance alloys for heating elements.
- (3) Technology of production of thermo-static bimetals.
- (4) Flux for submerged arc welding.
- (5) Manufacture of Alnico type permanent magnets.
- (6) Production of ceramic magnets.
- (7) Production of carbon-free ferro-alloys by alumino-thermic reactions.
- (8) Production of vanadium pentoxide and ferro-vanadium from vanadium bearing titaniferous magnetite.

Project Reports under the third category contain a general appraisal of all the features mentioned above and serve as a guide to a prospective entrepreneur for starting a new industry.

Extensive literature was surveyed in preparing a report entitled 'Project for Utilization of Zinc Wastes'. This gave short descriptions of dross, ash, flux skimmings and blowings and consisted of two parts. The first part dealt with the treatment of the wastes in plant provided with a galvanizing bath and the second part suggested methods for those plants without a galvanizing bath. The different processes are described and details are given on the following lines:

- (i) Cylinder method for the recovery of metallic zinc from ash.
- (ii) Recovery of zinc from residues containing chlorides.
- (iii) Electrolytic process from zincate bath.
- (iv) Dross distillation furnace.
- (v) Recovery of zinc from galvanizers' dross by modified aluminium process.
- (vi) Recovery of zinc from spent fluxes, skimmings and other wastes containing chlorides.
- (vii) Treatment of zinc blowings.

Technical Aid to Industries and Government Organizations

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 ad hoc short and long-term investigations besides specifications, tests, etc. The technical advice given to the industries covered a wide range of problems such as those relating to beneficiation of raw materials, composition and properties of metals and alloys for various applications to suit specific service conditions, techniques of heat treatment, technical know-how of industrial processes and production techniques to be adopted for small and medium-scale industries, remedial measures to overcome specific difficulties during manufacture, finishing stage and in storage conditions, utilization of waste products, recovery of metallics from drosses, etc., and a host of technical problems posed during manufacture. In addition, several hundreds of enquiries were attended on the application of the products developed by the NML for various end uses as also on the detailed technical data on the availability of raw materials for setting up of new industries.

A rough analysis has also been made on the size and the nature of industries seeking the advice of this laboratory, from which it was found that the industries, irrespective of their size and structure, do refer their technical problems to this laboratory and in fact technical enquiries have been received even from small units scattered at the remotest part of the country. A sample analysis was also made of the various types of technical enquiries referred to by the industries to assess the trend in the nature of enquiries received from year to year as well as to get an idea of the requirements of the industry from the information point of view. The results of the survey are presented in Table 98. From the

Table 98 — Analysis of Various Types of Enquiries Referred

Nature of enquiries attended	1960-61 April-March %	1961-62 April-March %	1962-63 April-March %
1. Availability of minerals, metals and alloys, equipment and machinery	22.8	12	15.8
2. Technique of production of metals and alloys in different composition and forms	18.0	17.6	8.5
3. General information and data on metallurgical subjects	10.8	5.1	9.6
4. Selection of metals and alloys for different metallurgical and engineering purposes and their specifications	10.0	14.4	16.2
5. Protective coatings (corrosion resistant), electroplating and metal finishing	10.0	6.8	10.0
6. Technique of founding, casting, defects in castings and remedies thereof	7.4	16.1	4.5
7. Refractory materials, compositions, their mechanical properties and uses	4.5	0.8	1.1
8. Technique of heat treatment, annealing and core hardening, etc., of metals and alloys	3.0	1.7	6.2
9. Mechanical working of metals and alloys, etc.	2.7	1.7	1.7
10. Technique of melting of metals and alloys	2.2	5.1	10.1
11. Utilization of byproducts of metals and mineral industries	2.0	2.5	0.6
12. Use of minerals, metals and alloys	1.8	1.7	3.4
13. Extraction and refining of metals from ores	1.8	1.7	3.4
14. Statistical data	1.7	2.5	0.6
15. Substitute metals and alloys, their composition and process of manufacture, etc.	0.9	3.4	4.5
16. Recovery of metals and alloys	0.4	5.1	4.5

table it can be seen that in the year 1960-61, 23. per cent of the enquiries received from the industries related to the availability of minerals, metals and alloys, equipment and machinery, while 18 per cent of the enquiries were on the technical production of metals and alloys, and 10 per cent each on general information and data on metallurgical subjects, selection of metals and alloys for different end uses and protective coating, electroplating and metal finishing, etc. The percentage of technical advice rendered in the various fields in 1960-61, 1961-62, 1962-63 show an interesting comparison especially in some of the fields which show an increasing trend. This sample survey although may not reveal quantitatively the real demand of the industries, but can provide a pointer to show in which of the field the information machinery is required to be strengthened and can also provide a micro-basis for subject evaluation.

During the period under review, total number of technical enquiries attended were 183, out of which 24 were from government organizations and 159 from private firms and individuals. Details of some enquiries are furnished in Appendix III.

Collaborative Work

The National Metallurgical Laboratory is collaborating with Commonwealth Committee on Mineral Processing in the field of mineral beneficiation work conducted in India as well as with the International Working Group on Bonding Clays and has undertaken investigations on samples of bentonite available in different countries of the world. Besides the above, National Metallurgical Laboratory is actively collaborating with Indian Standards Institution in formulating standards, Ordnance establishments towards development and production of

special alloys and metal products, Department of Atomic Energy, Geological Survey of India, Indian Bureau of Mines and other laboratories under C.S.I.R. in respect of utilization of indigenous raw materials, development of products and processes, etc. The research work done at the National Metallurgical Laboratory has been recognized by the following universities for the award of postgraduate degrees:

Agra University, for Ph.D. degree

Banaras Hindu University, for Ph.D. degree

Bihar University, for Ph.D. degree

Jadavpur University, for Ph.D. degree

Panjab University, for Ph.D. degree in Chemistry

Andhra University, for D.Sc. degree

Bombay University, for all research degrees

Madras University, for M.Sc. degree

Industrial Surveys and Collection of Statistical, Economic and Market Data

Latest statistics relating to production, consumption, demand and cost of various metals and metal products, ores and minerals, etc., in India and different countries of the world were collected and maintained on card index. All the leading newspapers and commercial dailies were scanned everyday for news of importance pertaining to metallurgical and mineral industries; research and development work in the field of metallurgy and allied subjects; national and international policies on production, export, etc., of metals, metal products, alloys, ores and minerals, etc., and a clipping service is being maintained.

Over 3000 patent specifications and technical articles were summarized from Indian and foreign publications and made available to research workers during the

year under review. The patent specifications were systematically classified and patent index maintained as lined catalogues which made it possible to readily trace relevant patents on diverse subjects and explore their fields of application.

Extension Work

Action has been initiated on the establishment of foundry regional stations one each in Howrah, Madras, Bombay and Batala, so that problems on a regional basis can be tackled more efficiently and the units in disseminating the technical know-how developed by the National Metallurgical Laboratory. The foundry regional station at Batala in Punjab will be ready in near future and the construction work of the foundry regional station at Madras has been undertaken.

The establishment of Marine Corrosion Research Station of the National Metallurgical Laboratory on the sea coast at Digha in West Bengal is nearing completion. This 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.

Training Classes, Demonstrations, etc.

Practical training facilities have been provided to government stipendary trainees and others sponsored by universities, technological institutions, industrial firms, etc., in various branches of metallurgical engineering. Besides, the laboratory imparts specialized training at graduate and artisan levels in various metallurgical fields. Twenty-eight graduate and fifty-two trade apprentices are at present undergoing training in the laboratory.

PATENTS

Patents filed during the period:

- (1) *Indian Patent No. 82191* — An improved jacketted electrolytic cell for the electro-deposition of metals and metallic oxides in general and manganese dioxide in particular (10-5-1962).
- (2) *Indian Patent No. 83652* — Improvements in or relating to magnesite refractories (10-8-1962).
- (3) *Indian Patent No. 83968* — A method for reconditioning the coated magnesium powders (3-9-1962).
- (4) *Indian Patent No. 84670* — Improvements in or relating to electrolytic cells (10-10-1962).

Patents accepted during the period:

- (1) *Indian Patent No. 76997* — Improvements in or relating to the production of copper powder by electrolytic process (28-11-1962).
- (2) *Indian Patent No. 76415* — Improvements in or relating to the modification of aluminium alloys containing silicon (18-7-1962).

Patents sealed during the period:

- (1) *Indian Patent No. 76997* — Improvements in or relating to the production of copper powder by electrolytic process (18-5-1963).
- (2) *Indian Patent No. 76415* — Improvements in or relating to the modification of aluminium alloys containing silicon (22-2-1963).

PUBLICATIONS

NML Technical Journal

The *NML Technical Journal*, a quarterly publication, has stepped into fifth 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 both in the standard of technical contents as well as in the presentation. The abstracts of the papers published in the *NML Technical Journal* are abstracted by leading abstracting services like *Chemical Abstracts*, *Journal of Iron & Steel Institute*, U.K., *American Society of Metals*, *Battelle Memorial Review*, etc. Over hundred and fifty Indian and foreign technical journals, publications, etc., are obtained on free exchange basis.

Proceedings of the Symposium

During the year under review, publication covering the proceedings of the Symposium on 'Ferro-alloy Industry in India', which was held under the auspices of the National

Metallurgical Laboratory in February 1962, was brought out within a record time. The publication contained twenty-nine authoritative technical papers along with full account of the discussions that took place.

Papers Published

During the period thirty-five scientific papers were published as per Appendix I.

Brochures, Booklets, Reports, etc.

A brochure on 'Integrated Mineral Beneficiation Pilot Plant' installed at the National Metallurgical Laboratory was brought out on the inauguration of the plant by Lady Goodeve. The scope of mineral beneficiation and its importance along with the description of the mineral beneficiation pilot plant equipment and techniques of beneficiation have been outlined in the brochure.

During the year under review, forty-one investigation and research reports were prepared. The details are furnished in Appendix II.

LIBRARY, DOCUMENTATION AND TRANSLATION SERVICE

Library and Documentation Service

The activities of the Library were further enlarged during the year both in the service facilities to the scientific staff and in the classification and indexing of the scientific articles published. During the year, detailed classification of research and investigation reports on the basis of UDC was completed. The abstract cards of the Iron & Steel Institute of the U.K. were reclassified and regrouped in accordance with the needs of the users of this Library, with suitable subheadings devised for this purpose.

Twenty-six issues of the Index of Technical Articles ((IOTA) were published, each containing more than 150 entries on different subjects of interest published in current journals all over the world. The entries were arranged under 200 subheadings which reflected the field of research and study of the laboratory. The abstract cards, on the basis of which the IOTA is printed, are filed according to subheading and provide an invaluable tool to the research staff in the preparation of bibliography and also as sources of information on diverse subjects. The IOTA was regularly circulated to the different divisions of the laboratory.

In the matter of acquisition, considerable stress was laid in completing the collection

of patent specifications relating to metallurgy. Patent journals from Germany, Japan, Belgium, France, etc., were subscribed in addition to those obtained from the U.K. and the U.S.A. These journals have added to the comprehensiveness of the collection of patents and have enhanced the value of the laboratory as the Regional Patent Inspection Centre.

The Library now subscribes to almost all reputable abstract journals in the field of its speciality. A large number of journals and books were received in exchange to the *NML Technical Journal*.

Translation Service

Articles in French, German and Russian of direct interest to the research work were translated into English for the reference of all concerned. Oral translations were also rendered to give the research worker a quick and approximate idea of the scope and results contained in technical publications and to enable him to exactly locate the portion of the text of direct interest to him. Besides, many other routine translation work were also attended to. About 250 technical papers published in French, German and Russian were translated during the year under review, each paper being of an average of 4000 words.

SYMPOSIUM

TO focus attention on the latest technological trends and research development in the production of iron and Steel, an International Symposium on 'Recent Developments in Iron and Steel Making with Special Reference to Indian Conditions' was organized by the National Metallurgical Laboratory and Indian Insti-

tute of Metals in collaboration with the Iron & Steel Institute (London). The Symposium was held at the National Metallurgical Laboratory from 4th to 8th February 1963. This joint meeting in India was unparalleled in many ways, not only in the useful exchange of ideas but also in the opportunity it provided for the



FIG. 63 — HON'BLE MR. C. SUBRAMANIAM, UNION MINISTER FOR STEEL AND HEAVY INDUSTRIES, GOVERNMENT OF INDIA AND CHAIRMAN, EXECUTIVE COUNCIL OF THE NATIONAL METALLURGICAL LABORATORY, DELIVERING THE INAUGURAL ADDRESS ON THE INAUGURATION DAY OF THE SYMPOSIUM ON 'RECENT DEVELOPMENTS IN IRON AND STEEL MAKING WITH SPECIAL REFERENCE TO INDIAN CONDITIONS'



FIG. 64 — DR. B. R. NIJHAWAN, DIRECTOR, NATIONAL METALLURGICAL LABORATORY, PROPOSING VOTE OF THANKS TO HON'BLE MR. C. SUBRAMANIAM, UNION MINISTER FOR STEEL AND HEAVY INDUSTRIES, GOVERNMENT OF INDIA, AND OTHER EMINENT AND DISTINGUISHED DELEGATES TO THE INTERNATIONAL SYMPOSIUM ON 'RECENT DEVELOPMENTS IN IRON AND STEEL MAKING WITH SPECIAL REFERENCE TO INDIAN CONDITIONS'

growth of professional bonds within the Commonwealth and beyond.

It is now generally acknowledged that steel is basic to heavy industrialization in any country. The problems that confront India in expanding its steel production are complex. In spite of vast iron ore reserves, estimated at 20,000 million tons, the rapid expansion of the steel industry has revealed some of the ore's basic deficiencies, such as high-alumina content and the high proportion of fines produced in mining and sizing. The resources of metallurgical coal are not limited, but also remain concentrated in

the eastern part of India and generally have high-ash contents which require expensive washing. At the same time, flux-grade limestones are high in insolubles which adds to the problems of steelmaking. These problems will be further aggravated as the annual capacity of the Indian steel industry, which is now being expanded from 6 million to 10 million tons by 1963, will further rise to 18-19 million tons by 1970.

Unless these difficulties are overcome by the application of the latest research results and technological developments, the

rationale of India's accelerated steel development will lose some of its force. It is generally recognized that India's future export prospects will depend upon her becoming the 'Workshop of the East', which again presupposes that assured supplies of steel will be available, and that India can maintain the position it once had as the cheapest steel producer in the world.

Viewed in this context, the Symposium provided a valuable forum for exchange of technical know-how on raw material problems, processing techniques, and size and layout of steel works, with special emphasis on problems of expansion in rapidly developing countries.

More than three hundred delegates from all over the world including representatives of the Iron & Steel Board, the British Iron & Steel Federation and the Iron & Steel Institute, known as the 'Holy Trinity' of steel organization in the United Kingdom, attended the Symposium. The delegation was headed by Mr. M. A. Fiennes, Chairman and Managing Director of Davy Ashmore Ltd. and President of the Iron & Steel Institute. It included many delegates who were not strangers to the Indian steel industry, such as Sir Charles Goodeve, Director of the British Iron & Steel Research Association; Mr. R. Mather, Chairman of Skinningrove Iron Co. Ltd.;

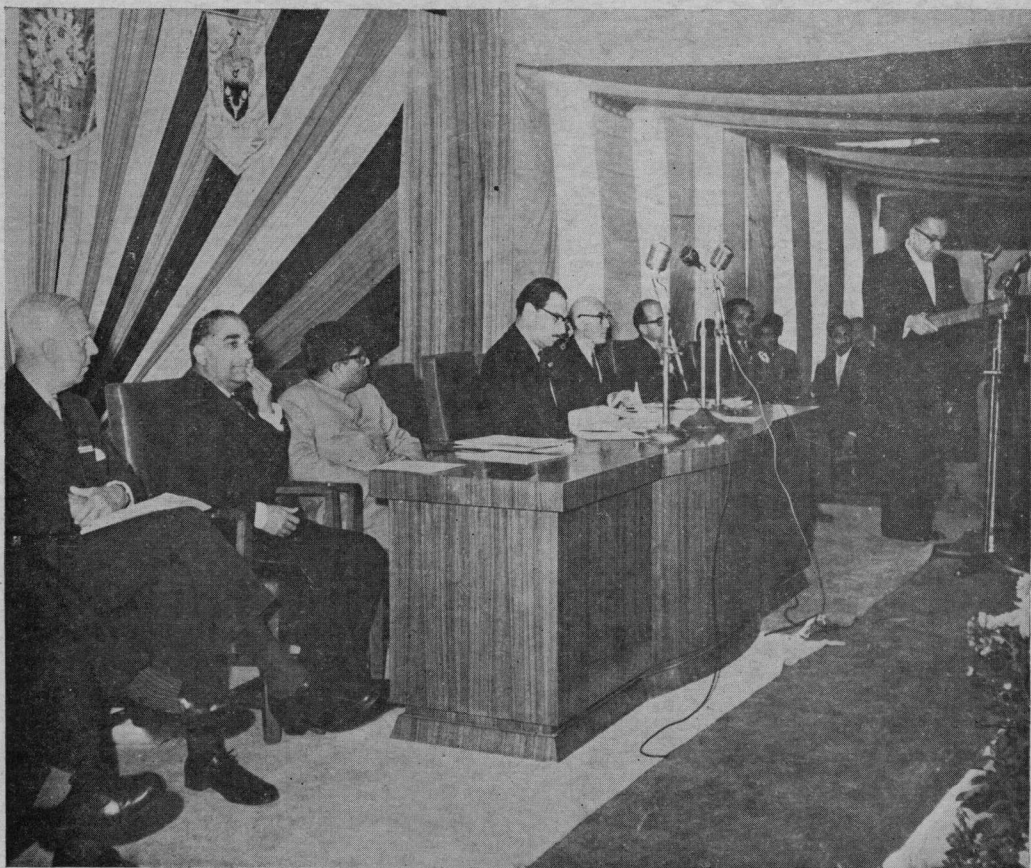


FIG. 65 — SIR JEHANGIR GHANDY, Kt.C.I.E., DIRECTOR-IN-CHARGE, TATA IRON & STEEL CO. LTD., ADDRESSING THE DISTINGUISHED DELEGATES AT THE INAUGURAL SESSION OF THE SYMPOSIUM



FIG. 66 — DR. B. R. NIJHAWAN, DIRECTOR, NATIONAL METALLURGICAL LABORATORY, BEING CONFERRED THE HONORARY VICE-PRESIDENTSHIP OF THE IRON & STEEL INSTITUTE (U.K.) BY MR. MAURICE FIENNES, PRESIDENT OF THE INSTITUTE

Mr. G. B. McMeans, Vice-President, Kaiser Steel Corp., U.S.A.; Prof. Crussard, Director of I.R.S.I.D., France; and Mr. G. Brandes, Managing Director, Ore Sintering Dept., Lurgi; other prominent delegates included Lord Clydesmuir, Director of Colvilles Ltd.; Sir Julian Pöde, Chairman of the Steel Company of Wales Ltd. and President of the British Iron & Steel Federation; Sir Henry Clay of McLellan & Partners; and Mr. K. Headlam Morley, Secretary of the Iron & Steel Institute.

The Symposium was inaugurated by Hon'ble Mr. C. Subramaniam, Union Minister for Steel and Heavy Industries. Welcoming the distinguished delegates from India and abroad, Sir Jehangir Ghandy, Director-in-Charge, Tata Iron & Steel Co. Ltd., said that steel played a vital role in modern civilization and constituted "the keystone of the economic arch of a nation". "It is, therefore, but appropriate", Sir Jehangir said, "that the development of the steel industry had been given the foremost place in India's industrial planning, forming, as it did, the hard core of the country's Second and Third Five Year Plans." In this connection, he complimented Mr. C. Subra-

maniam on his success in increasing the tempo of production in the public sector steel plants. The subject of the Symposium was in keeping with the spirit and needs of the times, even more so during the emergency created by the Chinese aggression on India's borders when the country had to strengthen both the military and economic fronts. In this connection Sir Jehangir, in complimenting Dr. B. R. Nijhawan, paid a warm tribute to him for the valuable research and development work done at the National Metallurgical Laboratory under his guidance; he also complimented Dr. D. P. Antia on his contributions to the growth of the Indian Institute of Metals.

Mr. C. Subramaniam, Union Minister for Steel and Heavy Industries, Government of India and Chairman, Executive Council, National Metallurgical Laboratory, in his Inaugural Address emphasized that the steel industry must pay more attention to research and training in future, which alone could ensure progress. The Minister called for a 'bold and imaginative plan' of steel development, and observed that there was a very close and obvious correlation between the amounts of steel consumed in an economy and the levels of economic development. In India, steel development had become one of the important pillars of economic planning. Commenting on the problems of low productivity in the Indian steel industry, the Minister said that coke rate per ton of iron produced was much higher than the level reached in other countries. He wanted the experts to devise ways to reduce coke consumption and overcome difficulties experienced due to high percentages of silica and alumina in iron ores. He was happy to note that NML had done highly useful work in developing techniques for the improvement of the raw materials for the steel plants. The Union Minister suggested that the Indian Institute of Metals might set up separate groups for studying various metals.

Mr. Maurice Fiennes in his Presidential Address paid a warm tribute to India's achievements in raising steel production in an incredibly short space of time. He stressed that India's task in research and development must be devoted towards development of its natural resources in an economic manner. He was, however, very much pleased to see the National Metallurgical Laboratory which had set a very fine example of the determination of India to produce her own technology by the use of her own technologists. Continuing, he said that if the forecasts about India's future steel expansion programme were to be implemented, one had to face the vast problems posed by such dynamic expansion. Leaving aside the question of finance, the most immediate problem would be the need for technological and experienced manpower. It was for this reason, he urged, that India would do well to develop its present steel plants to their maximum economic potential. This, he considered, need not, however, interfere with the construction of small or moderate size plants spread over a wider geographical area, provided these were not of a scale which would make such demand on the scarce skilled manpower resources. Regarding finance for rapid steel expansion, he urged greater emphasis on 'direct equity investment in good traditional terms'.

Dr. B. R. Nijhawan, Director, NML, proposed a vote of thanks to the participating delegates. He also thanked Dr. D. P. Antia, President, and Mr. R. D. Lalkaka, Secretary of the Indian Institute of Metals; Mr. S. K. Nanavati, Chairman, Symposium Reception Committee, and members of other Committees; Dr. T. Banerjee, Dy. Director, NML; Mr. R. M. Krishnan, Assistant Director, NML; and all others who had worked for the success of the Symposium.

Awards

The Iron & Steel Institute (U.K.) conferred the Honorary Vice-Presidentship

of the Institute on Dr. B. R. Nijhawan Director, National Metallurgical Laboratory, and Honorary Membership on Sir Jehangir Ghandy, Director-in-Charge, Tata Iron & Steel Co. Ltd.

Technical Sessions

During the five-day International Symposium, fifteen technical papers covering various aspects of research, production and planning on iron and steel were presented and discussed in three technical sessions relating to: (1) Preparation and Use of Indian Raw Materials; (2) Planning and Design of New Plants; and (3) Choice of Steelmaking Processes.

NML Mineral Beneficiation Pilot Plant

Synchronizing with the Symposium, the pilot plant for mineral beneficiation, set up by the National Metallurgical Laboratory at a total capital outlay of Rs. 5 million, was declared open by Lady Goodeve with Prof. Humayun Kabir, Vice-President, Council of Scientific & Industrial Research, presiding at the function. The pilot plant can treat up to 5 tons of ore per hour depending upon the specific treatment and optimum upgrading cycle and is one of the best equipped integrated plants in the world for undertaking beneficiation and upgrading of low-grade ores and minerals and for trials pertaining to agglomeration of concentrates. The equipment was procured with funds provided by the U.S. Export-Import Bank. The entire engineering design, layout, erection and installation of this pilot plant was done by the staff of the National Metallurgical Laboratory. The pilot plant can continuously treat any type of ore by classical ore-dressing methods including reduction roast treatments and agglomeration of concentrate fines. This



FIG. 67 — A VIEW OF THE DISTINGUISHED DELEGATES ON THE INAUGURAL DAY OF THE SYMPOSIUM

facility will not only enable speedy translation of processes developed on laboratory scale to industrial scale operation, but will also provide valuable economic and design data.

Dr. B. R. Nijhawan, in welcoming the delegates, invited Indian mineral and metallurgical industries to make full use of the comprehensive facilities provided by this integrated mineral beneficiation pilot plant. He thanked Mr. P. I. A. Narayanan, Officer-in-Charge (Ore-Dressing), and his colleagues for their hard and devoted work in commissioning the plant in a record time.

At the opening of the integrated mineral beneficiation pilot plant, Sir Charles Goodeve, Director, B.I.S.R.A., remarked that he had the opportunity of studying quite a number of pilot plants in his life but he had not

yet seen any that can stand above the pilot plants in operation at the NML. Sir Charles further stated that the British Iron & Steel Research Association who were planning to extend their mineral beneficiation pilot plant facilities had not even gone half the pace in comparison with the National Metallurgical Laboratory during the last five years. Sir Charles referred to the leadership and excellent ideas of Dr. Nijhawan for implementing the research results and the pilot plant trials. He further commented that he was seriously thinking of sending some of their own problems which could be worked out at the National Metallurgical Laboratory where the facilities in many respects exceed greatly their own in Britain.

Prof. Humayun Kabir, Vice-President, C.S.I.R., observed that the National

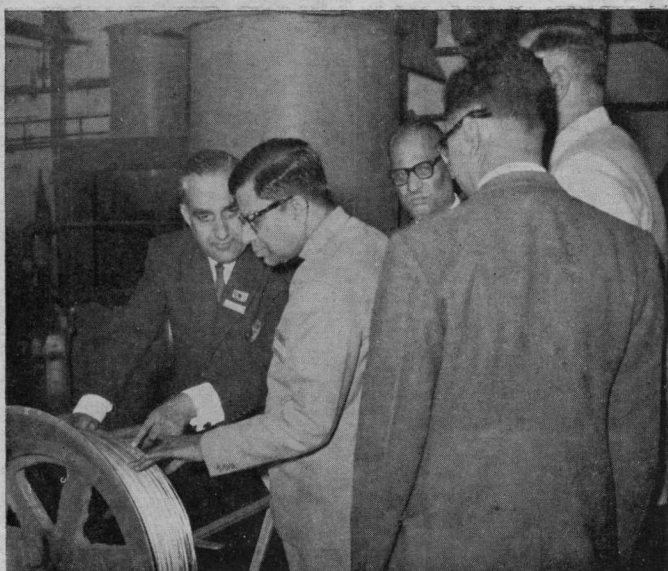


FIG. 68 — DR. B. R. NIJHAWAN, DIRECTOR, NATIONAL METALLURGICAL LABORATORY, SHOWING TO HON'BLE MR. C. SUBRAMANIAM, UNION MINISTER FOR STEEL AND HEAVY INDUSTRIES, GOVERNMENT OF INDIA, SOME OF THE PRODUCTS DEVELOPED AT THE NATIONAL METALLURGICAL LABORATORY

Metallurgical Laboratory during a short span of just over a decade of its existence had made very significant contributions to the national life of the country. Prof. Kabir expressed his personal appreciation and the appreciation of the Government of India to Dr. B. R. Nijhawan for the excellent work done at the National Metallurgical Laboratory under his able leadership.

Mr. P. I. A. Narayanan, Officer-in-Charge, Ore-Dressing Division, proposed a vote of thanks to Hon'ble Minister Prof. Humayun Kabir, Lady and Sir Charles Goodeve and other distinguished delegates and to Dr. B. R. Nijhawan, Director, National Metallurgical Laboratory, for his untiring efforts and valuable guidance in setting up the plant.

Concluding Session

At the conclusion of the technical session, Dr. Nijhawan gave a resume of the technical papers presented and discussed whilst

referring to the outlines of some of the major topics on the subject.

Sir Jehangir Ghandy expressed the hope that the visit of the distinguished members of the Iron & Steel Institute would, to some extent, serve to dispel any doubts that might exist in their minds regarding the potentialities of this country and that it would enable them to take a positive part in the exciting and still unfolding drama of Indian development.

Mr. Maurice Fiennes said that the Symposium had been a grand success and that it established 'a channel of communication for the future', adding that the quality of the papers submitted was of the highest order. He said the Symposium could be described as an 'International Agglomeration' and felt happy to find the tremendous progress in iron and steel technology in India.

Sir Charles Goodeve said that the Symposium had been of immense value to the visitors and that the meeting went on with clock-like precision. Other distinguished



FIG. 69 — DR. B. R. NIJHAWAN, DIRECTOR, NATIONAL METALLURGICAL LABORATORY, SHOWING TO PROF. HUMAYUN KABIR, MINISTER FOR SCIENTIFIC RESEARCH & CULTURAL AFFAIRS, GOVERNMENT OF INDIA, AND MR. MAURICE FIENNES, PRESIDENT, IRON & STEEL INSTITUTE, U.K., THE PRIMITIVE METHOD OF IRON MAKING WHICH WAS DEMONSTRATED AT THE NATIONAL METALLURGICAL LABORATORY

visitors — Prof. Crussard, Mr. George McMeans and Mr. Gregory — expressed satisfaction at the ideas exchanged and contacts established.

The Symposium Supplement

On the inaugural day of the Symposium, a special supplement was brought out by *Economic Times*. The supplement contained leading papers from eminent scientists

and technologists in the field of iron and steel industry. The day-to-day coverage of the Symposium, including its inaugural session and opening of the Mineral Beneficiation Pilot Plant, was fully covered by all the leading dailies of the country like *Hindustan Standard*, *Amrita Bazar Patrika*, *Hindu*, *Statesman*, *Hindustan Times*, *Times of India*, *Economic Times*, *Financial Express*, *Searchlight*, etc., and also many vernacular papers.

GENERAL

THE extension work to the main building comprising the eastern, western and central wings was completed. The side wings will provide an extra length of 160 ft. to the main building which is now 480 ft. long, and an additional plinth area of 43,000 sq. ft. Installation work of all the service facilities in the side wings of the main laboratory has also been completed. Equipment relating to vacuum melting units, magnesium reduction furnace, installation of high-frequency melting furnaces and electronic instrumentation section are being installed as also the X-ray fluorescence equipment, namely Philips Autrometer which is believed to be the first of its kind in this country. Equipment relating to the Extraction Metallurgy will be housed in the new side wing and the research work on ferrous and non-ferrous extractive metals including pressure leaching and hydro-metallurgy will be stepped

up. The Corrosion Research Laboratory at the National Metallurgical Laboratory has now been housed in a separate complete wing and the newly constituted Iron and Steel and Alloy Steel Divisions will be housed in the side wing. Installation of their specialized equipment and their fabrication in NML Workshops have also been stepped up.

The raising of the main foundry bay roof with the main base of the technological block is expected to be completed during this year which will put the foundry melting and related research facilities at par with other service sections. Work relating to the integrated Mineral Beneficiation Pilot Plant such as gas holder, gas producer, gas-linkage lines connecting Low-shaft Furnace Pilot Plant and the Mineral Beneficiation Pilot Plant is under progress and these facilities will enable to undertake the investigations on pre-reduction of iron and steel in long rotary kiln of Mineral

FIG. 70 — A VIEW OF THE MAIN BUILDING OF THE NATIONAL METALLURGICAL LABORATORY AFTER THE CONSTRUCTION OF NEW WINGS





FIG. 71 — A VIEW OF THE EXTENDED WESTERN WING OF THE MAIN BUILDING OF THE NATIONAL METALLURGICAL LABORATORY

Beneficiation Pilot Plant employing low-shaft furnace clean gas and subsequent smelting of pre-reduced metals in a small shaft furnace, design and fabrication of which has been completed. The shaft furnace will be in close vicinity to the direct reduction rotary kiln of the Mineral Beneficiation Pilot Plant. Likewise, research work covering agglomeration techniques such as pelletizing, briquetting and sintering are being expanded to cover both the fundamental and applied fields. The continuous sintering unit, being obtained from Messrs Simon Carves Ltd., is expected to be installed shortly. This will complete the facilities of the Pilot Plant which would be one of the best installed anywhere.

The Central Engineering Design Section of the National Metallurgical Laboratory has now been housed on the top floor with adequate facilities provided for making pilot plant scale models and preparation of detailed working drawings for different project reports under preparation at the National Metallurgical Laboratory. It is hoped that equipment for the iron and steel pilot plant studies to be obtained under the United Nations Agency will now make due headway.

The Marine Corrosion Research Station at Digha is expected to be in full occupation towards the end of this year and the research staff will also be transferred to Digha. Very close linkage will be maintained at Digha between other corrosion



FIG. 72 — A VIEW OF THE EXTENDED EASTERN WING OF THE MAIN BUILDING OF THE NATIONAL METALLURGICAL LABORATORY

research stations such as their departments located at Bombay and elsewhere to undertake the coordinated researches of Navy and Defence authorities. Construction of the residential houses for the staff at the Marine Corrosion Research Station will also be taken in hand. The foundry regional station at Batala will also be in full operation towards the end of this year and construction work is in progress for the foundry regional station to be established in Madras.

Purchase and Stores sections of the National Metallurgical Laboratory kept unceasing activity for procurement of equipment, raw materials and supplies. It is gratifying to report that the procurement of

basic equipment for the Mineral Beneficiation Pilot Plant was effectively handled and completed according to schedule. The Administrative work of the laboratory was efficiently maintained and the heavy budgetary accounts of the laboratory were also handled with utmost efficiency and speediness. Details of expenditure are furnished in the following page.

The construction of D-II type multi-storeyed blocks for the NML staff consisting of 10 flats and E type multistoreyed blocks consisting of 30 units were completed and all the flats have been occupied. The construction work relating to two more C-II type quarters and 64 F, G and H type quarters are nearing completion.

ACTUAL EXPENDITURE OF THE NATIONAL METALLURGICAL LABORATORY FOR THE YEAR 1962-63

*(Figures in
lakh of Rupees)*

RECURRING

P-1	Pay of Officers	...	...	...	...	6.464
P-2	Pay of Establishment	...	...	...	...	8.315
P-3	Allowances	...	...	...	...	2.422
P-4	Contingencies	...	...	...	...	3.993
P-6	Maintenance	...	...	...	...	0.341
P-7	Chemicals, Apparatus and Equipment for Research	...	...	...	...	10.242
F.S.S.—	Fellowships and Scholarships	...	...	...	...	0.407
TOTAL						32.184

CAPITAL

P-5	(1) Works	...	...	...	...	6.857
	(2) Services	...	...	...	...	1.383
	(3) Apparatus and Equipment	...	...	...	...	5.623
	(4) Miscellaneous	...	...	...	...	0.916
TOTAL						14.779

PILOT PLANT

PP-1	(i)	Equipment and Installation	...	...	...	6.872
	(ii)	Building and Services	...	...	...	4.744
PP-2		Provision of Staff	...	...	...	3.297
PP-3		Purchase of Raw Materials	...	...	...	0.619
PP-4		Miscellaneous Contingencies	...	...	...	2.354
TOTAL						17.886

LOW-SHAFT FURNACE PROJECT

PP-1	(i)	Equipment and Installation	...	...	...	1.588
	(ii)	Building and Services	...	...	...	0.030
PP-2		Provision of Staff	...	...	...	2.712
PP-3		Purchase of Raw Materials	...	...	...	2.747
PP-4		Contingencies	...	...	...	4.423
TOTAL						11.500

Construction of Staff Quarters	...	...	...	...	7.269
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GRAND TOTAL 83.618

APPENDIX I

SCIENTIFIC PAPERS PUBLISHED

1. The Role of National Metallurgical Laboratory in the Research and Development of Ferro-alloy Industry in India — B. R. Nijhawan, *Proceedings of the Symposium on Ferro-alloy Industry in India*, (42-51), 1962 and *NML Technical Journal*, IV (1), (41-50), 1962.
2. Production of Low-carbon Ferro-chrome — V. S. Sampath and P. P. Bhatnagar, *Proceedings of the Symposium on Ferro-alloy Industry in India*, (77-82), 1962.
3. Production of Carbon-free Ferro-alloys by Alumino-thermic Reactions — P. P. Bhatnagar and B. R. Nijhawan, *Proceedings of the Symposium on Ferro-alloy Industry in India*, (83-88), 1962.
4. Smelting of Ferro-alloys in Low-shaft Furnace — J. Goswami, A. B. Chatterjea and B. R. Nijhawan, *Proceedings of the Symposium on Ferro-alloy Industry in India*, (95-101), 1962.
5. Studies on the Electrolytic Preparation of Iron-chromium Alloys from Chromite Ore — S. C. Aush, P. B. Chakravarty and T. Banerjee, *Proceedings of the Symposium on Ferro-alloy Industry in India*, (108-117), 1962.
6. Efficient Utilization of Ferro-alloys in the Production of Stainless Steels — R. K. Dubey and P. K. Gupte, *Proceedings of the Symposium on Ferro-alloy Industry in India*, (118-126), 1962.
7. Scope of Development of Ferro-alloy Industry in India — B. R. Nijhawan and P. P. Bhatnagar, *Proceedings of the Symposium on Ferro-alloy Industry in India*, (127-135), 1962 and *Eastern Metals Review*, Independence Number, 1962.
8. Physico-chemical Aspects of Ferro-alloy Production — M. Subramanian, R. N. Misra and P. P. Bhatnagar, *Proceedings of the Symposium on Ferro-alloy Industry in India*, (174-186), 1962.
9. Symposium on Ferro-alloy Industry in India — A Review — P. K. Gupta and M. R. Kulkarni, *NML Technical Journal*, Vol. IV (2), (4-11), 1962.
10. Structure of Electrolytic Silver-copper Alloys — P. L. Ahuja and T. Banerjee, *NML Technical Journal*, Vol. IV (2), (15-20), 1962.
11. Mineralogy and Liquidus Determination of Some Low-shaft Furnace Slags by Hot-stage Microscopy — M. R. K. Rao, *NML Technical Journal*, Vol. IV (2), (21-25), 1962.

12. Thermal Beneficiation of Low-grade Ferruginous Manganese Ores — C. Sankaran, M. C. Sen and P. P. Bhatnagar, *NML Technical Journal*, Vol. IV (2), (26-33), 1962.
13. Some Raw Materials' Problems for Iron-making in India — B. R. Nijhawan, *NML Technical Journal*, Vol. IV (2), (34-38), 1962.
14. Failure of a Cold Rolling Mill Roll — A Case Study — B. N. Das, P. Basak and B. R. Nijhawan, *NML Technical Journal*, Vol. IV (3), (4-7), 1962.
15. Corrosion of Mild Steel in Saturated Brine Solution — K. P. Mukherjee, A. K. Lahiri and T. Banerjee, *NML Technical Journal*, Vol. IV (3), (8-12), 1962.
16. Interface Energy Studies of Some Copper-nickel Alloys Containing a Liquid Phase — B. Halder and Ved Prakash, *NML Technical Journal*, Vol. IV (3), (13-17), 1962.
17. Constitution and Variation of Lattice Parameters of Some Indian Chromites with Magnesia Addition and Subsequent Firing — M. R. K. Rao, N. V. Naidu and H. V. Bhaskar Rao, *NML Technical Journal*, Vol. IV (4), (4-11), 1962.
18. Thermal and Electrical Conductivity of Copper-manganese Alloys — M. K. Gupta and T. Banerjee, *NML Technical Journal*, Vol. IV (4), (12-16), 1962.
19. Galvanic Corrosion of Ferrous and Non-ferrous Metals in the Industrial Atmosphere of Jamshedpur — S. Rao, Addanki, K. P. Mukherjee and A. K. Lahiri, *NML Technical Journal*, Vol. IV (4), (17-22), 1962.
20. Development of Corrosion Resistant Alloys — B. R. Nijhawan, *Proceedings of the Symposium on Corrosion of Metals*, 1962 and *Commerce*, Annual Number, 1962.
21. Dissolution of Steel and Absorption of Hydrogen by Steel during Acid Pickling in Presence of Inhibitor — A. K. Lahiri, N. G. Banerjee and T. Banerjee, *Proceedings of the Symposium on Corrosion of Metals*, 1962.
22. Corrosion of Metals and Alloys Exposed at Jamshedpur — S. Rao, Addanki, K. P. Mukherjee, A. K. Lahiri and T. Banerjee, *Proceedings of the Symposium on Corrosion of Metals*, 1962.
23. Refractory Properties of Indian Sea Water Magnesia — P. C. Sen, M. R. K. Rao and H. V. Bhaskar Rao, *Transactions of Indian Ceramic Society*, XXXI (3), (70-74), 1962.
24. Studies on Clays from Bansī, Mirzapur Dist. — A. Dutt and H. V. Bhaskar Rao, *Transactions of Indian Ceramic Society*, XXXI (3), (86-90), 1962.
25. Investigations into Oxygen Steel-making Process Employing Indian Raw Materials and Refractories — B. L. Sen, B. R. Nijhawan and A. B. Chatterjee, *Transactions of Indian Institute of Metals*, Vol. XV (3), (55-75), 1962.
26. Recent Development in Iron and Steel Technology — C. Sharma and B. R. Nijhawan, *Current Engineering Practice*, Special Number, May 1962.
27. Role of National Metallurgical Laboratory in Developing Iron and Steel Technology in India — B. R. Nijhawan, *Iron & Steel Review*, Rourkela Number, 1962.

28. National Metallurgical Laboratory's Research and Development Activities—B. R. Nijhawan, *Power and Production*, March 1962.
29. Scope of Metallurgical Research in India — B. R. Nijhawan, *Eastern and Indian Engineer*, 104 Annual Number, 1963.
30. Development of Mineral Wealth of Rajasthan — B. R. Nijhawan, *Rajasthan Merchants' Conference Souvenir*, 13th March 1963.
31. Some Observations on Corrosion Resistance of Ancient Delhi Iron Pillar and Present Time Adivasi Iron Made by Primitive Methods — A. K. Lahiri, T. Banerjee and B. R. Nijhawan, *NML Technical Journal*, Vol. V (1), (46-54), 1963.
32. Some Problems of Iron and Steel Making in the Hindustan Steel Plants — B. R. Nijhawan and P. H. Kutar, Preprints of papers for presentation at the *International Symposium on Recent Developments in Iron and Steel Making with Special Reference to Indian Conditions*, (2/1-2/18), 1963.
33. Iron Production in Low-shaft Furnace Plants with Indian Raw Materials — A. B. Chatterjea and B. R. Nijhawan, Preprints of papers for presentation at the *International Symposium on Recent Developments in Iron and Steel Making with Special Reference to Indian Conditions*, (1/42-1/64), 1963.
34. Beneficiation, Sintering and Processing of Raw Materials for Iron and Steel Industry — P. I. A. Narayanan, G. V. Subramanya and G. P. Mathur, Preprints of papers for presentation at the *International Symposium on Recent Developments in Iron and Steel Making with Special Reference to Indian Conditions*, (1/29-1/41), 1963.
35. A Study of Clay from Kumbalam, Kerala State — K. N. Kartha and H. V. Bhaskar Rao, *Transactions of Indian Ceramic Society*, XXXI (4), (95-101), 1963.

APPENDIX II

SCIENTIFIC INVESTIGATIONS COMPLETED AND REPORTS PREPARED

1. Reducibility of Bolani Iron Ores and Sinters — M. Subramanian, C. Sankaran and P. P. Bhatnagar (I.R.237/62).
2. Pilot Plant Production of Concentrate from Low-grade Salem Magnetite — B. L. Sengupta, S. B. Dasgupta, G. P. Mathur and P. I. A. Narayanan (I.R.238/62).
3. Investigation on Blast Furnace Stove Refractories — S. C. Jain, T. V. Prasad and H. V. Bhaskar Rao (I.R.239/62).
4. Moulding Characteristics of Batala Black and White Sands — T. A. Beck, Jatinder Mohan and P. K. Gupte (I.R.240/62).
5. Beneficiation Studies on Lateritoid Manganese Ore from Malaya — R. Ganesh, G. V. Subramanya and P. I. A. Narayanan (I.R.241/62).
6. Recovery of Diamond from Diamondiferous Tuff and Gravel Sample from Panna Area, Madhya Pradesh, India (I.R.242/62).
7. A Study of Clay from Kumbalam (Kerala State) — K. N. Kartha and H. V. Bhaskar Rao (I.R.243/62).
8. Pilot Plant Studies on Beneficiation and Sintering of Kiriburu Iron Ore from National Mineral Development Corporation — S. B. Dasgupta, P. K. Sinha, S. K. Banerjee, G. P. Mathur, G. V. Subramanya and P. I. A. Narayanan (I.R.244/62).
9. Investigation on the Spanish Bentonite (Gardogel) under the International Working Group on Bonding Clays — A. T. Chirayath, Jatinder Mohan and P. K. Gupte (I.R.245/62).
10. Investigation on Bentonite Sample from Poland (Ground Silica Earth) under the International Working Group on Bonding Clays — A. T. Chirayath, Jatinder Mohan and P. K. Gupte (I.R.246/62).
11. Investigation on the English Bentonite (No. 168) under the International Working Group on Bonding Clays — A. T. Chirayath, Jatinder Mohan and P. K. Gupte (I.R.247/62).
12. Investigation on the Bentonite Sample from Germany under the International Working Group on Bonding Clays — A. T. Chirayath, Jatinder Mohan and P. K. Gupte (I.R.248/62).
13. Investigation on the Cutch Bentonite — A. T. Chirayath, Jatinder Mohan and P. K. Gupte (I.R.249/62).

14. Investigation on the Bahua Bentonite, Bhavnagar (Gujarat) — A. T. Chirayath, Jatinder Mohan and P. K. Gupte (I.R.250/62).
15. Investigation on the Bakudi Bentonite, Santhal Paraganas (Bihar) — A. T. Chirayath, Jatinder Mohan and P. K. Gupte (I.R.251/62).
16. Investigation on the Bentonite Samples Received from Indian Standards Institution — A. T. Chirayath, S. S. Bhatnagar and P. K. Gupte (I.R.252/62).
17. Beneficiation of Limestone Sludge from Titagurh Paper Mills Ltd.— R. Ganesh, G. V. Subramanian and P. I. A. Narayanan (I.R.253/62).
18. Batch and Pilot Plant Flotation Studies on Khetri Copper Ore — S. K. Banerjee, M. V. Ranganathan, S. K. Dhar, R. Ganesh, G. V. Subramanian and P. I. A. Narayanan (I.R.254/62).
19. Batch and Pilot Plant Beneficiation Studies on Low-grade Fluorspar from Rajasthan for Production of Acid and Metallurgical Grade Spar — P. V. Raman, B. L. Sengupta, R. Ganesh, M. V. Ranganathan, G. V. Subramanya and P. I. A. Narayanan (I.R.255/62).
20. Recovery of Metallic Values from Industrial Brass Dross — M. Subramanian, T. L. Srikrishna and P. P. Bhatnagar (I.R.256/62).
21. Agglomeration of Fluorspar Flotation Concentrate Obtained from Low-grade Fluorspar from Rajasthan — B. L. Sengupta, G. V. Subramanya and P. I. A. Narayanan (I.R.257/62).
22. Beneficiation of Low-grade Graphite from Kashmir — P. V. Raman, G. V. Subramanya and P. I. A. Narayanan (I.R.258/62).
23. Pilot Plant Studies on Beneficiation and Sintering of Rajhara Iron Ore for Bhilai Steel Plant of Hindustan Steel Ltd. — S. B. Dasgupta, P. K. Sinha, G. P. Mathur and P. I. A. Narayanan (I.R.259/62).
24. Pre-concentration of Lead-zinc Ore from Zawar Employing Heavy Media Separation — S. K. Banerjee, S. K. Dhar, G. V. Subramanya and P. I. A. Narayanan (I.R.260/63).
25. Beneficiation Studies on a Low-grade Nickeliferous Ore from Assam — R. Ganesh, G. V. Subramanya and P. I. A. Narayanan (I.R.261/63).
26. Thermal Beneficiation of Low-grade Chrome Ore from Kankauli Deposits, Ratanagiri — R. N. Misra and P. P. Bhatnagar (I.R.262/63).
27. Studies on the Hot Strength Characteristics of Some Indigenous Bentonites — A. T. Chirayath, V. S. Bhandary and P. K. Gupte (I.R.263/63).
28. Breakdown Characteristics of CO₂ Cores — B. V. Somayajulu, S. S. Bhatnagar and P. K. Gupte (I.R.264/63).
29. Report on the Operation of Low-shaft Furnace Pilot Plant at the National Metallurgical Laboratory for the Production of Pig Iron with East Punjab Raw Materials from Mahindergarh — A. B. Chatterjea and B. R. Nijhawan (I.P.R.2/62).
30. Report on the Operation of Low-shaft Furnace Pilot Plant at the National Metallurgical Laboratory for the Production of Pig Iron with Raw Materials from Maharashtra State — A. B. Chatterjea and B. R. Nijhawan (I.P.R.3/62).

31. Suitability of 'Hathi-ki-Dhani' Bentonite as a Binder for Use in Foundries — A. T. Chirayath, Jatinder Mohan and P. K. Gupte (R.R.126/62).
32. Thermal and Electrical Conductivity of Copper-manganese Alloys — M. K. Gupta and T. Banerjee (R.R.127/62).
33. Determination of Hydrogen in Metals and Alloys by Carrier Gas Method — P. C. Debnath, N. G. Banerjee and B. C. Kar (R.R.128/62).
34. Constitution and Variation of Lattice Parameters of Some Indian Chromites with Magnesia Addition and Subsequent Firing — M. R. K. Rao, N. V. Naidu and H. V. Bhaskar Rao (R.R.129/62).
35. Plasticizing of Non-plastic Refractory Raw Materials: Part I — Effect of Certain Factors in the Pot Milling of Sillimanite — H. P. S. Murthy, A. V. Subrahmanian and H. V. Bhaskar Rao (R.R.130/62).
36. Dissolution of Steel and Absorption of Hydrogen by Steel during Acid Pickling in Presence of Inhibitor — A. K. Lahiri, N. G. Banerjee and T. Banerjee (R.R.131/62).
37. Studies on Indian Refractory Clays: Part IV — Some Clays from Madras State — H. P. S. Murthy, A. V. Subramaniam and H. V. Bhaskar Rao (R.R.132/62).
38. Electrolytic Separation and Determination of Copper in High Manganese Brasses — P. C. Chatterjee and H. P. Bhattacharjee (R.R.133/62).
39. A Study of the Hot Extrusion Characteristics of Aluminium Alloys — B. N. Das and R. Choubey (R.R.134/63).
40. Galvanic Corrosion of Ferrous and Non-ferrous Metals Exposed in the Industrial Atmosphere of Jamshedpur — S. Rao, Addanki, K. P. Mukherjee and A. K. Lahiri (R.R.135/63).
41. Some Observations on Corrosion Resistance of Ancient Delhi Iron Pillar and Present Time Adivasi Iron Made by Primitive Methods — A. K. Lahiri, T. Banerjee and B. R. Nijhawan (R.R.136/63).

APPENDIX III

TECHNICAL ASSISTANCE RENDERED TO INDUSTRIES

1. Separation of lead from copper-lead-silver and gold alloys.
2. Manufacture of various kinds of sintered metal powder parts.
3. Chemicals to extract aluminium from dross and recovery of aluminium by electrolysis.
4. Composition of acid-proof cement.
5. Method of electro-polishing of stainless steel.
6. Process for melting aluminium.
7. Method of determination of carbon contents of steel wire.
8. Composition of non-acidic soldering fluxes and details of an alternative process of pickling.
9. Composition of the steel used for piston rings for diesel auto.
10. Possibilities of using aluminium and its alloys to replace copper in the manufacture of printing rollers.
11. Uses of limestone and calcite.
12. Properties and application of TISCROM steel.
13. Composition of free-cutting steel.
14. Application of silicon metal, silico-manganese and silico-chrome in production of alloys.
15. Method of electroplating of lead.
16. Composition of steel used for cutting dies.
17. Production technique of ferro-manganese from manganese ore.
18. Technique of production of barium metal from baryte ore and its uses in industry.
19. Use of the furnaces for melting aluminium alloys.
20. The technique of recovery of zinc from galvanizing plant zinc dross.
21. The technique of remelting of stainless steel.
22. Steel tape compounding varnish with phthalic acid base.
23. Various uses of high-purity graphite.
24. Materials for construction of acid and alkali resistant tanks.
25. Chemical composition and uses of aluminium alloys for builders' hardware products.

26. Specification for the chilled iron shots and grit used for shot-blasting.
27. The technique of production of aluminium powder.
28. The technique of production of synthetic graphite.
29. Detailed composition and properties of steel used for structural purpose.
30. Technique of modern nickel-plating process and nickel coating on aluminium.
31. Technique of annealing of cold-rolled mild steel strips.
32. Production technique of ferro-alloys in submerged arc furnace.
33. The composition and technique of making metal polishes for brass, nickel, chromium and steel.
34. Techniques of anodizing of aluminium.
35. Technique of manufacture of titanium dioxide from ilmenite ore.
36. Uses of powder coal in foundries.
37. Composition, technique of casting, heat treatment and mechanical properties of manganese steel used for stone crushers.
38. The technique of vacuum metallization of glass, ceramics and plastic for imitation jewellery.
39. The process for making constantan wire with nickel-copper wire.
40. Uses of iron core plates in A.C. electric machines and its composition.
41. Technique of remelting of stainless steel scrap.
42. Production technique of copper-zinc alloy.
43. Composition of liquid non-acidic flux for soldering.
44. Type of metal suitable for use in sewage pump.
45. Technique of colouring of steel.
46. Composition of soldering flux and soldering metal for soldering aluminium.
47. Melting practice to be adopted for obtaining controlled porosity in non-ferrous metal.
48. Nature of baths used for colouring of brass.
49. Technique of manufacture of iron-cobalt alloy powders.
50. Specification of quartzite used for ferro-silicon manufacture.
51. Procedure to be adopted for ramming the lining in rotary urnace.
52. Method of manufacture of stainless steel sheet and utensils casting from the scrap.
53. Process for manufacture of porous bronze bearing and copper powder.
54. Utility of hardened zinc.
55. Production technique of ferrites and sintered soft alloys.
56. Properties of steel at sub-zero temperature.
57. Methods of manufacture of metal powder and raw materials and machinery required for the purpose in the industry.
58. The production technique of zinc, aluminium and bronze powder.

59. The process of separation of lead and copper from their alloys.
60. Technique of making of iron, zinc, brass, lead and tin powders.
61. Mechanical properties of meehanite.
62. Recovery of silver from lead-silver alloy.
63. Recovery of copper from scrap.
64. Composition of nickel-bronze, silicon-bronze, manganese-bronze and other types of bronzes.
65. Composition and availability of pig irons in India.
66. Uses of tungsten steel for permanent magnets.
67. Percentage losses of metal in melting, oxidation, slagging, etc., in utensil making.
68. Method of casting stainless steel alloy of 316 and 321 type.
69. Steel for cold-forging nut plant.
70. Occurrence of phosphate rocks in India and its composition.
71. Method of manufacture of welding rods for electric welding.
72. Technique of melting of pyrophoric alloy to make cigarette lighter flints.
73. Specifications of coal and coke for different kinds of foundries.
74. Substitute of 'woods' metal.
75. Use of bell metals containing less than 10 per cent tin for making utensils.
76. Technique of detinning to obtain tin.
77. Corrosive behaviour of mono-nitro-toluene.
78. Technique of sand casting of aluminium.
79. Composition of metal polish.
80. Standard tests for castings.
81. Equipment for remelting of stainless steel scrap casting, annealing and machining.
82. Electroplating of aluminium and its alloys.
83. Technique of heat treatment of high-speed steel and composition and other data of heat treatment salts.
84. Technique of case hardening of steel.
85. Types of steel to be used for the fabrication of emulsion vessels.
86. Surface hardening technique for metals.
87. Moulding technique of cement sand in steel foundries.
88. Technique of case hardening of cast iron casting.
89. Composition, mechanical and physical properties and casting characteristics of aluminium alloys used for auto-pistons and other parts.
90. Case hardening of steel in salt bath.
91. Composition of the flux and rods for arc welding electrodes.
92. The effects of smoke from silicate industry on public.

93. Blackening of the steel-back of the engine bearing as a protection against rusting.
94. The technique of tinning of aluminium.
95. Composition of cast iron for moulds for moulding tiles.
96. Composition of melting of nickel silver.
97. The technique of use of bright nickel bath for nickel plating.
98. Grade of master alloys to be used for various types of bronze.
99. Process for drawing of copper wire.
100. Method of casting and rolling of lead.
101. Technique of using side-blown converter for alloy steel plant.
102. Composition of the steel for surgical blades.
103. Composition of steel used for worm gears and worm for pulley block and tackles.
104. Technique of extraction of cadmium.
105. Welding electrode-composition of iron and steel.
106. Technique of surface finishing on cold-drawn copper wire.
107. Technique of remelting of copper scrap and its rolling and drawing.
108. Annealing technique for malleable casting.
109. Equipment and technique of drawing copper wire.