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NATIONAL METALLURGICAL LABORATORY
COUNCIL OF SCIENTIFIC & INDUSTRIAL RESEARCH
JAMSHEDPUR, INDIA

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FOREWORD

IN presenting the report of the Director, National Metallurgical Laboratory, for the year 1965-66, I derive particular satisfaction from the fact that its many-sided activities have acquired wider dimensions, and that it has constantly sought and found new avenues of scientific study and research.

Metallurgical industries in India, both in public and private sectors, have their share of national problems and difficulties, emanating mainly from the acute foreign exchange crisis and scarcity of imported raw materials, particularly of ferrous and non-ferrous alloys. The sense of urgency that the import substitution programme assumes at present for taking us towards our cherished goal of self-sufficiency and self-reliance, places added responsibility on institutions engaged in metallurgical research. The performance of the National Metallurgical Laboratory has been impressive from this stand-point. Many of its research studies on substitute alloys have been fruitful, and are now being implemented on a commercial scale. It organized an international symposium on ' Metallurgy of Substitute Ferrous and Non-Ferrous Alloys' earlier this year, and thus gave an opportunity to experts in the field to get together and compare notes on this all-important subject.

Metallurgy being the hard core of our planned progress, the importance of research in this vital field of national endeavour cannot be over emphasized. The way NML has served the mineral and metal industries strengthens our faith in the future of this institution. It has successfully undertaken a number of metallurgical research projects related to the Third Five-Year Plan, and helped many public and private undertakings by evolving new processes and providing technical know-how. The painstaking work undertaken by the Laboratory on Kiriburu iron ores and their beneficiation, agglomeration and sintering for the Bokaro Steel Plant has been described by the Soviet experts to be of ' a high technical level '.

Similarly, the NML Project Report on the Foundry Pig Iron Plant, employing low-temperature carbonized coke made out of Singareni non-coking coals, has its own importance in view of the limited reserves of coking coal in the country. This Report has been prepared after comprehensive investigations at the Low-shaft Furnace Pilot Plant.

On behalf of the National Mineral Development Corporation, intensive pilot plant investigations were undertaken on the beneficiation studies of low-grade copper ores from Khetri, Kolihan and Rakha Mines. N.M.D.C. are now developing these low-grade ores with a view to setting up copper smelting plants.

In collaboration with the Central Design and Engineering Unit of the Council of Scientific & Industrial Research, the National Metallurgical Laboratory is setting up a Magnesium Plant at Jamshedpur. A full Project Report is ready and preparatory steps for setting up the plant are well under way. To be built at a cost of Rs. 67 lakhs on a seven-acre plot generously given by the Tata Iron & Steel

Company Limited, this plant is expected to produce 250 tons of magnesium a year on a semi-commercial scale.

Foundry technology is yet another field in which the Laboratory has taken tremendous strides. In order to provide technical help and guidance to small-scale foundries situated in different parts of the country, NML is setting up a network of foundry stations. The second NML Foundry Station, which was formally opened at Guindy, Madras, by Shri R. Venkatraman, Minister for Industries, Government of Madras, in December 1965, marked yet another milestone in this direction. Steps are also being taken to start the third foundry station at Howrah, the fourth at Ahmedabad and the fifth at Agra.

Particularly heartening is the initiative that NML has taken in organizing the 33rd International Foundry Congress in New Delhi towards the end of the current year in collaboration with the Institute of Indian Foundrymen under the aegis of the Union Ministry of Industry. To be held in India for the first time, this Congress will be attended by scientists from more than 24 countries of the world. Besides, the United Nations, for the first time, will sponsor observers from the developing countries. Considering the rapid advances that are being made in foundry technology all over the world, an international gathering of this kind should be of immense benefit not only to India, but also to other developing countries.

Fundamental research at NML has kept pace with applied development projects. Several research theses have been submitted on these fundamental studies, and eight scientists have so far been awarded Ph.D. degrees by different Indian Universities.

In keeping with the aims of the Government policy on scientific research, the National Metallurgical Laboratory is contributing its utmost towards securing for the people of the country ' the benefits that can accrue from the acquisition and application of scientific knowledge '. This outstanding work has received wide appreciation not only within the country but also outside, and the credit goes to Dr. B. R. Nijhawan and his able colleagues.

Dr. Nijhawan's valuable contributions to metallurgical research find a fitting recognition in the Honorary Membership of the Societe Francaise de Metallurgie, Paris, recently conferred on him, and in his selection as Senior Adviser to the Centre for Industrial Development at the United Nations in New York. After about 19 years of dedicated service to the Laboratory and to the country, he will be leaving India shortly for New York to take up his new assignment, and, I am sure, many under-developed countries will benefit by his rich experience in mineral and metallurgical fields.

On behalf of the Members of the Executive Council, and on my own, I would like to record our sincere appreciation of the sustained work that has been done at NML under Dr. Nijhawan's able leadership.

J. J. GHANDY
Chairman
Executive Council
National Metallurgical Laboratory
Jamshedpur (*India*)

July 31, 1966

INTRODUCTION

THE National Metallurgical Laboratory has passed through another significant year of all-round progress in the multiple fields of research and development work.

During the year under review, the National Metallurgical Laboratory has implemented some of the important development themes of the Third Five-year Plan whilst giving significant support to existing major Public and Private Sector metallurgical enterprises in their industrial programme. Some major highlights relate to the preparation of raw materials for non-ferrous industries such as the extensive flotation studies made on low-grade copper ore samples from Khetri and Kolihan in Rajasthan and Rakha in Bihar on behalf of National Mineral Development Corporation with a view to subsequent establishment of new copper smelting plants in the country. The work for Bokaro Steel Project on beneficiation and agglomeration of Kiriburu iron ore has been highly commended by the Soviet Experts. Flowsheets developed for the beneficiation and sintering of Dalli Pahar iron ore from Bhilai Steel Plant; beneficiation and sintering of Meghataburu iron ore for National Mineral Development Corporation; beneficiation and pelletization of iron ore fines from Redi mines for Government of Maharashtra and the detailed Investigation Project Reports prepared on these subjects after extensive pilot plant trials are some examples of the systematic and painstaking work of the National Metallurgical Laboratory.

The setting up of the semi-commercial Magnesium Pilot Plant has made further progress and the plant is expected to be set up well before the schedule.

Injection of light naphtha trials in the Pilot Low-shaft Furnace to the Laboratory has yielded very useful and encouraging results. Employing low-temperature carbonized coke, the National Metallurgical Laboratory has successfully produced pig iron in the Low-shaft Furnace which holds considerable promise in the establishment of regional pig iron plants. High carbon ferro-chrome and silico-chrome using Giridih coke along with wood charcoal were successfully produced on tonnage scale in the Pilot Submerged Arc Smelting Furnace and the processes were found to be commercially feasible.

The long range research projects on development of substitute alloys and products have yielded highly encouraging results some of which are now being implemented on industrial scale. The work in the field of non-ferrous has been further enlarged and steady progress has been maintained in development of various types of aluminium-based alloys suitable for use in engineering and structural purposes in place of the conventional alloys made of copper, tin, zinc, lead, etc. The techniques of refining hypo- and hyper-eutectic aluminium-silicon alloy developed in the Laboratory has been licensed to a West German firm.

Top priority has been given to the Defence requirements and close co-ordination is maintained with the various Defence Establishments of the country. On behalf of Indian Standards Institution, a number of projects were undertaken with a view to formulation and rationalization of Indian Standard specifications.

The foundry extension units of the Laboratory are rendering valuable help and assistance to the small-scale foundries, and this assistance has been recognized by the Directorate of Industries. Apart from the Foundry Stations at Batala and Madras which are functioning in full swing, the stations at Howrah and Ahmedabad will start soon and a fifth one is under establishment at Agra. A number of short and long range research projects are under way at the Marine Corrosion Research Station, Digha; and the scope of work on corrosion, both in the main Laboratory and Digha Station, has been considerably enlarged.

The study on fundamental problems has maintained its steady progress. Theses based on the study have been submitted and scientists were awarded the Ph.D. degree.

The National Metallurgical Laboratory organized an International Symposium on ' Metallurgy of Substitute Ferrous and Non-Ferrous Alloys ' with a view to stress upon the development and production of substitute alloys in the background of acute foreign exchange shortage and the utilization of indigenous raw materials particularly in times of emergency. The results of researches done in the National Metallurgical Laboratory on development of substitute alloys and products along with distinguished research contributions from Indian and Overseas scientists were presented and discussed.

During the year under review the Laboratory has taken the following patents.

Patents filed

(i) Indian Patent No. 102483 — An improved method for the recovery of metallic values from waste metallic fines (12-11-1965).

(ii) Indian Patent No. 102481 — A method for recovering volatile metals from their waste metallic fines.

Patents Accepted

(i) Indian Patent No. 96661 — A novel method for separating iron from ilmenite and its application in upgrading ferruginous ores (11-5-1966).

(ii) Indian Patent No. 94769 — An improved device for the continuous hot-dip coating of metallic strip and wire (15-1-1966).

(iii) Indian Patent No. 94768 — An improved device for the isolation of dross in molten baths during continuous hot-dip processing of strip or wire (22-4-1966).

Patents Sealed

(i) Indian Patent No. 82191 — An improved jacketted electrolytic cell for the electrodeposition of metals and metallic oxides in general and manganese dioxide in particular (10-5-1966).

The following processes have been released to the industries to date for commercial exploitation:

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. Production of electrical resistance alloys for heating elements.
5. Technology of production of thermostatic bi-metals.
6. Production of alnico type permanent magnents.
7. Hot-dip aluminizing of ferrous materials.
8. Refractory compositions comprising graphite and silicon carbide (carbon-bonded graphite crucibles).
9. Refractory compositions comprising graphite and alumino-silicate materials and glazes to render such compositions resistant to oxidation (clay-bonded graphite crucibles).

10. Production of carbon-free ferro-alloys by aluminothermic reactions.
11. Production of iron powder for autogenous cutting.
12. Preparation of liquid gold.
13. Production of flux for submerged arc welding.
14. Electroplating of metals on aluminium or its alloys.
15. Chemical polishing of aluminium.
16. Metallization of non-conductors.
17. Brass plating from non-cyanide bath.
18. Production of dense carbon aggregate suitable for being used as base material for carbon products in general and Soderberg paste in particular.
19. Production of electrolytic copper powder.
20. Production of ceramic magnets.
21. Production of modified aluminium alloys containing silicon.

A brief résumé of the progress of important projects underway 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 a Mixed Iron Ore Sample from Kondekasa Block of Dalli Pahar Deposits for Bhilai Steel Plant

Four different types of iron ore samples from Dalli Iron Ore Mines, each weighing about 50-55 tonnes, were received for conducting beneficiation and sintering studies by mixing the four different samples in proportion to their ore reserves.

The mixed sample as received assayed Fe, 62·54; SiO_2, 2·59; Al_2O_3, 4·30 and loss on ignition, 3·9 per cent. Screenability tests performed with different moisture contents in the sample indicated that screening was least efficient when the moisture content of the sample was 5 per cent. Scrubbing followed by wet screening of the crushed ore (−2 in./−50·8 mm. size) indicated that a clean, sized, washed product of $+\frac{1}{2}$ in. size (+12·7 mm.) free from adherant ore fines, for direct charging into the iron blast furnace, and a $-\frac{1}{2}$ in. size (−12·7 mm.) classifier sand free from slime for sintering could be obtained. The $+\frac{1}{2}$ in. (+12·7 mm.) washed lumps constituting 68·0 per cent by weight assayed Fe, 65·15; SiO_2, 1·97 and Al_2O_3, 3·14 per cent with a recovery of 70·6 per cent of Fe. The slime produced was 5·6 per cent by weight with the rejection of about 26·1 per cent of the total silica and 16·9 per cent of the total alumina originally present in the ore, for a corresponding iron loss of 3·7 per cent only.

'Wet screening' the ore (without prior scrubbing) at −2 in. (−50·8 mm.) size could reject 24·1 and 13·6 per cent of the total silica and alumina respectively, for an Fe loss of 3·1 per cent only. The amount of slime produced was slightly lower, i.e. 4·8 per cent by weight. The $+\frac{1}{2}$ in. (+12·7 mm.) wet screened lumps constituting 69·5 per cent by weight assayed Fe, 64·68; SiO_2, 2·14 and Al_2O_3, 3·7 per cent with an iron recovery of 71·9 per cent.

The $-\frac{1}{2}$ in. (−12·7 mm.) classifier sand obtained from washing at −2 in. (50·8 mm.) size, constituting 26·4 per cent by weight, assayed Fe, 60·9; SiO_2, 3·51 and Al_2O_3, 4·92 per cent and contained 25·7 per cent of the total iron content of the ore. Since this product was high in insolubles, sinters made therefrom would not offer any metallurgical advantage. Hence, this product was upgraded by jigging. The beneficiated jig concentrate (combined) 20·2 per cent by weight assayed Fe, 64·6; SiO_2, 1·98 and Al_2O_3, 3·27 per cent with an iron recovery of 20·9 per cent with respect to the original.

Sintering characteristics of the $-\frac{1}{2}$ in. (−12·7 mm.) washed and upgraded classifier sand were studied for making unfluxed and fluxed sinters. In the case of unfluxed sinters, it was found that 7 per cent water and 4·5 per cent coke were the optimum values for producing a good sinter. The coke content had to be varied from 3·5 to 5·0 per cent in the case of high basicity sinters (basicity ratio ranging from 1·0 to 2·4) to produce good strong sinters. The results also showed that solid fuel coke could be substituted by gaseous fuel (producer gas) up to 40 per cent of the total coke needed for production of unfluxed sinters. With regard to fluxed sinters with basicity up to 1·6, saving in the coke could be only up to 30 per cent. In the case of sinters with higher basicity, gaseous fuel could not be used to replace

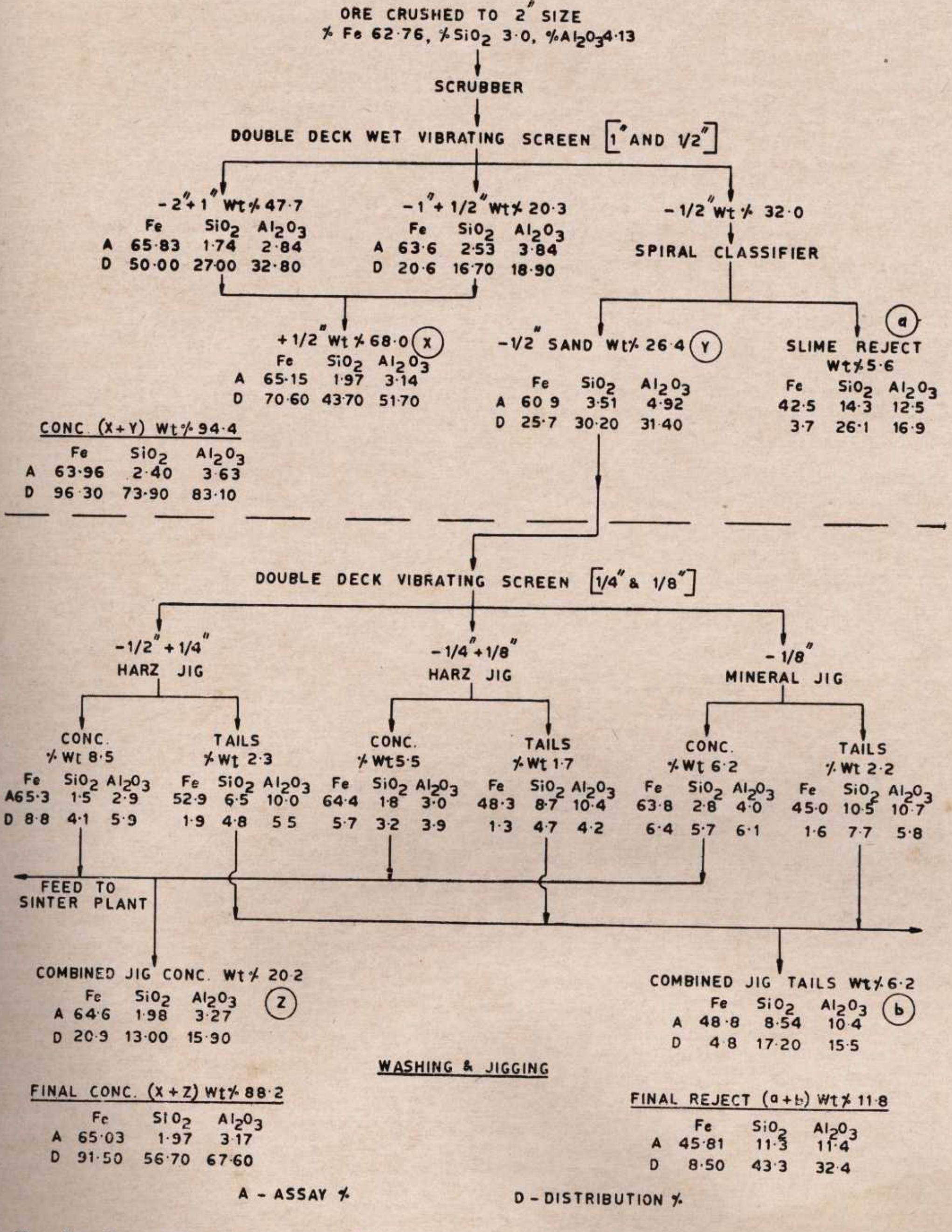


FIG. 1 — SUMMARY OF RESULTS OF WASHING AND JIGGING OF DALLI IRON ORE MIXED SAMPLE

the coke. Highly oxidized sinters were produced when mixed firing was employed, whereas unfluxed sinter was invariably high in FeO. Weathering tests performed with fluxed sinters showed that the amounts of $-\frac{1}{2}$ in. (−12·7 mm.) fines generated from sinters during storage for 24 hours varied from 1·29 to 3·51 per cent by weight depending upon basicity (disintegration being more with higher basicity).

The results of washing and jigging are given in Fig. 1. Based on these results a general flowsheet recommended is given in Fig. 2.

The economic evaluation of beneficiation treatment of iron ores has followed certain well-accepted parameters for improved productivity and lowered fuel and flux rates vis-à-vis prepared and beneficiated ore burdens apart from incidental much desired benefits of uniform smelting operations and lower slag volumes. Table 1 gives the economic evaluation of beneficiation of iron ores assessed on the following basis which has uniformly been kept in view of Indian iron ores.

(i) The cost of raw ore as mined in this particular case is taken at Rs. 4·28 per ton. Transport charges for ore are not included in this.

(ii) Beneficiation plant is assumed to operate with a capacity of 14,000 tonnes of run-of-mine ore per day.

(iii) Capital cost of the plant does not include cost of mining equipment and township.

(iv) For every one per cent decrease in the alumina content of the ore, the

Table 1 — Economic Evaluation of Beneficiation of Dalli Mixed Sample

	Crushing to −2″ dry Screening and utilization of $-\frac{1}{2}''$ fines			Crushing to −2″ dry screening and rejection of $-\frac{1}{2}''$ fines			Crushing to −2″ scrubbing wet screening de-watering of $-\frac{1}{2}''$ fines			Crushing to −2″ scrubbing wet screening beneficiation of $-\frac{1}{2}''$ fines		
	Fe	SiO_2	Al_2O_3	Fe	SiO_2	Al_2O_3	Fe	SiO_2	Al_2O_3	Fe	SiO_2	Al_2O_3
Analysis of final beneficiated ore %	62·34	2·59	4·28	64·21	2·23	3·78	63·96	2·40	3·63	65·03	1·97	3·17
Yield tonnes/day	14,000 100			10,290 73·5			13,216 94·4			12,348 88·2		
Capital cost of ore treatment plant	Rs. 2·50 crores			Rs. 2·50 crores			Rs. 3·36 crores			Rs. 4·5 crores		
Cost of treatment per tonne of beneficiation ore												
(a) Capital cost	Rs. 0·894			Rs. 1·216			Rs. 1·273			Rs. 1·825		
(b) Operating cost	Rs. 0·180			Rs. 0·245			Rs. 0·257			Rs. 0·367		
(c) Cost of raw ore as mined	Rs. 4·280			Rs. 5·823			Rs. 4·534			Rs. 4·852		
Total cost per tonne of prepared ore	Rs. 5·354			Rs. 7·284			Rs. 6·664			Rs. 7·044		
Tonnes of ore required for tonne of pig iron	1·545			1·500			1·507			1·482		
Total cost of the ore required per tonne of pig iron	Rs. 8·271			Rs. 10·926			Rs. 9·139			Rs. 10·440		
Difference in alumina per cent	—			0·50			0·65			1·11		
Expected saving in production cost per tonne of pig iron in blast furnace iron smelting due to decrease in alumina by beneficiation	—			2·50			3·25			5·55		

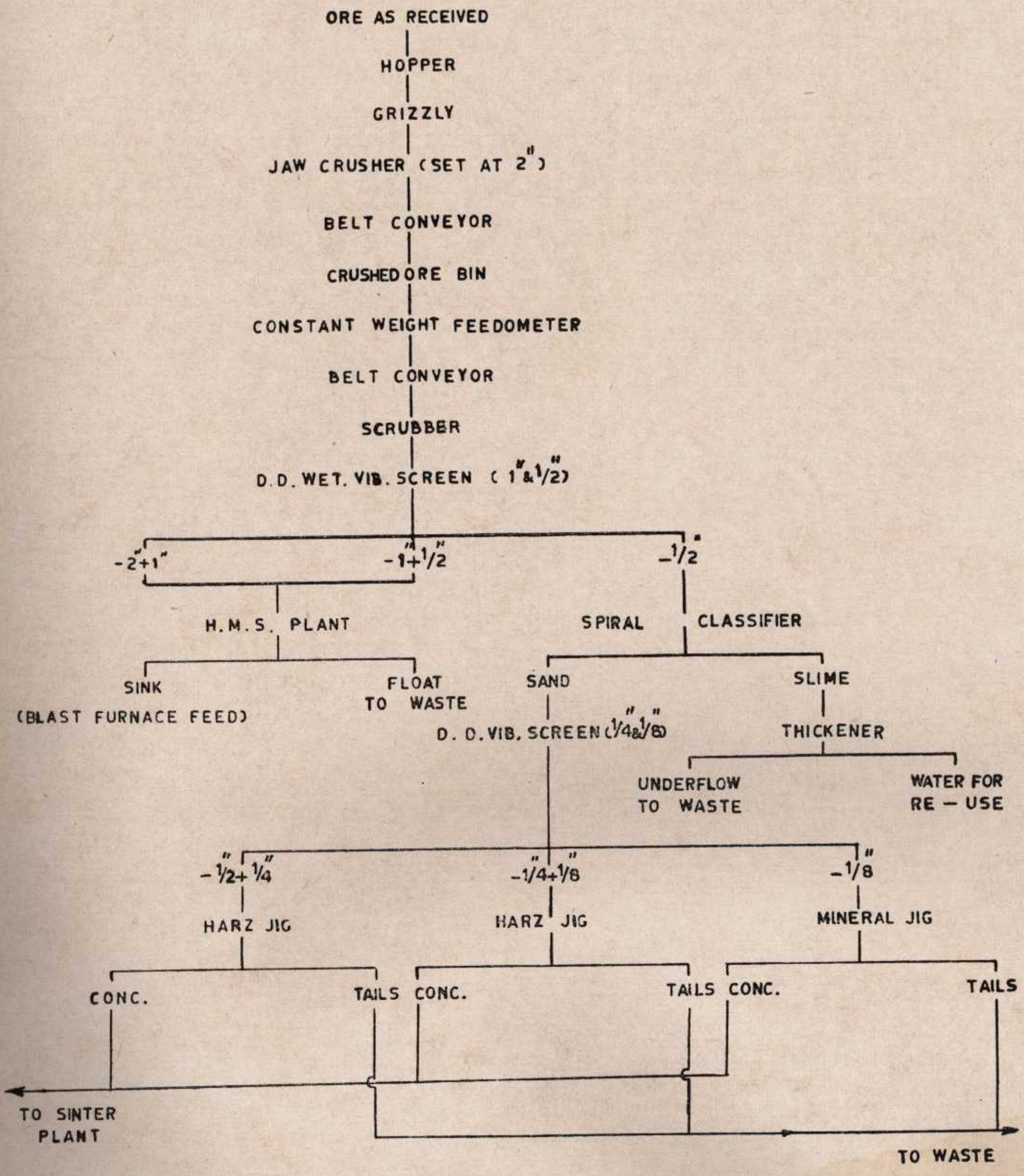


Fig. 2 — Flowsheet Employed for the Beneficiation of Dalli Iron Ore Mixed Sample

coke rate and flux rate would decrease by 40 and 60 kg., respectively, per tonne 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 tonne of pig iron produced would be Rs. 5·0 after allowing for depreciation, etc.

1.1 Pilot Plant Studies on (i) Beneficiation of an Iron Ore from the Kiriburu Mines of National Mineral Development Corporation, (ii) Production of Additional — 10 mm. Fines, and (iii) Production of High-basicity Sinters for the Bokaro Steel Project

Pilot plant investigation was undertaken with a mixed ore sample collected from the lower and upper portion of the main haulage road from Bench No. 3 of Hill No. 1 at the instance of the Bokaro Steel Plant authorities for determing the optimum beneficiation cycles and study of sintering characteristics including formulation of a suitable procedure for the production of additional —10 mm. fines which will make up the required 70 per cent of ore fines for sintering production and the remaining 30 per cent in the form of —40 mm. +10 mm. size lumps; the two together constituting the blast furnace burden.

Three different schemes were studied for the production of additional amount of fines by crushing and wet and/or dry screening the crushed product. The results obtained are given in the flowsheet (Fig. 3). A simplified flowsheet is furnished in Fig. 4.

Table 2 gives the chemical analyses of the —10 mm. products for sintering obtained by employing the three schemes. The above results indicated that the best grade of —10 mm. ore fines was obtained by employing scheme (III), namely, wet screening of the jaw-crushed ore followed again by wet screening at 10 mm. of the cone-crushed fines, although the grades of fines produced by schemes I and II were not very much lower. There being no appreciable difference between the grades of ore fines obtained with the three schemes separately, the final choice of the method of production of fines will depend mainly upon the availability of large quantities of water for wet screening. This method will also need equipment for thickening, upgrading and filtration of slimes. On the other

Table 2 — Chemical Analysis of —10 mm. Products

Products	Wt % with respect to original	Assay %			Distribution %		
		Fe	SiO_2	Al_2O_3	Fe	SiO_2	Al_2O_3
Scheme (I): II+VII+X (dry/dry screening) and without jigging	59·4	61·7	2·7	4·2	61·4	41·0	51·5
Scheme (I): IIa+VII+X (dry/dry screening and after jigging)	54·5	63·2	1·85	3·5	57·8	26·0	40·3
Scheme (II): II+VII+VIII (dry/wet screening and without jigging)	57·5	61·8	2·7	4·1	59·5	40·1	49·2
Scheme (II): IIa+VII+VIII (dry/wet screening and after jigging)	52·6	63·3	1·8	3·6	55·9	24·4	38·0
Scheme (III): II+III+IV (wet/wet screening and without jigging)	55·8	62·0	2·7	4·14	57·9	38·3	47·1
Scheme (III): IIa+III+IV (wet/wet screening and after jigging)	50·9	63·6	1·7	3·4	54·3	22·6	35·9

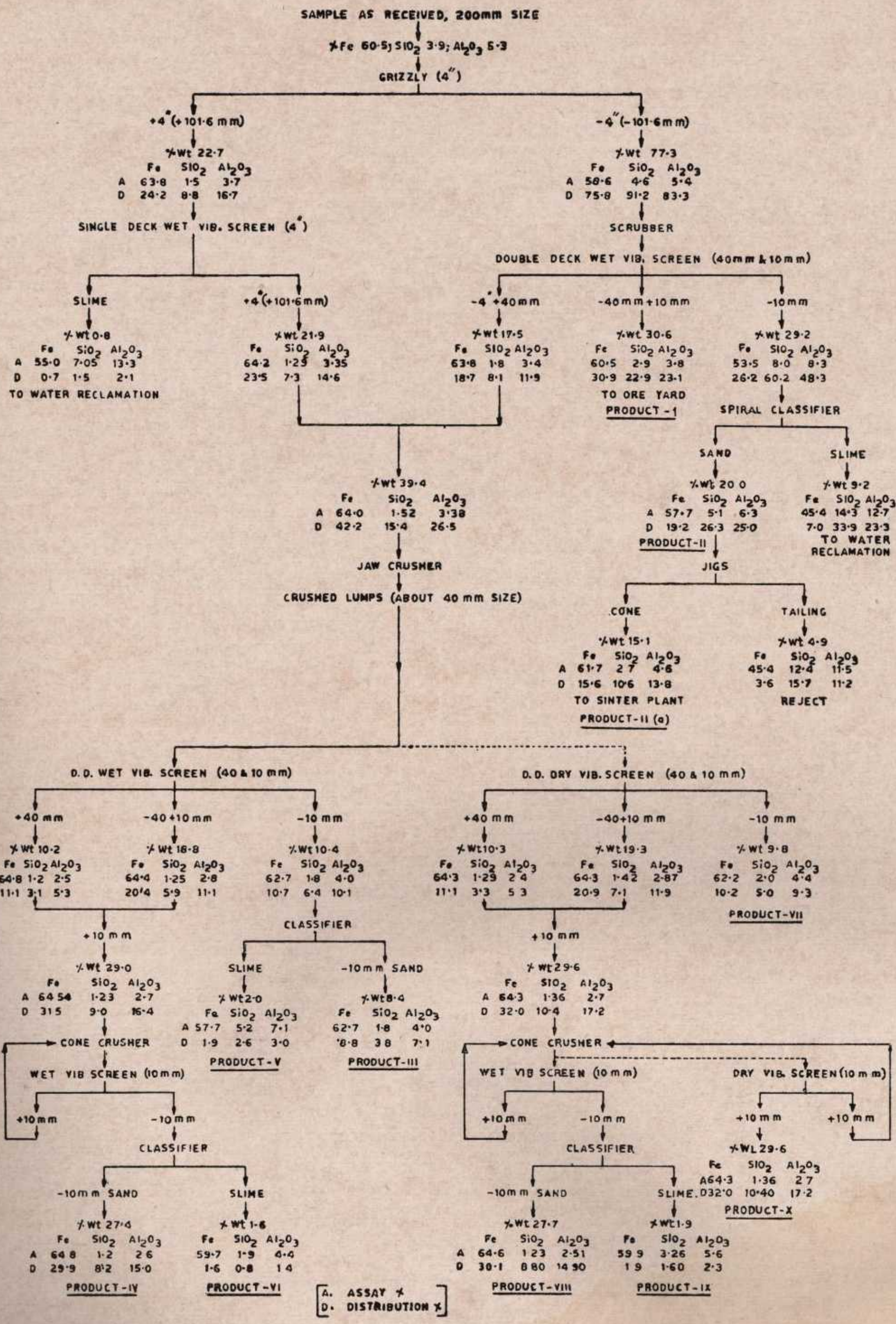


FIG. 3 — FLOWSHEET FOR PRODUCTION OF ADDITIONAL QUANTITY OF —10 MM. KIRIBURU IRON ORE FINES

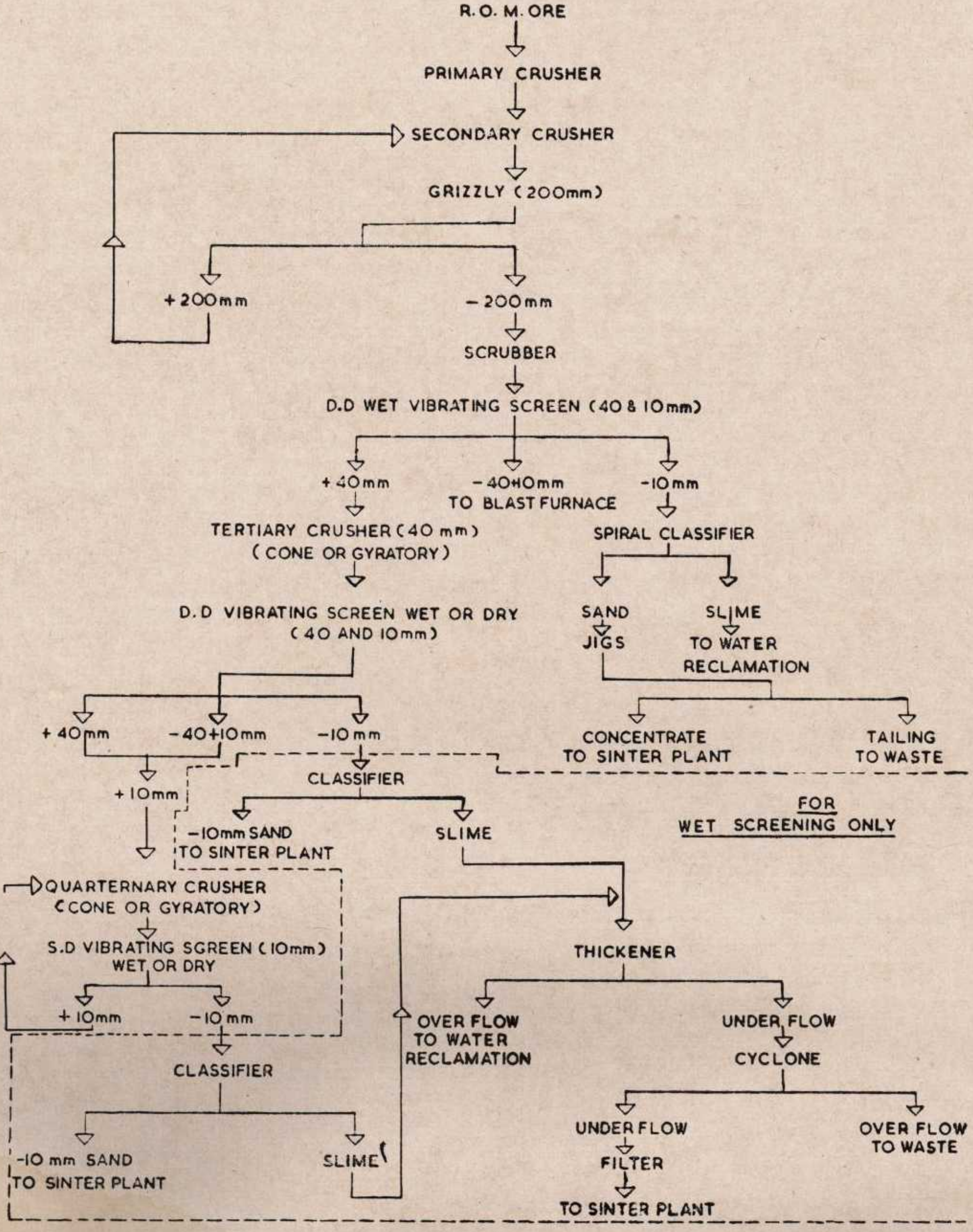


FIG. 4 — SIMPLIFIED FLOWSHEET FOR PRODUCTION OF 70:30 KIRIBURU IRON ORE FINES AND LUMPS

hand, for schemes I and II, the removal of dust generated during dry screening will be the major problem.

The original −10 mm. fraction obtained after scrubbing assayed only Fe, 57·7 per cent; whereas the fines produced by crushing the lumps assayed over Fe, 64·0 per cent; for mixing the former with the latter, it was felt advisable to jig the former to improve its grade to Fe, 61·7 per cent; for an iron loss of only 3·6 per cent in the tailing. The final fines for sintering was of a better grade by nearly Fe, 2 per cent; if jigging was employed only for the original −10 mm. fines, which constituted 20 per cent by weight of run-of-mine ore.

The analyses of the slime produced at the various stages are given in Table 3.

Table 3 — Chemical Analyses of Slimes

Product	Wt with respect to original %	Assay Fe %	Distribution Fe %
1. Primary slime produced during scrubbing and before jaw-crushing			
−4 in.	9·2	45·4	7·0
+4 in.	0·8	55·0	0·7
2. Product V: Slime after jaw crushing	2·0	57·7	1·9
3. Product VI (wet/wet): Slime after cone crushing	1·6	59·7	1·6
4. Product IX (dry/wet): Slime after cone crushing	1·9	59·9	1·9

The primary slime obtained after scrubbing the sample as received was of the poorest grade due to the presence of laterite and may be rejected, for the loss of 7·7 per cent of the total iron. On the other hand, the slimes obtained after crushing the ore in jaw and cone crushers were of medium grade due to the presence of hematite fines and could be easily improved in grade, if required, to over Fe, 61/62 per cent, by a simple cyclone treatment after thickening. Thus the improvements in iron recovery by adding the rich cyclone-treated slime to the sinter feed (−10 mm.) will be about 1·5 to 3 per cent depending upon the flowsheet employed.

The results also showed that a total of 54·5 to 59·4 per cent by weight of −10 mm. ore fines including cyclone-treated slimes from jaw and cone crushed ores could be obtained for sintering. By rejecting 9·2 and 0·8 per cent by weight of primary slimes obtained during wet screening after scrubbing the sample as received, an additional 3·6 to 5·1 per cent by weight of the original −40 mm. +10 mm. washed lumps (constituting 30·6 per cent by weight) would have to be crushed to −10 mm. to make up the ratio of fines to lumps as 70 : 30, depending upon the flowsheet adopted. The above results indicate that even if the entire quantity of +40 mm. fraction obtained from the ore after scrubbing was crushed to −10 mm., there might still be some marginal shortage of −10 mm. fines, if a final lump/fines ratio of 30 : 70 is aimed at.

The overall economics of (i) washing of 200 mm. size ore as received, and (ii) beneficiation of original −10 mm. fines are furnished in Table 4 taking the cost of raw ore mined as Rs. 5·0. The other parameters being the same as referred to earlier.

1.2 Pilot Plant Studies on Beneficiation and Sintering of Laminated Iron Ore from Dalli Mines of Bhilai Steel Plant

A sample of laminated iron ore from Dalli Mines of Hindustan Steel Limited was received for beneficiation and sintering studies at the National Metallurgical Laboratory. The sample assayed Fe, 58·3; SiO_2, 4·1 and Al_2O_3, 7·4 per cent with 5·2 per cent loss on ignition.

Table 4 — Economics of Beneficiation of −10 mm. Fines and Washing of 200 mm. Size Ores

	Run-of-mine ore crushing to 200 mm.			Crushing to 200 mm. scrubbing, wet screening and dewatering of −10 mm. fines			Crushing to 200 mm., scrubbing, wet screening and beneficiation of −10 mm. fines		
	Fe	SiO_2	Al_2O_3	Fe	SiO_2	Al_2O_3	Fe	SiO_2	Al_2O_3
Analysis of final beneficiated iron ore %	60·5	3·9	5·3	61·5	2·8	4·2	62·3	2·2	3·7
Yield tonnes/day	14,000 (100%)			12,600 (90%)			11,914 (85·1%)		
Capital cost of ore treatment plant	Rs. 2·50 crores			Rs. 3·36 crores			Rs. 4·50 crores		
Cost of treatment per tonne of beneficiated ore:									
(a) Capital cost	Rs. 0·894			Rs. 1·333			Rs. 1·891		
(b) Operating cost	Rs. 0·180			Rs. 0·262			Rs. 0·424		
(c) Cost of raw ore as mined	Rs. 5·000			Rs. 5·556			Rs. 5·876		
Total cost per tonne of prepared ore	Rs. 6·074			Rs. 7·151			Rs. 8·191		
Tonnes of ore required for tonne of pig iron	1·592			1·567			1·546		
Total cost of ore required per tonne of pig iron	Rs. 9·674			Rs. 11·200			Rs. 12·670		
Difference in Alumina per cent	—			1·1			1·6		
Expected saving in production cost per tonne of pig iron in blast furnace iron smelting due to decrease in alumina by beneficiation	—			Rs. 5·5			Rs. 8·0		

The ore was crushed to 2 in. in a jaw crusher and screened through ½ in. (12·7 mm.) (i) dry, (ii) wet, and (iii) wet after a scrubbing treatment (washing). The +½ in. lump ore fractions obtained by the dry screening, wet screening and washing operations assayed 59·1, 59·75 and 61·5 per cent Fe, respectively. Twenty-one per cent of the total silica and 15·1 per cent Al_2O_3 present in the ore were rejected in the slimes during washing for an iron loss in this product of only 3·9 per cent. The −½ in. (−12·7 mm.) classifier sand obtained after scrubbing and wet screening assayed Fe, 58·66; SiO_2, 3·8 and Al_2O_3, 6·7 per cent. The insolubles were rather high and sinters made from this product were not expected to offer any metallurgical advantage due to their high insolubles content. Jigging of the sized fractions of the classifier sand yielded a jig concentrate, weighing 25·1 per cent of the original ore and assaying Fe, 63·2; SiO_2, 2·21 and Al_2O_3, 4·20 per cent. The jig tails assayed Fe, 45·8 and insolubles 20·9 per cent. Jigging of the washed fines thus showed substantial improvement in grade.

Sintering characteristics of jig concentrate produced from −½ in. classifier sand obtained by washing the ore at 2 in. size were studied for the production of unfluxed as well as fluxed sinters. In the case of unfluxed sinter, it was found that 6 per cent water and 4·5 per cent coke were optimum for producing strong sinters,

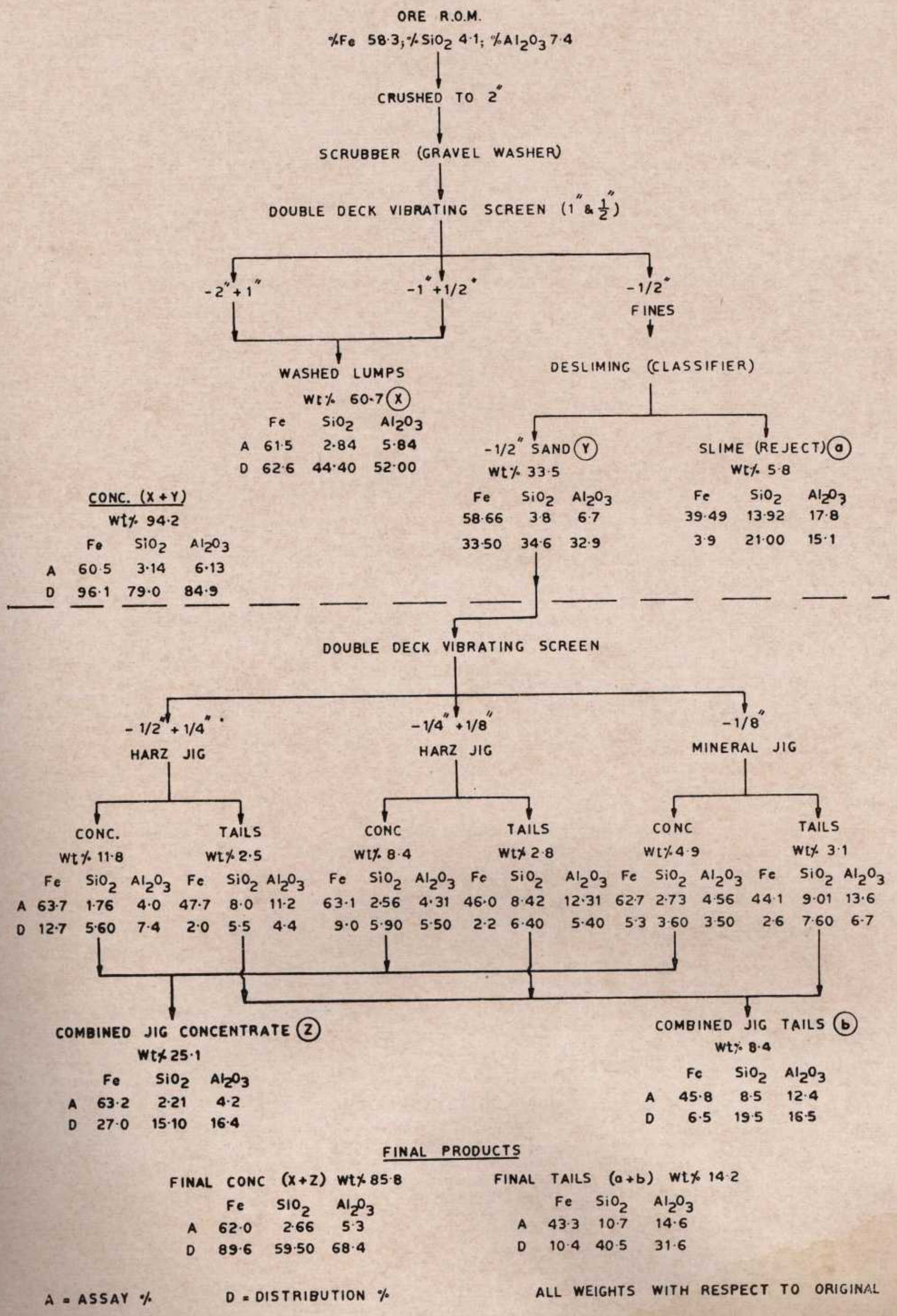


FIG. 5 — SUMMARY OF RESULTS OF WASHING AND JIGGING OF LAMINATED IRON ORE FROM DALLI MINES

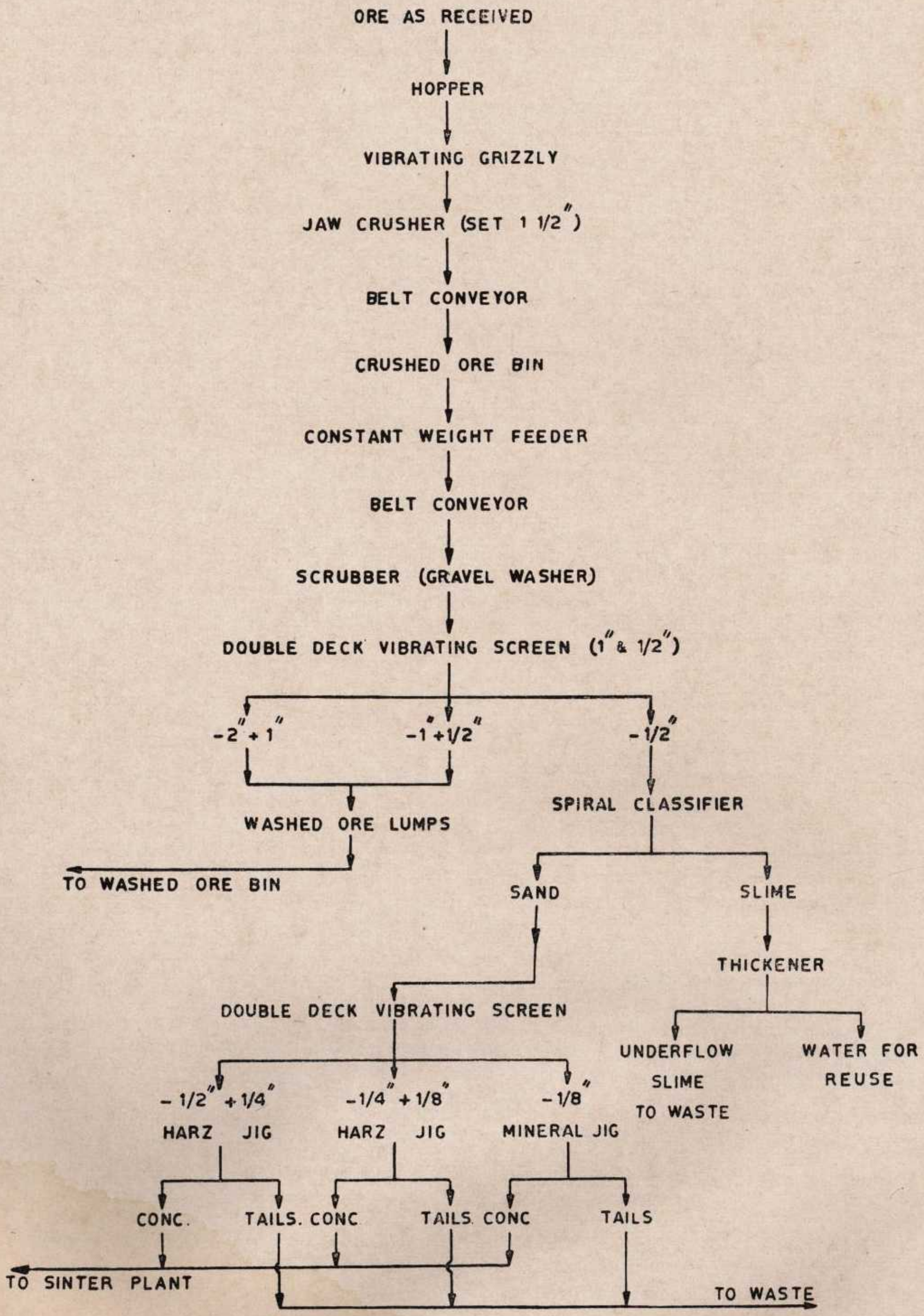


FIG. 6 — FLOWSHEET EMPLOYED FOR THE BENEFICIATION OF IRON ORE FROM DALLI MINES

though metallurgically inferior to fluxed sinters. Good quality fluxed sinters up to a basicity of 2·4 could be produced by suitable additions of limestone. As the limestone (from Nandini Mines) employed for production of fluxed sinters was high in MgO content, no separate dolomite additions were made in the sinter mix. The results further showed that solid fuel could be partially substituted by gaseous fuel (coke oven gas) up to 30 per cent of total amount of coke needed for proper sintering in the case of both fluxed and unfluxed sinters. The sinters produced had good strength and also possessed a high degree of oxidation. Weathering tests performed with fluxed sinter showed that the amount of $-\frac{1}{2}$ in. ($-12{\cdot}7$ mm.) fines generated from sinters during storage for 24 hours varied from 2·1 to 3·8 per cent by weight.

A summary of the results of washing followed by jigging of fines is given in Fig. 5. The flowsheet employed for beneficiation of the sample is given in Fig. 6.

1.3 Pilot Plant Studies on Beneficiation and Sintering of Massive Iron Ore Sample from Dalli Mines of Bhilai Steel Plant

A sample of massive iron ore from Dalli Mines received for beneficiation and sintering studies, assayed Fe, 66·0; SiO_2, 2·3; and Al_2O_3, 2·1 per cent. The sample being high grade and low in insoluble content, scrubbing and upgrading of fines were not considered essential. Dry and wet screening studies with the ore after crushing to 2 in. size were made. Results obtained by the two methods of screening were not very much different from each other owing to the sample being of high grade. However, the products obtained after wet screening were of somewhat better grade than those obtained by dry screening.

Sintering characteristics of a $-\frac{1}{2}$ in. classifier sand obtained after wet screening at 2 in. size were studied for the production of fluxed as well as unfluxed sinters. In the case of unfluxed sinters, it was found that 6 per cent moisture and 4·5 per cent coke were optimum for producing strong sinter, even though unfluxed sinters were metallurgically not to be recommended for industrial usage. Good quality fluxed sinters up to a basicity of 2·4 could be produced by calculated additions of limestone. As the limestone employed for the production of fluxed sinters was high in MgO content, no separate dolomite addition was made in sinter mix.

The results of the mixed firing studies showed that in the case of fluxed sinters, solid fuel could be partially substituted by gaseous fuel (producer gas) up to 20 per cent of the optimum coke addition needed for proper sintering. The unfluxed sinters produced by this treatment were brittle and had low shatter values. On the other hand, the fluxed sinters had good strength as well as higher degree of oxidation. Weathering tests performed with fluxed sinter showed that the amount of $-\frac{1}{2}$ in. ($-12{\cdot}7$ mm.) fines generated from sinters during storage for 24 hours varied from 1·31 to 2·68 per cent.

A summary of results of washing is given in Fig. 7. The flowsheet employed for beneficiation of the sample is given in Fig. 8.

1.4 Pilot Plant Studies on Beneficiation and Sintering of a Compact Laminated Iron Ore Sample from Dalli Mines of Bhilai Steel Plant

A sample of compact laminated iron ore from Dalli Mines of Hindustan Steel Ltd, received for beneficiation and sintering studies assayed Fe, 66·45; SiO_2, 1·94 and Al_2O_3, 2·0 per cent. As the sample was of high grade and low in insolubles, scrubbing and upgrading of fines were not considered necessary. Dry screening of the sample after crushing to 2 in., yielded two products, viz. the $+\frac{1}{2}$ in. fraction assaying

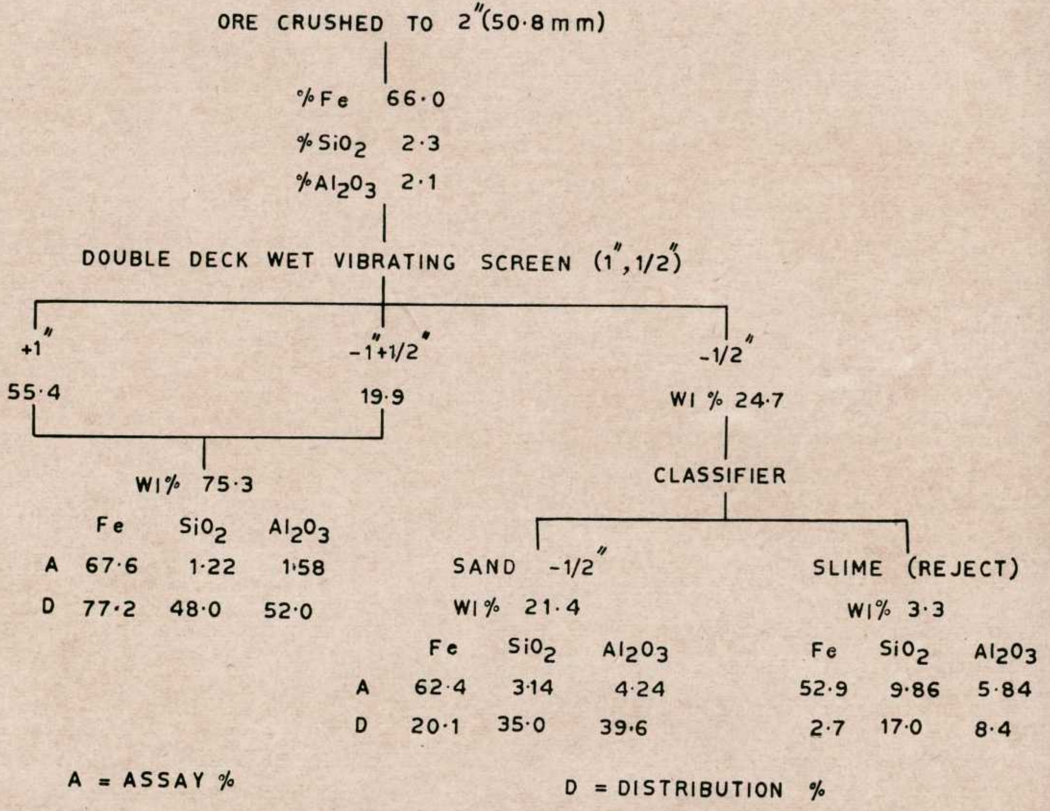


FIG. 7 — SUMMARY OF RESULTS OF BENEFICIATION OF MASSIVE IRON ORE SAMPLE FROM DALLI MINES

Fe, 67·5; SiO_2, 1·35 and Al_2O_3, 1·6 per cent representing an iron recovery of 77·7 per cent and the $-\frac{1}{2}$ in. fraction assaying Fe, 61·3; SiO_2, 4·08 and Al_2O_3, 3·5 per cent with a distribution of 22·3 per cent Fe in it.

Wet screening of the 2 in. size ore followed by desliming of $-\frac{1}{2}$ in. fraction, yielded a $+\frac{1}{2}$ in. washed lump fraction, $-\frac{1}{2}$ in. classifier sand and a slime. The $+\frac{1}{2}$ in. wet screened material, as compared to the dry screened $+\frac{1}{2}$ in. product, was of somewhat better grade assaying Fe, 68·2; SiO_2, 1·1 and Al_2O_3, 1·31 per cent but with a slightly lower Fe recovery of 76·3 per cent. Since there was rejection of 23·2 and 14·3 per cent of the total silica and alumina, respectively, in the ore through the slime during wet screening, the washed classifier sand as compared to $-\frac{1}{2}$ in. dry screened material also had an improved iron and lower insolubles content; this product assayed Fe, 64·7; SiO_2, 2·72 and Al_2O_3, 3·52 per cent.

Sintering characteristics of $-\frac{1}{2}$ in. classifier sand were studied for making unfluxed as well as fluxed sinters. In the case of unfluxed sinters it was found that 7 per cent water and 4·5 per cent coke were the optimum values for producing sinters of good strength although metallurgically unfluxed sinters would not be acceptable. Fluxed sinters up to a basicity of 2·4 were prepared by suitable additions of limestone which possessed adequate strength and good metallurgical characteristics. Mixed firing studies undertaken on the sample further showed that solid fuel could be substituted by gaseous fuel up to 20 per cent of the total

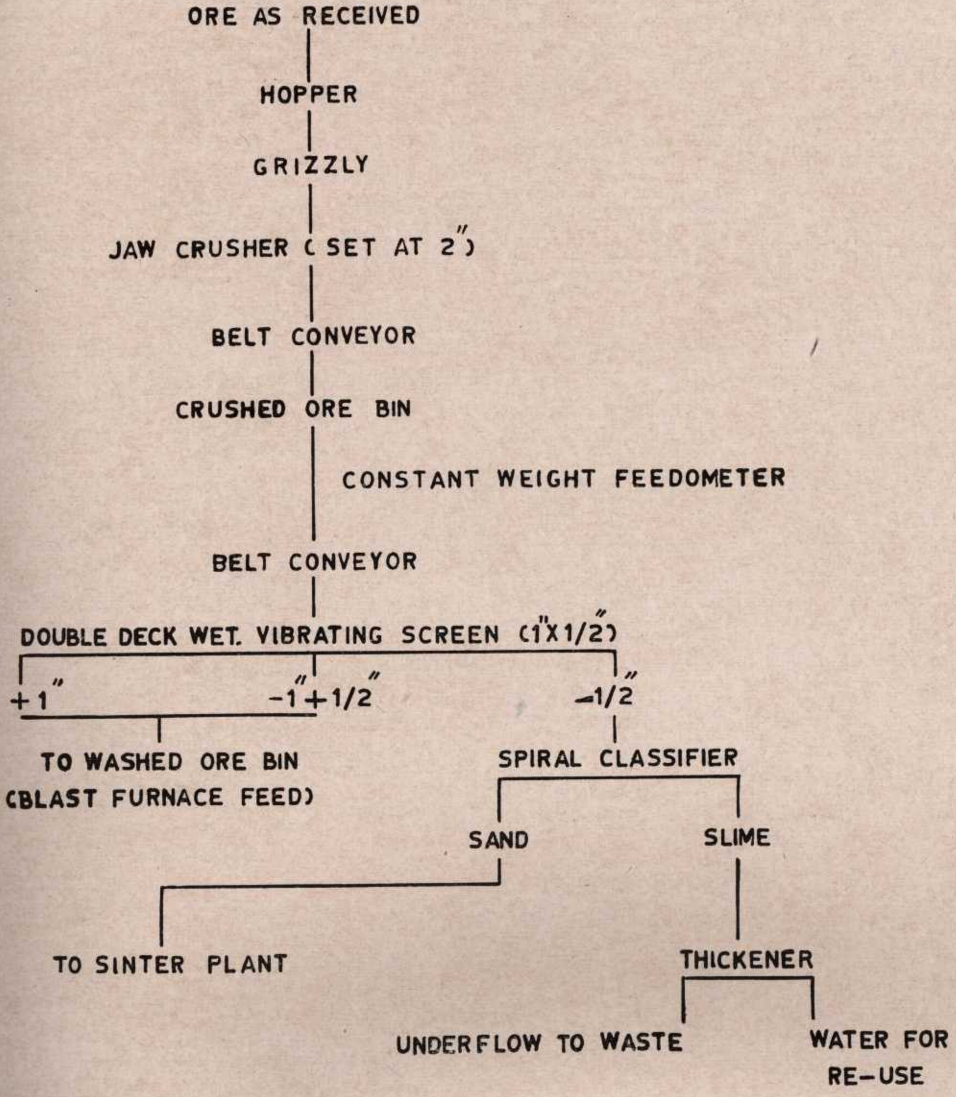


FIG. 8 — FLOWSHEET EMPLOYED FOR THE BENEFICIATION OF MASSIVE IRON ORE SAMPLE FROM DALLI MINES

amount of coke breeze needed for proper sintering in the case of unfluxed sinters and up to 30 per cent in the case of fluxed sinters. The sinters produced by this technique had good strength and possessed optimum metallurgical characteristics as such, higher degree of oxidation than unfluxed sinters.

The flowsheet employed for beneficiation of the sample is given in Fig. 9. A summary of results of washing is given in Fig. 10.

1.5 Pilot Plant Studies on Beneficiation and Sintering of a Sample of Hydroxide Iron Ore from Dalli Mines of Bhilai Steel Plant

A sample of hydroxide ore from Dalli Mines of Hindustan Steel Ltd, received for beneficiation and sintering studies, assayed Fe, 49·6; SiO_2, 7·6 and Al_2O_3, 11·3 per cent;

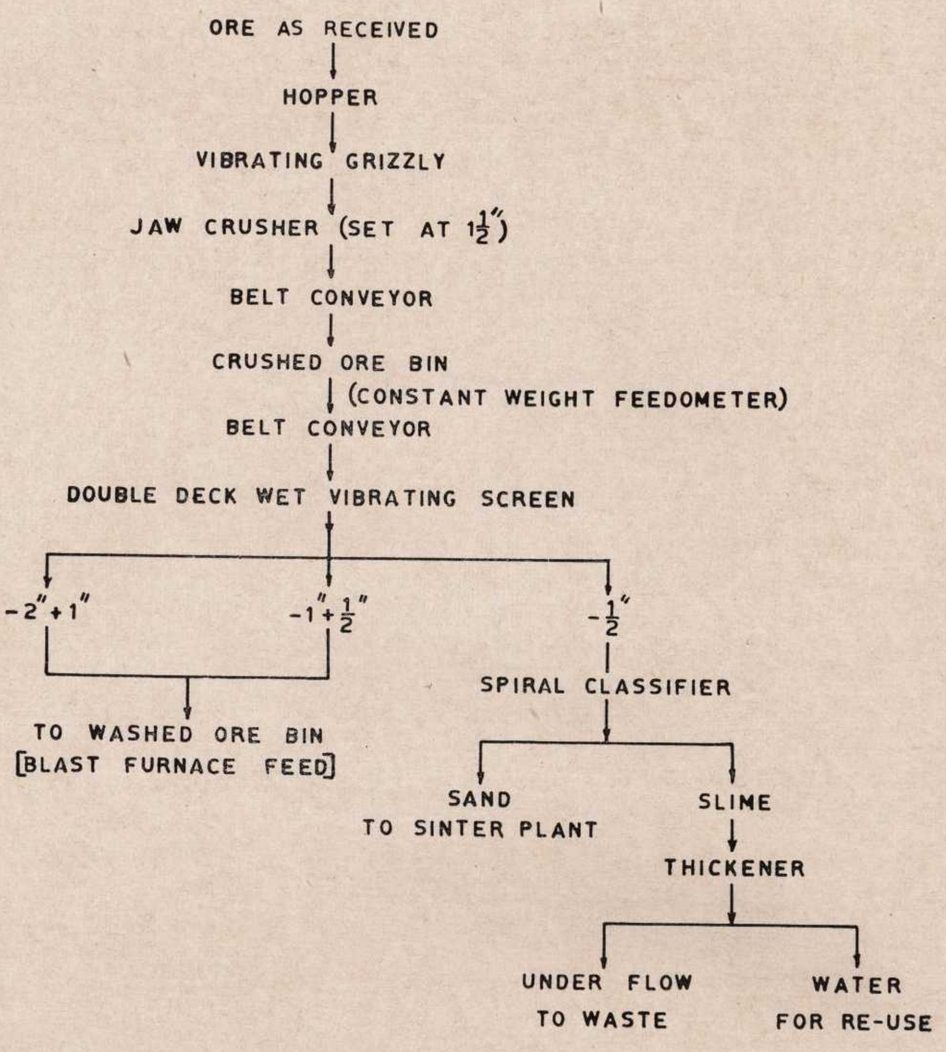


Fig. 9 — Flowsheet Employed for the Beneficiation of Compact Laminated Iron Ore from Dalli Mines

this was the poorest among the 4 samples received from Dalli, as it was very high in its laterite content.

Screenability tests showed that screenability was poorest at a moisture content of 7·5 per cent. Sieve analysis of the run-of-mine sample, which contained lumps 200 mm. to fines, showed Fe values, varying from 53·5 per cent in the +6 in. (+152·4 mm.) fraction to 44 per cent in the $-\frac{1}{2}$ in. fraction. Dry screening, wet screening, and scrubbing followed by wet screening of the sample crushing to 2 in. (50·8 mm.), yielded $+\frac{1}{2}$ in. lump ore fractions assaying 51·7, 52·07 and 52·5 per cent Fe respectively. The three products were not very much different from one another. This was due to fact that the sample received for testing was completely dry and hence, did not create any screening difficulties. But during rainy season, the ore would become very sticky because of high clay and laterite content and thereby create serious difficulties during transporting, screening, etc. However, scrubbing followed by wet screening of the crushed ore yielded

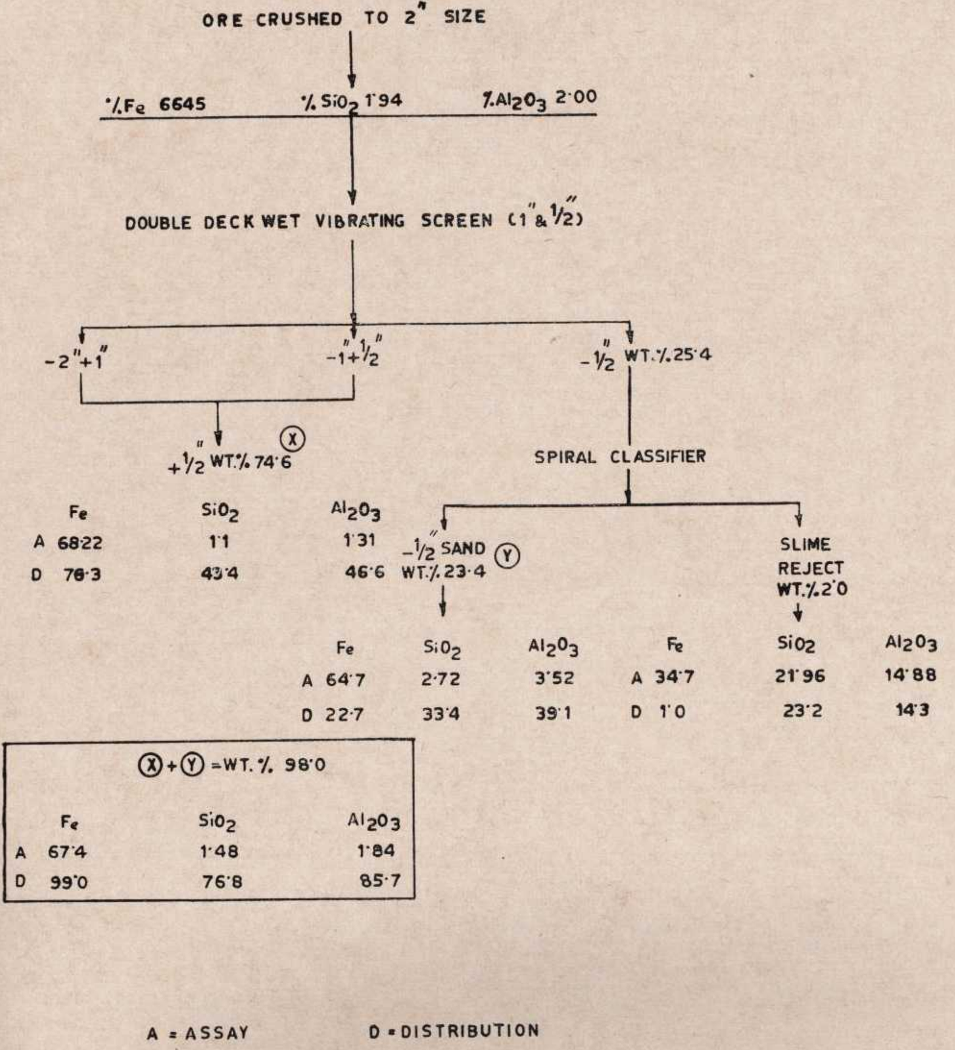


FIG. 10 — SUMMARY OF RESULTS OF BENEFICIATION OF COMPACT LAMINATED ORE FROM DALLI MINES

the best result. Only 15·2 per cent of total SiO_2 and 13·2 per cent of total alumina present in the sample were rejected in the slime. As the original sample itself was highly lateritic and of a low grade, washed lumps and classifier sand were also high in SiO_2 and Al_2O_3 contents, necessitating further upgrading employing gravity methods.

Heavy media separation of washed lumps, improved its grade to Fe, 63·3; SiO_2, 2·1 and Al_2O_3, 2·56 per cent but with an iron recovery of only 19·2 per cent with respect to the original sample. The high loss of iron in the tailing (float product) was due to the original sample being of a low grade and high in its laterite content. The silica and alumina contents of the classifier sand, obtained after scrubbing and wet screening, were reduced from 6·96 and 11·68 per cent to 4·2 and 7·1 per cent, respectively, in a combined jig concentrate assaying 53·7 per cent Fe with an iron recovery of only 12·4 per cent with respect to the original sample. The low Fe recovery in this case was also due to the reasons stated above.

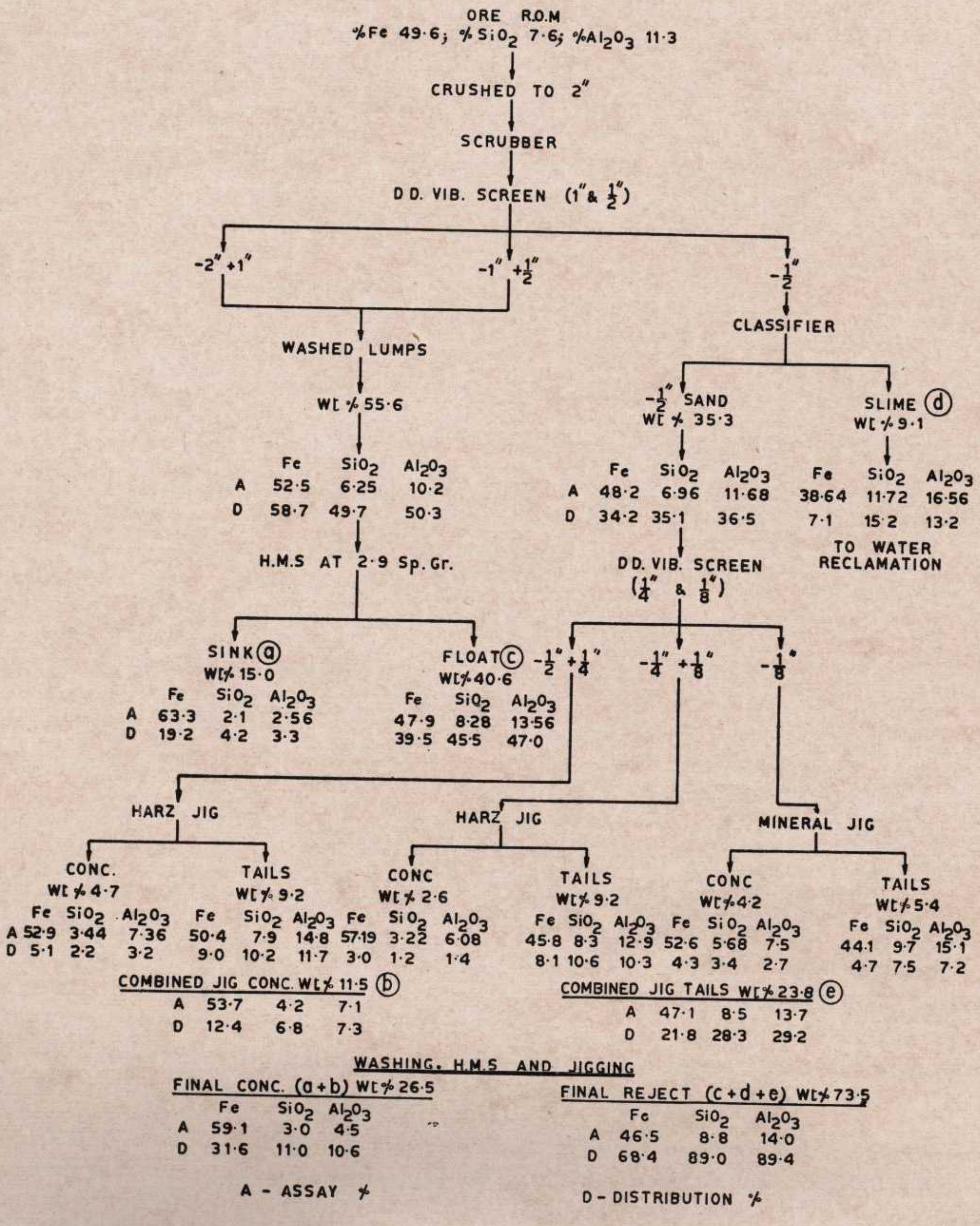


FIG. 11 — SUMMARY OF RESULTS OF WASHING AND UPGRADING OF HYDROXIDE IRON ORE FROM DALLI MINES

Sintering characteristics of −½ in. jig concentrate obtained from the classifier sand after washing the ore at −2 in. size were studied for making unfluxed as well as fluxed sinters. In the case of unfluxed sinters, it was found that 7 per cent water and 4·5 per cent coke were the optimum values for producing a strong sinter, although metallurgically unfluxed sinter was not desired. Good quality fluxed sinter up to a basicity of 2·03 could be produced by suitable additions of limestone. The results further showed that solid fuel could be partially substituted by gaseous fuel

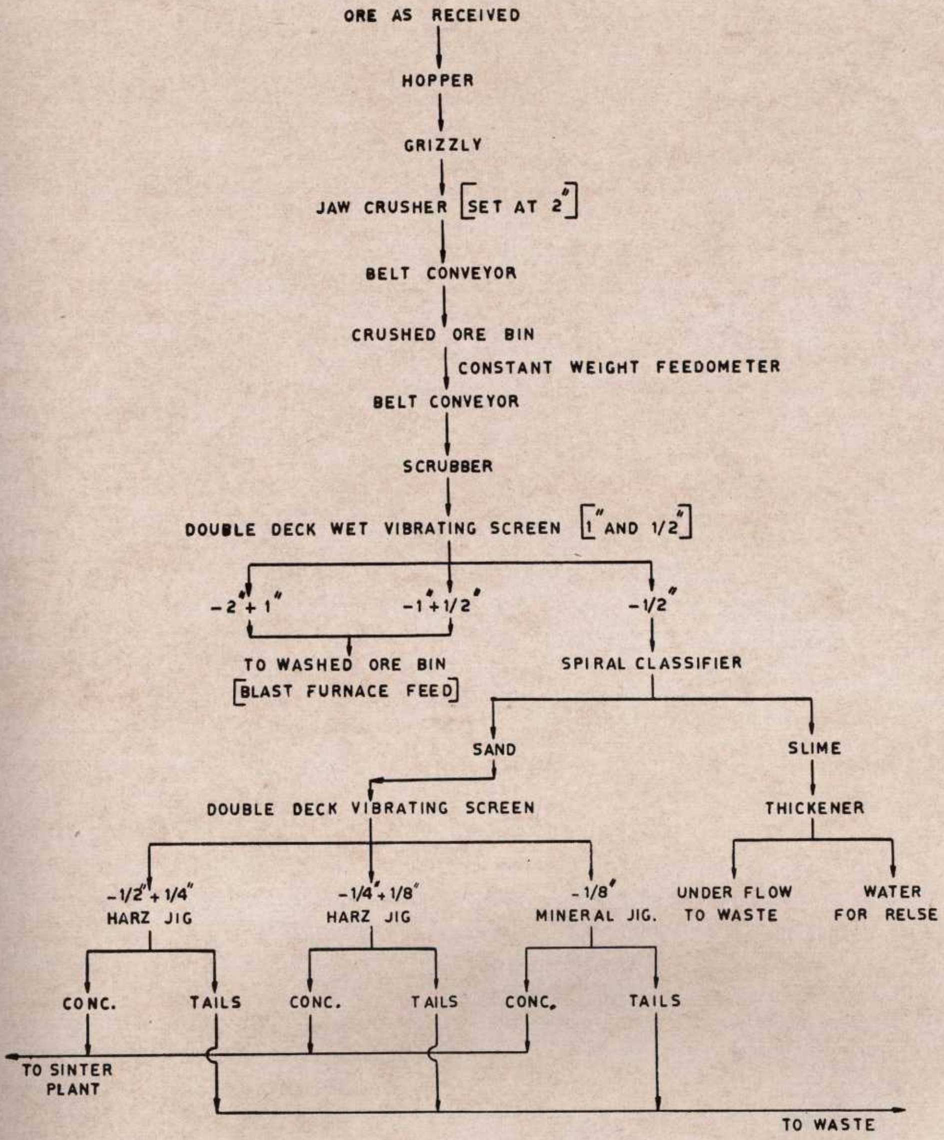


FIG. 12 — FLOWSHEET FOR BENEFICIATION OF HYDROXIDE IRON ORE FROM DALLI MINES

(producer gas) up to 30 per cent of the total amount of coke needed for proper sintering in case of unfluxed sinters and up to 20 per cent in case of fluxed sinter. The sinters thus produced were of good quality and high strength and also possessed a high degree of oxidation when compared with those made by conventional sintering process. Weathering tests performed with a sinter of basicity 2·03 showed that the amount of $-\frac{1}{2}$ in. (−12·7 mm.) fines generated during storage for 24 hours was 2·9 per cent by weight.

A summary of results of washing, followed by upgrading of washed products, is given in Fig. 11. The flowsheet employed for beneficiation of the sample is given in Fig. 12.

1.6 Beneficiation and Pelletization Studies on Iron Ore Fines from Redi Mines, Ratnagiri District, Maharashtra

A sample of iron ore fines from Redi Mines, Ratnagiri District, Maharashtra, was received for beneficiation and pelletizing studies. The sample assayed Fe, 63·0; SiO_2, 1·5; Al_2O_3, 2·0 and CaO, 0·1 per cent, and MgO trace, S trace, P, 0·01 and L.O.I., 5·7 per cent. Normally a sample of this nature, low in insolubles and high in combined water, may not need beneficiation before pelletization, but washing test was performed to see the extent to which the clayey gangue (laterite) could be eliminated in the event of possible contamination with laterite during mining.

Simple washing of the ore and cyclone treatment of the slime yielded beneficiated fines assaying Fe, 64·4; SiO_2, 1·3 and Al_2O_3, 1·7 per cent with a recovery of 90·3 per cent Fe in the lumps and fines. Though the grade of beneficiated fines in relation to the insolubles had not appreciably improved after scrubbing, wet screening and cycloning, this treatment had, however, rejected a slime fraction containing the bulk of laterite contaminating the ore. The +9·5 mm. lumpy fraction assaying 62 per cent Fe and constituting 12·2 per cent by weight, was separated for marketing as lumpy ore.

The concentrate fines were ground to three different sizes, viz. 69, 78 and 82 per cent −270 mesh, in a ball/rod mill. Extensive studies on pelletization with these three grinds, both in a batch drum pelletizer and in a continuous disc pelletizer, indicated that strong pellets could not be produced without addition of binder. Addition of 0·5 per cent bentonite as a binder considerably improved the strength of the pellets. It was further observed that the feed to the pelletizer need not be ground finer than 78 per cent −270 mesh. The green pellets were strong enough to withstand rough handling during conveying from the pelletizer to the firing equipment. Firing of the pellets at about 1350°C. for heat-hardening yielded strong pellets satisfactory for handling and transport over long distances. The fired pellets assayed Fe, 66·3; FeO, 0·6; SiO_2, 1·4 and Al_2O_3, 2·0 per cent and had good physical properties. These pellets should make an ideal feed for iron smelting in a blast furnace.

1.7 Pilot Plant Studies on Beneficiation and Sintering on a Sample of Iron Ore from Meghataburu Mines of N.M.D.C. Ltd

A 100 tonnes representative sample of run-of-mine ore from Meghataburu Iron Ore Mines received for beneficiation tests assayed Fe, 61·87; SiO_2, 2·08; Al_2O_3, 4·00 and L.O.I., 5·7 per cent.

Scrubbing followed by wet screening of the crushed ore (at −2 in. size) yielded a clean sized washed product of $+\frac{3}{8}$ in. size (+9·5 mm.) free from adherent ore fines for direct charging into the blast furnace and for export purposes and $-\frac{3}{8}$ in. (−9·5 mm.) slime-free classifier sand product for sintering. The results obtained from both wet screening tests as well as scrubbing followed by wet screening were more or less identical but were better than those obtained by dry screening. The washed lumps assayed Fe, 63·60; SiO_2, 1·2 and Al_2O_3, 3·36 per cent; 32·3 per cent of the total SiO_2 and 18·1 per cent of the total Al_2O_3 could be rejected in slime weighing 6·7 per cent. Though the insoluble contents of the $-\frac{3}{8}$ in. (−9·5 mm.) classifier sand obtained after washing was not considered high, it was subjected to jigging. The combined jig concentrate assayed Fe, 62·50; SiO_2, 2·33 and Al_2O_3, 2·90 per cent with an iron loss in the jig tailing of only 2·8 per cent with respect to the original. This indicated the beneficial effects of jigging the fines before sintering.

The sintering characteristics of the $-\frac{3}{8}$ in. ($-9{\cdot}5$ mm.) washed and beneficiated jig concentrate were studied for making fluxed and unfluxed sinters. The optimum water, coke and sinter return fines were found to be 7, 4·5 and 25 per cent, respectively, in the case of unfluxed sinters. Good quality superfluxed sinters up to basicity ratio 2·4 could be produced by suitable additions of limestone.

The results further showed that coke breeze used for sintering could be substituted by gaseous fuel (producer gas) to the extent of 30 per cent of the total coke needed. The sinters produced by such mixed firing technique were of good quality and strength and also possessed a very high degree of oxidation when compared with those made by the conventional sintering process. Mixed firing technique was also adopted to make sinter with basicity ratio up to 1·8.

1.8 Pilot Plant Studies on Beneficiation and Sintering of a Mixed Laminated Iron Ore Sample from Rajhara Mines of Bhilai Steel Plant, Hindustan Steel Ltd

Three samples of iron ore (2 laminated iron ore and 1 massive ore) were received from Rajhara Mines of Bhilai Steel Plant, for detailed beneficiation and sintering studies at the National Metallurgical Laboratory. The two laminated iron ore samples were mixed in the proportion of 9·1 and a mixed sample prepared for tests. The representative sample of mixed laminated iron ore assayed Fe, 62·2; SiO_2, 3·65; Al_2O_3, 3·47 and L.O.I., 4·5 per cent.

Screenability tests with varying moisture contents in the ore indicated that screening efficiency would be minimum when the moisture content was 10 per cent. Dry screening tests performed after crushing the sample to -2 in. size ($-50{\cdot}8$ mm.) showed that the $+\frac{3}{8}$ in. ($+9{\cdot}5$ mm.) lumps, constituting 46·6 per cent by weight, assayed Fe, 63·8; SiO_2, 1·4 and Al_2O_3, 2·51 per cent. Washing test (scrubbing followed by wet screening) carried out with the sample at -2 in. ($-50{\cdot}8$ mm.) size indicated that a clean sized, washed product free from adhered ore fines and $-\frac{3}{8}$ in. ($-9{\cdot}5$ mm.) classifier sand product free from undesirable slime could be obtained. The results showed that (i) the improvement in the Fe grade of washed lumps was 2 per cent, and (ii) the rejection of SiO_2 and Al_2O_3 through slime was 51·7 and 34·2 per cent, respectively, for a corresponding Fe loss of 15·1 per cent.

The $-\frac{3}{8}$ in. ($-9{\cdot}5$ mm.) classifier sand was almost slime-free and was of a better grade than the dry screened fines, and assayed 63·5 per cent Fe with 5·7 per cent insolubles. The washed sand being of a good grade could be used for sinter production as such. However, attempts were made to see if the SiO_2 and Al_2O_3 content could be brought down by jigging. Jigging of the above product yielded a combined jig concentrate constituting 26·9 per cent by weight and assaying 65 per cent Fe with 4·16 per cent insolubles. The slime obtained from washing constituted 17·3 per cent by weight. It assayed Fe, 54·2; SiO_2, 10·4 and Al_2O_3, 6·8 per cent. The loss of Fe in the slime was 15·1 per cent. Attempts were, therefore, made to concentrate the slime by employing a cyclone which gave a concentrate constituting 12 per cent by weight with respect to original and assaying Fe, 63·65 and 5·16 per cent insolubles recovering 12·4 per cent of Fe from a total of 15·1 per cent. This product could be mixed with the $-\frac{3}{8}$ in. washed sand for sinter production.

Sintering characteristics of $-\frac{3}{8}$ in. ($-9{\cdot}5$ mm.) washed classifier sand, both unbeneficiated and beneficiated but without the slime concentrate, were studied for making fluxed as well as unfluxed sinters. Good metallurgical grade fluxed sinter up to a basicity ratio of 2·4 could be produced from unbeneficiated fines whereas a basicity ratio of even 2·7 could be reached with beneficiated ore-fines. The results further showed that solid fuel coke could be substituted by gaseous fuel (producer gas) up to 30 per cent of the total amount of coke needed for proper sintering, for high basicity, and even up to 40 per cent for basicity of 1·6 with beneficiated fines. The sinters thus produced were of good quality and high strength and possessed a high degree of oxidation when compared with those made by conventional sintering process.

1.9 Pilot Plant Studies on Beneficiation and Sintering Characteristics of a Mixed Sample of Massive and Laminated Iron Ore from Rajhara Mines of Bhilai Steel Plant, Hindustan Steel Ltd

Three samples of iron ore, viz. (i) soft-laminated iron ore reported to have been collected from pits No. 1, 2, 4, 5 and 6, (ii) compact-laminated iron ore reported to be from pit No. 3, and (iii) massive iron ore, were received at National Metallurgical Laboratory from Rajhara Mines of Bhilai Steel Plant. Tests were carried out on a 40 tonnes mixed sample prepared in NML by mixing the massive iron ore [sample (iii)] with mixed laminated iron ore [samples (i) and (ii) mixed in the ratio 9: 1] in the proportion of 45: 55. The mixed sample of massive and laminated ore was of a good grade assaying Fe, 65·6; SiO_2, 2·1; Al_2O_3, 2·51 and L.O.I., 2·7 per cent.

Screenability tests showed that screenability was least efficient at 5·0 per cent moisture. Dry screening (i), and scrubbing (ii), followed by wet screening (washing) of the sample after crushing to 2 in. (50·8 mm.) size yielded $+\frac{3}{8}$ in. (+9·5 mm.) lump ore fraction assaying 67·0 and 67·5 per cent Fe respectively. The two products were not very much different from each other. This was due to the fact that the sample received for testing was completely dry and hence, did not create any screening difficulty. But during rainy season, the ore would become wet and sticky and thereby create serious screening difficulties. However, scrubbing followed by wet screening at 2 in. size yielded better results. The $+\frac{3}{8}$ in. (+9·5 mm.) washed lumps constituting 59·5 per cent by weight assayed 67·5 per cent Fe, with 2·28 per cent insolubles. About 48·8 per cent of the total SiO_2 and 34·8 per cent of the total Al_2O_3 present in the sample were rejected in the slime for a corresponding Fe loss of 8·8 per cent only. The $-\frac{3}{8}$ in. (−9·5 mm.) washed classifier sand was almost slime-free and was of a better grade than the dry screened $-\frac{3}{8}$ in. fraction. The product assayed 65·7 per cent Fe with 4·52 per cent insolubles, was of a good grade and its further upgrading for sinter production by jigging was considered unnecesssary. The rejection of slime by washing was 10·6 per cent by weight. The slime, assaying 4·04 per cent Fe, contained a high percentage of insolubles (SiO_2, 10 $+Al_2O_3$, 8·06 per cent) with a Fe loss of 8·8 per cent. Attempts were, therefore, made to beneficiate the same by cyclone treatment. The concentrate (cyclone underflow) obtained was 7·0 per cent by weight with reference to original and assayed 64·95 per cent Fe and 5·16 per cent insolubles with a Fe recovery, with reference to original, of 7 per cent. This concentrate could be mixed with the washed sand for sinter production. The loss of Fe in the cyclone overflow was only 1·8 per cent.

Sintering characteristics of $-\frac{3}{8}$ in. (−9·5 mm.) washed classifier sand but without the cyclone concentrate were studied for making fluxed as well as unfluxed sinters. It was found that 7 per cent water and 5 per cent coke were the optimum values for producing unfluxed sinters. Good quality fluxed sinters up to a basicity of 2·4 could be produced by suitable addition of limestone. The results further showed that solid fuel coke could be substituted by gaseous fuel (producer gas) up to 30 per cent of total amount of coke needed for proper sintering. Sinter thus produced were of good quality and also possessed a high degree of oxidation when compared with those by the conventional sintering process. Weathering tests with highly basic sinters showed that the percentage of fines produced during the period of observation was low or within tolerable limit.

1.10 Pilot Plant Studies on Pelletization of Mixed Iron Ore Sample from Goa

Four different iron ore (lumpy) sample were received from M/s. Salgoakar, Goa. The samples are from Sallitho, Surla, Pale and Velguem mines, weighing 31, 25, 17 and 17 tonnes respectively. The problem was to find out whether these ores were amenable to pelletization and also whether any beneficiation was at all needed before the pelletization. The samples consisted of −6 in. lumps. The four samples were mixed up in the ratio of 48: 40: 26: 26 as desired by the firm. The mixed sample was crushed to about $-\frac{3}{8}$ in. size. The crushed material was divided into two representative halves. One half was taken for jigging and the other half was kept for cycloning. The results showed that jigging was quite effective in obtaining concentrate with high iron value and low alumina. The cyclone treatment consists of grading the ore to −20 mesh, and rejecting the low-grade cyclone overflow. Further work is in progress.

1.11 Beneficiation of Iron Ore from Kudermukh, Mysore

The sample as received assayed Fe, 42·17; FeO, 4·34; SiO_2, 36·29; Al_2O_3, 1·06; S, 0·074; P, 0·028 and L.O.I., 2·71 per cent. Magnetite, hematite and goethite were the chief iron minerals and these were free from quartz, the principal gangue, at about 100 mesh. Straight tabling at 48 mesh size yielded a table concentrate assaying Fe, 63·66 and SiO_2, 7·2 per cent with a recovery of 71·7 per cent Fe in it. Spiralling followed by tabling of the spiral tailing yielded a combined concentrate assaying Fe, 60·63 and SiO_2, 9·9 per cent with 73·2 per cent Fe recovery. Straight flotation using petroleum sulphonate at a *p*H of 3·5 followed by one cleaning yielded a concentrate assaying Fe, 63·8 and SiO_2, 5·8 per cent with a recovery of 87·5 per cent Fe. Wet magnetic separation followed by flotation of the non-magnetic fraction, at *p*H 3·5 as before, yielded a mixed concentrate assaying Fe, 62·9 and SiO_2, 6·2 per cent with a recovery of 90·1 per cent Fe in the product. Further work is in progress.

1.12 Beneficiation of Iron Ore from Goalpara, Assam

The sample as received assayed Fe, 46·45; FeO, 4·41; SiO_2, 32·0; Al_2O_3, 0·60; CaO, 0·22; MgO, 0·60; S, 0·16 and P, 0·07 per cent. Straight tabling at −48 mesh after hydroclassification yielded a combined concentrate assaying 66·8 per cent Fe with a recovery of 89·3 per cent Fe. Flotation using starch as depressant for iron minerals and aeromine 3037 as the collector for gangue yielded a tailing assaying 67·4 per cent Fe with a recovery of 93·4 per cent Fe. Flotation using sodium sulphonate at *p*H 4 yielded a concentrate assaying 66·21 per cent Fe with a recovery of 79·4 per cent Fe. Spiral and table treatment of the sample at −48 mesh size after hydroclassification yielded a combined concentrate assaying 66·92 per cent Fe with a recovery of 90·9 per cent Fe in it.

2.0 Flotation Studies on a Copper Ore Sample from Ghatiwalli Adit, Khetri, Rajasthan, for National Mineral Development Corporation

At the instance of National Mineral Development Corporation Ltd flotation studies were undertaken in the National Metallurgical Laboratory on a copper ore sample from the Ghatiwalli adit of Khetri Project, Rajasthan.

The sample as received assayed Cu, 0·99; Fe, 23·1; S, 6·08; SiO_2, 42·38, Al_2O_3, 11·4; and MgO, 5·96 per cent, and 0·14 dwt/T Au and 2·26 dwt/T Ag. The principal opaque minerals observed in the sample were pyrrhotite, magnetite, chalcopyrite and pyrite in order of predominance followed by traces of chalcocite. The bulk of the ore was composed of siliceous gangue. Chalcopyrite was mostly free below 150 mesh size.

Grindability studies employing feeds of fineness, namely 59, 72 and 83 per cent —200 mesh, indicated that a 72 per cent —200 mesh feed was optimum for maximum recovery of copper in the concentrate when 0·1 lb./T of potassium ethyl xanthate and 0·074 lb./T of pine oil were used during roughing. The optimum *p*H for effecting maximum recovery of copper during roughing was found to be 7·6. Increase or decrease in the collector addition did not improve the results, indicating that 0·1 lb./T was the optimum. Rougher flotation, under optimum conditions, followed by three cleanings of the bulk concentrate yielded a product assaying Cu, 20·46; Fe, 33·3 and S, 30·1 per cent, and 2·0 dwt/T Au and 23·6 dwt/T Ag with recoveries of Cu, 82·0; Ag, 41·8 and Au, 57·1 per cent in the final concentrate. Regarding the rougher concentrate to —200 mesh followed by three cleanings at a *p*H of 9·5 improved the grade of copper to an appreciable extent. The concentrate assayed Cu, 29·72; Fe, 31·1 and S, 33·4 per cent, and 2·2 dwt/T Au and 27·8 dwt/T Ag with a copper recovery of 80·2 per cent. Recirculation of the cleaner tailings, as would be the case in industrial scale practice, is expected to yield better recovery. The concentrate and the third cleaner tail when combined would improve the recovery of copper to 84·3 per cent in a concentrate of grade Cu, 24·6; Fe, 30·4 and S, 28·5 per cent.

2.1 Flotation Studies on a Copper Ore Sample from Shaft No. 3, Khetri, Rajasthan, for National Mineral Development Corporation

One ton of low-grade copper ore sample from shaft No. 3 of Khetri mines, Rajasthan, assaying Cu, 0·79; Fe, 20·16; S, 2·85; SiO_2, 52·82 and Al_2O_3, 10·2 per cent, and Trace-CaO, MgO, 5·09 per cent, 0·08 dwt/T Au and 1·7 dwt/T Ag., was received from the National Mineral Development Corporation for flotation studies, for production of a concentrate suitable for use in the flash smelter to be set up at Khetri, Rajasthan.

Petrological examination of the sample indicated the presence of magnetite as the predominant opaque mineral followed by chalcopyrite, pyrrhotite and pyrite and traces of marcasite. The siliceous gangue was chiefly composed of quartz, chlorite, ferro-magnesium minerals (chiefly enstatite) and biotite in the order of abundance. The chalcopyrite was found to be fairly liberated only below 150 mesh. Grindability tests on the sample showed that the ore was of a medium nature between medium hard and medium soft, according to hardness classification of Denver Equipment Co., U.S.A. (Fig. 13).

Maximum recovery of copper value (about 96 per cent) was obtained at natural *p*H (7·9) in the Fagergren cell employing an optimum grind of 69·5 per cent —200 mesh and using 0·1 and 0·07 lb./T of potassium ethyl xanthate as collector and pine oil as frother, respectively. Two-cycle locked tests with one, two and three cleanings

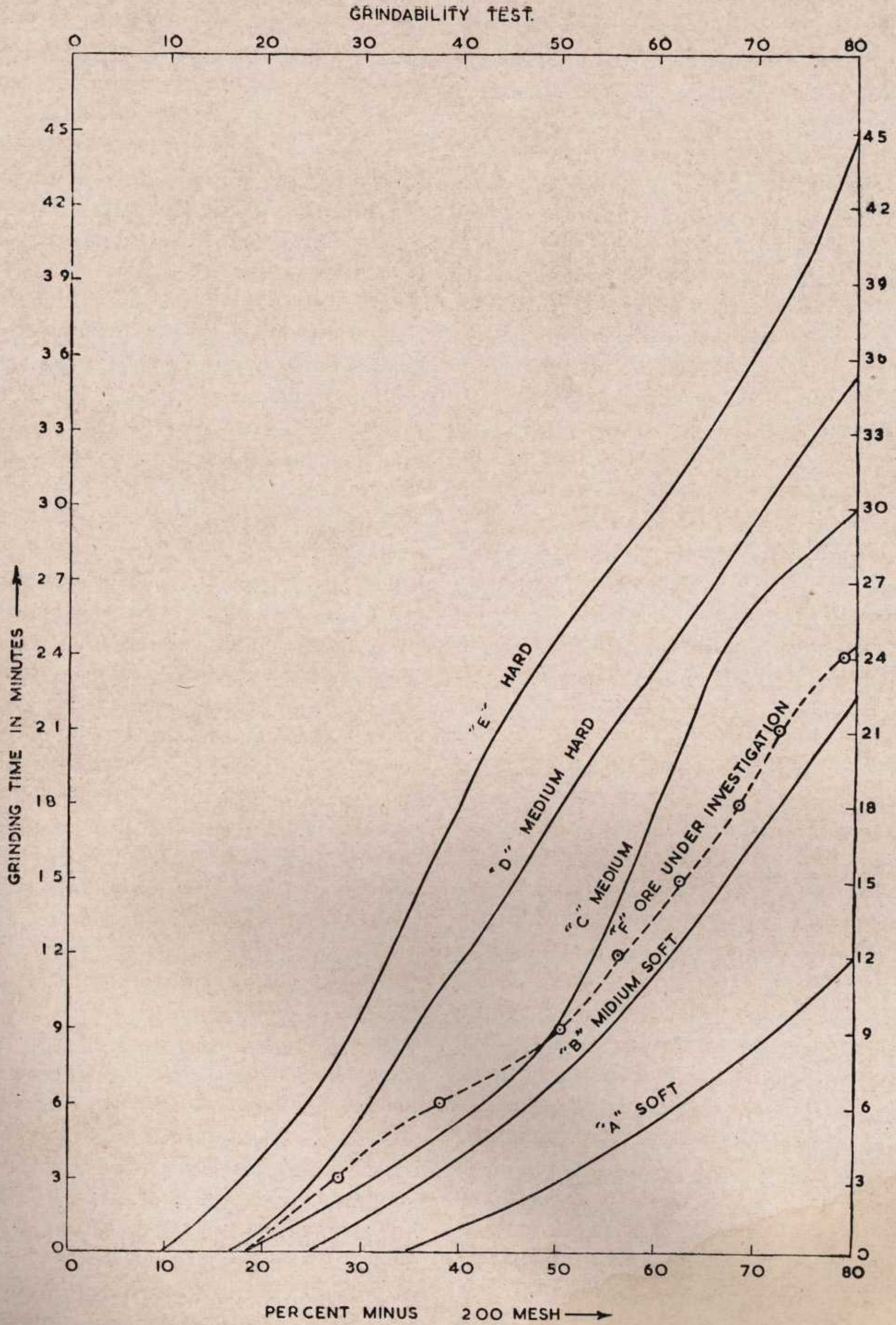


FIG. 13 — GRINDABILITY TESTS OF COPPER ORE FROM KHETRI

produced concentrates assaying between 7 and 15 per cent Cu with recoveries ranging from about 88 to 63 per cent Cu, respectively. The object of the locked tests was to determine the effect of returning the middling products to the flotation circuit to simulate plant practice. Locked tests employing two cleanings with two composite samples prepared by mixing 10 and 25 per cent of Kolihan copper ore (2·69 per cent Cu) with the ore under investigation, respectively, did not improve the grade of concentrate to any appreciable extent.

As suggested by French consultants to the National Mineral Development Corporation, fresh flotation tests were also carried out to obtain a bulk concentrate assaying about 9 per cent Cu with silica content below 10 per cent. A bulk concentrate, assaying Cu, 9·77; Fe, 36·97; S, 28·29; SiO_2, 14·60; Al_2O_3, 2·8; CaO, 0·62 and MgO, 1·9 per cent, was obtained by rougher flotation in a Denver No. 7 flotation cell under optimum conditions, with a recovery of 92 per cent Cu. This bulk concentrate was refloated to yield a final bulk concentrate, assaying Cu, 12·28; Fe, 37·39; S, 34·02; SiO_2, 8·58; Al_2O_3, 1·04; CaO, 0·33; MgO, 1·17; dwt/T Au 0·8 and dwt/T Ag, 8·9 per cent with a recovery of 90 per cent Cu. This product should be a suitable feed to the proposed flash smelter to be set up at Khetri, Rajasthan.

Setting and filtration tests were carried out with (i) Selective Cu concentrate samples (about 15 per cent Cu) obtained from locked tests, (ii) Composite samples representing final tailings obtained from locked tests, (iii) Bulk concentrate assaying 9·77 per cent Cu, and (iv) Rougher tailing as obtained in (iii). In case of (i) and (ii) use of separan increased the settling rate of the tailing considerably and to a lesser extent in case of concentrate. In case of (iii) and (iv) use of separan up to 0·02 lb./T increased the settling rate of the concentrate by about 2·5 ft/hr and by about 0·5 ft/hr with the tailing. The filtration rates for the bulk concentrate and rougher tailing varied from 0·38 to 2·33 tons and 0·53 to 2·3 tons/ft^2 of filter area per 24 hours respectively.

2.2 Flotation Studies on a Copper Ore Sample from Kolihan, Rajasthan, for National Mineral Development Corporation

At the instance of the National Mineral Development Corporation, flotation studies were undertaken in the National Metallurgical Laboratory on a copper ore sample from Kolihan, Rajasthan. As received the copper ore assayed Cu, 2·69; Fe, 15·5; S, 8·18; SiO_2, 50·18; Al_2O_3, 8·87; dwt/T Ag 2·6 and dwt/T Au 0·19 per cent.

The sample contained chalcopyrite and pyrrhotite in almost equal proportions. Other metallic minerals present in minor amounts were magnetite and pyrite. The principal non-metallic minerals present were quartz and chlorite. Liberation of chalcopyrite occurred at about −150 mesh size. Grindability tests carried out with the ore followed by comparison with the standard graph indicated that the sample could be designated as a medium ore, according to the classification of Denver Equipment Co., U.S.A. For flotation tests a grind with 52·2 per cent −200 mesh was found to be optimum.

For maximum recovery of copper during roughing, 0·07 kg./ton of potassium ethyl xanthate and 0·01 kg./ton of pine oil were necessary. Flotation followed by one cleaning of the rougher float yielded a final concentrate assaying Cu, 27·41; S, 33·7; Fe, 31·6; Al_2O_3, 1·42; MgO, 0·2; CaO, 0·16 and SiO_2, 2·17 per cent, and 2·0 dwt/T Au and 13·5 dwt/T Ag with recoveries of Cu, 93·8; Au, 96·8 and Ag, 47·7 per cent, respectively. The copper recovery would be expected to improve further in actual plant practice when the cleaner tailings would be recirculated.

The optimum *p*H for roughing and cleaning was found to be 9·5 which was maintained by addition of lime. Flotation of the copper tailings in acid circuit at a *p*H of 5·8 followed by one cleaning using the same collector and frother produced a pyrrhotite-pyrite concentrate assaying 38·8 per cent S with a recovery of 40·4 per cent S. The ore being rich in pyrrhotite, a concentrate with higher sulphur value was unlikely. Setting and filtration tests with the concentrate as well as tailing had shown that these products could be made to settle fast with flocculant and be filtered at a high rate.

2.3 Flotation Studies on a Copper Ore Sample from Rakha Mines, Bihar, for National Mineral Development Corporation

At the instance of National Mineral Development Corporation Ltd flotation studies were undertaken in the National Metallurgical Laboratory on a copper ore sample from Rakha Mines in Singhbhum district, Bihar.

The sample as received assayed Cu, 2·73; Fe, 5·54; SiO_2, 73·08; Al_2O_3, 10·14; S, 3·96; Mo, 0·025; Co, 0·02; Ni, 0·072 and U_3O_8, 0·030 per cent. Bulk of the ore was composed of quartz and chlorite. The chief copper mineral was chalcopyrite which was well liberated from gangue at about 100 mesh. Pyrite, magnetite and pyrrhotite were present in smaller amounts. Grindability tests carried out with the ore and compared with standard graphs indicated that the sample could be designated as a medium to medium-soft ore, according to the classification of Denver Equipment Co., U.S.A.

Optimum conditions for rougher flotation of copper were determined using K.E. xanthate as collector and pine oil as frother. Tests employing feeds of different fineness indicated that a flotation feed containing 54·8 per cent −200 mesh gave the optimum grind for rougher flotation. Optimum *p*H for rougher flotation was found to be 8·6-8·8 and the rougher float obtained with this *p*H assayed 24·3 per cent Cu and represented a recovery of 96·9 per cent Cu in the product. Use of higher xanthate in place of K.E. xanthates improved the grade and recovery of Cu in the rougher float to a little extent. Use of methyl isobutyl carbinol (MIBC) in place of pine oil resulted in slightly improved grade of concentrate for nearly the same recovery in the float. The rougher float assayed 25·26 per cent Cu with a recovery of 97·1 per cent in the product.

Since the copper minerals in the sample exhibited natural flotability, it was noticed that pine oil addition alone would be sufficient to recover bulk of the Cu minerals in the float. Use of small amount of K.E. xanthate together with frother in the last stages would effect the recovery of most of the remaining copper in the tailing. The mixed rougher float assayed 24·25 per cent Cu with a recovery of 97·6 per cent Cu. Single reflotation of the rougher float obtained under optimum conditions, yielded a refloat concentrate assaying 29·2 per cent Cu and recovering 85·2 per cent Cu in it. However, regrinding the rougher float to −150 mesh size followed by one cleaning improved the grade of the refloat concentrate to 30·1 per cent Cu with an improved recovery of 90·1 per cent Cu in it. Recirculation of the refloat tailing, as would be the case in practice, to the circuit is expected to improve the recovery of copper in the concentrate further. Practically all the MoS_2 present in the sample, reported in the refloat concentrate, assayed 0·32 per cent Mo.

Since Mo in the copper concentrate was in the form of molybdenite, attempts were next made to produce a Mo concentrate. The copper refloat concentrate was subjected to five cleanings when a Mo concentrate assaying 35·95 per cent Mo (MoS_2, 60 per cent), and with a recovery in the product of 68·4 per cent Mo was obtained. Recirculation of the refloat tailings back to the circuit is expected to improve the recovery of Mo in the concentrate. Further cleanings of the Mo float could not be performed for want of sufficient quantity of the Mo float for reflotation. MoS_2 being an easily flotable mineral and capable of easy separation from chalcopyrite, it could be reasonably expected that a Mo concentrate fulfilling the grade requirements for use in ferro-molybdenum could be produced without difficulty, with still higher recoveries than that indicated above. Filtration tests undertaken with the Cu concentrate had shown that, depending on the percentage solids in the pulp, the rate varied from 2·2 tons to 10·5 tons/ft^2 of filter area per 24 hours. Settling tests with the concentrate as well as tailing had shown that separan would increase settling rate of the concentrate by about 2 ft/hr and to a lesser extent (1 ft/hr) in the case of tailing.

3.0 Production of Upgraded Limestone Pellets for M/s. Tata Iron & Steel Co. Ltd, Jamshedpur

A 1200 tonnes sample of low-grade limestone, assaying CaO, 43·44 and insolubles, 12·56 per cent, was received from M/s. Tata Iron & Steel Co. Ltd for producing upgraded limestone pellets for charging in steel-making furnaces on a trial basis so as to study its effect on the production of steel.

Following the flowsheet developed earlier which broadly comprises grinding the ore to 80 per cent −200 mesh and floating off limestone using sodium oleate as collect ore and sodium silicate as depressant for siliceous gangue; a concentrate, assaying CaO, 50·5 and insolubles, 2·08 per cent, was obtained. Using molasses as binder the concentrate was pelletized. The heat-hardened pellets of optimum strength and size thus produced, assayed CaO, 48·5 and insolubles, 3 per cent. About 264 tonnes of dry pellets were produced out of which about 215 tonnes were supplied to Tisco so far.

3.1 Beneficiation Studies on Limestone Sample from Salem, Madras

A limestone sample with over 10 per cent SiO_2 content was received from the Geologist, Neyveli-Salem Steel Project, Hasthampatti, Salem, for beneficiation tests. The sample as received assayed CaO, 43·05; insolubles, 17·97 and P_2O_5, 0·19 per cent. Straight flotation yielded a concentrate assaying CaO, 51·7; insolubles, 3·28 and P_2O_5, 0·22 per cent; with a recovery of 98·5 per cent CaO.

Variation in feed fineness indicated that a grind with 51·5 per cent −200 mesh would be the optimum. The concentrate assayed CaO, 51·0 and insolubles, 4·70 per cent with a recovery of 99·1 per cent CaO. Flotation tests using varying amounts of sodium silicate as depressant for siliceous gangue indicated that best results were obtained with 0·35 kg./ton of sodium silicate. Flotation followed by one cleaning of the rougher float yielded a concentrate assaying CaO, 52·9 and insolubles, 2·0 per cent with a recovery of 97·1 per cent CaO in it. Further work is in progress.

3.2 Beneficiation Studies on Limestone from Rohtas Industries, Dalmianagar

A limestone sample was received from Rohtas Industries, Dalmianagar, for producing a concentrate suitable for cement manufacture. The sample as received assayed CaO, 38·68; MgO, 3·13; SiO_2, 17·92; Al_2O_3, 4·03; Fe_2O_3, 1·54; Fe, 1·07; CO_2, 30·48; P, 0·065 and total insolubles, 33·84 per cent.

Flotation employing a 96 per cent —200 mesh feed yielded a concentrate assaying CaO, 43·56 and insolubles, 14·52 per cent with a recovery of 90·0 per cent CaO. Flotation followed by two cleanings of the rougher float yielded a concentrate assaying CaO, 47·43; SiO_2, 7·7 and insolubles, 9·55 per cent with a recovery of 85·2 per cent CaO.

4.0 Reduction of Silica Content in a Magnesite Sample from Salem, Madras

A raw magnesite sample, assaying MgO, 45·3; CaO, 1·46; CO_2, 48·5; SiO_2, 3·91; Al_2O_3, 0·3; Fe_2O_3, 0·55 and S, 0·12 per cent, was received from M/s. Dalmia Magnesite Corporation for reducing the silica content in the sample to below 1·5 per cent, and incidentally CaO also as low as possible.

Quartz, serpentine, felspar, ferro-magnesium minerals and opaques were the principal gangue in the sample. The gangue was liberated at about —35 mesh size. Only flotation was employed for elimination of the gangue minerals from magnesite. Rougher flotation was done with a feed containing 76 per cent —200 mesh and using oleic acid emulsion (0·55 kg./ton) and pine oil (0·02 kg./ton). Cleaning of the rougher float produced magnesite concentrates assaying from 0·9 to 1·4 per cent SiO_2 with weight recoveries between 60·3 and 73·4 per cent, depending upon the number of cleaning operations performed. A magnesite concentrate assaying SiO_2, 1·4 and CaO, 0·2 per cent with a yield of 73·4 per cent by weight could be produced by two cleanings of the rougher float. Recirculation of the cleaner tailings, as is done in practice, is expected to yield higher recoveries. Use of cationic reagents for flotation of siliceous gangue did not yield satisfactory results.

4.1 Pilot Plant Studies on Reduction of Silica Content of a Magnesite Sample from Salem, Madras, employing Crushing and Screening Methods

About 48 tonnes of magnesite, assaying 4·1 per cent SiO_2, were received from Salem through M/s. Belpahar Refractories Ltd, to investigate the possibility of reducing the silica content employing only crushing and screening methods. Sieve and chemical analyses of samples (i) as received, (ii) after crushing to about 1 in. (25·4 mm.) size, and (iii) after crusing to about ½ in. (12·7 mm.) size were performed. None of the sieve fractions analysed less than 2 per cent silica indicating that crushing followed by rejection of fine fractions high in silica will not be able to yield a low-silica product acceptable to the refractory industry. The investigation is almost completed.

5.0 Pilot Plant Studies on Beneficiation of a Peroxide Manganese Ore

About 11·3 tonnes of peroxide manganese ore was received to reduce the iron content from 5·32 per cent Fe to about 3·0 per cent Fe. The ore as received assayed Mn, 50·6; Fe, 5·32 and SiO_2 4·72 per cent. The sample was ground to about 35 mesh in the ball mill and passed through drum-type magnetic separator producing a non-magnetic concentrate assaying Mn, 51·94; Fe, 3·69 and SiO_2, 4·39 per cent. In order to lower the Fe content still further, the non-magnetic concentrate was passed through a cross-belt magnetic separator at 3·2 amp. Further work is in progress.

6.0 Beneficiation of Low-grade Kyanite Sample from Barabil, Orissa

A sample of low-grade kyanite was received from S. Lal & Co., Barabil, Orissa.

The sample as received assayed Al_2O_3, 51·6; SiO_2, 41·2; Fe_2O_3, 1·1; TiO_2, 1·4; CaO, 0·44; MgO, 0·41; K_2O_2 and Na_2O, 1·6 and L.O.I, 1·7 per cent. Tabling of classified feed at −35 and −65 mesh sizes did not yield satisfactory results. Variation in the feed fineness during flotation indicated that a grind with 36 per cent −200 mesh was optimum. The flotation concentrate assayed 60·3 per cent Al_2O_3, with a recovery of 67·0 per cent Al_2O_3. Flotation studies at different *p*H showed that the optimum *p*H was 4·9 for best results. Use of 0·2 kg./ton of lactic acid and 0·6 kg./ton of sodium sulphonate during primary flotation followed by 0·15 kg./ton of lactic acid during cleaning yielded a concentrate assaying 60·7 per cent Al_2O_3, with a recovery of 67·4 per cent Al_2O_3. Further work is in progress.

7.0 Pilot Plant Studies on Reduction of Insoluble Content of a Selenite Sample from Rajasthan

A sample of selenite weighing about 3 tonnes was received from M/s. Bikaner Gypsums Ltd, assaying SO_3, 45·16; CaO, 31·7; insolubles, 1·2 and CO_2, 0·29 per cent, and traces of Nacl, MgO, etc. The impurity present was clay as inclusions of pockets and stringers within the crystal aggregates of selenite and are liberated mostly in about 6-10 mm. size. For complete liberation grinding to −28 mesh was indicated. Batch tests carried out showed that bulk of clay could be eliminated on coarse crushing to $-\frac{3}{4}$ in. and crushing followed by desliming. For further improvement finer grinding to about −20 mesh followed by washing was necessary. Based on the batch tests, pilot plant scale tests were conducted. The $-\frac{3}{4}$ in. washed sand assayed 0·63 per cent insolubles which, on further grinding and desliming in two stages, gave two sand products assaying 0·42 and 0·38 per cent insolubles. The slime collected in the two stages assayed average 1·15 per cent insolubles. The mixed sand product representing 73·2 per cent by weight of the original feed assayed 0·41 per cent insoluble corresponding to about 99·6 per cent selenite equivalent and is of an acceptable grade to meet the specific requirement.

8.0 Pilot Plant Studies on Beneficiation of an Apatite Sample from Singhbhum District, Bihar

Four samples of low-grade apatite each weighing about 20 tonnes and drawn from different localities of Singhbhum district, Bihar, were received from the Government of Bihar for beneficiation studies so as to produce a concentrate for use as natural fertilizer. The samples from Itagarh, Garidhi, Samaidhi and Sungri as received contained 15·18, 22·45, 13·65 and 25·87 per cent of P_2O_5, respectively.

To assess the advantage of using an upgraded apatite concentrate rather than the raw apatite by actually using it as fertilizer in field, the Government of Bihar requested NML to supply about 400 kg. of upgraded apatite and 700 kg. of raw apatite ground to −100 mesh. Accordingly, the low-grade apatite sample from Itagarh area was ground to 80 per cent −200 mesh and floated using 0·69 kg./ton soap solution producing 400 kg. of fine concentrate assaying P_2O_5, 30·30 and Fe, 6·72 per cent. The same sample from Itagarh was passed through jaw crusher and roll crusher followed by coffee grinder producing 700 kg. of pulvarized sample of raw apatite ground to 80 per cent −100 mesh. For beneficiation studies on pilot plant scale a mixed apatite sample was prepared by mixing 30 tonnes of ore from Itagarh, 5 tonnes from Garidhi, 9 tonnes from Samaidhi and 12 tonnes from Sungri. Arrangements are in progress to conduct the pilot plant tests.

9.0 Grindability Tests on a Fluorspar Sample from Rajasthan

A sample of fluorspar ore from Mandoki-pal, Dungarpur district, Rajasthan, was received from the Government of Rajasthan for grindability tests in connection with their Fluorspar Project. The samples as received were ground to −10 mesh for grindability tests.

Batches of −10 mesh ore were wet ground for varying lengths of time in a batch ball mill under identical conditions. Sieve analyses of the ground products were performed and the percentage of −200 mesh produced determined in each case.

A curve was drawn on a standard graph by plotting the per cent weight of −200 mesh material produced against corresponding grinding time in minutes and the relative hardness of fluorspar sample compared with standard curves. The curve for the fluorspar sample under investigation was found to lie between two curves, designated as medium soft and medium hard, indicating that the sample could be designated as medium ore (Fig. 14).

10.0 Beneficiation of a Phosphate Rock Sample from Singhbhum District, Bihar

A sample of low-grade phosphate rock was received from M/s. Dhalbhum Phosphate Co. Ltd, Calcutta, to find out whether it could be beneficiated to a grade suitable for superphosphate manufacture.

The sample as received assayed P_2O_5, 23·26 (BPL, 50·8 per cent); SiO_2, 19·31; Fe, 11·32; Al_2O_3, 9·15; CaO, 30·0; F, 2·1 and Cl, 0·24 per cent with traces of Cu and SO_3. Microscopic studies undertaken on a representative −10 mesh sample and its sieve fractions indicated that apatite was the only phosphate mineral present in it and chlorite, felspar, quartz, biotite and magnetite constituted the gangue. It was also noticed that apatite was freed of magnetite even at 28 mesh, whereas it was fairly freed of siliceous gangue only at about 48 mesh. Only soap flotation studies were attempted to separate the apatite from siliceous and magnetite gangue. Oleic acid emulsion was employed as collector for apatite while the gangue was depressed with or without sodium silicate. Variations in size of the flotation feed indicated that grinding the sample to 76·1 per cent −200 mesh would be necessary to attain optimum results. The rougher apatite float assayed 41 per cent P_2O_5, with a recovery of 95·5 per cent P_2O_5 in it.

Studies on the effect of sodium on the flotation of apatite indicated that addition of this reagent slightly improved the grade of apatite float, but the recoveries of P_2O_5 in the floats were lower. Use of pine oil in conjunction with collector showed the tendency for some of the magnetite to float along with apatite and hence the grade of the rougher float dropped to 35·2 per cent P_2O_5. One cleaning of the rougher float yielded a refloat apatite concentrate assaying P_2O_5, 41·7; SiO_2, 0·65; Fe, 0·4; Al_2O_3, 1·8; F, 2·9 per cent and CO_2 trace with a recovery of 84·4 per cent P_2O_5 in it. This recovery should improve in practice by recirculation of the refloat tailing back to the circuit. The concentrate fulfills the grade and size requirements laid out for its use in superphosphate manufacture.

11.0 Separation of Mica from Jamuna Sand received from National Building Organization, New Delhi

A sample of Jamuna sand was received from National Building Organization, for removal of mica from the sand. Flotation test in the acid range (*p*H 3·0) using Armac CD as collector for mica has been completed and the products sent for petrological examination. Further work is in progress.

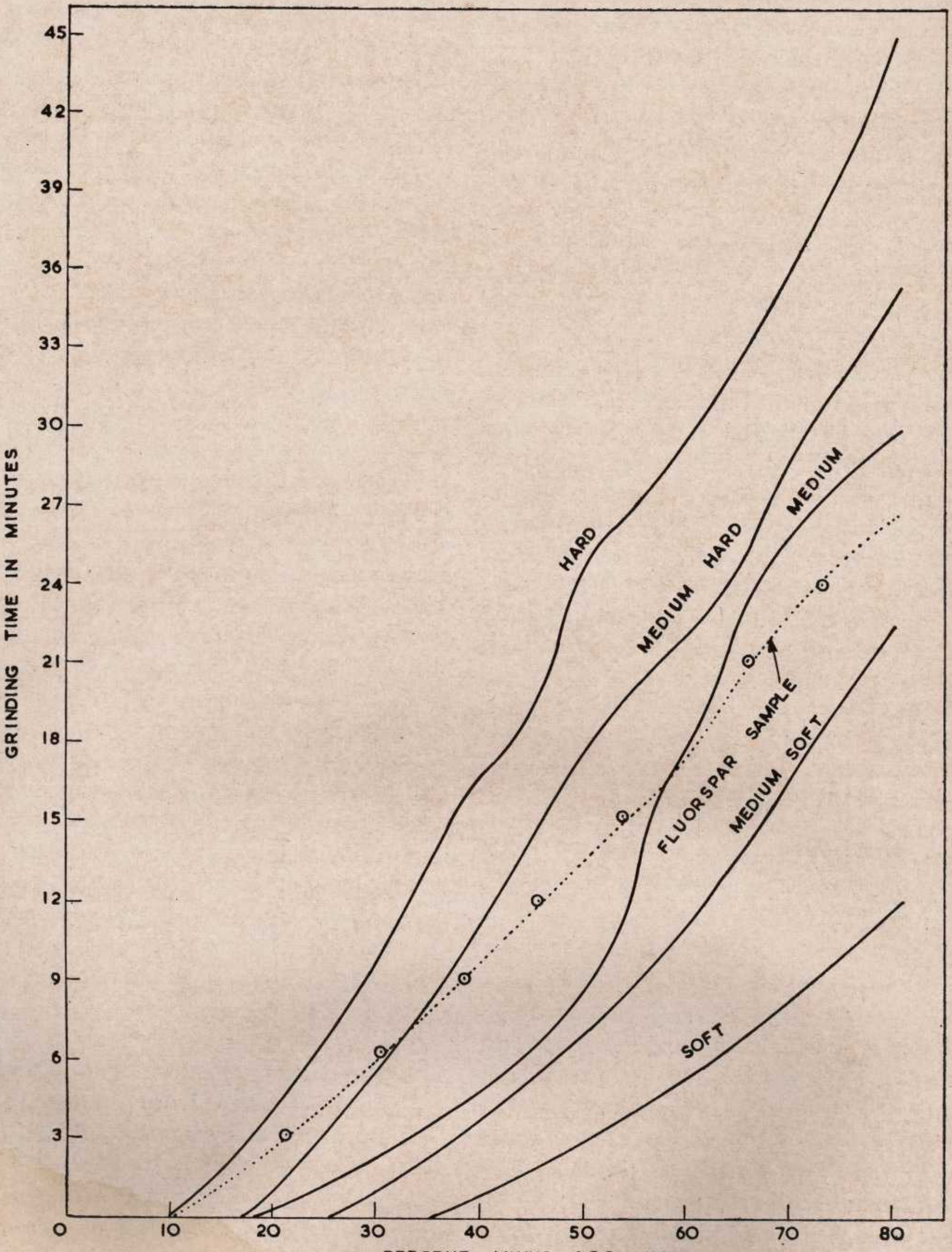


FIG. 14 — GRINDABILITY TESTS OF FLUORSPAR FROM RAJASTHAN

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

The Pilot Submerged Arc Furnace has been installed at the National Metallurgical Laboratory for formulating the technical know-how for the production of diverse ranges of ferro-alloys and for developing electro-metallurgical technique for the smelting of indigenous ores. The furnace has a production capacity of 1-3 tons of ferro-alloys per day depending on the type of the ferro-alloy required. Raw materials are accepted on contract for researches on their amenability to electrothermal 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, the Pilot Plant was operated for the production of high carbon ferro-chrome and silico-chrome.

The feasibility of using chromite ore from the Orissa Mineral Development Corporation, Giridih coke and wood charcoal in the production of high-carbon ferro-chrome was studied in the 500 kVA Pilot Submerged Arc Furnace. Operating conditions were arrived at to produce high-carbon ferro-chrome containing 60-65 per cent chromium, 3-8 per cent silicon and 5-7 per cent carbon. It was observed that Giridih coke along with wood charcoal could be successfully used in the production of high-carbon ferro-chrome. Similarly, silicon-chrome containing 40 per cent silicon was produced by the slagless method. It was observed that Giridih coke could be used as a carbonaceous reducer with partial replacement by wood charcoal in the production of silico-chrome containing Si, 40 and C, 0·05 per cent.

Raw Materials

(i) *Chromite* — The chrome ore supplied for the trials from one of the deposits in Orissa was received in size ranging from 2 in. to fines. The chemical and screen analysis of the washed ore are given in Tables 5 and 6, respectively.

Table 5 — Chemical Analysis of the Chromite

Cr_2O_3	51·68%
FeO	14·85%
Fe_2O_3	0·59%
SiO_2	9·8%
Al_2O_3	9·27%
MgO	11·57%
CaO	1·7%
S	0·064%
P	0·009%
Cr: Fe	2·95: 1
MgO: Al_2O_3	1·25: 1

Table 6 — Screen Analysis of Chromite

−2"	+	1½"	...	10·73%
−1½"	+	1"	...	33·52%
−1"	+	¾"	...	7·37%
−¾"	+	½"	...	25·75%
−½"	+	¼"	...	15·19%
−¼"	+	⅛"	...	3·35%
—	−⅛"	—	...	4·09%
				100·00%

(ii) *Reducing Agent* — The carbonaceous reducing agents employed in the campaign were:

(a) Hard coke from Giridih, supplied in lump form in sizes varying from 6 to 1 in.

(b) Charcoal.

The coke and charcoal were crushed and their screen analysis during different periods is given in Table 7.

The chemical analysis of Giridih coke and charcoal are given in Table 8.

(iii) *Fluxes* — The fluxes used were quartzite, magnesite, bauxite and fluorspar.

(a) *Quartzite* — The quartzite was supplied from a local deposit and contained lot of clay matter. The quartzite was washed and screened. The fraction −1½ + ⅛ in. was used as a flux and it contained 98 per cent silica.

Table 7 — Screen Analysis of Giridih Coke and Charcoal used During Various Stages of the Campaign

Sizes	Giridih coke Period					Charcoal
	12-7-65	19-7-65	29-7-65	3-8-65	15-8-65	
−2″ +1½″	26·31%	—	—	—	—	12·52%
−1½″+1″	63·16%	17·3%	20·0%	14·36%	21·92%	25·79%
−1″ + ¾″	2·95%	39·5%	23·2%	13·73%	13·26%	9·33%
−¾″ + ½″	4·32%	20·2%	21·3%	51·71%	40·30%	31·16%
−½″ + ¼″	1·22%	11·01%	22·3%	15·49%	14·08%	15·64%
−¼″ + ⅛″	0·33%	7·32%	5·4%	2·90%	3·55%	5·56%
−⅛″	1·71%	4·67%	7·8%	1·81%	6·89%	—

Table 8 — Chemical Analysis of Giridih Coke and Charcoal

	Coke	Charcoal
Moisture	2·2%	10·18%
Volatile matter	1·72%	32·00%
Fixed carbon	74·12%	48·12%
Ash	21·94%	9·70%
Ash analysis		
SiO_2	60·75%	10·48%
Al_2O_3	21·6%	5·56%
CaO	0·33%	51·4%
MgO	5·15%	6·60%
Fe_2O_3	7·6%	5·64%
S	1·1%	0·28%
P	0·13%	0·65%

Table 9 — Chemical Analysis of Various Fluxes

Constitutent in %	Quartzite	Bauxite	Magnesite	Fluorspar
SiO_2	98·00	22·40	2·41	—
Al_2O_3	0·50	55·35	0·27	—
MgO	0·05	—	93·90	—
CaO	0·60	—	0·21	—
CaF_2	—	—	—	—
Fe_2O_3	0·30	5·31	0·95	—
Loss on ignition	—	14·58	1·90	—
TiO_2	—	2·90	—	—
CaF_2	—	—	—	75%

(b) *Fluorspar* — The fluorspar used contained about 75 per cent CaF_2.

(c) *Bauxite* — The bauxite contained 55·35 per cent alumina and was crushed to −1½ in.

(d) *Magnesite* — Dead-burnt Salem magnesite of the first size containing 94 per cent magnesia was used.

The chemical analysis of various fluxes used during the campaign is indicated in Table 9.

Smelting Campaign in the Pilot Submerged Arc Furnace

Premixed charge, each unit consisting of 100 kg. of chromite, 35 kg. of coke and 9 kg. of quartzite, was charged at regular intervals at the rate of 1 charge per half an hour. The phase-to-phase voltage was maintained at 55-60 volts with the electrode current at 4400 amp. Tapping of the metal was carried out at regular intervals of 4 hours duration. The metal produced during the period contained Cr, 60-61 and Si, 7·5-10 per cent, and the average power consumption was 7150 kWH/tonne of ferrochromium. The slag was, however, viscous and the effect of various fluxes like bauxite, fluorspar and magnesite were studied and it was observed that the addition of magnesite at the rate of 1 kg. per unit charge improved the fluidity of the slag. The MgO/Al_2O_3 ratio was improved from 0·7 to 1·1.

Effect of Wood Charcoal

In order to improve the furnace operation, a part of the coke was replaced by wood charcoal. The proportion of wood

Table 10 — Raw Material Consumption for the Charcoal Periods

	Period			
	11	12	13	14
Chromite ore per unit charge	100 kg.	100 kg.	100 kg.	100 kg.
Coke per unit charge	28 ,,	28 ,,	28 ,,	25 ,,
Charcoal per unit charge	10 ,,	13 ,,	13 ,,	12 ,,
Quartzite per unit charge	12 ,,	12 ,,	11 ,,	10 ,,
Magnesite per unit charge	1 ,,	1 ,,	1è ,,	2 ,,
Total chromite ore	2900 ,,	6200 ,,	6700 ,,	2700 ,,
Total coke	784 ,,	1716 ,,	1742 ,,	675 ,,
Total charcoal	260 ,,	806 ,,	851 ,,	324 ,,
Total quartzite	396 ,,	532 ,,	737 ,,	269 ,,
Total fluorspar	20 ,,	—	—	—
Total magnesite	29 ,,	62 ,,	93 ,,	54 ,,
Duration of the period	12 hours	39 hours	55 hours	18 hours
Total power for the period	6690 kWH	10680 kWH	17200 kWH	6410 kWH
Total metal	677 kg.	3061 kg.	2768 kg.	1359 kg.
Total slag	998 ,,	2483 ,,	2347 ,,	1265 ,,
Metal analysis %	Cr 60-62 Si 4·5-6·0 C 5·0-6·3	Cr 60-63 Si 5·0-6·2 C 5·5-6·0	Cr 62·5-65·5 Si 3·3-3·8 C 4·9-6·0	Cr 65·0-67·8 Si 2·1-3·8 C 5·0-6·5
Slag analysis %	SiO_2 38·0-41·0 Cr_2O_3 2-4 Al_2O_3 22-25 MgO 31-32·5	SiO_2 38-41 Cr_2O_3 2-3 Al_2O_3 22-25·5 MgO 31·2-32·5	SiO_2 35·8-38·8 Cr_2O_3 3·5 Al_2O_3 23-26 MgO 32-33	SiO_2 38-41 Cr_2O_3 3-4 Al_2O_3 22·4-23·8 MgO 30·8-33·8
Power consumption/ton of metal	9881 kWH	3489 kWH	6213 kWH	4718 kWH

charcoal addition ranged between 25-30 per cent of the total reductant. The proportion of charge, raw material consumption and the operating data are given in Table 10. The table shows that standard grade ferro-chromium could be produced successfully by replacing a part of the reductant with charcoal. The power consumption per ton of the alloy produced was decreased. The slag fluidity and the slag metal separation were also good. Slag volume was low and indicated less loss of metallics in the slag. The ratio of magnesia to that of alumina in the slag varied between 1·24 and 1·5 which was higher than the coke periods. This improvement may be attributed to the lower alumina content in the charcoal ash. In the last period, when the magnesia to alumina ratio exceeded 1·5 in certain tappings, the slag became viscous again which indicated that this ratio should be within 1·0-1·5 for smooth operation of the furnace and easier tapping of slag and metal.

Based on the exhaustive pilot plant investigation conducted, basic performance data were obtained for producing different grades of high-carbon ferro-chromium containing Cr, 60-65; Si, 3-8 and C, 6 per cent using Giridih coke and wood charcoal as the reductants. It was observed that for proper fluidity and neat separation of metal and slag, the SiO_2 content of the slag must be about 35 per cent. The ratio of alumina to that of magnesia was also found to be critical to favour better operating conditions and the value should be less than 0·9. Wood charcoal was used sucessfully by replacing 25 and 33 per cent of the Giridih coke in the production of high-carbon ferro-chrome containing chromium, 60-65; silicon, 3-5 and carbon, 6 per cent. The ratio of Al_2O_3 to MgO was 0·72 to 0·75. It was observed that about 35-40 per cent SiO_3 should be present in the slag for smooth furnace operation. Since wood charcoal had a very low ash content, it was found necessary to increase quartzite in the burden.

12.1 Trials on the Production of Silico-chrome

Systematic smelting trials were undertaken in 500 kVA Submerged Electric Arc Furnace *to assess the commercial feasibility and* operational advantages in the production of silico-chrome by the two-stage process or slagless process.

Raw Materials

The high-carbon ferro-chrome containing 7-9 per cent Si obtained in the production of ferro-chrome was broken and crushed to the size of −50 +6 mm. in a jaw crusher. The composition of ferro-chrome averaged Cr, 62·28; Si, 7-98 and C, 6·08 per cent. The other raw materials like quartzite, Giridih coke, charcoal, Durgapur coke, magnesite, etc., were the same as those used in the production of high-carbon ferro-chrome.

Smelting Trials

Initially, smelting trials were done with Durgapur coke as the reducing agent and the unit charge mix consisted of the following:

High-carbon ferro-chrome	...	25	kg.
Quartzite	...	44	,,
Durgapur coke	...	25	,,
Magnesite	...	1·5	,,

During this campaign, a silico-chrome containing Si, 42·7; Cr, 37·74; Fe, 18·92 and C, 0·22 per cent was produced with a power consumption of 5659 kWH/tonne. The furnace was operated with phase-to-phase voltage of 62·5 per cent.

During Campaign II, the Durgapur coke was replaced by Giridih coke and the carbon balance was maintained the same. The average analysis of the silico-chrome tapped was:

Si	...	43·08 - 44·70	per cent
Cr	...	36·93 - 39·22	,, ,,
Fe	...	17·36 - 18·20	,, ,,
C	...	0·06 - 0·11	,, ,,

The average power consumption amounted to 5251 kWH/tonne. During Campaign III, a part of Giridih coke was replaced by charcoal to improve the penetration of the electrodes as well as to increase the per*meability of the charge mix.* The unit charge consisted of :

High-carbon ferro-chrome	...	25	kg.
Giridih coke	...	20	,,
Charcoal	...	10	,,
Quartzite	...	44	,,
Magnesite	...	1·25	,,

The average power consumption during this campaign was 5641 kWH/tonne of silico-chrome made. It was observed that the operational troubles like bridge and crust formation was not encountered as a result of charcoal addition and the electrode penetration were considerably improved. The average analysis of the metal tapped was

Si	...	43·09 - 43·22	per cent
Cr	...	38·49 - 37·00	,, ,,
Fe	...	0·06 - 0·09	,, ,,

It was found that holding the silico-chrome after tapping in a refractory-lined ladle prior to casting, decreased the carbon content to less than 0·05 per cent.

It was concluded that the production of silico-chrome containing Cr, 37-38; Si, 40-44; and C, 0·05 per cent using Giridih coke and charcoal by the two-stage process was feasible on a commercial scale. Assuming an average power consumption of 5200 kWH/tonne of high-carbon ferro-chrome during the first stage, an overall power consumption of 7400 kWH/tonne of silico-chrome was obtained by the two-stage process. Very little slag was produced during this operation, thereby facilitating the smooth operation of the furnace.

13.0 Electrolytic Recovery of Tin from Tinplate Scrap in Sulphuric Acid Electrolyte

Prior to this an investigation regarding electrolytic detinning and simultaneous recovery of tin in alkaline bath had been completed and the Report had been published in the last Annual Report (1964-65).

This investigation was taken up with a view to study the feasibility of detinning and simultaneous recovery of tin from tin plate scrap from sulphuric acid electrolytic bath. The electrolyte was composed of sulphuric acid, ferrous sulphate and tin sulphate. Tin sulphate was introduced in the electrolyte by dissolving tin foil in the hot mixture of sulphuric acid and ferrous sulphate solution. The spent pickle liquor from the pickling of mild steel sheets from a tinplate manufacturing plant can very profitably serve as a source of electrolyte.

The electrolysis in the case of acid-bath was carried out at room temperature. Stainless steel sheets were used as cathode and the tinplate scrap packed in the form of rectangular bundles served the purpose of the anode. Experiments are being carried out to determine the optimum conditions for recovery of tin, e.g. concentration of M_2SO_4, $FeSO_4$, tin sulphate, etc., in the electrolyte, the precipitation of SnO_2 in the electrolyte, temperature of the bath, current density, amount of scrap to be charged, time of detinning and other factors like dissolution and deposition of tin from the electrolyte under circulation of electrolyte or otherwise. This bath does not require any regeneration except occasional filtration to remove SnO_2 precipitations.

The detinning and recovery of tin in the experiments carried out so far were quite efficient and satisfactory. Regarding the purity of this tin deposit some contamination of the deposit with iron was found. Results of a few experiments are given below:

Composition of the electrolyte:

H_2SO_4	...	60 g./l.
$FeSO_4$	...	167 ,,
Tin	...	5·1 ,,

Electrodes:

Anode — 250 g. of tin plate scrap pressed into a rectangular bundle measuring about 4 × 3 × 1 in. containing 4·03 g. tin.

Cathode — Two stainless steel plates, measuring 5 × 3 × $\frac{1}{16}$ in. each.

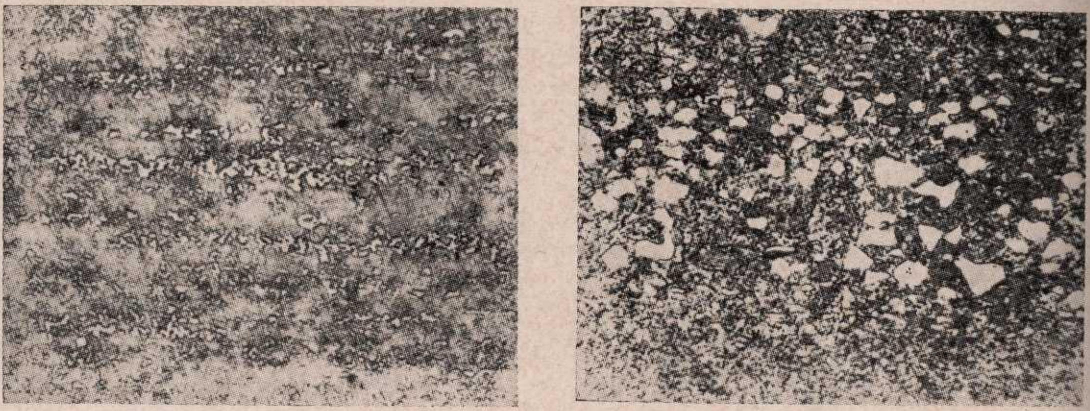
Time of electrolysis — 60 minutes at 2 amp. and 0·6 volts.

Tin recovery as metal	...	96 per cent
C.E. at the cathode	...	90 ,, ,,
C.D. at the cathode	...	9·4 amp./sq. ft

14.0 Electrodeposition of High-purity Chromium from CrO_3 Acid and Fluoride-bath

This investigation was taken up with a view to electrodeposit pure metallic chromium. The main impurities of the chromium deposit are oxygen, nitrogen and hydrogen. These gaseous impurities render chromium brittle. In this investigation a bath containing 300-400 g./l. of CrO_3 and 3-4 g./l. of Na or K-fluoride, at boiling temperature, was used. The electrodes used were an alloy of lead and tin (7 per cent tin) as anode and pure electrolytic copper as cathode. Cylindrical anode were found to give better current distribution and efficiency. Electrolysis was carried out at the boiling temperature of the electrolyte (100°-110°C.). The loss of solution due to evaporation was made up by adding hot distilled water from time to time.

Current efficiency of pure Cr deposit in the usual $CrO_3 + SO_4$ bath range from 6-16 per cent. In this investigation, using CrO_3 +fluoride bath, the C.E. had ranged from 18 to 26 per cent in runs of one hour and more. The deposit of Cr-metal from the cathode was stripped by dissolving the copper in hot dilute nitric acid. Since the bath and the fumes were very corrosive and toxic, the experiments were suspended for some time. Use of aluminium in place of copper for cathode material would be taken up. The results of a few experiments are given in the following page.



(a) High Frequency Melt ×570 (b) Electro-slag Remelt ×570

FIG. 15 — PHOTOMICROGRAPHS OF 18:4:1 HIGH-SPEED STEEL SHOWING THE SHAPE AND DISTRIBUTION OF CARBIDE

Composition and concentration of electrolyte:

CrO_3 ... 300 g./l.
NaF ... 5·3 „

Electrodes:

Anode — 7 per cent tin lead alloy (cylindrical)

Cathode — Pure copper concentric cylinder to anode, weighing 130 g., surface area 2·8 sq. in.

Time of electrolysis ... 1 hr
Temperature of electrolyte 102°C.
Weight of cathode-set after the electrolysis ... 131·5 g.
Weight of Cr deposit ... 1·5 g.
Current efficiency of deposit (Hexavalent chromium) 26·3 per cent

The purity and ductility of the Cr deposited were being determined.

15.0 Electro-slag Melting and Refining

Modern techniques such as vacuum stream degassing, vacuum casting, vacuum melting and casting, consumable electrode vacuum arc remelting and electric slag remelting are employed for producing clean alloys possessing optimum fatigue properties for use in power engineering and other specialized applications. These processes except electric slag remelting require high capital cost investment and complex vacuum equipment. Electro-slag refining is today employed industrially in U.S.S.R. and other countries for the production of alloys such as, ball-bearing steels, heat-resistant alloys, stainless steels, etc. The case of operation of the process has aroused great interest in the metallurgical field. Since the process has potential possibilities in the manufacture of special steels with indigenous resources, a project was taken up at the National Metallurgical Laboratory to develop requisite technical know-how for electro-slag refining treatment.

The fundamental principle of the electro-slag process consists in melting and refining through a synthetic slag. The only investment in this process is a suitable low-voltage transformer. In this work a single-phase 24 kW transformer was used. For industrial application, single-phase as well as three-phase transformers have been used.

During the process of melting the temperature of the working slag was of the order of 1700°-1900°C., which resulted in the melting of the electrode and metal while dripping through the molten slag in the form of droplets gets refined. Sulphur contents were thereby lowered by 30-60 per cent and the non-metallic inclusions in the remelted products were fine and most of them were in the form of harmless globules. During the process, melting and solidification proceeded simultaneously and the electro-slag cast ingots were free from columnar structure, carbide seggregation and axial porosity. The surface finish of the electro-slag refined ingot was highly smooth due to absence of metal mould reaction and because of the intermediate layer of slag formed in the water-cooled mould walls.

The ingot mould (may be round or square type) made of mild steel formed one terminal of the output line from the transformer and electrode the other. In order to initiate the reaction, a small quantity of self-fusing flux containing Al-Mg alloy and KNO_3 was used. The flux produced the initial molten slag on arcing which ultimately formed an electrically conducting medium for the current to flow between electrode and mould bottom. Due to resistance offered by the slag to the electric current flow, the slag attained very high temperature which helped in the melting of the electrode. Calcium fluoride formed a major proportion of the working flux in order to impart electrical conductivity. The mixture normally contained 70 per cent calcium fluoride and 30 per cent alumina or in some cases, part of the alumina was replaced by other constituents, such as lime, according to the refinement required.

A mild-steel, closed-bottom, cylindrical mould, 2 in. inside diameter and ¼ in. thick, was used for all the experiments. Mould walls were cooled by a circular water spraying ring along the mould wall. The feeding of the electrode was done manually with the help of a pulley. Due to manual control, the feeding rate could not be kept uniform which resulted in frequent make and break contacts thus prolonging somewhat refining time. It had been reported earlier that the electro-slag cast ingots were free from columnar structure, carbide segregation

Table 11 — Chemical Analyses of Steels used and Their Final Analyses after Electro-slag Remelting (percentages)

Steel sample	Electrode									Electro-slag remelt from the electrode									Percentage reduction of 'S'
	C	S	P	Si	Mn	W	Cr	V	Al	C	S	P	Si	Mn	W	Cr	V	Al	
				Per cent									Per cent						
Commercial mild steel	0·22	0·02	0·03	0·09	0·69	—	—	—	0·01	0·23	0·006	0·03	0·12	0·64	—	—	—	0·1	70
EF 36	0·44	0·016	0·028	0·47	0·77	—	—	—	Tr	0·39	0·005	0·028	0·47	0·77	—	—	—	Tr	70
EF 119	0·45	0·009	0·027	0·37	0·94	—	—	—	Tr	0·44	0·006	0·027	0·47	0·84	—	—	—	Tr	33
EF 120	0·61	0·01	0·029	0·29	0·81	—	—	—	Tr	0·59	Tr	0·03	0·54	0·74	—	—	—	Tr	—
HF 505	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
C, 1 and Cr, 1·4 per cent	0·98	0·04	0·02	0·46	1·02	—	1·30	—	—	0·95	0·01	0·04	0·43	0·98	—	1·27	—	0·20	75
Ball-bearing steel																			
HF 520																			
18/4/1	0·7	0·009	0·008	0·32	0·37	18·25	4·4	1·28	—	0·7	Tr	0·012	0·24	0·26	18·25	4·12	1·28	—	—
High-speed steel																			

and axial porosity. The forged sections of 18: 4: 1 high speed steel made in high frequency furnace and by electro-slag remelt showed a remarkable change in their carbide distribution as shown in Figs. 15(a) and (b).

The chemical analysis of different steels before and after electro-slag remelting are given in Table 11.

The inclusion contents of different steels after electro-slag remelting were found to be considerably reduced as shown in Table 12.

Table 12 — Comparison of Inclusion Counts (L/T 'Lineal Traverse' Count Method) of the Steel made by Electric Arc and High Frequency Method and Electro-slag Remelting

Steel			L/T count inclusions cm.	Reduction in inclusion count %
Arc furnace	EF	36	25·4	64
ESR	EF	36	9·08	—
Arc furnace	EF	119	14·2	24·7
ESR	EF	119	10·7	—
Arc furnace	EF	120	14·1	44
ESR	EF	120	7·9	—
High frequency C, 1 and Cr, 1·4%	HF	505	40·0	52
ESR ,,	HF	505	19·2	—

The inclusion study of ball-bearing steel was also made by A.S.T.M. standard and (based on J. K. Chart) their values are given in Table 13.

Electro-slag refined steel possesses better mechanical properties compared with the conventionally manufactured steels particularly in relation to toughness and ductility. Table 14 shows the results of Charpy impact tests conducted at room temperature (21°C.) on 18: 4: 1, and C, 1 and Cr, 1·4 per cent steel.

Table 14 — Change in Notched Bar Impact Values of 18 : 4 : 1 High-speed Steel and C, 1 and Cr, 1.4 per cent Ball-bearing Steel at Room Temperature (21°C.) Before and After Remelting

Sample		Impact values at room temperature (21°C.) ft-lb.
H.F. 520	(a) High frequency melt	(i) 3·61 (ii) 3·8 (iii) 3·9
	(b) E.S.R.	(i) 4·33 (ii) 5·2 (iii) 3·9
H.F. 505	(a) High frequency melt	(i) 6·0
	(b) E.S.R.	(ii) 9·0

Vacuum gas analysis had shown considerable reduction in the hydrogen and oxygen content of the electro-slag remelted steels. Electro-slag remelting produces ingots free from axial porosity and seggregation. The process of electro-slag remelting definitely improved the quality of steel with respect to reduction of sulphur to very low levels, reduction of non-metallic inclusions, production of much superior ingot surface finish, better mechanical and impact properties. The work was completed.

Table 13 — Inclusion Study of Ball-bearing Steel based on J. K. Chart (A.S.T.M. Standards E. 45-51)

Sample H.F. 505	A Sulphide		B Alumina		C Silicate		D Globular oxide	
	T	H	T	H	T	H	T	H
High frequency melt	0·38	0·65	0·50	0·16	nil	0·24	0·80	1·34
Electro-slag melt	nil	nil	0·54	0·40	nil	nil	1·50	0·92

T = Thin. H = Heavy.

16.0 Production of Ferro-silico-zirconium by Alumino-thermic Process

The investigation was taken up with a view to study feasibility of commercial production of ferro-silico-zirconium by reduction of zircon sand with aluminium and a suitable energizer.

Zircon sand, containing ZrO_2, 63·87 and SiO_2, 35·34 per cent blended with iron ore, was reduced with aluminium powder using energizers. It was found that the reaction can be made autogenous with good slag/metal separation only by suitable adjustments of energizer. The composition of the alloy so obtained was Zr, 19·78; Si 14·77 and Al 14·14 per cent. Experiments were carried out with decreased amounts of aluminium powder than that required theoretically, and flux additions.

17.0 Nitriding of Ferro-chrome

The investigation was taken up with a view to produce nitrogen bearing ferro-chrome and to study the effect of absorption of nitrogen with respect to particle size, temperature time, grain size, effect of composition, etc.

Carbon-free ferro-chrome produced by the alumino-thermic process containing Cr, 73; Al, 0·2 and Si, 2·7 per cent was nitrided with high-purity oxygen-free nitrogen gas in a tubular furnace maintained at the desired temperature. The alloy was crushed and graded to 1 and ½ in., and −3 +4, −6 +8 −8 +10, −10 +12 and −14 +20 mesh sizes. Each sample was nitrided at 800°, 900°, 1000° and 1100°C. for different periods varying from 4 to 48 hours. The extent of nitrogen absorption at 1000°C. are given in Table 15.

It was observed that the amount of nitrogen absorbed increases with the decreasing particle size and increasing temperature. The nitrogen absorption increased linearly with duration up to a certain period beyond which the rate of absorption was not significant.

The rate of absorption increased with increasing temperature and the duration of nitriding decreased with increasing temperature for the same amount of nitrogen absorption. It was also observed that the rate of nitrogen absorption for a given particle size and for a given duration rapidly increased between 950° and 1000°C.

With 1 and ½ in. sizes of ferro-chrome, 2·25 and 3·48 per cent of nitrogen were absorbed at 1100°C. in 24 hours. Further experiments with the same sizes at various temperatures and periods were in progress. Studies on the influence of particle size, temperature and duration at temperatures beyond 1100°C. were being continued.

18.0 Reducibility of Kiriburu Iron Ores and Sinters

Reducibility characteristics of lumpy Kiriburu iron ores and their sinters were studied at the instance of M/s. Hindustan Steel Ltd.

Table 15 — Percent Nitrogen Contents of Various Particle Sizes for varying Periods at 1000°C.

Time hr.	Nitrogen %					
	−3+4	−4+6	−6+8	−8+10	−10+12	−12+20
4	1·184	1·320	4·364	6·282	—	—
8	1·918	2·101	2·383	2·738	3·286	4·429
16	5·450	5·830	7·040	—	7·480	8·620
24	6·020	—	7·730	8·240	8·560	8·620
48	6·990	—	7·110	7·630	8·470	9·110

The samples were received after their pilot plant studies on beneficiation and sintering at National Metallurgical Laboratory. Reducibility tests were carried out on iron ore lumps of different sizes of −40 +25, −50 +40, −70 +50 and −100 +70 mm. In each size 500 g. of the sample was reduced with hydrogen at 800°C. with a flow rate of 3·5 litres per minute. The time for 90 per cent reduction is given in Table 16.

Table 16 — Effect of Size on Iron Ore Lump of Massive Laminated Type on the Rate of Reduction

Weight of sample ... 500 g.
Temperature ... 800°C.
Hydrogen flow ... 3·5 l./min.

Sl. No.	Size in mm.	Time for 90% reduction in minutes	Remarks
1	−100+70	270	Slightly cracked during reduction
2	−100+70	150	Disintegrated
3	−70+50	250	Cracked
4	−70+50	220	Disintegrated
5	−70+50	155	Disintegrated during heating
6	−50+40	160	Cracked
7	−40+25	145	Cracked

It was observed that the time for 90 per cent reduction increased with increase in size. Whereas the iron ore lump of −40 +25 mm. took 145 minutes for 90 per cent reduction and the −70 +50 mm. sample took 155-250 minutes, the −100 +70 mm. sample took 150-270 minutes.

The variation in reduction rate in each size range was found to be due to varying decrepitation of the sample ore during heating, as well as during the course of reduction. The formation of cracks and decrepitation of laminated layers during the course of reduction led to faster reduction of large-sized iron ore lumps. It was concluded that the size of lumps for Kiriburu iron ores for blast furnace burden can be even higher than 50 mm. so far as reducibility characteristics are concerned.

Reducibility studies were undertaken on some typical sinters made at the National Metallurgical Laboratory. Sinters weighing 150 g. and of −0·263 +0·185 in. (−6·68 +4·7 mm.) size were reduced at 800°C. with hydrogen flowing at 3·5 litres per minute. The following conclusions were drawn from the experimental results:

(i) Unfluxed sinters prepared from washed and beneficiated ore fines were more easily reducible than the unfluxed sinters prepared from ore fines that had been merely washed but unbeneficiated. The reasons can be attributed to the increased Fe content and decreased gangue content of the washed and beneficiated ore.

(ii) Self-fluxed sinters prepared with limestone additions showed better reducibility than the non-fluxed sinters both made from washed and beneficiated ore fines. As the basicity, expressed as $\frac{CaO+MgO}{SiO_2}$ was increased from 0·8 to 1·6 by increasing the amount of limestone additions, the reducibility was accelerated at a basicity value between 1·2 to 1·4 beyond which there was no further improvement. With the addition of limestone, the FeO content of the sinters decreased to almost half the quantity present in non-fluxed sinters. The improved degree of oxidation from 90 to 95·3 resulted in improved reducibility of the sinters.

(iii) Self-fluxing sinters prepared using limestone and dolomite in the ratio of 4: 1 and having different basicities showed lower reducibility characteristics than the self-fluxing sinters prepared with limestone additions alone at each of the basicity level. The degree of oxidation in both cases was of the same order.

(iv) The addition of blue dust for self-fluxing sinters up to 30 per cent with respect to ore fines in the sinter mix increased the reduction rate of the self-fluxing sinters.

(v) Self-fluxing sinters prepared by mixed firing technique in which the solid fuel was substituted by gaseous fuel showed a high degree of oxidation of 97 per cent and very fast reduction rates.

(vi) The reducibility was found to be fast if the FeO content was maintained below 15 per cent.

19.0 Thermal Beneficiation of Low-grade Ferruginous Chrome Ores

The large reserves of low-grade Indian chrome ores with Cr: Fe ratios less than 2·8: 1 cannot be used for production of standard grade ferro-chrome. These ores are not amenable to conventional ore-dressing treatments for removal of iron, since iron and chromium occur in chemically combined state in the chromite spinel. The process developed earlier consisted in preferential solid state reduction of iron from the chrome ore and subsequent removal of iron by acid leaching leading to yield high Cr/Fe ratio in the residue. Further studies have now been made to develop a more economical method for removal of iron than acid leaching.

A chrome ore table concentrate containing Cr_2O_3, 40·83; Fe (total), 22·19; Al_2O_3, 16·9; SiO_2, 3·32; MgO, 7·5 and CaO, 1·2 per cent and initial Cr: Fe ratio of 1·23: 1 was reduced with coke at 1250°C. The reduced ore was given an aqueous corrosive leaching while the reduced iron was oxidized which could be separated by gravity separation. The various factors studied include the effect of controlled ore: coke ratio on reduction, effect of intermittent grinding, electrolytes, *p*H, concentration of electrolytes, as well as kinetics of leaching on the extent of iron removal from the ore. It was observed that the reduced ore obtained on heating chrome ore with 7 per cent coke could be upgraded to a Cr: Fe ratio of 4: 1 without acid leaching.

20.0 Beneficiation of Ilmenite by the Removal of Iron

The treatment of ilmenite with sulphuric acid for the production of pigment grade TiO_2 results in considerable acid consumption owing to the high iron content of the ilmenite. The large quantities of ferrous sulphate solution has its own disposal problem. Investigations were, therefore, undertaken on the preferential removal of iron from ilmenite either by roast reduction or by chlorination technique.

Roast reduction — It was observed that iron could be removed from ilmenite by reduction and subsequent aqueous oxidation. Based on the earlier experiments, carbonaceous reduction of briquetted and preoxidized samples were carried out and studies were undertaken to study the effect of temperature, kinetics of reduction, particle size, etc.

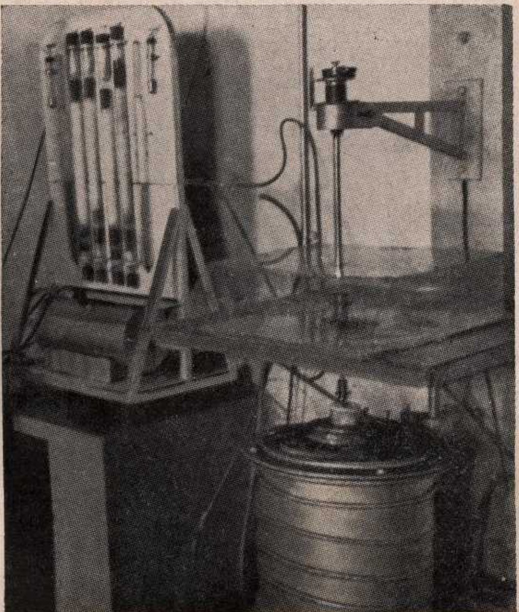


FIG. 16 — HIGH TEMPERATURE SLAG VISCOMETER, DESIGNED AND FABRICATED AT THE NATIONAL METALLURGICAL LABORATORY

Different aspects on gaseous reduction of ilmenite was studied. Gases used for reduction included hydrogen, coke-oven gas and preheated coke-oven gas. A temperature within 925° to 1000°C. was found to be optimum for gaseous reduction of ilmenite. Investigation on reduction of ilmenite was complete and a Report was under preparation.

Chlorination of Ilmenite with HCl gas

The ilmenite concentrate employed in the investigation analysed TiO_2, 52·92; FeO, 26·49; Fe_2O_3, 18·3; SiO_2, 0·98; V_2O_5, 0·18 and Cr_2O_3, 0·084 per cent. Chlorination was carried out in a vertical silica tube with HCl gas passing downwards from the top. The reaction products were collected continuously at the bottom. Ilmenite was briquetted using 1 per cent dextrine as binder. Systematic studies were carried out on the effect of temperature, particle size and period on the chlorination of iron, effect of pre-reduction and preoxidation of ilmenite on the chlorination of iron, effect of partial pressures of H_2 and HCl as well as Cl_2 and HCl, the extent of the removal of V and Cr and the loss of TiO_2 on chlorination with HCl gas.

Chlorination studies carried out at 600° to 1075°C. indicated that more than 94 per cent iron removal was possible at and above 800°C. and the extent of removal of iron increased to 99·9 per cent with increase in temperature to 1075°C. Chlorination of iron increased from 94·4 to 98·2 per cent as the particle size of ore decreased from 0·1936 to 0·0451 mm. progressively. Volatilization of iron was found to be virtually complete in 3 hours with a −36 mesh (B.S.S) size of ore at 800°C. Along with 2 per cent dextrine binder 20 per cent excess of stoichiometric amount of coke was used to prepare the briquettes and when reacted at 1150°C. for 4 hours, 88 per cent of iron was reduced to metallic state.

Prior to reduction, ilmenite was oxidized in a current of air in a fluidized bed reactor at 1050°C. for 2 hours. The X-ray diffraction studies and chemical analyses indicated the presence of only a trace of FeO, while most of the iron was present as Fe_2O_3. This preoxidized ilmenite was observed to be more easily reducible than briquetted sample and reduction could be effected at lower temperatures and shorter periods. The effect of temperature on the reduction of iron oxide, on the briquetted and preoxidized samples is indicated in Table 17.

Table 17 — Effect of Temperature on Reduction

Ilmenite: coke ratio ... 1:0·15
Period of reduction ... 4 hours

Sl. No.	Ilmenite sample	Temperature of reduction °C.	Metallic iron %
1	Briquetted	1100	84·92
2		1150	88·10
3		1180	87·65
4		1200	88·04
1	Preoxidized	950	3·84
2		1000	13·26
3		1050	58·86
4		1100	85·58
5		1150	90·93

Kinetics studies on briquetted and preoxidized ilmenite indicated the optimum period of reduction as 3½ and 2½ hours, respectively.

Pre-reduction of ilmenite with hydrogen at 950°C. followed by chlorination at 800°C. resulted in chlorination of 99 per cent of iron, but the product was a less volatile ferrous chloride. Preoxidized ilmenite at 1000°-1050°C. with more than 98 per cent iron as Fe_2O_3 was chlorinated at 8000°C., to remove only 32·58 per cent iron, while chlorination at 1100°C. removed 94 per cent iron as ferric chloride.

Chlorination with a mixture of H_2 and HCl gas indicated that as the H_2 content in the gas mixture was increased, the chlorination of iron in ilmenite was decreased owing to the lowering of the partial pressure of the chlorinating agent. Use of chlorine instead of HCl at 800°C. removed 43·43 per cent iron as compared to 84·68 per cent iron for a two-hour long chlorination. Chlorine alone in the absence of a reducing agent was not as effective as HCl which combines the effect of a reducing agent like H_2. Further work was in progress with various $HCl-Cl_2$ ratios in the gas mixture.

The extent of chlorination of the impurities like V and Cr had been studied and it was found that 92 per cent V could be volatilized at 1075°C. and 63·2 per cent at 800°C., whilst Cr could be removed to the extent of 21·25 per cent at 1075°C. and 17 per cent at 800°C. Even the addition of 3 per cent C followed by chlorination at 950°C. failed to chlorinate the chromium oxide in ilmenite. Further work is in progress to decompose the iron chlorides with steam to regenerate the HCl. Attempts were also being made to continuously chlorinate ilmenite with HCl gas.

21.0 Recovery of Vanadium from Calcium Complex Salts

The bauxite deposits of Bihar contain about 0·05 per cent vanadium and nearly 30-35 per cent of vanadium enters the Bayer's process liquor during alumina extraction. The presence of vanadium in aluminium affects the electrical properties of the latter and the elimination of vanadium from the process liquor becomes inevitable. On cooling the process liquor to 25°C., vanadium separates out as a complex salt containing phosphorous and alkali. The sodium complex salt thus obtained is treated with lime to regenerate alkali for use in the extraction process, whereas the complex is converted to a calcium salt which is normally disposed off. Systematic studies were undertaken to recover vanadium from the calcium complex salt preferably in the form of chemically pure V_2O_5.

The salt was treated with 20 per cent sulphuric acid, whereby all the vanadium and phosphorous was dissolved. Neutralization of the leach liquor to *p*H 6 resulted in almost complete precipitation of phosphorous leaving 96·4 per cent of the vanadium in solution. Further neutrtlization of the acidic liquor led to lower vanadium recovery. The precipitated phosphate could be used for phosphatic fertilizer. The vanadium containing filtrate was acidified to *p*H 2 with sulphuric acid and the precipitated red cake contained 78 per cent V_2O_5. Ammonium chloride addition to the vanadate solution at *p*H 6 quantitatively precipitated vanadium as an orange coloured mixed vanadate, which could be filtered out, redissolved and reprecipitated with NH_4Cl from ammoniacal solution to yield chemically pure ammonium metavanadate.

22.0 Extraction of Nickel from Nickeliferous Ore

Recent explorations by the G.S.I. has brought to light several low-grade nickel deposits of both lateritic and serpentinous origins. These reserves are reported to be widespread. In view of the strategic importance of nickel, extraction of nickel from these low-grade ores are of considerable importance. Systematic investigations were taken up on two different samples of nickel ores, one from Rajasthan and the other from Orissa, to study the extraction characteristics of nickel.

(i) *Serpentinous Ore from Rajasthan*

The ore from Rajasthan was serpentinous in nature and contained NiO, 0·33; MgO, 37·18; SiO_2, 38·45; Fe (total), 5·75; Al_2O_3, 1·83 and L.O.I., 16·19 per cent. The ore contained iron as magnetite and on magnetic separation yielded 96·95 per cent non-magnetic and 3·05 per cent magnetic fractions, but the nickel contents of both were found to be of the same order.

Attempts were made to reduce the ore with hydrogen and the reduced nickel was leached with ammoniacal liquor in presence of oxygen gas. At the optimum reduction temperature of 700°C., the nickel recovery was 44 per cent. The nickel recovery did not improve further on prolonged reduction of leaching time beyond 2 and 8 hours, respectively. Pressure leaching of the reduced ore with ammoniacal ammonium sulphate solution in presence of oxygen at 5 atmospheric pressures at 70°C. gave no better recovery than 54 per cent.

(ii) *Lateritic Ore from Sukinda, Orissa*

Two grades of nickel bearing laterite samples were received from Sukinda area of Orissa. One of these was light brown light weight, while the other deep brown and dense in character. The light brown light weight variety was taken up for studies and was found to contain NiO, 1·8; Fe (total), 47·04; Cr_2O_3, 6·2; SiO_2, 6·8; MgO, 1·1; Al_2O_3, 12·36 and MnO, 2·9 per cent. Hydrogen reduction of this ore at temperatures ranging from 500° to 800°C. was investigated and it was observed that reduction at 750°C. followed by ammoniacal leaching converted 82 per cent of nickel to soluble nickel-ammine. The reduction was found to be complete within 90 minutes and an overall 80 per cent nickel recovery had so far been achieved.

23.0 Utilization of Kashmir Bauxite for the Production of Alumina

Bauxite sample received from Kashmir contains high proportions of silica and the alumina occurs as diaspore and clay. The conventional Bayer's process cannot be adopted with this ore due to high alumina and soda losses and the necessity of high pressures and caustic concentration needed to dissolve the diaspore. The object of the present investigation is to preferentially remove silica from the bauxite by calcination followed by digestion with dilute caustic solution. The desilicated residue may be subjected to alumina extraction either by the conventional Bayer's process or any other suitable treatment consistent with the nature of the bauxite sample under investigation.

The bauxite sample received analysed Al_2O_3, 55·35; SiO_2, 22·4; Fe_2O_3, 5·31; TiO_2, 2·9 and L.O.I., 14·56 per cent. The sample was calcined for 4 hours at different temperatures and the calcine leached with 15 per cent NaOH solution at 95°C. for 1 hour for removal of silica. The results are shown in Table 18.

Table 18 — Results of the Tests on Alkali Leaching of Samples Calcined at Different Temperatures

Temperature of calcination °C.	Silica left in the residue %	Silica removed %
800	14·50	59·29
830	6·36	85·30
850	4·40	89·76
900	4·20	89·90

Table 18 indicates that the bauxite had to be calcined at 850°C. or higher for efficient removal of silica and the silica content in the residue was reduced to below 5 per cent. Calcination was found to be virtually complete in 1 hour.

Bauxite calcined at 850°C. for 1 hour duration was leached in different concentrations of alkali in the leach solution and it has been observed that a minimum of 15 per cent concentration is required to keep the silica level below 5 per cent in the residue for subsequent treatment by the Bayer's method. A few experiments were carried out to study the effect of leaching the calcine with different proportions at

15 per cent NaOH solution and it was observed that desilication was more effective with increasing alkali content. Studies on kinetics of leaching and temperature of leaching were under way.

24.0 Reconditioning of Magnesium Powder

Magnesium powder during storage for long periods deteriorates due to the formation of oxide coating on the metallic surface and cannot be used in pyrotechniques. The investigation was taken up at the instance of the Defence establishments for improving the metallic content to over 99 per cent from powder containing less than 80 per cent metallics. For use in the Defence establishment, 2400 kg. of magnesium powder, grade V, with metallic magnesium content varying from 76·5 to 93·3 per cent, were reconditioned to over 99 per cent metallic content. For further use by the Defence establishments, 195 kg. of magnesium-manganese alloy powder that was oxidized during long storage periods, was also reconditioned from its original metallic content of 93 to over 99 per cent.

25.0 Preparation of Anhydrous Magnesium Chloride

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

By various drying techniques, taking special care to prevent oxide and oxychloride formation, a product $MgCl_2$ containing approximately 20-22 per cent moisture was obtained without much oxide. Attempts to eliminate water further resulted in increase of the oxide content of the product. Consequently, attempts were made to prepare anhydrous magnesium chloride from magnesium oxide by chlorination methods. The magnesium oxide was obtained in the form of fine powder. It was briquetted with calcined petroleum coke and magnesium chloride solution to make the briquettes sufficiently strong to resist storage and weather condition as well as to have furnace stability. It was found that crushing strength of the briquettes prepared with calcined petroleum coke and magnesium chloride solution could not be increased beyond 15 lb./in.^{2}. Therefore, decision was taken to improve the quality of the briquettes by the addition of pitch with coke. Following results were obtained.

Composition of the briquetts:
  Magnesium oxide powder ... 100 g.
  Petroleum coke blended with pitch ... 30 g.
  Binder —45 —100 cc. of $MgCl_2$ solution of 22°Be′

Crushing strength of the dried briquetts.

Drying temperature °C.	110	170	274	350	425
Crushing strength lb./in.^{2}	2500	2120	2000	35	25

Attempts are being made to examine the suitability of the briquettes by actual chlorination experiments.

26.0 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 imports. In view of the expanding demand of this material in the country, need for its synthetic preparation is 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. The indigenous flourspar is low grade and requires beneficiation in the first instance. Extensive work on laboratory and pilot plant scale on beneficiation of fluorspar was conducted.

The process was investigated earlier in decomposing the metallurgical grade fluorspar in an aqueous medium with sulphuric acid and boric acid. Calcium fluoride of the fluorspar reacted to yield fluoboric acid and gypsum (insoluble). The liquor rich in fluoboric acid was neutralized with hydrated alumina and sodium carbonate which resulted in forming cryolite and boric acid. Cryolite precipitate was filtered out and the spent liquor was reused for leaching of fluorspar after addition of sulphuric acid.

The results reported earlier indicated that the overall fluorine recovery was 75 per cent approximately. Further experiments were carried out to improve the overall fluorine recovery by varying the conditions. The results of one such experiment are given in Table 19 which indicate overall fluorine recovery as 85 per cent, an improvement over the previous experiments.

Table 19 — Results of Experiments

No. of cycles	Fluorspar	Sulphuric acid g.	Hydrated alumina	Sodium carbonate	Cryolite obtained
1	600	709	228	259	440
2	600	709	228	259	432
3	600	709	228	259	430
4	600	709	228	259	430
5	600	709	228	259	437
6	600	709	228	259	434
7	600	709	228	259	446
8	600	709	228	259	412
9	600	709	228	259	369
	5400 g.	6381 g.	2052 g.	2431 g.	3828 g.

Fluorspar: CaF_2, 89·18; SiO_2, 3·2; Al_2O_3, 0·49; Fe_2O_3, 0·4; S, 0·16; Pb, 0·22; $CaCo_3$, 3·75 and L.O.I., 2·44 per cent.

H_2SO_4 : 36 Normal

Sodium carbonate: Anhydrous C.P.

Hydrated alumina: Sandard Bayer's Product

Leaching conditions: Temperature, 95°C.; Time, two hours; stirring in 5-litre beaker with glass stirrer

Volume: 3 litres

Boric acid: 120 g./l.

Sodium sulphate: 25 g./l.

Analysis of spent liquor: Al, 20·6 g., Na, 158·0 g., SO_4, 507 g., Fe, 4·14 g., F, 60 g. and H_3BO_3, 339 g.

$$CaF_2 \text{ in 5400 g. of fluorspar} = 89{\cdot}18 \times 54 = 4816 \text{ g.}$$

$$F \text{ in 4816 g. of } CaF_2 = \frac{38 \times 4816}{78} = 2320 \text{ g.}$$

$$F \text{ in Cryolite} = \frac{52 \times 3818}{100} = 1990{\cdot}56 \text{ g.}$$

$$\text{Overall fluorine recovery} = \frac{1990{\cdot}56 \times 100}{2320} = 85{\cdot}8 \text{ per cent}$$

Cryolite average analysis: Na, 24·5; Al, 18·52; F, 52·01; SiO_2, 0·03; Fe, 0·04 and SO_4, 3·5 per cent; and moisture, trace.

The salient feature of this experiment is that the residual sulphuric acid after leaching was not removed at all. Theoretical amounts of all the ingredients were used. The product is well within specifications for the aluminium industry although the sulphate content of the cryolite is slightly higher than that previously prepared.

Experiments were also carried out to utilize the spent liquors which contain boric acid, fluorine, sodium, etc. Sodium and potassium fluoborates which are used as fluxed can be prepared from these liquors. Potassium fluoborate can be prepared quite pure as it precipitates out by the addition of K^+ ions into the liquors. Recovery of boric acid from the spent liquor was also tried. The volume of the spent liquor was reduced by evaporating the liquor. The spent liquor was then cooled to 10°C. The liquor (3 litres) contained 339 g. of boric acid; 210 g. of boric acid crystallized out on cooling. The crystallized boric acid was filtered, washed and dried. The rest of the boric acid can be utilized for the preparation of fluororates. Further experiments for the improvement of the process and utilization of the spent liquors are in progress.

27.0 Nickel-free Austenitic Stainless Steels

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 high temperature 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 production costs, yield value and range of industrial products obtainable under Indian market conditions and requirements. Tests were conducted to determine the low-temperature and sub-zero tensile and impact properties, high-temperature tensile and impact properties, high-temperature tensile strength, weldability, oxidation resistance and aging characteristics of different compositions of the nickel-free austenitic stainless steels.

During the period under review, work was carried out on 10 kg. heats made in a 20 kW high-frequency furnace using low-carbon steel scrap, low-carbon ferro-chrome, nitrogen containing low-carbon ferro-chrome and nitrided electrolyte manganese. High-grade ferro-silicon was used as deoxidant. The pouring temperatures and holding periods of the heats after attaining pouring temperatures were carefully controlled to obtain desirable nitrogen content in steel. To prevent gas evolution during solidification of the ingot, top of ingot was capped with a thick steel plunger immediately after teeming. The ingots were homogenized at 1150°C. for periods of 4 hours and were forged into bars and slabs. The slabs were machined or ground to remove the surface defects and rolled into sheets. Results of various tests carried out are given below.

Tensile tests were carried out on flat specimens of 2·54 cm. (1 in.) gauge length made from sheets solution-treated at 1050°C. Results are given in Table 20.

Table 20 — Tensile and Hardness Values

Composition %	Ultimate tensile strength		Elongation on 2·54 cm. (1″) G.L. %	Hardness V.P.N.
	kg./mm.^{2}	Tons/in.^{2}		
Heat No. B-2				
Cr, 20·9 Mn, 13·8 N, 0·68 C, 0·12 Si, 0·70	94·85	(60·22)	45·3	270
Heat No. A-44				
Cr, 16·68 Mn, 14·13 C, 0·05 N, 0·49 Si, 0·21	94·80	(60·20)	48·4	260
Type 18/8				
Cr, 18·34 Ni, 8·23 C, 0·09 Si, 0·32	61·43	(39·00)	65·6	175

Elevated temperature tensile tests were carried out at temperatures of 200°, 300° and 450°C. using Hounsfield tensometer standard specimen No. 13. Results are given below in Table 21.

Table 21 — High Temperature Tensile Strength

Composition %	Test temperature °C.	Ultimate tensile strength		Elongation (gauge length 1·605 cm.) (0·632 in.) %
		kg./mm.^{2}	Tons/in.^{2}	
Heat No. E-68/2				
Cr, 21·5	200	68·84	(43·7)	50
Mn, 13·2	300	65·06	(41·3)	32
N, 0·78	450	61·90	(39·3)	37
C, 0·06	—	—	—	—
Si, 0·27	—	—	—	—

A special fixture was made for sub-zero tensile testing. Specimens of 1 in. G.L. were tested immersed in petroleum ether cooled to desired temperature by liquid air additions. Results of the tests are given below in Table 22.

Table 22 — Results of Sub-zero Tensile Tests

Composition %	Treatment temp. °C.	Ultimate tensile strength		Elongation G.L. 2·54 cm.(1″) %
		kg./mm.^{2}	Tons/in.^{2}	
Heat No. B-2	25	97·06	(61·5)	43·7
Cr, 20·9	0	94·18	(59·8)	50·0
Mn, 13·8				
N, 0·68	−10	95·92	(60·9)	50·0
C, 0·12				
Si, 0·70	−20	93·29	(60·5)	49·9
	−30	101·90	(64·7)	46·9
	−40	94·81	(60·2)	50·0
	−50	96·55	(61·3)	50·0
	−60	97·62	(61·98)	50·0

Oxidation resistance of steel B$_2$ (Cr, 20·9; Mn, 13·8; N, 0·68 and C, 0·12 per cent) was determined in a thermal balance in air at different temperatures.

Impact tests were carried out on V-notch charpy specimens made from steel No. E-28, solution-treated at 1050°C., both at room temperature and sub-zero temperatures up to −70°C. and the results are given in Table 23.

Table 23 — Impact Properties

Composition %	Temperature of test °C.	Energy of fracture	
		kg./mm.	ft/lb.)
Heat No. E-28	25	28·15	(204)
Cr, 21·3	0	25·95	(188)
Mn, 14·1			
N, 0·64	−20	23·46	(170)
C, 0·05			
Si, 0·30	−40	20·97	(152)
	−50	14·35	(104)
	−57	10·49	(76)
	−60	9·936	(72)
	−70	8·142	(59)

Deep drawing tests were performed in Olsen cupping machine and also in Swift cupping press of 6½ in. capacity. Tests in the Swift cupping machine were carried out with flat bottom mandrel using grease-graphite mixture as lubricant. A pressure of 80 lb./in.^{2} was maintained on the pressure plate during the tests. Results of the Olsen cupping tests are given in Table 24.

Corrosion tests were carried out on 1·5 × 0·5 in. specimen prepared from solution-treated sheets. Corrosion tests performed included boiling 65 per cent nitric acid and salt spray; constant immersion were also carried out in vinegar, lime juice plus 1 per cent NaCl, 5 per cent citric acid

Table 24 — Olsen Cupping Tests

Composition %	Thickness of sheet mm.	Depth of cup mm.	Load	
			kg.	lb.
Heat No. 48				
Cr, 21·56 Mn, 15·32 C, 0·08 N, 0·69 Cu, 1·09	0·9143	9·897	4540	10,000
Heat No. 43				
Cr, 17·56 Mn, 13·62 C, 0·09 N, 0·56 Cu, 0·70	0·8915	10·90	4903	10,800
18/8 *Cr-Ni*				
Cr, 18·63 Ni, 10·07	0·9143	12·45	3541	7800

plus 1 per cent NaCl, 5 per cent sulphuric acid (aerated) and 1 per cent hydrochloric acid (aerated). The Cr-Mn-N stainless steels develop high mechanical strength and have good ductility. On 30 per cent cold reduction, the tensile strength was increased to 85 tons/in.^{2} These steels have excellent deep drawing properties. X-ray diffraction analysis indicated the stability of austenite even after cold reduction up to 30 per cent. High temperature tensile tests show high order of strength and ductility at elevated temperatures. The Cr-Mn-N steels are as corrosion resistant as AISI 304 in atmosphere, 65 per cent boiling nitric acid test, salt spray and weak organic acids. These steels, when aged at different temperatures and time, showed that the corrosion resistance up to 400°C. remained unimpaired but started deteriorating rather fast above this temperature. The corrosion rate reached maximum at about 600°C. and thereafter it was reduced till 900°C. when it reached again the original corrosion rate. The oxidation resistance of these steels is inferior to 18/8 stainless steel.

27.1 Aging Characteristics of Nickel-free Austenitic Stainless Steels

With the formulation of a satisfactory explanation of the mechanism of age-hardening in aluminium alloys, similar studies are being made widely on other ferrous and non-ferrous alloys with a view to improve their mechanical properties. As nickel-free austenitic stainless steel has considerable potential application in India, a project has been initiated to study the hardness change, nucleation and growth process of grain boundary precipitate on aging at temperatures ranging from 400° to 900°C., with interval of 50°C., the time ranging from 5 min. to 100 hr. at each temperature.

Specimen of nickel-free stainless steel were given austenitizing treatment. Before austenitizing, the specimens were vacuum-sealed in silica tube and after soaking at 1050°C. for $\frac{1}{2}$ hr., were quenched in water by breaking the vacuum seal. Aging treatments were given at 600° and 800°C. for 1 to 100 hr. A pearlitic type of precipitation starting at grain boundary was noticed on aging at higher temperature. The higher was the temperature of aging, the coarser were the lamellae of the pearlitic type of precipitate. Effect of temperature was much more on the amount of precipitation than the effect of time of aging. Remarkable hardness increase was noticed on aging up to 5 hours at both 600° and 800°C. where the amount of precipitate was not much, specially in case of 600°C. But increase of hardness is more or less same in both the cases, irrespective of the amount of precipitate. The amount of grain boundary precipitate increased on aging with further time, but hardness remained fairly constant.

Future work will envisage the extraction of the precipitate both by electrolytic and chemical method and to identify the compound by X-ray diffraction or chemical method. Electron microscopy study will also be made with specimen aged at lower temperature and lesser time where no appreciable change in micro-structure was visible in optical microscopy but remarkable hardness change was noticed. More heats of steel of similar composition will be made to study the impact and tensile properties which are mostly affected by boundary precipitation.

27.2 Isothermal Transformations in 17 per cent Cr-Mn-N Stainless Steels

The object of this investigation was to study the formation of different phases at various holding temperatures after solution treatment, and correlate the structures thus observed with the mechanical properties of these steels. The effect of the variation of manganese and nitrogen on the phase-formation and their stabilization under different conditions was also under investigation.

Six heats of 20 lb. ingot capacity each were prepared, composition of which are given in Table 25.

Table 25 — Composition of the 17% Cr Steels under Investigation

	Mn %	N %	C %
S_3	6·50	0·34	0·027
S_4	8·30	0·39	0·027
S_5	10·30	0·38	0·054
S_6	12·70	0·37	0·063
S_7	15·69	0·31	0·072
S_8	16·91	0·41	0·108

The ingots were hot-forged and rolled to $\frac{1}{16}$ in. thick sheets and $\frac{3}{4}$ in.^{2} bars for examination. The test pieces for structure analysis were solution-treated at 1050°C. for 15 minutes and water-quenched, and examined by X-ray diffraction, optical and electron-microscopic techniques. The residues were extracted electrolytically and subjected to the same tests. Some results are given in Table 26.

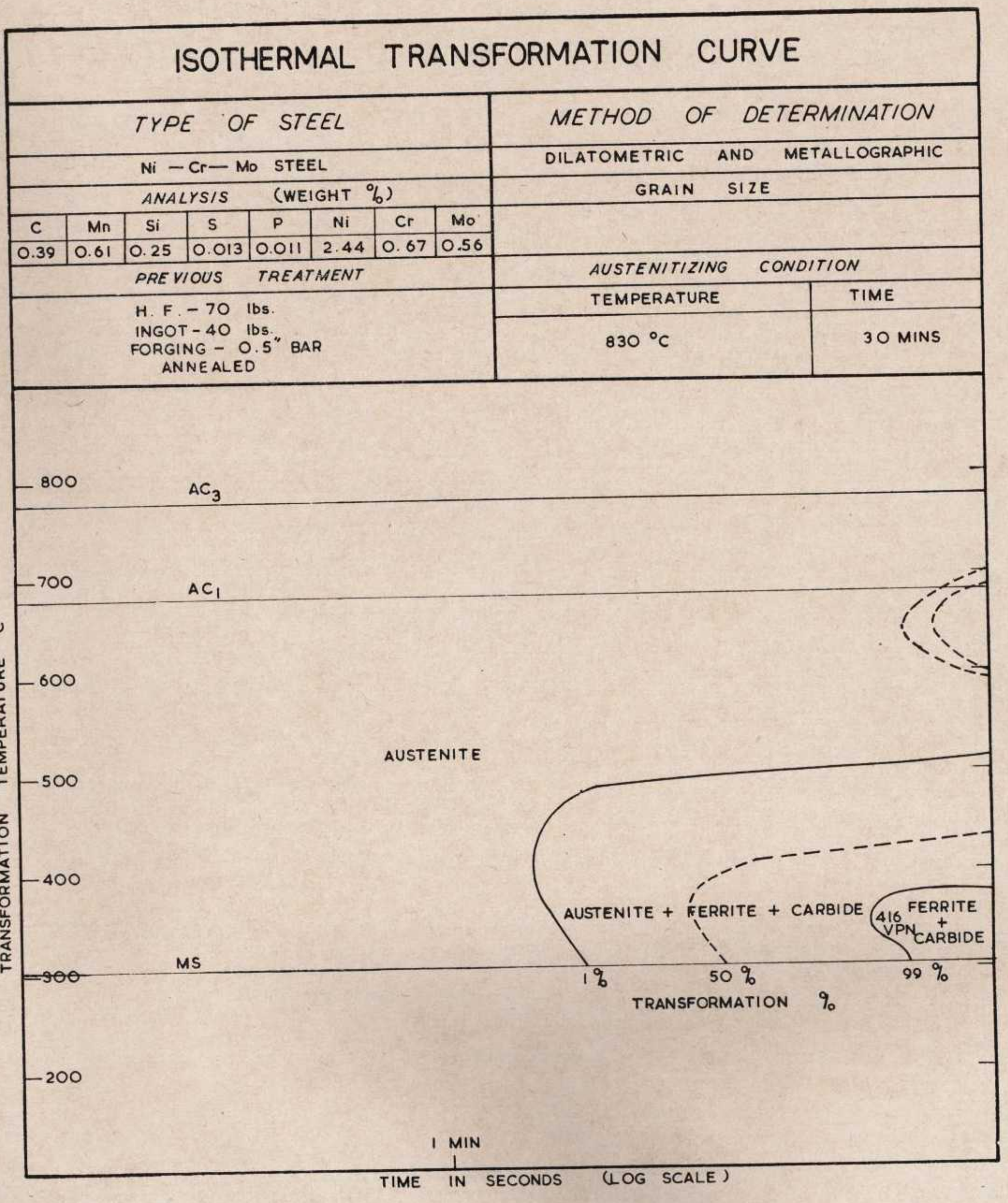
Table 26 — X-ray and Hardness Data of Some of the Steels

Samples No.	Solution treated 1050°C.-15 min. w.q.		900°C.-2 hr w.q.		750°C.-100 hr w.q.	
	Structure	V.P.N. 30 kg.	Structure	V.P.N. 30 kg.	Structure	V.P.N. 30 kg.
S_3	Gamma + Alpha	236	Gamma + Alpha	277	Alpha	490
S_4	Gamma + Alpha	248	Gamma	256	Gamma + Alpha	283

The unusual hardness of 490 V.P.N. exhibited by S_3 at 750°C.-100 hr. treatment is very interesting and offers its feasibility for engineering applications. Further work is in progress.

28.0 Development of Substitute Alloy Steels

Objectives of this Project were based on the basic themes to develop indigenous substitute alloy steels eliminating as far as possible alloying elements, such as nickel, moybdenum, tungsten, cobalt, etc., resources of which do not exist in India; such substitute alloy steels through judicious combinations of indigenous alloying elements and optimum heat treatment should conform to the requirements of physical properties and specific service performance characteristics of standard alloy steels. The project covers the important fields of formulating time-temperature-transformation and continuous-cooling-transformation curves, hardenability studies vis-á-vis ruling sections and optimum heat-treatment cycles and fabrication techniques.



ISOTHERMAL TRANSFORMATION CURVE

TYPE OF STEEL

Ni—Cr—Mo STEEL

ANALYSIS (WEIGHT %)

C	Mn	Si	S	P	Ni	Cr	Mo
0.39	0.61	0.25	0.013	0.011	2.44	0.67	0.56

PREVIOUS TREATMENT

H. F. – 70 lbs.
INGOT – 40 lbs.
FORGING – 0.5" BAR
ANNEALED

METHOD OF DETERMINATION

DILATOMETRIC AND METALLOGRAPHIC

GRAIN SIZE

AUSTENITIZING CONDITION

TEMPERATURE	TIME
830 °C	30 MINS

Fig. 17

(i) *Tool Steels*

Twenty-five heats of high-speed steel were made in 25 lb. basic-lined electric induction furnace substituting non-indigenous element tungsten by indigenously available metals like aluminium, titanium, boron and even abundant nitrogen either singly or in proper combination of two or three elements. Table 27 shows the composition of tool steel. Hardness of these steels were investigated after forging,

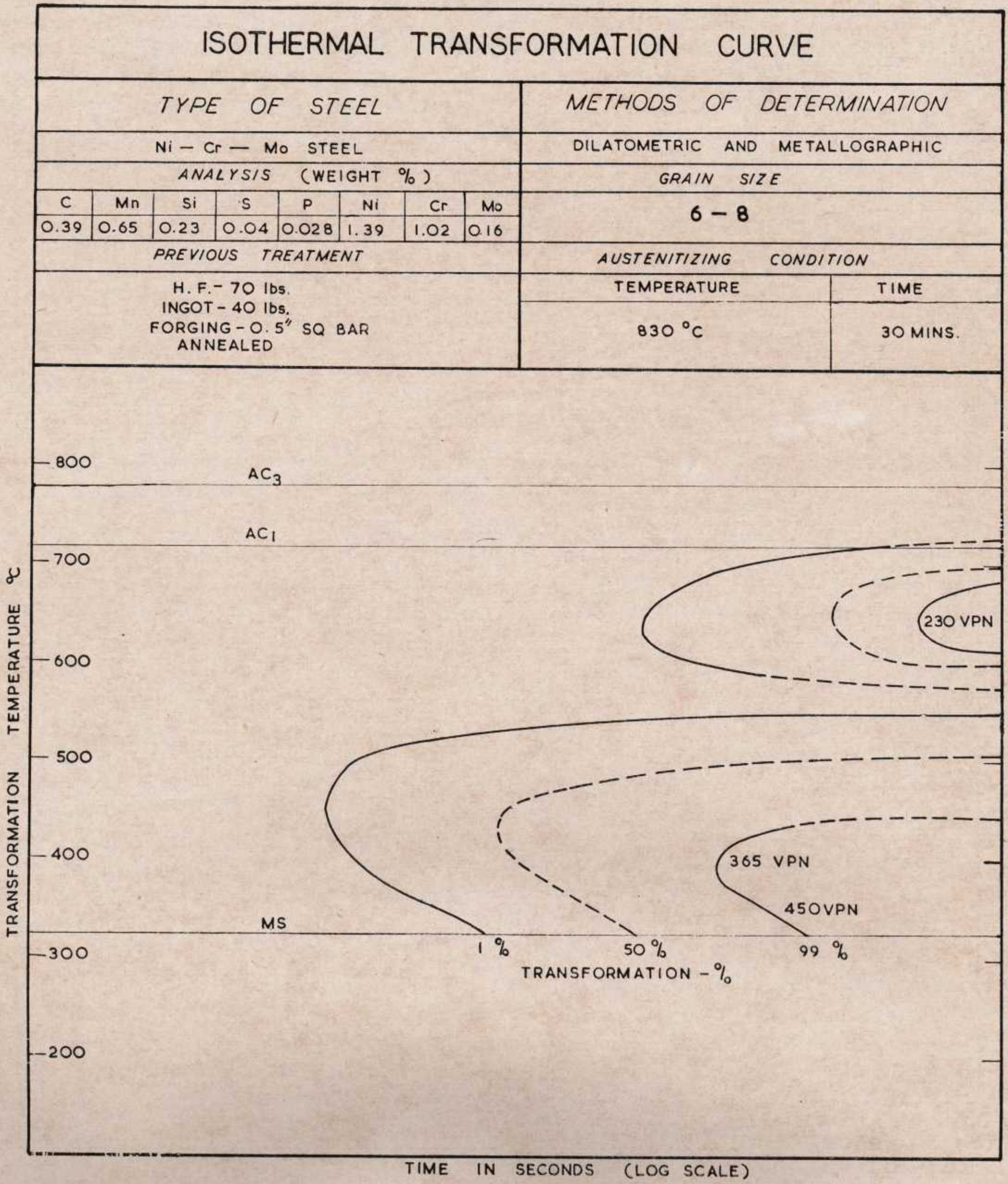


FIG. 18

quenching and tempering. Results of the above steels giving optimum hardness level after single and double tempering are given in Table 28.

(ii) *Ausforming of High-speed Tool Steels*

The effect of mar-working on the 18/4/1 high-speed steel was studied in relation to various percentages of deformation, tempering and sub-zero treatment. It was observed that by application of the new process, a substantial increase in hardness was obtained. Table 29 shows the hardness results obtained after various degrees of deformation

Table 27 — Chemical Composition of Steels

Steel No.	C %	Mn %	Si %	W %	Cr %	V %	Al %	Ti %	N %	B %	Zr %	Cb %
TS 1	0·81	0·54	0·23	6·4	4·50	1·35	0·3	—	0·08	—	—	—
TS 2	0·83	0·43	0·20	6·6	4·42	1·41	0·6	—	0·12	—	—	—
TS 3	0·80	0·54	0·74	6·3	4·48	1·5	0·8	—	0·13	—	—	—
TS 4	0·85	0·59	0·28	6·7	4·40	1·56	—	0·1	—	—	—	—
TS 5	0·84	0·54	0·31	6·6	4·5	1·52	—	0·3	—	—	—	—
TS 6	0·82	0·53	0·32	6·8	4·45	1·49	—	0·48	—	—	—	—
TS 7	0·85	0·53	0·35	18·1	4·6	1·56	—	—	—	—	—	—
TS 8	0·78	0·48	0·25	6·4	4·3	1·2	1·0	0·5	—	—	—	—
TS 9	0·80	0·51	0·27	6·3	4·2	1·15	—	—	—	0·09	—	—
TS 10	0·81	0·58	0·31	5·96	4·1	1·4	—	—	—	0·17	—	—
TS 11	0·79	0·57	0·29	5·9	4·3	1·2	—	—	—	0·26	—	—
TS 12	0·82	0·61	0·28	6·5	4·3	1·1	—	—	—	—	0·16	—
TS 13	0·85	0·55	0·23	6·4	3·96	1·5	—	—	—	—	0·43	—
TS 14	0·90	0·57	0·25	6·1	3·4	1·3	—	—	—	—	—	0·40

Table 28 — Hardness after Tempering (V.P.N.)

Steels	At 450°C.		At 550°C.		At 600°C.		At 650°C.	
	Single	Double	Single	Double	Single	Double	Single	Double
TS 1	735	755	804	810	755	790	625	700
TS 2	800	810	851	856	805	825	670	750
TS 3	770	750	790	804	770	765	700	695
TS 4	785	790	842	850	775	775	655	655
TS 5	802	812	870	880	806	790	675	670
TS 6	776	775	841	845	760	770	635	635
TS 7	810	820	852	862	800	815	670	675

Table 29 — Hardness Results Obtained

Percentage of reduction	Hardness (V.P.N.)							
	After ausforming	After sub-zero treatment	Tempering temperature °C					
			475		525		575	
			Single tempering	Sub-zero treatment	Single tempering	Sub-zero treatment	Single tempering	Sub-zero treatment
6	810	832	823	836	842	854	850	866
10	818	835	838	852	850	852	856	868
35	850	858	858	864	868	875	876	881
55	860	860	863	864	875	—	880	880

and subsequent tempering and sub-zero treatment.

(iii) *Development of Die Steels*

Ausforming technique was applied to the high-carbon high-chromium cold work die steels to study the strengthening effect. The process consists in heating the sample of 1 in. cross-section and 2½ in. length to the austenitizing range and after allowing the samples to be plastically deformed in the stable austenitic region below the critical temperature and quenching in air to develop martensite product, it was observed that the hardness of the steel increased by 104 V.P.H.N. at 60 per cent

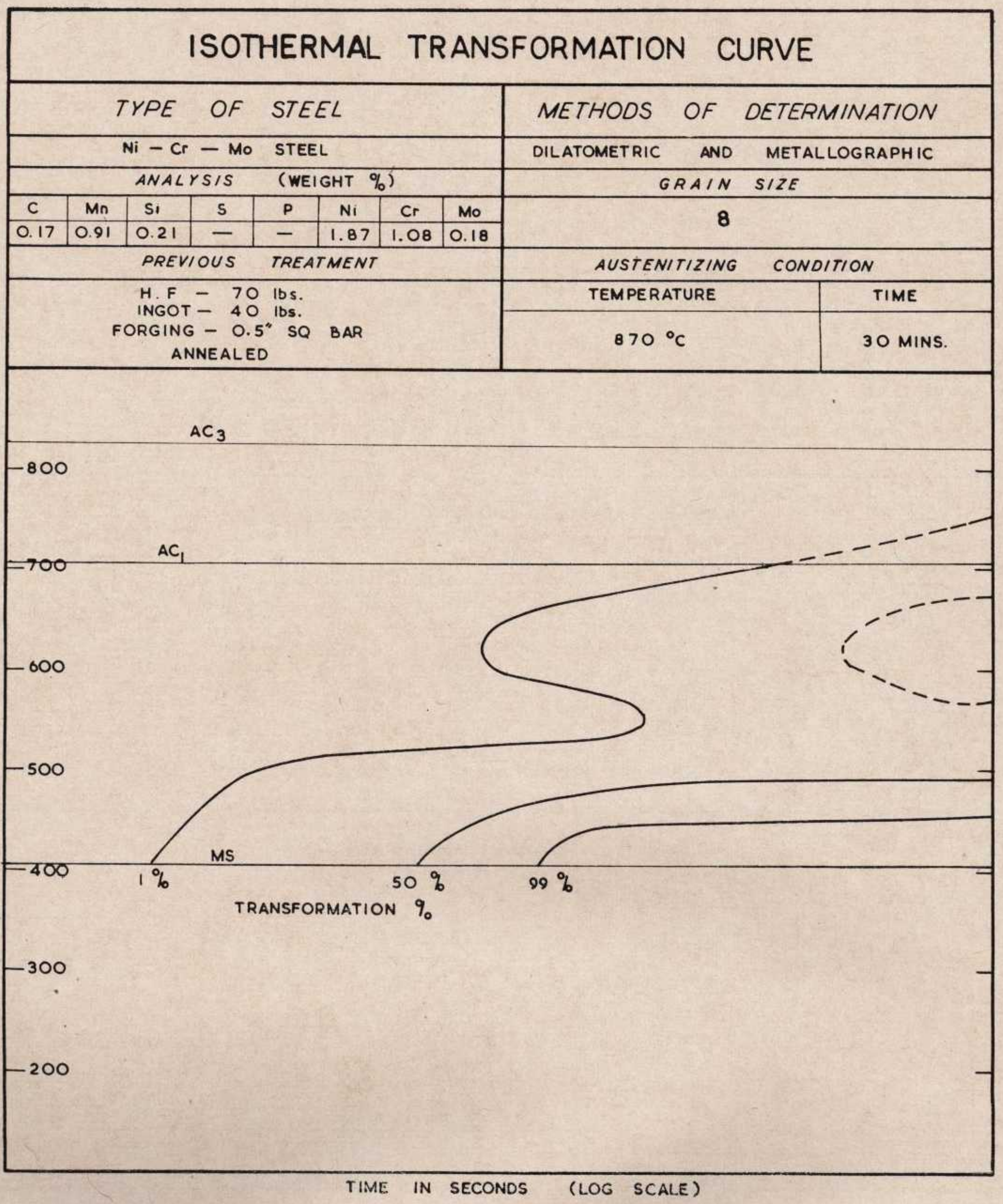


FIG. 19

deformation level. Table 30 gives the hardness achieved at different percentages of deformation.

Other characteristics, such as effect of single and double tempering, effect of sub-zero treatment, etc., are being studied.

Table 30 — Hardness obtained at Different Stage of Deformation

Percentage of reduction	Hardness (V.P.H.N.)
0	688
40	775
60	792

<table>
<tr><td colspan="10">ISOTHERMAL TRANSFORMATION CURVE</td></tr>
<tr><td colspan="8"><i>TYPE OF STEEL</i></td><td colspan="2"><i>METHODS OF DETERMINATION</i></td></tr>
<tr><td colspan="8">Ni — Cr — Mo STEEL</td><td colspan="2">DILATOMETRIC AND METALLOGRAPHIC</td></tr>
<tr><td colspan="8"><i>ANALYSIS</i> (WEIGHT %)</td><td colspan="2"><i>GRIN SIZE</i></td></tr>
<tr><td>C</td><td>Mn</td><td>Si</td><td>S</td><td>P</td><td>Ni</td><td>Cr</td><td>Mo</td><td colspan="2" rowspan="2">8</td></tr>
<tr><td>0.16</td><td>0.56</td><td>0.27</td><td>—</td><td>—</td><td>2.01</td><td>1.51</td><td>0.20</td></tr>
<tr><td colspan="8"><i>PREVIOUS TRETMENT</i></td><td colspan="2"><i>AUSTENITIZING CONDITION</i></td></tr>
<tr><td colspan="8" rowspan="2">H. F. — 70 lbs.
INGOT — 40 lbs.
FORGING - 0.5" SQ. BAR
ANNEALED</td><td>TEMPERATURE</td><td>TIME</td></tr>
<tr><td>860°C</td><td>30 MINS.</td></tr>
</table>

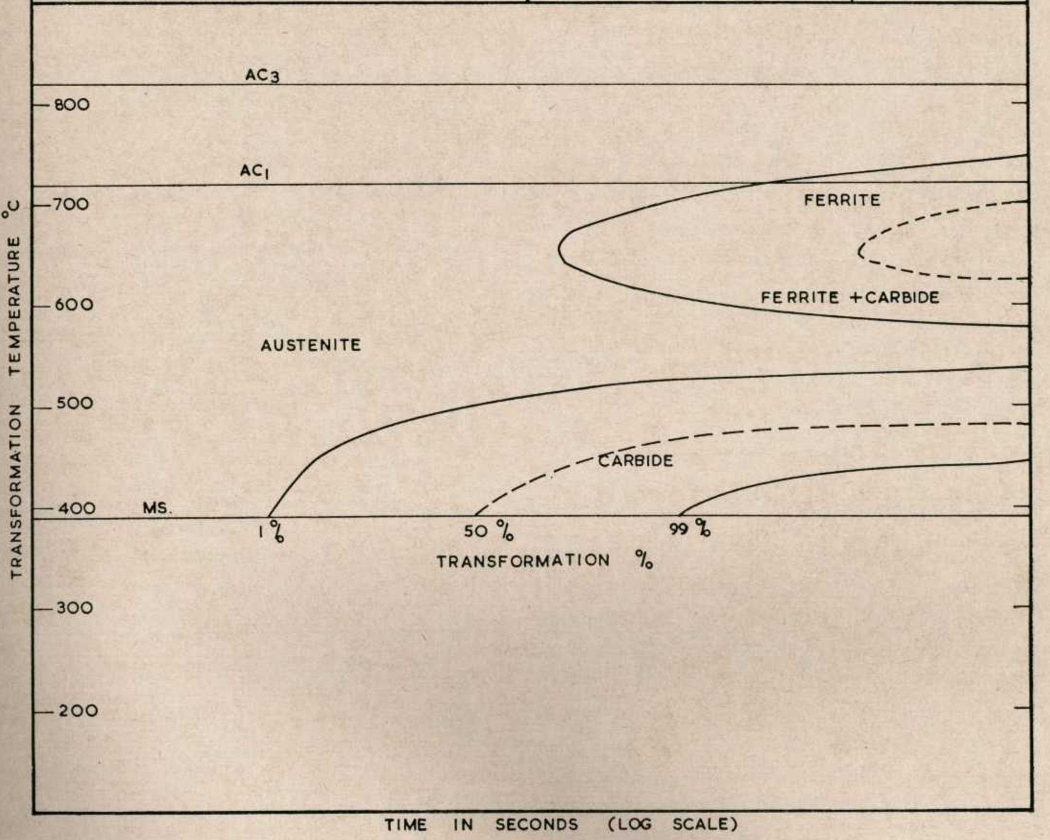


FIG. 20

(iv) *Isothermal Transformation Characteristics of Alloy Steels*

This project was taken up with a view to prepare an atlas of isothermal transformation diagrams of Indian alloy steels including substitute alloy steels under development at the National Metallurgical Laboratory.

Some of the isothermal transformation diagrams prepared from ISI steels are shown in Figs. 17 to 20, and Table 31 gives the chemical composition of steels.

Table 31 — Composition of Steels in Percentage Weight

	C %	Mn %	Si %	S %	P %	Ni %	Cr %	Mo %
1	0·39	0·61	0·25	0·013	0·011	2·44	0·67	0·56
2	0·39	0·65	0·23	0·040	0·028	1·39	1·02	0·16
3	0·17	0·91	0·21	—	—	1·87	1·08	0·18
4	0·16	0·56	0·27	—	—	2·01	1·51	0·20

(v) *Continuous-cooling-transformation of Alloy Steels*

This work was undertaken with a view to study the continuous-cooling-transformation characteristics of Indian alloy steel and to prepare an atlas of the continuous-cooling-transformation diagrams for use in Industry.

Some of the continuous-cooling-transformation diagrams prepared from ISI steels are shown in Figs. 21 to 23 and the chemical composition of the steels corresponds to 1, 2 and 3 in the preceding table.

(vi) *The Effect of Prior Treatments on Precipitation Reaction in Alloy Steels*

Studies were made on the effect of prestraining on the precipitation reactions in C, 0·45-W, 2·45 & Mo, 0·1; C, 0·94-W, 1·17 & Mo, 0·04; C, 0·88-W, 2·73 & Mo, 0·08 and C, 0·63-W, 1·13 & Mo, 0·046 steels. Specimens were given austenitizing treatment at 1050°C. for 1 hr in argon atmosphere followed by quenching in water. A pretempering treatment at 250°C. for 1 hr was required for straining the specimens for studying their subsequent tempering characteristics.

Mechanical properties such as tensile strength, ductility (elongation per cent, reduction in area per cent), impact strength and hardness were studied before and after tempering by varying the amount of prestraining and subsequent tempering. Some of the results are given in Tables 32 to 35.

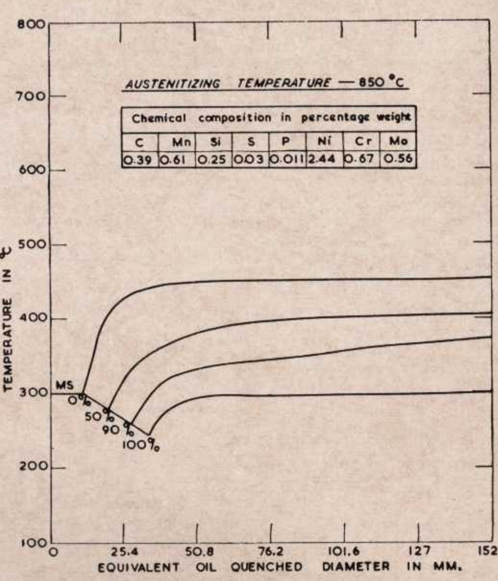


FIG. 21 — CONTINUOUS-COOLING-TRANSFORMATION DIAGRAM FOR STEEL

Table 32 — Composition of Steels

Sample No.	Composition C %	W %	Mo %
Rx_1	0·95	2·45	0·100
Rx_2	0·94	1·17	0·040
Rx_3	0·88	2·73	0·046

Arrangements were being made to study the nature and structure of the precipitated phases formed during tempering with and without straining both by electron microscopic and X-ray diffractometroanalysis. Extraction replicas from the pretempered and strained samples were examined under

Table 33 — Mechanical Properties after Prestraining and Tempering

Pretempering temperature ... 250°C., 1 hour
Retempering temperature ... 450°C., 1 hour

Sample No.	Amount of prestrain %	Elongation %	R.A. %	T.S. Tons/ in.^{2} %
Rx_1	0·5	1·7	3·7	80
Rx_2	0·5	2·1	4·4	70
Rx_3	0·5	1·2	2·6	74
Rx_1	0·4	1·9	3·9	76
Rx_2	0·4	2·2	4·6	64
Rx_3	0·4	1·4	2·8	72
Rx_1	0·7	1·5	3·1	82
Rx_2	0·7	1·9	3·9	71
Rx_3	0·7	1·1	2·2	80

Table 34 — Mechanical Properties after Prestraining and Retempering

Pretempering temperature ... 250°C., 1 hour
Degree of prestrain ... 0·5%

Sample No.	Retempering temperature °C.	Elongation %	R.A. %	T.S. tons/ in.^{2} %
Rx_1	400	1·5	3·5	86
Rx_2	450	1·7	3·7	80
Rx_3	500	2·1	4·0	75
Rx_1	400	1·9	3·8	72
Rx_2	450	2·1	4·4	70
Rx_3	500	2·4	4·8	62
Rx_1	400	1·0	2·5	78
Rx_2	450	1·2	2·6	74
Rx_3	500	1·5	3·1	66

Table 35 — Mechanical Properties after Direct Tempering

Direct tempering ... 450°C., 1 hour

Sample No.	Tempering temperature °C.	Elongation %	R.A. %	T.S. tons/ in.^{2}
Rx_1	450	1·8	3·8	70
Rx_2	450	2·2	4·6	58
Rx_3	450	1·2	2·6	65

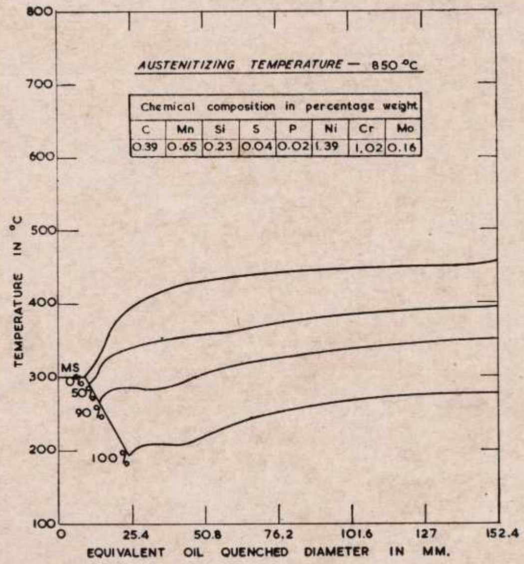


Fig. 22 — Continuous-cooling-transformation Diagram for Steel

electron microscope for the possible precipitations. Few selected diffraction patterns were also taken. In order to isolate the precipitates for their X-ray diffraction analysis an electrolytic carbide extraction set up under inert atmosphere is being fabricated.

29.0 Development of Low-alloy High-tensile Structural Steel

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

The previous experimental heats were made with a view to improve the properties of the existing low-alloy steels in use by judicious modification in the percentages of alloying elements. The main alloying elements of these heats were manganese, chromium, silicon and copper. To improve the yield strength, elements such as aluminium, vanadium and titanium, which act as grain refiners and strengthen the material by going into solution and in some cases give rise to precipitate hardening were added. To achieve better weldability, carbon, the principal cause of welding trouble, was maintained at a low level of about 0·15 per cent. To impart fair resistance against atmospheric corrosion copper of the order of 0·4 per cent was added to most of the steels. Phosphorus which is known to improve corrosion resistance in a better way along with copper was also added in some of the steels. During the period under review, work in connection with the determination of various properties of the steels were continued. Some of the sub-zero impact test results are given in Table 36.

Table 36 — Charpy Impact Values (ft/lb.) of Some Experimental Heats

Heat No.	Room temperature	0°C.	−20°C.	−40°C.	−60°C.	−70°C
39/1	56	50	40	38	38	38
43	94	25	20	9	7	—
44	90	54	32	16	19	—
—	—	—	48	—	—	—
45	60	22	—	14	19	—
54	26	18	8	4	5	—
—	—	14	—	—	—	—
41/3	90	79	60	42	—	—

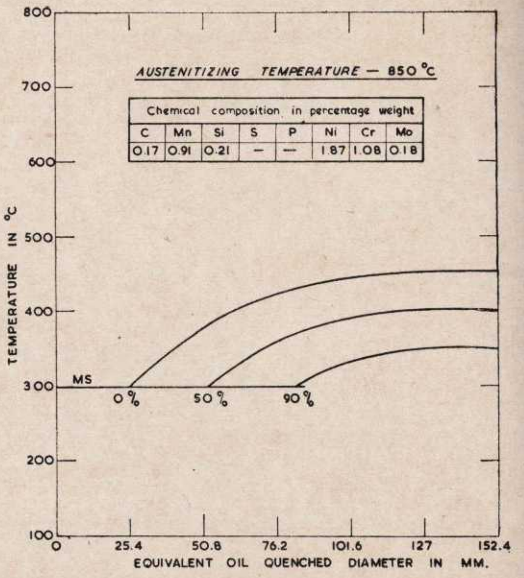


FIG. 23 — CONTINUOUS-COOLING-TRANSFORMATION DIAGRAM FOR STEEL

Further, a few fresh heats were made with small amount of nitrogen and nitride forming elements (Al, V, Ti) which markedly improved the tensile properties, specially the yield point, while preserving adequate ductility. These were 10 kg. heats made in the high-frequency furnace. The ingots were hot-forged from 1150°C. The ingots which contained aluminium were forged from still higher temperature to allow all the aluminium nitride to go into solid solution.

The improvement in mechanical properties by the nitrides is associated with grain refinement and depends on the extent to which the nitrides are dispersed. Normalizing after hot working from the proper temperature is reported to be essential to get desired micro-structure and high-strength properties. To determine the proper temperature of normalizing, small pieces from the hot-forged bars were normalized from different temperatures and prepared for hardness measurements, micro-structure examination and grain-size measurements. Table 37 gives the hardness values of the specimens normalized from different temperatures. To examine the effect due to solution hardening, micro-hardness measurements on the ferrite grains were also made. Charpy impact tests and sub-zero temperatures will be carried out when the proper normalizing temperatures of the steels are determined. Tensile tests after normalizing from different temperatures are in progress. A few results of heat No. 58 are given in Table 38.

Bigger heats of the type mentioned above will be taken up after results of tests of the small heats show a satisfactory trend.

Table 37 — Hardness Values (V.P.N.) of Some Nitrogen Bearing Compositions at Various Normalizing Temperatures

Heat No.	Normalizing temperature						
	920°C.	950°C.	1000°C.	1050°C.	1100°C.	1150°C.	1200°C.
56	150	157	155	155	145	150	156
58	169	181	172	225	230	230	229
59	142	135	130	133	127	137	148

Table 38 — Tensile Test Results of Heat No. 58 when normalized from 1000°, 1150° and 1200°C.

Heat No. 58	U.T.S. (T.S.I.)	Y.P. (TSI)	Elongation %	R. Area %
Normalized 1000°C.	39·4	32·5	33	68
Normalized 1100°C.	43·5	—	28	65
Normalized 1150°C.	48·2	—	28	60

30.0 Improved Mild Steel for Structural Purposes

The mechanical properties of mild steels can be vastly improved with alloying additions of residual quantities and these improved varieties have the desirable combination of properties for structural applications. The desirable properties of low-alloy structural steels are (i) high yield strength, (ii) low impact-transition temperature, and (iii) good welding properties. The yield strength can be increased by grain refinement, or precipitation hardening or solid solution strengthening. Low-impact transition temperature can usually be achieved by grain refinement. Weldability can be improved by reducing the carbon content to a minimum.

Heats having low C-Mn base were made with Nb-V-N and Ti-Nb additions. The compositions of the steels and the properties obtained after normalizing at various temperatures are shown in Table 39. The results were analysed on the basis of Petch's relationship and its derivations and the factors responsible for the change in properties were accounted for. Further work is in progress.

31.0 Development of Electrical Resistance Alloys for Heating Elements

Conventional heating elements used for domestic or industrial heating appliances contain high content of nickel and some cobalt. Resources of nickel and cobalt do not exist in India. With increase in the availability of electric power for consumer use and industrial growth, demands for such heat-resistance electrical elements for domestic and industrial heating appliances have substantially risen in India. With a view to develop electrical heat-resistance elements containing no nickel or cobalt, research and development work was taken up which led to the development of indigenous electrical heating elements.

Major work on the development of heating element alloys had already been completed and the process was leased to the industry. During the period under review, the work chiefly related to study of weldability of the alloy developed. For the production of heating element wires, it is often necessary to butt-weld the wire when these break during wire-drawing operation. Welding of the Fe-Cr-Al alloys is generally difficult due to excessive grain-coarsening and also due to oxidation of the constituent elements Cr and Al during the welding operation. This led to formation of weak and brittle weld zone which results in failure of the joint during subsequent wire-drawing operation.

Table 39 — Properties of the Steels Studied

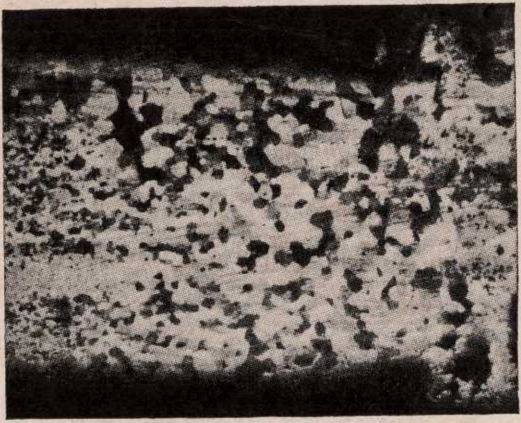
Cast No.	Composition	Normalizing temperature °C.	U.T.S. t.s.i.	L.Y.S. t.s.i.	Yield/tensile ratio	Percentage pearlite	Grain diameter micron	Transition temp. °C.		V.P.N. 30 kg. load
								40 ft/lb. value	50% fibrons	
3	C, 0·07 Mn, 1·55	850	37·0	27·00	0·73	4·8	7·63	—	—	161·0
	V, 0·20 Nb, 0·06	900	41·0	28·75	0·70	8·3	12·05	−48	−42	167·0
	Si, 0·018 S, 0·045	950	42·5	29·50	0·70	10·0	13·05	−40	−30	183·0
	P, 0·018 N, 0·009	1000	42·5	30·00	0·70	11·5	14·01	−24	−14	188·0
		1050	44·0	—	0·70	11·0	27·00	—	—	192·0
4	C, 0·10 Mn, 1·64	850	35·0	28·50	0·81	6·4	8·60	—	—	158·0
	V, 0·19 Nb, 0·06	900	37·0	29·50	0·80	8·0	9·70	−68	−56	161·0
	Si, 0·018 S, 0·042	950	37·4	29·70	0·90	9·0	9·80	−50	−41	163·0
	P, 0·016 N, 0·006	1000	38·0	30·00	0·79	10·0	11·00	−35	−35	166·0
		1050	41·0	32·50	0·79	10·0	28·00	—	—	185·0
5	C, 0·10 Mn, 1·64	1000	37·0	28·75	0·78	13·3	16·00	−6	—	164·0
	Nb, 0·04 Ti, 0·06	1050	38·5	29·75	0·79	8·6	19·40	0	—	170·0
	Si, 0·08 P, 0·02	1100	39·5	30·00	0·77	8·0	21·30	+8	—	180·0
	S, 0·03	1150	48·25	37·00	0·77	4·6	21·10	—	—	205·0

To overcome this difficulty, a study was undertaken to evolve a suitable method of butt-welding the alloys. For this purpose, an electric resistance micro-welding machine was employed. Varied conditions of pressure and heating current were used with and without flux. The end of the wires to be joined was either kept flat or in tapered condition with varying degrees of taper.

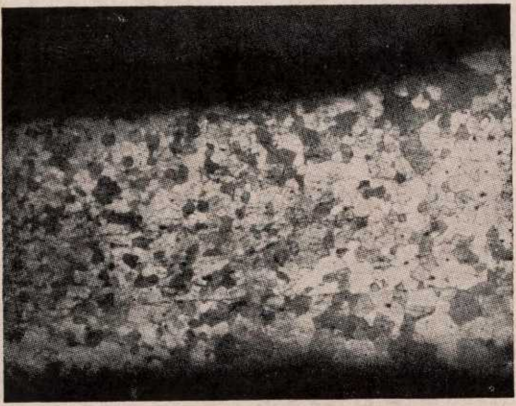
With tapered ends the welds produced were found to be quite ductile provided neither the heating current nor the pressure applied were too excessive. Under optimum conditions of these two variables, the weld produced could be successfully cold-drawn from 13 to 19 SWG size without any breakage. Some tests were conducted to study the effect of argon atmosphere on the quality of the weld. Under the present experimental arrangement some oxidation was noticed even with argon application. These aspects are further being examined.

As seen from the micro-structure of two typical samples, one with the tapered end and the other for the flat end [Figs. 24 (a) & (b)], the grains just within the weld zone are very fine. On either side of it the grains are coarse. This coarse grain zone covered extended area in case of specimens with flat end joints as compared to the tapered end joints. This was one of the reasons for the latter to give ductile welds. The correlation of micro-structure with the welding parameters was being made.

Loss of ductility of the wires under prolonged soaking at elevated temperature was studied by the bend ductility tests on the experimental alloys as well as a few common heating element alloys for comparison purposes. The alloy wires of 28 SWG size were



(a) With tapered end



(b) With flat end

FIG. 24 — MICRO-STRUCTURE OF THE WELDED ZONE

tested after annealing for ½ hr. at 750°C. and further soaking at 1000°C. for 15 hr. The test results are given in Table 40 where it would be seen that for the temperature and soaking period employed, the new alloys behaved almost in the same way as conventional heating elements. However, further tests for longer period of soaking at still higher temperatures are yet to be carried out.

Further experiments are under way to study the effect of trace additions of misch metal with or without Zr and Ti on the working and other physical properties of the alloys.

Table 40 — Bend Ductility of the Heating Element Wires under Various Heat Treatments

Material (Wire size) (0·376 mm.)	Number of 180° reverse bends on 2·5 mm. radius under condition of following treatment		
	Cold drawn	Annealed 750°C. ½ hr	Soaked at 1000°C. for 15 hr
E 31	11	16	11
E 31	15	18	12
E 6	7	14	8
2 E 28	10	16	10
Kanthal DS	—	16	8
Nichrome	—	15	8

32.0 Development of Magnetic Materials

(i) *Hard Ferrites*

During the period under review, work on development of magnets from strontium and iron oxides was taken up. A number of compositions containing strontium oxide 0·2, 0·4, 0·6, 0·8 and 1·0 mols and 6 mols of iron oxide were prepared and calcined at 1000°-1100°C. after thoroughly mixing in an edge-runner mill. The calcined mass was pulverized to 300 mesh in a pulverizer. Raw compacts of about 2 cm. dia. × 1 cm. were prepared from the fine powder by pressing it in a hydraulic press under a pressure of about 5 tons/in.^{2}. The pressed compacts were then sintered at 1080° to 1280°C. for 2 hr. and subsequently tested for their magnetic properties. In case of ferrites containing 0·8 mols. of strontium oxide the coercive force value ranged from 100-1400 oersteds and remnant from 1600-2000 gauss. The shrinkage values for strontium-ferrites containing 0·8 mols strontium oxide ranged from 8·7 per cent at 1080°C. to 33·5 per cent at 1280°C.

Practical demonstration was arranged for the licensees to appraise them of the technical know-how of the process of manufacturing barium, ferrite magnets.

(ii) *Study of Binary and Ternary Alloys of Manganese-aluminium Iron*

During the period under review, experiments were carried out to remove the fragility of the cast rods of manganese-aluminium alloys having aluminium content in the range of 28 to 30 per cent. It was observed previously that the cast rods disintegrated into a fine powder on storage. This was considered due to either eventual escape of gases absorbed during melting or reaction of atmospheric gases and moisture with alloy constituents or phase changes. It was observed that the eventual disintegration of the cast ingots disappeared if the alloy was doped with carbon up to 0·5 per cent or small percentages of other elements like copper, nickel and iron. Such cast ingots developed ferro-magnetism when they were either cooled from 1100° to 600°C. at the rate of 12° to 15°C./sec., or if they were tempered at 500°-550°C. after water-quenching from 1100°C.

The X-ray diffraction studies of the ferromagnetic phase showed that it was tetragonal in structure having lattice parameter $a = 2{\cdot}96$ Å, $C = 3{\cdot}52$ Å. The specimens cooled at other rates showed lines for a hexagonal structure also. The specimens were tested ballistically for the saturation induction values. In the quenched and tempered condition the specimens had saturation induction 2000 gauss, while in the critically cooled state the value was slightly less.

(iii) *Soft Ferrites*

Work on soft ferrites was undertaken to develop a technology and to study the suitability of indigenous raw materials for their manufacture. In the previous report work on preparation of nickel-zinc ferrites was described. During the period under review, preparation of manganese-zinc ferrite was undertaken with a view to use them as radio antenna rods. Iron oxide, zinc oxide and manganese oxide in molar proportion of 50: 30: 20 were taken and mixed thoroughly. One of the lots of the mixture was directly extruded into rods, dried at 250°C. and sintered at temperature of 1200°, 1260°, and 1280°C. for 2 to 6 hours with heating and cooling cycle of about 32 hours. The other lot was first calcined, powdered and then extruded into rods for sintering. The rods prepared from both the lots were comparatively tested for their hysteresis loop with the help of ferrotester. The rods prepared from the calcined powder not only showed better magnetic properties than the ones prepared from uncalcined powder, but also had comparable loops with the nickel-zinc ferrites prepared in the National Metallurgical Laboratory and those imported from abroad.

(iv) *Study of Electrical and Magnetic Properties of Some Low-manganese Low-aluminium Steel*

During the period under review, a 20 kg. heat was made containing iron, 95·5; aluminium, 3 and manganese, 1·5 per cent in the high-frequency induction furnace. Calcium-aluminium alloy was used as a deoxidizer. The ingot was hot-forged and rolled to 0·137 in. thick sheets which, after pickling, were cold-reduced to 0·03 and 0·018 in. thickness. These were then annealed in air at 900°C. and in vacuum at 1100°C. Air-annealed sheets at 900°C. showed better permeability than vacuum-annealed ones, other magnetic properties like saturation induction and coercive force remaining almost the same. Air-annealed sheets had $\mu = 5000\text{-}7000$, $Hc = 0{\cdot}4\text{-}0{\cdot}8$ oersteds and $Bs = 14\text{-}15000$ gauss. Vacuum-annealed sheets had $\mu = 3000\text{-}5000$ $Hc = 0{\cdot}5\text{-}0{\cdot}8$ oersteds and $Bs = 1300$ to 15000 gauss.

Attempts were made to improve the value of permeability by development of texture in the sheets. Sheets of 0·137 in. thickness were reduced to 0·03 in., annealed at 900°C. for 30 seconds to 30 minutes, cold-reduced to 0·01 in. and finally annealed at 1100°C. for 5 hours. The tests showed that such sheets actually were inferior in properties to hot-rolled sheets. The permeability was hardly 3000. Failure to develop proper texture associated with high permeability may be due to large number of alumina inclusions present as coarse particles throughout the grains. Attempts are now being made to make more clean ingots and study the recovery and recrystallization temperatures in these sheets.

33.0 Light Metals and Their Alloys

This project was taken up with a view to develop aluminium-based alloys possessing high strength to weight ratio and adequate resistance for stress corrosion, covering the use of rare earth group of elements. Investigations were conducted in different fields of study relating to production of aluminium alloys with high percentages of magnesium, alloying effects with rare earth metals, working characteristics, stress corrosion and aging characteristics at room and elevated temperatures.

Work on light metal alloys, was further continued on the following lines:

(i) Hot- and cold-working characteristics of aluminium alloys with high percentages of magnesium.

(ii) Stress corrosion characteristics of Al-Mg alloys.

(iii) Age-hardening characteristics of Al-Mg alloys.

In earlier studies on hot-working characteristics of Al/7-10 per cent Mg alloys, it was observed that straight aluminium alloys with high percentages of magnesium were very difficult to hot-roll. It was felt that the failures experienced in the hot-forging and hot-rolling stages might have been caused by inherent degassing and pouring practices. So, in order to verify the previous results, further experiments were made to rule out the possibility of defects arising during different stages of alloy preparation. Different fluxes were tried. Rare earth fluoride was also tried. Good results were obtained in comparison with proprietory fluxes of the market. The effect of mould material and pouring temperature was also studied. Graphite moulds with cross-section of 1$\frac{3}{4}$ × 4 in. and 20 in. in length were prepared from used electrodes and hot metal was poured in this mould. Cast billets obtained from such graphite moulds showed better hot-rolling characteristics. Micro-structures of the experimental alloys in 'as cast' condition and in different heat-treated conditions were studied. Grain size studies showed big grains. Solution heat treatment at different temperatures followed by varied types of quenching were tried to improve grain size. Misch metal was added in the melts in order to study the effects of rare earths on grain size.

As a result of all these experiments, it was concluded that aluminium-7 per cent magnesium alloy without any additional element cannot be hot-forged and hot-rolled satisfactorily. In most cases edge cracks and crocodile cracks appeared during rolling. However, with 1-3 per cent misch metal additions hot-working of the alloy was satisfactory and sheets were cold-rolled to 3·2 mm. thickness. The proof stress, tensile strength, percentage elongation, etc., were determined in the cold-rolled condition and also after aging at 150°C. for different periods of time. Stress-corrosion characteristics of the alloy are being studied. Specimens had been subjected to static stress in artificial sea water medium for a period exceeding one year. Stress-corrosion characteristics of the alloy in cold-rolled, heat-treated and aged condition (aged at 150°C.) is under investigation.

It had been observed that corrosion-resistance characteristics of the alloy are very good. Only in a few samples corrosion products were deposited heavily. Cracks developed in a few samples were metallographically examined. Intergranular cracks were observed in most cases.

33.1 Aluminium-Silicon Alloys

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

Some more work on Al-Si alloys, specifically on eutectic composition, was done. These alloys were modified by different modifying agents singly and in combination and the resulting micro-structure, mechanical properties and fluidity patterns were studied. The technique of modifying Al-Si alloys both in hypo- and hyper-eutectic stage by a single modifying agent has been licensed to a West German party.

33.2 Development of Aluminium-base Bearing Alloys

Due to the short production of lead and the absence of tin deposit in the country, work has been initiated on the development of substitute aluminium-base bearing alloy. Some small bearings are being made which will be put into service trial. The wear and lubrication tests of the test samples are being conducted at the Chittaranjan Locomotive Works.

33.3 Aluminium Bronze

The project was undertaken to study the melting and casting characteristics of the alloy to attain the maximum mechanical properties comprising ultimate tensile strength and proof stress coupled with high elongation in the following range of composition:

Aluminium	...	8·5-11·0 per cent
Iron plus manganese		10 per cent max.
Copper	...	Balance

The work on the project was completed and a brief account is furnished below.

Experimental melts were made in gas- and oil-fired furnaces, indirect arc and direct arc furnaces. Different melting techniques were adopted to improve the quantity of the ingot with respect to porosity, oxide inclusions and good surface finish. Melt were conducted with different flux covers to study their effect in metallic losses during melting and the mechanical properties with the change in composition.

The first series of metals were conducted in gas-fired and oil-fired crucible furnaces of 5 to 6 kg. capacity and the following raw materials were used:

(a) Fire refined copper ingot.
(b) Master alloy of copper-aluminium (50 per cent Al).
(c) Master alloy of iron-aluminium (70 per cent Al).
(d) Master alloy of copper-manganese (30 per cent Mn).

The use of these master alloys greatly helped in reducing dross formation, metallic losses and melting period. Three kilogram heats were made in all the cases and normal time required to complete melting and casting operation was about 50-60 minutes with cold charge and about 45 minutes with pre-heated copper scrap. In crucible melts, attempts were made to add iron as such in the place of iron-aluminium master alloys, but it was observed that the melting period was unduly extended, for taking iron into the solution even though iron was used as hoop iron along with the aluminium addition to take advantage of heat evolved by the exothermic reaction of aluminium oxide formed with the oxides in the molten alloys of copper and manganese alone. No flux was used in these experiments, as the tenacious aluminium oxide formed during the experiment was sufficient to avoid any gas pick-up from the furnace atmosphere. The temperature measurement was made by immersion thermocouple and pouring temperature (1080°-1180°C.) was maintained in all the melts. The alloys were cast in special type chill mould in order to provide small ingot test pieces to be taken from the wings of the ingot. Use of a few drops of carbon tetrachloride was made in the mould prior to tap to reduce the formation of oxide inclusions and to get good surface finish of the cast ingot, as it was revealed that the vapours of carbon tetrachloride evolved at the time of pouring envelop the pouring stream and reduce the oxides which are entrapped due to turbulance in the normal atmosphere.

The second stage of the experimental work on aluminium-bronze was made in 5 kg. capacity indirect electric arc furnace in order to study the melting conditions with and without fluxes with respect to porosity, oxide inclusions and melting losses in the cast product. Carbon tetrachloride was added in the mould as usual to prevent the pouring stream from oxidation. Further melting experiments were conducted to produce aluminium-bronze casting using copper wire (used scrap cable), aluminium ingot, electrolytic manganese, ferro-manganese, iron-aluminium master alloy and hoop iron. Using NaCl plus KCl as a flux cover, the melting losses in these experiments were found quite low and ranged from 2 to 5 per cent. In some cases slightly higher percentage of losses were obtained which was due to rocking of the furnace though all efforts were made to keep a regular atmosphere of NaCl and KCl fumes in the furnace. In these experiments pouring temperatures were attained easily and the melting period ranged from 25-30 minutes. A few heats were made in a magnesite-lined 40 kW direct arc furnace to produce larger melts. The use of KCl+NaCl flux was made in these heats and the average loss in the melt was also found to be less in spite of the fact that, each time any addition was made, the furnace lid had to be opened.

In order to improve the overall mechanical properties and particularly the proof stress value, some of the 3 kg. heats in the indirect arc furnace were degassed with oxygen-free dry nitrogen at a flow rate of 30 litres/hour. Degassing was also tried by holding (for 2 minutes) marble chips at the bottom of the 3 kg. melt which caused quite a good turbulance in the molten bath due to the evolution of carbon dioxide gas, but no improvement in the mechanical properties was found specially in the proof stress values. Melting of the alloy in indirect arc furnaces was carried out with different flux covers such as 50: 50 (NaCl +KCl), borax and cryolite to attain the required proof stress. Cryolite cover provided minimum losses in the melt with better surface finish; borax made a very viscous slag, and alloys that were made under borax appeared to form some hard constituents in the alloys which required sharpening of the tools very often during machining but there was no appreciable improvement in mechanical properties.

The alloys in the specification range of Al 8·5, 11·0 per cent; Fe+Mn, 10 per cent max. and balance, copper could be made with minium ultimate tensile strength of 60 kg./mm.^{2}; 0·2 per cent proof stress values 20-24·5 kg. and minimum elongation of 20 per cent. The micro-structures of different alloys were studied to see the effect of lammelar and globular structure on mechanical properties. Proof stress values of different alloys at varying contents of aluminium, iron, iron plus manganese were studied and showed considerable scatter on graphical representation.

34.0 Study on the Indigenous Foundry Moulding Materials

This project was taken up with a view to assess the moulding characteristics of Indian foundry sands and bonding clays and to determine their suitability for various types of castings. The sands were obtained through the Geological Survey of India and private foundries. During the period under review, investigations were completed on nine sand samples. A summary of the work done is given below.

(*a*) *Glass Sands*

These samples, received from the Directorate of Geology & Mining, U.P., Lucknow, were collected from different locations.

(i) *Bhainsahi Location* (*Lower Unit*); *White in Colour*

It was a high-silica sand having A.F.S. G.F. No. 67·4 and clay content of 3·6 per cent. It contained 96·47 per cent silica. It contained fine-grained sand of sub-angular to sub-rounded shape. The sample largely consisted of quartz with small amounts of felspar, hornblende, ferro-magnesium minerals, and few micaceous flakes. The sintering range was found to be above 1350°C. In view of its sub-angular to sub-rounded shape, two-sieve distribution, moderate permeability and high refractoriness, the sand is suitable for core making purposes in steel casting jobs. It can also successfully be made use of for making moulds for medium to heavy steel castings.

(ii) *Bhainsahi Location* (*Lower Unit*); *Yellow in Colour*

It was also high-silica sand having A.F.S. G.F. No. 72·06 and clay content of 3·76 per cent. It contained 96·82 per cent silica. The sand grains were fine, and sub-angular to sub-rounded in shape. The sample largely consisted of quartz with minor amounts of felspar. Ferro-magnesium minerals, muscovite and dark opaque grains were also present in traces. Sintering temperature of the sample was found to be above 1350°C. In view of the above properties and the study of the moulding characteristics, the sand was found to be suitable for core and mould making in medium and heavy steel foundries.

(iii) *Kanchanpur Location* (*Lower Unit*); *White in Colour*

The sample received from D.G.M., Lucknow, was a high-silica sand having A.F.S. G.F. No. 56·9 and contained 1·4 per cent A.F.S. clay. Petrological examination revealed that the sample consisted predominantly of quartz and traces of ferro-magnesium minerals. Shape of the grains was sub-angular to sub-rounded. Sample contained 99·3 per cent silica. Sintering temperature was found to be above 1350°C. In view of the high refractoriness, good permeability and three-sieve distribution, the sand can be made use of for light and medium steel castings.

(iv) *Kanchanpur Location* (*Lower Unit*); *Yellow in Colour*

The sample was a high-silica sand having A.F.S. G.F. No. 58·1 and contained 1·3 per cent clay. Petrological examination showed that it consists predominantly of quartz with minor amounts of felspar, traces of calcite and ferro-magnesium minerals. It contained 98·7 per cent silica. The shape of the sand grains was sub-angular to sub-rounded. In view of its high refractoriness and good permeability, this sand can be made use of for light and medium steel castings.

(*b*) *Maharashtra Silica Sands*

Eight silica sand samples were received from the Directorate of Geology & Mining, Maharashtra, and were collected from different locations.

(i) *Sample No. MSS*-1

The sample was a high-silica sand having A.F.S. G.F. No. 67·12 and contained 2·06 per cent A.F.S. clay. Petrological examination of the sample revealed it to consist mainly of quartz and jaspar, and oxides of iron were present in the traces only. Shape of the grains was found to be sub-angular to sub-rounded. The sample contained 95·07 per cent silica. The sintering range of the sample was found to be above 1350°C. In view of the above properties, the sand can be used in iron and steel foundries with the proper use of mould washes to reduce the metal penetration and to improve the surface finish.

The sand was collected from North Sand Stone Ridge at Phandaghat, in Kankavali Mahal, District Ratnagiri, Maharashtra.

(ii) *Sample No. MSS*-8

The sand was collected from the working pits of M/s. Samant Mines in the village Tandoli. It was a high-silica sand having A.F.S. G.F. No. 56 and contained 1·58 per cent A.F.S. clay. It assayed 98·9 per cent silica. The sample mainly consisted of quartz and minor amounts of felspar. Ferro-magnesium minerals were present in traces. Zircon and iron oxide were also found in minute amounts. Grain shape was sub-angular to sub-rounded. Sintering range was found to be above 1350°C. Judging from the above properties, the sand can be used in steel foundries.

(iii) *Sample No. MSS*-6

It was a high-silica sand containing 97·91 per cent silica in the 'as received' condition and 98·36 per cent silica in the 'washed state' and having A.F.S. G.F. No. 45·9. The clay content was 0·82 per cent. It contained coarse to fine-grained sand of sub-angular to sub-rounded shape. The sample essentially consisted of quartz with minor amounts of felspar along with fine flakes of muscovite and zircon. Traces of ferro-magnesium minerals were also present. Most of the quartz grains were coated with ferruginous and clayey material. Some non-mineralogical impurities and oxides of iron were also present. Sintering range of the sand was above 1350°C. Study of the moulding characteristics of the sand showed that the sand can be used in steel foundry paractice and is specially suitable for light and medium steel castings.

(iv) *Sample No. MSS*-3

It was a high-silica sand containing 98·08 per cent silica in the 'as received' condition and 98·12 per cent silica in the 'washed state'. The clay content was 4·18 per cent. It contained coarse to fine-grained sand sub-angular to sub-rounded in shape. The sample mainly consisted of quartz. Felspar, hornblende, tourmaline, muscovite and stanrolite were present in small amounts. Some of the quartz grains were coated with clayey and ferruginous materials. Few dark opaque grains and oxides of iron were also present. Sintering range of the sand was above 1350°C. Study of the moulding characteristics of the sand showed that the sand can be used in steel foundry practice and is specially suitable for light and medium steel castings.

(v) *Sample No. MSS*-2 (*under progress*)

Investigation work on the above sand sample received from the Directorate of Geology, Maharashtra, is in progress.

(vi) *Sample No. MSS*-4

The sand was collected from Shuriwadi Sand Stone Belt in village Math Vengurla Mahal, District Ratnagiri, Maharashtra. It is a high-silica sand having A.F.S. G.F. No. 26·7 and A.F.S. clay content 1·44 per cent.

(*c*) *Production of sized Sand for Foundry and Glass making from Quartzite Sands received from Chaibasa, Bihar*

Three different quartzite samples marked A(ASS), B(ASQ) and C(KTBJ) were received from Shri Lakshmiprasad Sao, Chaibasa, for production of sized sand for foundry and/or glass making. The principal gangue minerals were felspars in all the samples. The samples assayed: A(ASS) — SiO_2, 94·44; Al_2O_3, 2·26; Fe_2O_3, 0·87; CaO, 0·15; MgO, 0·41 and alkalies as chloride, 1·46 per cent; B(ASQ) — SiO_2, 92·48; Al_2O_3, 4·10; Fe_2O_3, 0·56; CaO, 0·22; MgO, 0·63 and alkalies as chloride, 1·50 per cent; and C(KTBJ) — SiO_2, 88·91; Al_2O_3, 7·45; Fe_2O_3, 0·53; CaO, 0·27; MgO, 0·96 and alkalies as chloride, 1·39 per cent, respectively. Hydraulic sizing of the sand samples, after grinding to −14 mesh, produced clean, more or less sized and well-graded sand products. The classified products assayed between SiO_2, 95·41-96·77 and Fe_2O_3, 0·85-0·46 per cent in sample A, and SiO_3, 93·09-97·45 and Fe_2O_3, 0·22-0·34 per cent in sample C. The combined washed products obtained from samples A and B assayed SiO_2, 96·34; Fe_2O_3, 0·58 and SiO_2, 96·32; Fe_2O_3, 0·34 per cent, respectively, with yields of 87·5 and 84·6 per cent by weights. These sands, containing minor amounts of felspars, may be utilized as grade 'B' foundry sand where specifications are not very rigid. The quality of sample C could not be improved to the foundry grade by washing alone and would require further beneficiation.

High intensity magnetic separation of classified sand products from samples A and B yielded non-magnetic fractions assaying SiO_2, 98·1; Fe_2O_3, 0·3 and SiO_2, 97·52; Fe_2O_3, 0·17 per cent, respectively, suitable for making ordinary glass. The possibility of further reducing the iron content will need more study. The non-magnetic fraction obtained from sample C, assaying SiO_2, 91·0 and Fe_2O_3, 0·46 per cent, was unsuitable for glass making, but satisfied the specifications laid down for grade 'C' foundry sand.

(*d*) *Production of sized Fraction from a Quartz Sand Sample from Ennore District, Madras*

Studies were undertaken on a sample of quartz sand from Ennore, Madras State, to find out a suitable method of obtaining the −28 +48 mesh fraction (Tyler equiv. of −30 +50 ASTM) for use as standard foundry sand. As received, the sample contained 79·3 per cent by weight of −28 +48 mesh fraction. Straight screening of the sample in a double-deck vibrating screen at 28 and 48 mesh sizes did not yield a clean −28 +48 mesh size fraction as it contained as high as 9 per cent of −48 mesh material. Appreciable amounts of oversize and under-size fractions were present in the −28 +48 mesh product obtained by straight tabling of the sample as received. Screening out the +28 mesh sand followed by tabling of the −28 mesh fraction yielded cleaner −28 +48 mesh size fraction constituting 68·3 per cent by weight and containing only a small amount of undersize material in the product.

34.1 Shell Moulding

The shell moulding process is considered as one of the greatest technological developments in the foundry industry. It has got several advantages over the conventional sand casting and expected to become very popular with the Indian foundries. The advantages of shell moulding process

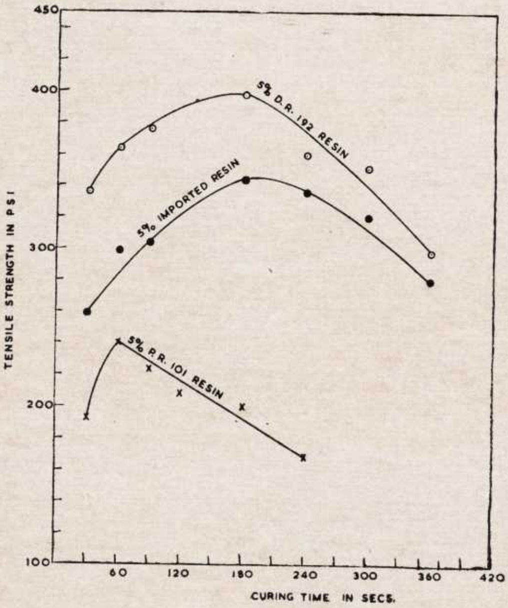


FIG. 25 — EFFECT OF CURING TIME ON TENSILE STRENGTH

are exceptionally good surface finish and dimensional accuracy, hence elimination of machining operations, decreased casting weight variation and less clearing cost. Work was taken up on this project with a view to enumerate the availability of the raw materials, such as sand, resin, etc., and study their suitability for this process.

The process consists of mixing a thermosetting resin with a clay-free, high-silica sand of which fineness between G.F. No. 90 to 140 which are then applied on preheated metallic pattern plate. The phenolic type of resin partially thermosets and builds up coherent sand shell next to the pattern giving a true replica of the pattern. The thickness of this shell is related to pattern temperature, dwell time on the pattern, etc. The cope and drag portion of the shells are then assembled and fused by a gum.

Tests were conducted on three phenolic resins, viz. P.R 101, D.R. 190 and an imported resin, for their suitability in shell moulding. It was found that the former two indigenous resins can fairly replace the imported resin. A comparative study of the tensile strengths of the three resins was conducted and the results are plotted in a graph (Fig. 25). Effect of investment time on shell thickness was also studied and the results are shown in graph (Fig. 26). All the moulding experiments were conducted in Croning F.6 type machine. Casting trials were conducted by pouring magnet alloy in the shell moulds for horseshoe magnets. The castings are shown in Fig. 27.

34.2 Hot Blast Cupola

The Indian foundry industry is faced with the problem of poor quality raw materials. The high-ash coke available in India is unsuitable for foundry use while the high phosphorous pig irons are not suitable for the production of high quality malleable irons. For foundries in India specializing in the malleable iron and also high quality grey iron, the hot blast cupola is of particular interest owing to relatively large proportion of coke they are forced to incorporate in the charge. With this point in view, NML has set up an experimental hot blast cupola and is engaged in carrying out an extensive research work, the benefits of which will be made available to the Indian foundry industry.

In the National Metallurgical Laboratory, a hot blast cupola has been installed where the blast temperature can be raised up to 500°C. by means of a radiation recuperator. From the charging door to the bottom the cupola measures 18 feet while the overall length including the chimney is 29 feet. It has been acid lined to 18 in. inside diameter. The capacity of the blower is 800 cu. ft/min. With the cold blast the melting rate is 1 ton/hr. The flue gas from the cupola is taken off just below the charging door and is led to the refractory lined combustion chamber of the recuperator. As the flue

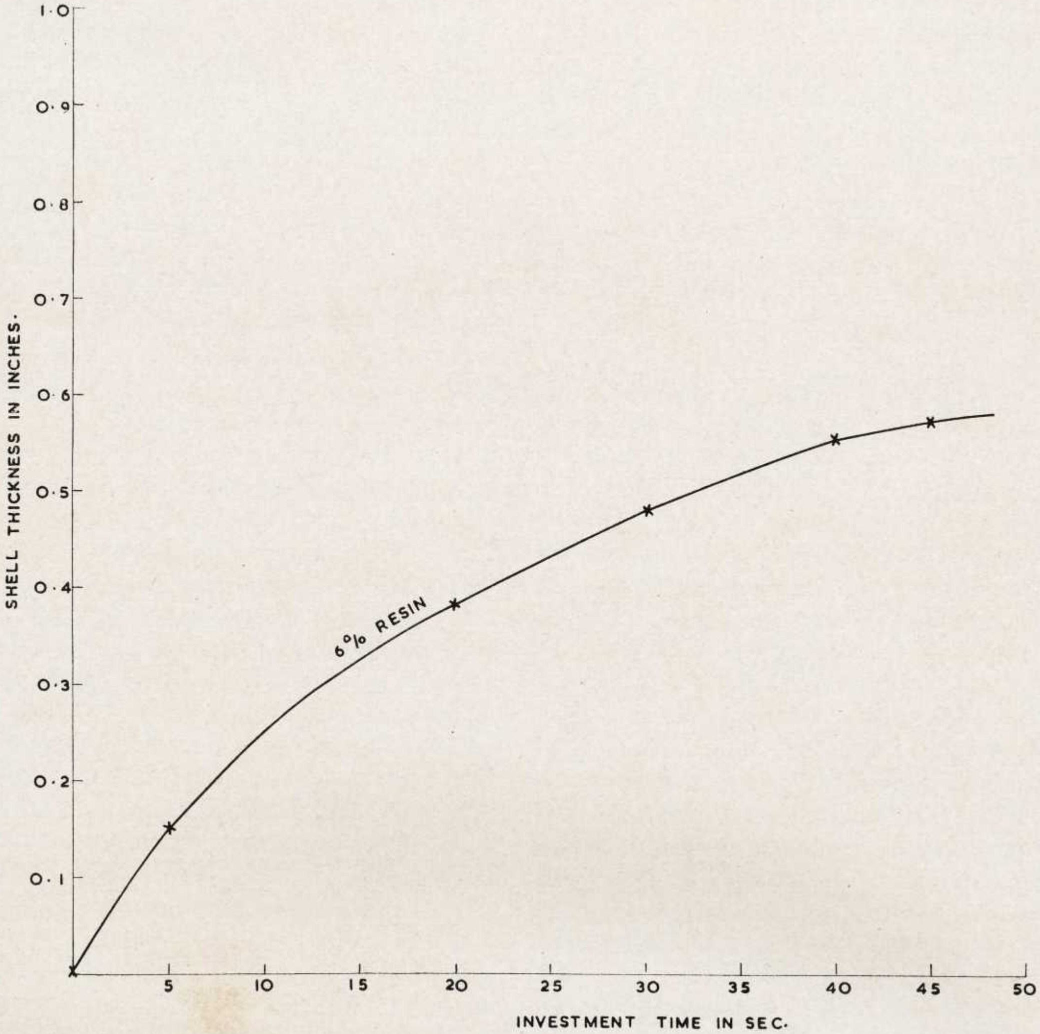


Fig. 26 — Effect of Investment Time on Shell Thickness

Fig. 27 — Castings made by Shell Moulding Process

gas contains about 10 to 12 per cent CO, this is burnt to CO_2 by supplying extra air and the heat developed by this exothermic reaction is employed to heat up the recuperator. The 43 in. diameter and 34 ft high recuperator consists of two concentric steel shells made of heat-resistant steel which is insulated with glass wool from the outside. Through the inside shell of the recuperator the flue gases pass upwards whilst the cold air from a blower enters the recuperator, and while travelling downwards in the annular space gets heated and is then led by means of insulated pipes to the wind box, and thence to the tuyers of the cupola. Necessary gas burners have been provided for preheating the recuperator at the beginning of the run as well as to raise the temperature at the combustion zone to the desired level.

A separate blower has been fitted to supply extra air for burning CO as well as gas in the initial stage. The cupola can be run either on hot or cold blast by adjusting the values leading air to the recuperator and the cupla. By means of dampers fitted in the waste gas duct of the recuperator and the stack of the cupola, adjustments can be made in the percentage of the flue gas passing through the recuperator and the cupola stack. Adequate provisions have been made to measure the temperatures in the combustion chamber waste gas, leaving the recuperator and the preheated air entering the tuyers.

Experiments with the hot blast cupola were undertaken on the following aspects:

(a) Increasing the metal temperature.

(b) Saving in the coke consumption.

(c) Increasing the silicon content of the metal by injection of silica flour through the tuyers.

(d) Reduction of sulphur content on account of using steel scrap as well as by increasing the reactivity of the slag.

(e) Improvement on the melting rate.

So far, the blast temperature was raised up to 400°C. and by means of external water cooling arrangement recently installed in order to reduce the erosion of refractory lining around the combustion zone of the cupola, it is proposed to increase the temperature further. With the blast temperature at 400°C. the metal temperature was increased to 1400°C. The coke consumption was cut down by 37·5 per cent. Silica injection through the tuyeres has been proved fruitful in obtaining higher silicon content in the metal. In the trial heats, silica injection as above, silicon content of the metal increased by 0·8 per cent maximum. By charging foundry pig and steel scrap in 1 : 1 ratio, sulphur content was lowered to 0·03 per cent in the tapped metal as compared to 0·05 per cent with the foundry pig alone. Further work was in progress.

35.0 Powder Metallurgy — Production of Metallic Powders

Metal powders of aluminium, zinc, tin, copper, brass, lead, etc., are used in different grades for a veriety of purposes including paint manufacture, chemical reduction and other powder metallurgy applications. Studies were under way to assess different methods for making metallic powders and evaluate conditions for production of different grades of metal powders. Examination and elimination of possible hazards during production of metal powders was also being considered.

A molten metal atomizer unit with interchangeable nozzles was designed and fabricated. Studies were initiated with production of aluminium powder. The molten metal was atomized and quenched in water. A series of experiments were carried out by atomizing molten aluminium at a temperature of 800°C. at various pressures through nozzles having different orifice diameters. The atomized samples were collected and sieved through a set of screens and the pattern of their distribution were recorded. It was observed that the distribution of various particle sizes follow a definite pattern. For a particular pressure keeping all other factors constant with smaller nozzle diameters the particles were rendered finer. Further studies were being made with other nozzle diameters. It was proposed to study the influence of the temperature of the molten aluminium on the final particle size of the powder. The process on production of copper powder developed earlier was leased out to a copper smelting firm.

35.1 Production of Iron Powder by Direct Reduction

The work on direct reduction characteristics of iron ore samples received from the Director of Geology & Mining, Government of Maharashtra, Nagpur, was completed. After completing the studies with paper packet method of packing the iron ore, pelletizing of the iron ore was carried out in a disc type pelletizing machine for direct reduction studies. Pelletizing of iron ore is desirable for mechanized operation and high production. It was found that crushing of the ore to 78 per cent below 270 mesh and addition of 0·5 per cent bentonite with moisture content of about 7 per cent produced pellets having satisfactory green and dry strength. The results obtained on reduction of these pellets showed low iron content in the reduced sponge. Pellets were then made by crushing the ore to 100 mesh and 3 per cent molasses was added as a binder. These pellets gave satisfactory results on direct reduction at different temperatures and varying time. The results obtained are given in the Table 41.

Results indicate that the iron ore can be successfully reduced by direct reduction using coke breeze and a suitable degree of reduction can be achieved for the manufacture of iron powder for autogenous cutting and scarfing. The analysis of iron ore shows SiO_2, 1·5 and Al_2O_3, 2·0 per cent, which are slightly high.

Investigation on direct reduction of iron ore supplied by M/s. Salgaocar E. Irmao Ltd, Goa, is in progress. The average

Table 41 — Results of Experiments with Iron Ore received from the Government of Maharashtra

Temperature °C.	Time hr	Analysis of the reduced sponge				
		Fe %	FeO %	C %	S %	P %
900	16	93·24	0·72	1·15	0·06	0·10
900	20	91·84	3·60	0·81	0·10	0·05
900	24	90·44	4·68	1·10	0·10	0·05
1000	8	92·12	1·44	1·08	0·08	0·05
1000	24	91·56	2·16	1·28	0·08	0·07
1100	6	94·36	2·16	1·10	0·02	0·08
1100	8	94·08	1·44	0·95	0·05	0·05
1100	16	93·80	Trace	1·27	Trace	0·06
1100	20	95·76	Trace	1·26	0·19	0·08
1100	24	93·80	0·72	1·24	0·02	0·09

Table 42 — Results of Experiments with Iron Ore received from M/s. Salgaocar E. Irmao Ltd, Goa

Temperature °C.	Time hr.	Analysis of the reduced sponge				
		Fe %	FeO %	C %	S %	P %
900	6	92·68	1·44	0·98	—	—
900	8	92·12	1·44	1·03	—	—
900	16	91·85	2·16	1·00	—	—
900	20	92·27	1·44	0·59	—	—
1000	6	90·72	2·34	0·80	0·040	0·09
1000	8	91·43	2·16	1·00	0·010	0·06
1000	20	91·28	2·16	0·93	0·010	0·05
1100	8	91·84	0·72	1·15	0·025	0·06
1100	20	91·84	2·88	1·13	0·020	0·07

analysis of the crushed and mixed iron ore is given below:

Fe %	63·86
FeO %	0·43
Al_2O_3 %	2·84
SiO_2 %	1·40
TiO_2 %	0·20
MnO %	0·35
S	0·03
P	0·03

The crushed ore containing about 75 per cent below 325 mesh was pelletized in a disc-type pelletizing machine. The pellets were dried at 200°C. for 2 hours. Direct reduction experiments were carried out at different temperatures for different length of time with dried pellets. The results obtained so far are given in Table 42.

As the iron ore was low in iron content and slightly high in gangue, the metallic iron in the reduced sponge obtained was rather low for the production of high-grade iron powder.

36.0 Direct Reduction of Iron Ores by Naphtha

The investigation was taken up to study the direct reduction of iron ores by naphtha, a petroleum by-product. Naphtha, being a mixture of paraffin hydrocarbon, consists of 10-14 per cent H_2 and 80-85 per cent C by weight, both of which act as reducing agents. The iron ores of all grades can be reduced to metallic iron by this process and the gangue material is taken care of by suitable fluxing agents. This process can also be utilized for upgrading iron ore, which cannot be economically beneficiated by gravity, magnetic, froth flotation or electrostatic separation techniques.

Cracked paraffin hydrocarbons (C, 80-85 and H_2, 10-15 per cent) when allowed to react with heated compacted iron ores, produced sponge iron in the form of powder. The exit gases were recirculated to increase the reaction efficiency. In the modified series of experiments critical amounts of air or oxygen was passed over heated compacted iron ores along with the saturated vapours of naphtha inside a similar reduction tube. This critical amount of air assisted in increasing the reaction rate along with complete utilization of cracked hydrocarbons. The pressure of gas at the inlet and outlet of the tube was kept nearly the same.

One hundred grams of iron ore with composition Fe_2O_3, 92·76; SiO_2, 1·74 and Al_2O_3, 4-8 per cent was taken in 100 mesh powder form. This was compacted with small addition of moisture and heated to 1050°-1100°C. in the reduction tube. Measured amounts of air at a pressure of 150-200 mm. of water gauge was bubbled through naphtha. The vapours were then passed through the reduction tube and the exit gases were passed through lime water. In 60-90 minutes about 20-28 cc. naphtha was passed producing the reduced product of the composition, Fe, 188·94 and C, 0·02-0·04 per cent. This product can be suitably used for applications of iron powder. Large-scale batch experiments were being projected to assess the suitability of the process further.

37.0 Kinetics of Hydrogen Reduction of Iron Ores by Electrical Conductivity Method

Kinetics of hydrogen reduction of iron ores can be studied by determining the rate of reduction of iron ores by hydrogen and incorporating the data on the effect of temperature, hydrogen flow rate, particle variation and composition on the rate of reduction of iron ores. This is normally determined by ascertaining the loss of oxygen from the ore by measuring the water formed due to reaction and/or the loss in weight of iron ore. Changes in electrical conductivity of iron ores during their reduction by hydrogen suggested a method of determining the rate of reduction of iron ores thereby eliminating the possibilities of inaccurate measurements. The project was taken up to develop this method and to make a complete study of kinetics of hydrogen reduction of iron ores.

Studies on four iron ores were conducted to assess their reducing characteristics with hydrogen by electrical conductivity method. This was done in two sets of experiments.

(a) Study on the effect of temperature on the electrical conductivity of iron ores: The electrical resistance of iron ores was measured by means of OSAW potentiometer when the ore sample was heated from room temperature to 950°C. As the temperature increased, the resistance decreased at a fast rate. In certain cases the electrical resistance was 12000 ohms at room temperature and 16 ohms at 950°C. This may be one of the inherent properties of iron ores, although the porosity of iron ores also plays an important part. The results on different ores are shown in Fig. 28.

(b) The iron ore sample (19·5 $mm.^2$ × 38 mm.), held between two copper discs supported by two copper rods was heated to 800°-950°C. in an electrical tubular furnace kept in vertical position. Purified hydrogen was passed through the tube for about 3 hours at a flow rate of 800-1000 cc./min. The water vapour so formed was passed through a cooled glass spiral and collected in a measuring cylinder. The resistance of the sample was measured during reduction. The resistance of iron ore at 950°C. was 2-15 ohms and after reduction 0·002-0·007 ohm. The experiments were repeated by varying hydrogen flow rate, temperature and time, etc. The results at constant temperature and hydrogen flow of different ores are shown in Table 43. The method serves the dual purpose of the determining the reducibility of iron ores by a

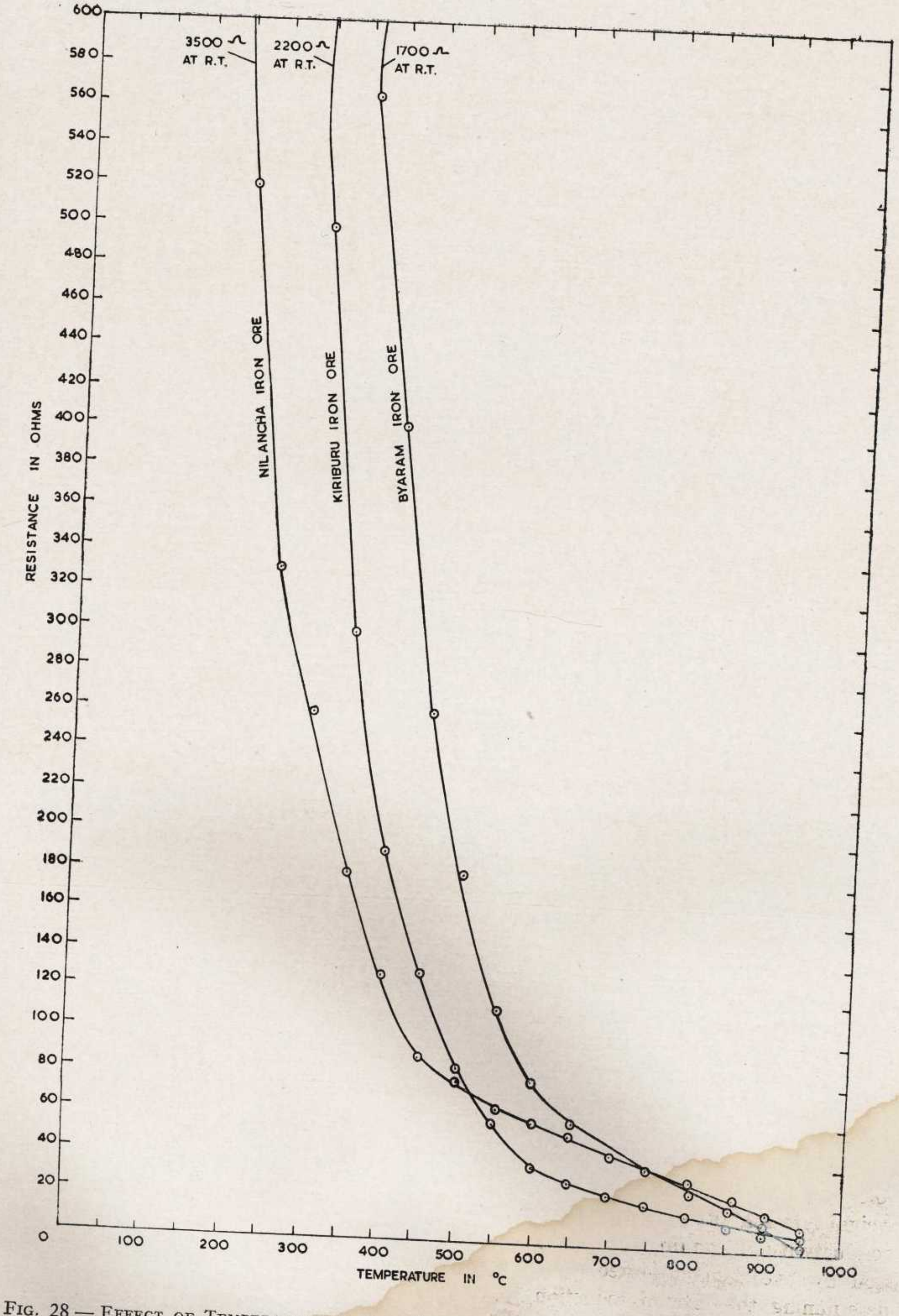


FIG. 28 — EFFECT OF TEMPERATURE ON THE ELECTRICAL RESISTANCE OF DIFFERENT IRON ORES

more precise method as well as for the study of the kinetics of hydrogen reduction of iron ores.

38.0 Solidification of Cast Iron

(i) *Aluminium Cast Iron*

The addition of aluminium to cast iron increases its heat resistance but diminishes its mechanical properties. Addition of aluminium up to 30 per cent has two graphitic zones, one up to a maximum limit of 8 per cent and the other in the range of 18-25 per cent. It has been reported that the addition of aluminium in the second graphitic zone imparts better heat resistance without appreciably deteriorating its mechanical properties.

Attempts were made to nodularize aluminium cast iron containing 18-25 per cent Al by addition of magnesium. Experiments were conducted to introduce magnesium by addition of aluminium magnesium alloys and magnesium salts. Microstructure, and mechanical and heat resistance properties of these cast irons were being studied.

(ii) *Effects of Alloying Additions in Cast Iron*

The investigation was taken up with a view to obtain higher tensile and impact strength along with hardness by the addition of alloying elements and also to study the mechanism of graphite formation. It has been reported that addition of tin increases tensile, impact and hardness values. It is a pearlite promoter and stabilizer, and does not influence the graphite structure. Up to 0·1 per cent tin is beneficial in tensile and impact properties but up to 0·3 per cent tin can be added with advantage. It also imparts beneficial effect on the scaling resistance of cast iron.

The cupola iron having the typical composition C, 3·4; Si, 2·43; Mn, 0·72; P, 0·28; and S, 0·08 per cent was taken up

Table 43 — Results of Hydrogen Reduction of Iron Ore at Constant Temperature

Sl. No.	Occurrence of Iron ore	Chemical analysis of iron ore					Electrical resistance of iron ore at room temp. ohm	Electrical resistance of iron ore at 950°C. ohm	Hydrogen flow rate in cc./min.	Time of hydrogen flow in min. and temp.	Resistance of reduced product ohm	Reduction %
		Fe_2O_3 %	Fe_3O_4	Al_2O_3 %	SiO_2 %	L.O.I. %						
1.	Nilancha iron ore Khamman (A.P.)	82·2	Trace	2·50	7·80	3·60	3500	2·30	1000	180 min. 950°C	0·002 ohms.	98·20
2.	Bayaram iron ore Worrangel (A.P.)	82·8	2·3%	2·20	6·90	2·50	1700	7·00	1000	180 min. 950°C.	0·002	98·50
3.	Tomka Iron ore (Orrissa)	79·6	18·5%	0·55	0·70	0·50	12,000	16·50	1000	150 min. 950°C.	0·003	94·50
4.	Kiriburu iron ore (Orissa)	86·3	—	5·35	2·80	4·90	2200	3·06	1000	180 min. 950°C.	0·003	94·40

Table 44 — Results of Experiments on Tin Addition in Cast Iron

Carbon equivalent	Chemical composition	Si content	Ti content	Max. tensile strength obtained	Max. Impact strength obtained	Fluidity	Hardness (BHN)
3·985	3·4%C, 1·68% Si, 0·66% Mn, 0·33% P, 0·054% S	nil	—	17·50 tons/in.²	20 ft/lb.	9·0 in.	196(2·5 cm. section)
3·730	3·19% C, 1·46% Si, 0·81% Mn, 0·35% P, 0·039% S, 0·042% Sn	0·042%	—	16·90 tons/in.²	18 ft/lb.	27·5 in.	210(2·5 cm. section) 404(0·5 cm. section
4·078	3·48% C, 1·71% Si, 0·67% Mn, 0·34% P, 0·057% S, 0·096% Sn	0·096%	—	16·66 tons/in.²	16 ft/lb.	31·5 in.	226(2·5 cm. section) 286(0·5 cm. section)
4·217	3·6% C, 1·93% Si, 0·68 %Mn, 0·27% P, 0·03% S, 0·91% Sn	0·910%	—	11·57 tons/in.²	6 ft/lb.	19·1 in. (with 1:1% Sn addition and 3·33 C E value)	—
3·60	2·92%C, 1·96% Si, 0·65% Mn, 0·38% P, 0·04% S, 0·12% Ti	—	0·120%	15·20 tons/in.²	14 ft/lb.	18 in.	—

for investigation. Cu in the range of 0·1-0·001 per cent and Ti, 0·001 per cent were present as trace elements and carbon equivalent value was 4·148.

Tin additions varying from 0 to 1 per cent were made. The data are given in Table 44. It was observed that tin has little effect on tensile strength. The impact strength was found to be affected appreciably in the same fashion as in the above case but the increase in hardness was seen in 2·5 cm. thick section step specimen as the percentage of tin increased. The fluidity was determined by measuring the length of the fluidity spiral and expressed in inches. The fluidity was found to increase with increase of tin content at the above mentioned compositions of cast iron but showed decrease at about 1 per cent tin addition at the composition. The eutectic cell was determined after etching the specimens with the Stead's reagent and viewing through Vicker's projection microscope at low magnification. The size of the eutectic cell was found to increase with decrease of tensile strength and increase of section thickness of castings. Thus in solidification, as the cooling rate increases, the eutectic cell size decreases, thereby increasing the number of eutectic cells per unit length or area. The addition of titanium is also being done to understand its effects, singly or in combination, on the properties of cast irons. Further investigation is in progress.

39.0 Gaseous Malleabilization of White Cast Iron

Presence of trace amounts of elements can exert a profound effect on the annealing rate of white iron. Some elements, such as Ni, Cu, Ti, Al and B, favour graphite formation and others, like Cr, Mo, Pb, Mg, Sn and Sb, retard the breakdown of cementite. So, study was being carried out to see the effect of Cu on graphitization under inert medium of nitrogen in the temperature range 850°-950°C. without using packing material. It was observed that C:Si ratio also played an important part on malleabilization. When it was 0·9-3·1, the malleabilization took place and beyond this range no effect was observed.

The procedure was to heat the white iron specimens of size of 1 in. approximately to the desired temperature step by step in electric furnace for 3 hours. The temperature was then maintained for 25 and 30 hours. Then the furnace was switched off and specimen allowed to cool. The specimen was then taken out and polished, and hardness and micro-structure studies were made. The time period was fixed at 25 to 30 hours as the effect of graphitization below this time was observed to be very low. The following composition of white iron was studied with varying amounts of Cu.

C, 2·36; Si, 1·24; Mn, 1·30; S, 0·02; P, 0·25 and Cu being 1·44, 1·49 and 1·79 per cent.

Some of the microstructures are given in Figs. 29, 30 and 31, and the hardness is indicated in the Table 45.

Table 45 — Hardness of Original and Heat-treated Samples

Annealing temp. °C.	Time hr	Sample I containing 1·44% Cu	Sample II containing 1·49% Cu	Sample III containing 1·79% Cu
Original sample	—	480	437	514
850	25	301	239	288
850	30	278	244	268
900	25	280	246	266
900	30	274	236	244
950	25	275	241	245
950	30	274	250	286

FIG. 29(a) — PHOTOMICROGRAPH OF ORIGINAL SAMPLE WITHOUT HEAT TREATMENT (Cu, 1·44 per cent) ×240

FIG. 29(b) — PHOTOMICROGRAPH AFTER HEAT TREATMENT ×240

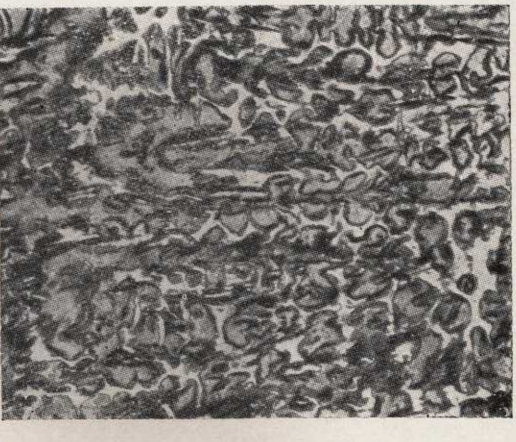


FIG. 30(a) — PHOTOMICROGRAPH OF ORIGINAL SAMPLE WITHOUT HEAT TREATMENT (Cu, 1·49 per cent) ×240

40.0 Dephosphorization of Indian Irons by Additions of Mixtures in Ladle

It has been reported that some measure of dephosphorization can be obtained by treatment of cupola metal with soda ash in amounts similar to those normally used for

FIG. 30(b) — PHOTOMICROGRAPH AFTER HEAT TREATMENT ×240

FIG. 31(a) — PHOTOMICROGRAPH OF ORIGINAL SAMPLE WITHOUT HEAT TREATMENT (Cu, 1·79 per cent) ×240

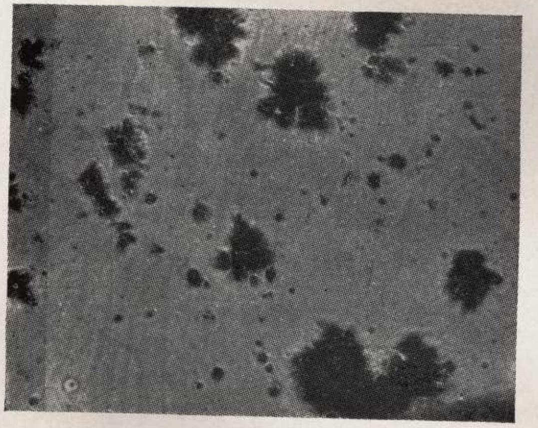


FIG. 31(b) — PHOTOMICROGRAPH AFTER HEAT TREATMENT ×240

desulphurizing. Attempts were made to add some dephosphorization mixture like iron oxide, lime and soda ash in particular sequence to study the effect on dephosphorization of Indian pig iron. The results are highly encouraging.

In a pre-heated ladle 20 kg. batches of molten iron containing 0·3 per cent phosphorous were treated. When the metal was nearly half full in the ladle, layers of iron oxide, lime and soda ash were spread over the metal surface and were allowed to react. It was repeated after pouring the remaining metal. Intense reactions took place. The metal was then allowed to be solidified after removing the slag. In one of the experiments metal composition was C, 4·09; Si, 3·08; P, 0·22; S, 0·03; and Mn, 0·68 per cent. The analysis of the treated metal was C, 4; Si, 2·5; P, 0·058; S, 0·02 and Mn, 0·66 per cent. Further experiments are in progress for large-scale trials.

41.0 Study of Temper Brittleness

The object of this work was to examine whether 'retrogression' shown by steels was susceptible to temper brittleness in relation to precipitation which had been put forward to be one of the causes responsible for temper brittleness in steels.

To understand the mechanism of temper brittleness the phenomenon of retrogression shown by precipitation hardening alloys has been investigated with a nickel-chrome steel known to be susceptible to temper brittleness. Specimen blanks were embrittled for different periods of time up to 1000 hours at 400° and 500°C. and impact tests carried out after cutting the notch to determine the amount of embrittlement. The variation of the amount of embrittlement with time bears resemblance to hardness-time curve of age hardening alloys as shown by curves 1 and 2 of Fig. 32 suggesting that the mechanism controlling temper brittleness is a process

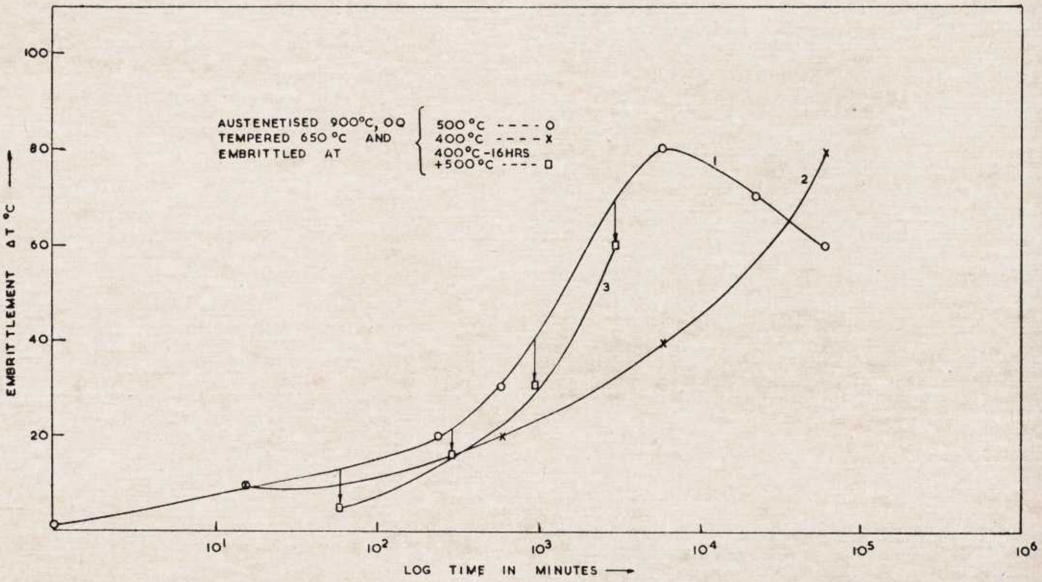


Fig. 32 — Temper Embrittlement plotted Against Log Time of Treatment

of nucleation and growth. Retrogression experiments carried out by embrittling at 400°C. followed by embrittlement at 500°C. shown by curve 3 of Fig. 32 also suggests that the controlling mechanism for temper brittleness appears to be nucleation and growth. While there was no difference in hardness between embrittled and unembrittled specimens, embrittled specimens showed intergranular fracture. Microscopic examination of specimens etched with aqueous picric acid showed preferential grain boundry attack, the grain boundry attack increasing with increasing amount of embrittlement.

42.0 Curie Temperature of Iron Alloys

The object of the investigation is to study the variations of curie temperature of iron with alloying elements of different valencies and to study if possible the electron distribution of iron.

Based on the concept advanced for explaining the variation of curie temperature of iron-aluminium and iron-silicon alloys, viz. absorption of electrons contributed by solute in the holes in the 3d band in iron, some of the existing data of these alloys were examined. Occurrence of superlattice structures Fe_3Al, Fe_3Si were accounted on the basis of the above concept and bonding considerations. Atomic radii data of Fe_3Al, Fe_3Si and lattice spacings, and co-efficient of thermal expansion of these alloys suggest that in Fe_3Si, Fe-Si bond is changing over to covalent whereas in Fe_3Al, the Fe-Al bond is not changing over to covalent. Besides, accounting for the variation of electronic specific heat of these alloys on the basis of the above concept, the decrease in co-efficient of thermal expansion of Fe-Al alloys in the range 0-12 at Al per cent was reconciled with the increase in electronic specific heat in the same range.

The work had now been extended to cobalt alloys. Even though melting under vacuum eliminated the brittleness of cobalt, the curie temperature of vacuum-melted cobalt was lower than that reported in literature as the impurities probably present in cobalt affected the curie temperature. Efforts were directed to obtaining pure cobalt and prepare alloys for the investigation.

43.0 Conductivity of Commercial Aluminium and its Alloys

Silicon and titanium normally present in commercial aluminium as dissolved impurities exercise adverse effects on 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 of 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 bring on the subject a fundamental approach, study was undertaken on changes in conductivity as precipitation progresses in commercial aluminium.

In continuation of the work on the improvement of electrical conductivity of commercial aluminium by the addition of silicon and/or magnesium, the scope of the work was extended to study the effect of binary solute additions on the electrical conductivity of electric grade aluminium. Binary alloys of aluminium with silicon, iron, manganese and magnesium were made containing 0·25, 0·5, 0·75 and 1 per cent of individual solute additions. The alloys were melted in electric furnace and cast in metal mould. The cast ingots (3·5 × 20 cm.) were homogenized and hot-forged at 450°C. and cold-rolled into 0·3 cm. thick sheets. The electrical resistivity was measured on specimens made from the rolled sheets under the following conditions:

(i) Solution treated at 550°C. for 8 hours and then water-quenched.

(ii) Aged at 300°, 350°, and 400°C., respectively, up to 30 hr at each temperature.

It was noted that substantial improvenents in the conductivity of these alloys vere obtained only after prolonged aging up to 30 hr and more between 350°-400°C. Since the tensile strength of the aluminium conductor is also an equally important factor, it was determined for all specimens in correspondingly heat-treated condition. The values of conductivity and tensile strength in the (a) solution treated and (b) aged conditions, respectively, are given in Tables 46, 47 and 48.

Table 46 — Conductivity of Some Binary Aluminium Alloys in the Solution-treated Condition

Alloy	Nominal solute %			
	0·25	0·5	0·75	1·00
Al-Fe	34	32	31-32	31
Al-Mn	37	36	34	32
Al-Mg	44	42	40	39
Al-Si	49	46	45	45

Table 47 — Ultimate Tensile Strength in kg./mm.^{2} of Binary Aluminium Alloys in the Solution-treated Condition

Alloy	Nominal solute %			
	0·25	0·5	0·75	1·00
Al-Fe	7·3	8·6	8·5	9·5
Al-Mn	7·1	7·5	8·7	7·8
Al-Mg	8·6	9·9	9·3	9·8
Al-Si	9·8	10·2	10·5	11·7

Based on the research experience gained with binary aluminium alloys, the following three alloys were made from aluminium of different purities and the specimens were tested for the conductivity and tensile strength under different conditions:

(i) P.M. 1 ... ... 98·5 per cent
(ii) P.M. 2 ... ... Commercial
(iii) P.M. 3 ... ... ,,

Fig. 33 shows the variation of electrical conductivity of alloy P.M. 2 with aging time at 400°C. P.M. 2 was then made on commercial scale in the form of wirebar (4½ in.^{2} × 40 in. at the top) weighing 30 kg. by melting the constituent metals in an oil-fired furnace using fluxes and degassers, and casting in a metal mould.

Table 48 — Conductivity (IACS%) and Tensile Strength (kg./ mm.^{2}) of Binary Aluminium Alloys, Aged at 350° and 400°C. for 30 hr

Alloy	Temp. °C.	Nominal solute %							
		0·25		0·5		0·75		1·0	
		T.S.	Conductivity	T.S.	Conductivity	T.S.	Conductivity	T.S.	Conductivity
Al-Fe	350	7·4	48·98	8·7	47·00	7·6	46·23	8·1	45·25
	400	7·0	52·24	8·3	51·60	7·3	50·56	7·9	49·40
Al-Mn	350	7·1	52·40	7·4	51·70	7·1	50·00	7·6	48·00
	400	7·1	54·20	7·3	52·60	8·0	50·70	7·7	48·50
Al-Mg	350	8·5	55·62	8·4	54·30	8·9	52·62	9·6	51·60
	400	8·2	57·02	8·5	55·62	9·2	54·30	9·4	52·50
Al-Si	350	8·3	56·62	8·4	59·60	8·5	52·62	8·1	54·00
	400	8·8	58·84	9·0	61·62	9·8	56·70	10·3	54·73

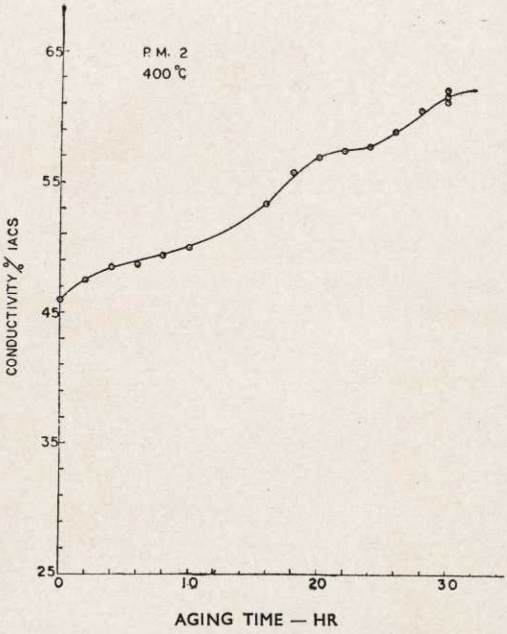


FIG. 33 — VARIATION OF ELECTRICAL CONDUCTIVITY WITH AGING TIME

Subsequently, P.M. 2 wire-bar was successfully rolled and drawn on an industrial scale by a leading cable manufacturer to the ACSR size (0·110 in. dia.) conductors without any alterations in the rolling and drawing sequences. The alloy stood all the tests specified in the Indian Standard Specifications. It is noteworthy that improvement in the conductivity was achieved along with a substantially increased strength of the conductor. Large-scale trial is in progress at present.

44.0 Structure of Liquid Metals

With a view to employ 'non-thermodynamic' experimental techniques for studying the structures of liquid metallic solutions and to obtain true understanding of the atomic interactions, investigations were taken up on the following lines:

(a) Study of interactions between Al-Cu, Al-Cu-Mg and Pb-Sn alloys in the liquid state.

(b) Study of the partitioning of magnesium between aluminium and lead.
(c) Enthalpy of liquid alloys.

Study of Interaction between Al-Cu Alloys in Liquid State

The work on the nature of atomic interactions and distribution of clusters in Al-Cu system was extended to the alloys containing 14, 33 and 40 per cent copper. The alloys were studied at 700° and 800°C. at 200 and 375 r.p.m., respectively, developing a centrifugal force of 40 g. and 141 g. The molten alloys were kept in stainless steel crucible lined with a graphite crucible in order to prevent the attack of the molten metal on the stainless steel crucible.

No macroscopic demarcation was observed in Al-14·0 per cent Cu alloys as was previously observed in Al-4 per cent Cu alloys. The Al-33 per cent Cu alloy showed the appearance of a phase presumably $CuAl_2$ after 5 hours of centrifuging at 700°C., 375 r.p.m. (141 g.). The macroscopic demarcation was observed in Al-40·0 per cent Cu alloy centrifuged at 700°C., 200 r.p.m. for 5 hours, which disappeared on centrifuging for 9 hours. But in the same alloy when the temperature of centrifuging was raised to 800°C., the demarcation appeared after 3 hours and disappeared after 7 hours of centrifuging. As reported earlier, copper was present in the form of clusters of $CuAl_2$ (Etaphase) composition. The cluster sizes were calculated for all the alloys. From the results it was concluded that

(i) cluster size decreased with increase in temperature of centrifuging at the same speed,
(ii) the increase of centrifugal force decreased the size of the cluster, and
(iii) the alloy of eutectic composition (Al-33·0 per cent Cu) had the minimum cluster size and increased in hypo- and hyper-eutectic alloys.

The cluster size of these alloys were correlated with the viscosity of the alloys.

Study of Interaction between Pb-base Alloys in Liquid State

Pb-Sb and Pb-Cd system were taken up for the study of atomic interactions in the liquid metallic Pb-base alloys. Eutectic Pb-Cd (17·5 per cent cd) alloys were centrifuged at 200 r.p.m. (i.e. at 40 times gravitational force) and at temperatures of 300° and 400°C. for periods of 1, 3 and 5 hours. Cluster sizes in the eutectic were calculated and further work on hypo- and hyper-eutectic alloys is in progress.

The work on Pb-Sb system is in progress in the high-speed centrifuge developed at National Metallurgical Laboratory. Three alloys of lead with 6, 11·2 and 30 per cent antimony were prepared. These alloys were centrifuged at 500 r.p.m. (62·5 times gravitational force) at 300°, 400° and 500°C. in the cases of 6 and 11·2 per cent antimony alloys and at 400°, 500° and 600°C. in the case of the 30 per cent Sb alloys. Chemical analysis showed a gradual change of composition across the solidified ingots in the case of hypo-eutectic and eutectics but no macroscopic segregation was observed. In the 30 per cent Sb alloy, however, the separation of a lead-rich layer was remarkably clear. The segregation of the Pb-rich phase at the further end of the crucible takes place even after an hour's centrifuging. The work is in progress to study the effect of (i) varying centrifugal force, (ii) temperature of liquid metal, and (iii) time of centrifuging on the atomic interaction.

Enthalpy of Liquid Alloys

The investigation to study the variation of heat capacity, Cp, with temperature in the case of eutectic, hypo- and hyper-eutectic lead-tin alloys was continued from the previous year to throw some light on the nature of interactions in the liquid state. The heat contents were determined in a precision ice calorimeter. The temperature dependence of the heat capacity for all the

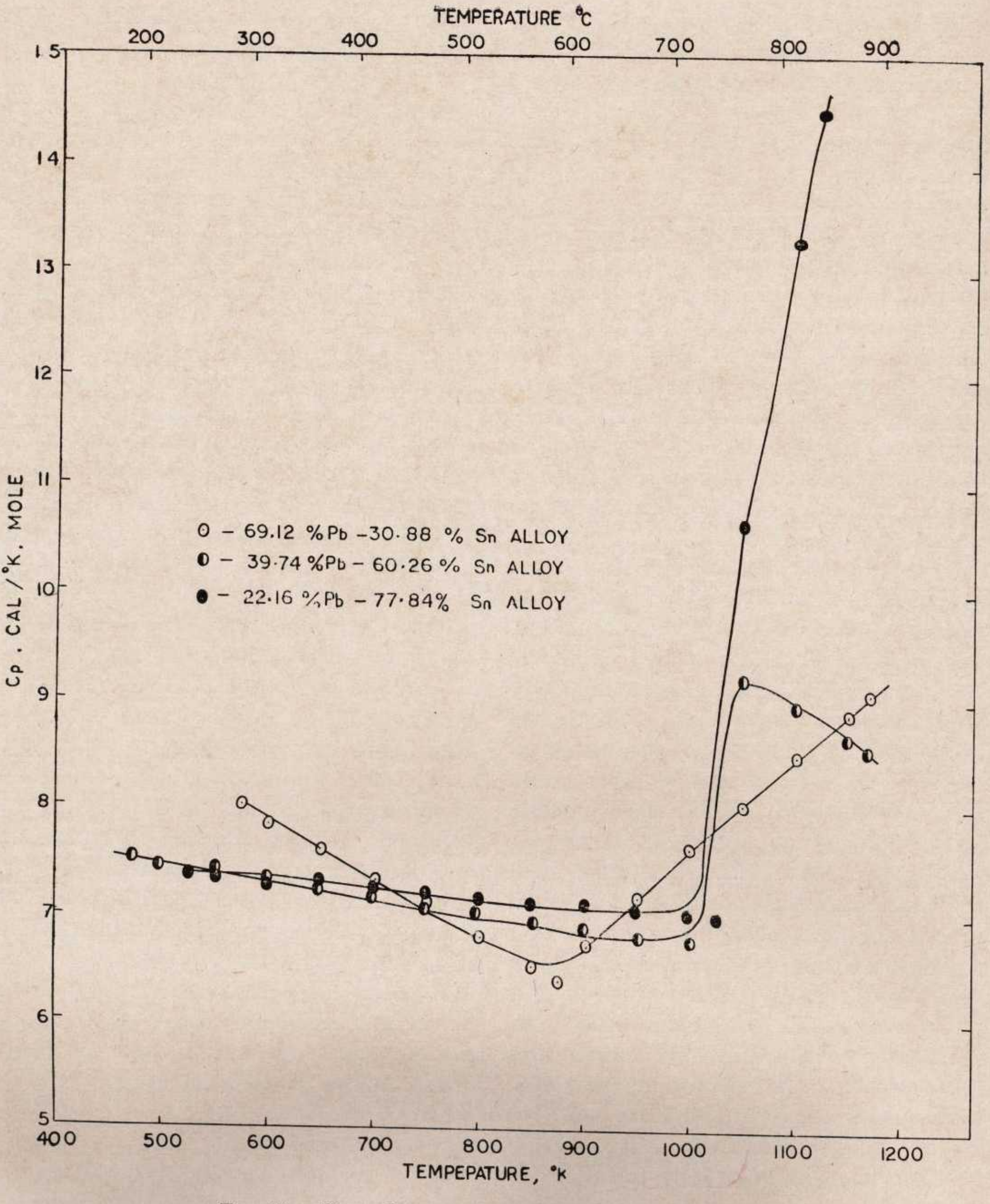


FIG. 34 — HEAT CAPACITIES OF LIQUID LEAD-TIN ALLOYS

three alloys is shown in Fig. 34. The Cp for all the three alloys initially decreased as the temperature was raised in the liquid state, and thereafter showed a marked increase up to 900°C. The decreasing Cp above the melting point has usually been ascribed to decrease in binding force and the break-up of quasi-crystalline atomic associations (or clusters) or to changes in the vibrational energy functions with temperature, or to both. The significant increase in the values of Cp for all the three alloys on considerable super-heating suggests that the binding force increased with temperature. An earlier investigation noted an increase in the size of Pb-Pb and Sn-Sn clusters in the Pb-Sn liquid alloys as the temperature was increased. The present investigation, therefore, lends support to these earlier results. Heat contents of eutectic, hypo- and hyper-eutectic alloys of lead-antimony system were also determined up to 900°C. A ternary alloy of lead-antimony-tin of eutectic composition was also investigated in the same temperature range. Detailed calculations are in progress to draw comprehensive conclusions.

45.0 Effect of Titanium on Iron and Steel

(i) *Titanium in Steel*

The project was undertaken to study the transformation and precipitation hardening characteristics of titamium steels with a view to develop high-strength ferritic steels. A brief literature survey has revealed that not much work has been done on steels having titanium as the primary alloying element although steels having titanium as the secondary alloying element have been rather extensively investigated. The phase diagram of iron-carbon-titanium systems shows that all the carbon can be fixed as titanium carbide if the amount of titanium exceeds four times that of carbon. With higher titanium contents, strength of the steel can be enhanced through (1) solid solution strengthening, and (2) precipitation of iron-titanide (Fe_2Ti) which appears in the alpha ferrite matrix due to decreasing solubility of titanium with falling temperature.

Five heats of titanium steels containing 0·06, 0·150, 0·39, 2·26 and 3·85 per cent titanium were made using steel scrap analysing 0·08 per cent carbon and adding ferro-titanium. The ingots were cast at 1650°C., into ingot moulds measuring $2\frac{1}{2}\times2\frac{1}{2}\times10$ in., and were homogenized at 1000°C. for 24 hours and forged at about 1100°C. to $\frac{1}{4}$ in.^{2}. Mechanical tests were carried out to determine the hardness, yield strength, ultimate tensile strength, percentage elongation and percentage reduction of area of all the titanium steels in cast, forged, annealed and normalized states. The grain coarsening tendency of 0·06 and 0·150 per cent titanium steels were also investigated. The age-hardening characteristics of 3·85 per cent titanium and 2·26 per cent titanium steel are now being investigated.

(ii) *Titanium in Cast Iron*

Work on this project was continued from the previous year. During the period under review, specimens of cast iron containing 0·5, 1·0, 5·0 and 7·0 per cent titanium were thermally cycled 72 times between 650°C. and 900°C. to study the effect of thermal treatment on the structure of cast iron. The specimens were metallographically examined and the carbides were electrolytically extracted and examined in X-rays. The tempering characteristics of the specimens quenched from 950°C. are being examined.

46.0 Mar-aging of Steels

The conventional process of hardening and toughening steel is based on formation of martensite and subsequent tempering. During tempering, different carbides are precipitated out. The precipitation reactions in an essentially low-carbon martensitic matrix can supplement the hardness of martensite, as the softening of martensite by the precipitation of carbides is avoided. Such mar-aged steels retain sufficient toughness in spite of the martensite and subsequent precipitation processes. During mar-aging different non-metallic compounds have been observed to precipitate, their precipitation causing the increase in the hardness. This work was taken up to study the precipitation process and the corresponding changes in the hardness of martensite in the Fe-Ni system in which varying amounts of aluminium were added.

The work indicated in the earlier Report is being carried out on similar lines but with new compositions of steels. In the previous work it was found that Fe-20 Ni base alloys with 3 per cent Al gave maximum hardness values. Hence alloys were made keeping Fe-20 Ni-3Al as base and adding 1 to 4 per cent Mn. Aging characteristics of these alloys were studied at 500° and 600°C. after soaking in the temperature range 900° to 1100°C. In these cases peak hardness values were much higher. Soaking temperature did not affect the aging behaviour significantly. The peak hardness maintained itself for over 100 hours at 500°C. but over-aging occurred much earlier at 600°C. This work has now been extended to Fe-Ni-Mn-Al steels in which some nickel has been replaced by manganese.

47.0 Grain Size of Magnesium and its Alloys

It is currently well known that mechanical performance of cast metals can greatly be improved by refining the 'as cast' grain size. Although the mechanism of grain refinement of steels or aluminium are well known, in the case of magnesium and its dilute alloys, it is based somewhat on an empirical basis. It is, however, known that grain refinement of certain magnesium alloys can be achieved either through (1) superheating the melt, or (2) inoculating the melt by carbon. However, the mechanism of grain refinement through either of these treatments is not completely understood. In particular, the following points remain obscure.

(a) Nature of the nuclei formed in the two process and the kinetics of their formation.
(b) The influence of alloying elements on the activation or poisoning of the embryos.
(c) Whether the same mechanism is operative in the two processes of grain refinement.

During the period under review, the influence of alloying elements on the activation or poisoning of the embryos for the liquid-solid transformation was studied. Magnesium-aluminium alloys containing respectively 0·5, 1, 2, 3 and 4 per cent aluminium, magnesium-chromium alloys containing 0·01, 0·05 and 0·1 per cent chromium, and magnesium-nickel and magnesium-manganese alloys at 0·01 and 0·05 per cent solute concentration were studied. The alloys were prepared by melting 50 g. of magnesium in stainless steel crucible under argon atmosphere and solute additions were made at 700°C. in all cases. The ingots were cast in graphite moulds at (1) room temperature, (2) 300°, and (3) 550°C. The moulds were preheated to avoid columnar structure to facilitate measurement of grain size. The effect of superheating was also studied by heating the melt to 900°C. and cooling it to 850°C. before casting in graphite moulds at (1) room temperature, (2) 300°, and (3) 550°C. For comparison, specimens of magnesium were also similarly cast. Following conclusions were drawn:

(i) The grain refining tendency increases in the order: nickel-manganese-aluminium.
(ii) Chromium has little effect on grain refinement but may eliminate the columnar crystallization.
(iii) In all cases superheat causes increased grain refinement though to varying extent.

Attempts are also being made to identify the solidification embryo by the liquid metal centrifuging technique.

48.0 Phase-transformation in Iron-manganese System

Work was taken up with a view to study the phase transformation in iron-manganese system.

In the earlier Report it was pointed out that there are two routes for the decomposition of Gamma phase. In Fe-Mn system these two frequently occur together, irrespective of whether the Epsilon phase finally decomposes into Alpha or not. In other words, austenite can either decompose directly to ferro-magnetic Alpha-martensite which is a bcc structure, or can decompose into ferro-magnetic Alpha phase through the intermediate formation of (i) stacking faults, and (ii) paramagnetic epsilon martensite. Since the stacking fault energy of Fe-Mn alloys is low, it should be possible to promote the Gamma to Epsilon transformation and to suppress the Gamma to Alpha transformation through the introduction of stacking faults in austenite. One method of introducing stacking faults, without change in composition, is to plastically deform the austenite. In this investigation the Fe-15 per cent Mn alloy was chosen for such a study as in this alloy Gamma ordinarily transforms to a mixture of Alpha-martensite and Epsilon-phase. Stacking faults were introduced through plastic deformation in a drop forging hammer and their effect on transformation is summarzied in Table 49.

Table 49 — Effect of Deformation of Gamma-Epsilon Transformation

Drop forging temperature °C.	Percentage of reduction	Response to a strong Magnet
750	28	Ferromagnetic
	37·5	Ferromagnetic
	75	Paramagnetic
700	28	Ferromagnetic
	37·5	Ferromagnetic
	80	Paramagnetic
650	37·5	Ferromagnetic
	90	Paramagnetic

It will be noted that the Gamma to Alpha transformation is completely suppressed after 75-90 per cent reduction in height in the temperature range 650°-750°C. Therefore, stacking faults nucleate and faciltate the Gamma-Epsilon transformation. When the deformation was carried out at higher temperature, Gamma transformed to Alpha, presumably because the stacking faults created by plastic deformation were instantaneously destroyed by thermal agitation.

49.0 Preferred Orientation in Extruded Rods

This project was taken up with a view to study the preferred orientation developed during extrusion as a function of (i) temperature of extrusion, (ii) speed of extrusion, and (iii) position in the rod and to study the influence of preferred orientation developed during extrusion on (a) mechanical properties, and (b) preferred orientation to be developed on subsequent cold drawing of the extruded rod.

During the period under review, the variation of the texture from the surface to the centre of the extruded rods of duralumin and commercial aluminium was studied using the conventional X-ray diffraction technique. The X-ray diffraction photographs were taken for 20 positions at a pitch of 0·0376 and 0·0392 in. (5 per cent of the original diameter of each of the extruded rods of duralumin and aluminium, respectively, from the surface to the centre). The results are summarized in Table 50. Further work is in progress.

50.0 Solubility of Nitrogen in Iron Alloys

Nitrogen considerably affects the properties of iron and steels, and the solubility of nitrogen in molten iron and the influence

Table 50 — Texture in Extruded Rods of Duralumin and Commercial Aluminium

Alloy	Zone	Texture	Remarks
Duralumin	Surface (Skin thickness, 0·113 in.)	[111] [100]	Prominent 80%
	Central*	[111] [100]	50% 50%
Commercial Aluminium	Surface (Skin thickness 0·157 in.)	[100]	Superimposed upon a random orientation. Comparatively the larger grains are oriented in a preferred manner showing the presence of [100] texture and the smaller grains are arranged randomly
	Central*	—	Randomly orientation

*The transition from surface to central texture occurs over an intermediate zone of 0·15° in.

of small alloying additions in iron on the solubility in liquid state has since been studied by several workers. The data on the solubility of nitrogen in alloys of iron in solid state, particularly below 1000°C., are, however, scanty and investigations are in progress to determine the nitrogen solubility in solid Fe-X-N alloys. The experimental set up comprising a silica reaction tube, manometers and a gas burrette with temperature control arrangements for both in the reaction zone and in ambient conditions was installed and calibrated at different temperatures and pressures up to 1000°C. Hot volume determinations at fixed temperatures and varied pressures in sub-atmospheric conditions gave rise to linear relationships and were used as calibration curves for solubility determinations. A vacuum melted iron-chromium alloy containing 4·01 per cent Cr was laminated to 0·01 in. thickness and nitrogen solubility at 800°C. determined. The solid-gas reaction was very slow and took very long to attain equilibrium. It was found that solubility of nitrogen in an iron-chromium alloy containing 4·01 per cent Cr obeyed Sievert's law up to 0·04 per cent nitrogen. Further experiments at higher temperatures and different pressures are being carried out.

51.0 Measurement of Activity of Carbon in Fe-Cr Alloys

The activity measurements of carbon in iron at 800°, 1000° and 1200°C. has been completed and the investigation is being extended to study the effect of alloying elements like Cr, Mn, Si, CO, Ni, V, etc., on the activity of carbon in iron. Studies on the effect of chromium on the activity of carbon in solid iron has initially been taken up.

The experimental set up earlier used for the studies with pure iron was used for the purpose. A sample of iron-chromium alloy containing 4·5 per cent chromium, by weight, was prepared in a vacuum induction furnace by melting Armco iron and electrolytic chromium. The iron-chromium alloy was then rolled to a 0·25 mm. thick sheet. The samples cut to 2·5 × 2·0 cm. from the rolled sheet were equilibrated with gaseous mixtures of carbon monoxide and carbon dioxide for a period of 72 hours or longer at 1000°C. The equilibrium condition was ascertained from the analysis of the equilibrated gas mixture at intervals of 12 hours, such that there was no change in the gas composition in two consecutive gas samples. Equilibrium was approached from carburizing side only, and the equilibrated iron-chromium samples were then analysed for carbon content by usual combustion method.

The equilibrium partial pressure ratio p^2CO: pCO_2 and the corresponding carbon content of the iron-chromium samples are given in Table 51. From the results of this investigation the carbon content of the sample corresponding to the particular

Table 51 — Results of Equilibration at 1000°C

Expt. No.	$p^2CO : pCO_2$	Wt C %
1	11·92	1·1510
2	19·08	1·1570
3	36·44	1·2990
4	56·99	1·4950
5	108·00	2·6230
6	80·83	3·4710
7	82·13	1·9027
8	80·39	2·0250
9	117·80	2·4880
10	135·40	2·8758

$p^2CO : pCO_2$ is slightly higher than that reported by previous workers. Further work is in progress.

52.0 Effect of Titania on the Viscosity of Blast Furnace Slags

The high alumina of the Indian blast furnace slags, ranging from 25-30 per cent Al_2O_3, results in a viscous as well as high-melting slag. Replacement of a part of lime by magnesia lowers the melting point of the blast furnace slags to some extent. On the basis of the constitution of liquid silicates, addition of small quantities of titania to the high-alumina blast furnace slags is expected to lower the viscosity of such slags due to the lower metal: oxygen ratio of TiO_2 as compared to lime or magnesia. Studies were undertaken to determine the effect of titania on the viscosity of high-alumina blast furnace slags.

A torsional type viscometer and a high-temperature molybdenum wound furnace were fabricated, and the unit was assembled. The viscometer was calibrated at room temperature and a linear relationship was observed between the fluid viscosity and the logarithmic decrement of the oscillatory system. High-temperature viscosity measurements of two CaO-MgO-Al_2O_3-SiO_2 slags at various temperatures up to 1600°C. were carried out and the viscosity-temperature relationships agreed with the results obtainted using a rotating type of viscometer. Viscosity measurements with titania containing slags are being initiated.

53.0 Photo-elastic Stress Analysis

Work on photo-elastic stress analysis covered the study of distribution of stresses in engineering components. A study is necessary from the point of view of understanding the behaviour of the components as well as for improving the design. Photo-elastic evaluation of stress concentration factor due to multiple notches was undertaken to obtain quantitative measure of the stress-relieving effect of neighbouring notches. Both theoretical and experimental determination of stress-concentration factors due to single notch is available. But the studies on stress-concentration factor due to multiple notches and studies on the influence of neighbouring notches on stress-concentration factor are very scarce. Photo-elastic evaluation of stress-concentration factor due to multiple notches with different spacing and different relative depth was undertaken to provide further data to assist the designer in utilizing stress-relieving effect of notches in design engineering.

Stress-concentration factor due to three notches in a beam under pure bending was measured using two sets of values for beam width, minimum section and depth of the central notch. In each series the stress-concentration factors were determined with varying notch separation and relative depth of the notches. On the basis of the two series of experimental results, the empirical formula giving the per cent reduction of stress at the root of the central notch in terms of notch separation and relative depth of the side notches were obtained; this is of the form:

$$Z = \frac{AB^x}{10^{C-Dx}-1}\left[10^{(C-DX)y}-1\right]$$

where

Z = per cent stress relief at the root of the central notch

$X = \frac{a}{2r}$, a = notch separations,

r = radius of the notches

$Y = \frac{b}{h}$, b = depth of the side notches;

h = depth of the central notch

The constants A, B, C, D were determined for the two series of notches. These constants are obviously function of d h and r, and the stress system, where d is the minimum beam width under the notch. Further work is in progress.

54.0 Study on Internal Friction of Metals

The aim of the project was to study the phenomenon of strain aging, temper brittleness and interactions of interstitial solute atom of nitrogen and carbon in iron through measurement of internal friction. Study of aging behaviour of Fe-N and Fe-Mn-Ni alloys was undertaken by means of internal friction technique. A simple low-frequency (1-2 cycles per sec.) torsion pendulum apparatus designed and fabricated at the National Metallurgical Laboratory was used. Three heats with different Mn and N contents were prepared using low-residue iron. The ingots were hot-forged to about $\frac{1}{4}$ in.^{2} bars which were subsequently machined and drawn to 19 SWG size wires. The final cold drawing was adjusted to give about 30, 50 and 80 per cent cold reductions in the final wires of 19 SWG size. The cold-drawn wires were quickly mounted in the internal friction apparatus and I.F. values were found. The specimens were subsequently given annealing treatment for 1 hr. at temperatures ranging from 100°-600°C. and the values of I.F. were measured. Variations in the results were observed for samples treated under similar conditions. Some of the tests are being repeated. In general, the I.F. values registered a drop on annealing up to 200°C. and after about 400°C. it showed rising tendency possibly due to recrystallization and re-solution of precipitates for annealing at higher temperature. Further experiments to confirm the earlier results and verify their reproducibility are in progress.

55.0 Study on Reflectivity and Hardness of Ore Minerals

To study the reflectivity and hardness of ore minerals about 80 samples were collected so far. It was observed that the temperature and pressure involved in the thermoplastic mounting process induced some change in physical properties as the specimens were mounted in a cold-setting plastic. Polishing technique which had been developed and being followed is as follows:

(i) Two stages of grinding in the Garton-Vanderwilt polishing machine.

(ii) Polishing with the two grades of diamond paste for rough and fine finish.

(iii) Buffing with chromic oxide for two to three minutes.

(iv) Chemical and mechanical polishing to remove fine scratches with a mixture of chromic acid, ferric oxide and distilled water. The proportion depends on the nature of the mineral. However, this was not effective for iron minerals.

Polished sections for about 50 ore minerals were prepared, and constituent minerals were identified with the conventional ore microscopic methods. X-ray diffraction techniques were employed to nearly 30 ore minerals. Evaluation of the inter-planar spacings for 20 minerals was completed and for the rest the calculations are being carried out.

Photo-electric cell housing was designed and fabricated. Baral section of pure quartz to be used as standard in reflectivity measurements was prepared. Micro-hardness values for the already identified minerals are being determined.

56.0 Development of Thermostatic Bimetals

Thermostatic bimetals are temperature-sensitive elements that are widely used in a great variety of ways for indicating, regulating and controlling temperature, and as thermal relays, circuit-breakers and overload protection devices. The entire requirements of bimetals are met today in the country by import. With the increase of the electrical and engineering industries in this country, the need for bimetal will be increasingly felt. This research project has been in progress to develop suitable manufacturing techniques for different types of bimetals.

Extensive studies were completed earlier on the physical and mechanical properties of various ferrous and non-ferrous alloys to assess their suitability for use in thermo-bimetals. Low-expansion alloys were mainly Fe-Ni alloys with 36-46 per cent nickel. Amongst the high-expansion alloys various austenitic Fe-Ni-Cr-Mn alloys, brass, silicon bronze and Mn-Cu-N alloys were studied.

The manufacturing technique of all ferrous bimetal was worked out in detail and the process was leased out to two firms for commercial exploitation. Practical demonstration was arranged to the representatives of the firms. The technical know-how for the production of thermo-bimetals and the associated problems in various stages of production were explained and clarified. The various stages of production involves (i) melting and casting of ingots, (ii) hot working of ingots to slabs, (iii) surface preparation and preparation of the composite, (iv) bonding of the composite, (v) hot-rolling of the bonded composite to sheets, (vi) pickling and cleaning, (vii) cold rolling, (viii) heat treatment, and (ix) testing.

The technique of production of bimetals with copper-base alloys, particularly brass, as high-expansion component was worked out in detail and samples of brass-invar bimetal were supplied to various interested parties. Mn-Cu-Ni alloys were studied for use as high-expansion component; the technique of melting and casting of these alloys and their hot-working characteristics were established. Some amount of thermo-bimetals of this type were produced in which bonding was effected by heat and pressure. Further standardization is necessary as regards production technology of this special type of thermo-bimetal. Work was also done for developing the technique of production of composite strips and wire for use as substitute for springs and conductors in the electrical industry. A preliminary Report was prepared regarding the formulation of a draft Indian Standard for General Requirements for Thermo-bimetals.

57.0 Study on Hot Working of Steel

This project was taken up with a view to study the overheating and burning in relation to hot working of steel. For this study, a definite programme of heating and forging sequence was worked out, and some steels were carefully selected. Over-heating and burning phenomena are known to be closely associated with the grain boundary of steel; hence, the study of the grain boundary is important along with other factors which affect over-heating and burning in steel.

The work on the low-alloy steel done earlier was completed. A study of high-phosphorus and sulphur in steel was taken up to correlate their effect on the overheating and burning characteristics of the steels. For this purpose a high-phosphorus steel of the composition C, 0·66; Mn, 0·73; Si, 0·3; S, 0·02 and P, 0·14 per cent was taken up. The effect of heating on the segregation of phosphorus at grain boundary was studied at varying temperatures ranging between 1000° and 1200°C. To study the diffusion of sulphides in this steel suitable specimens

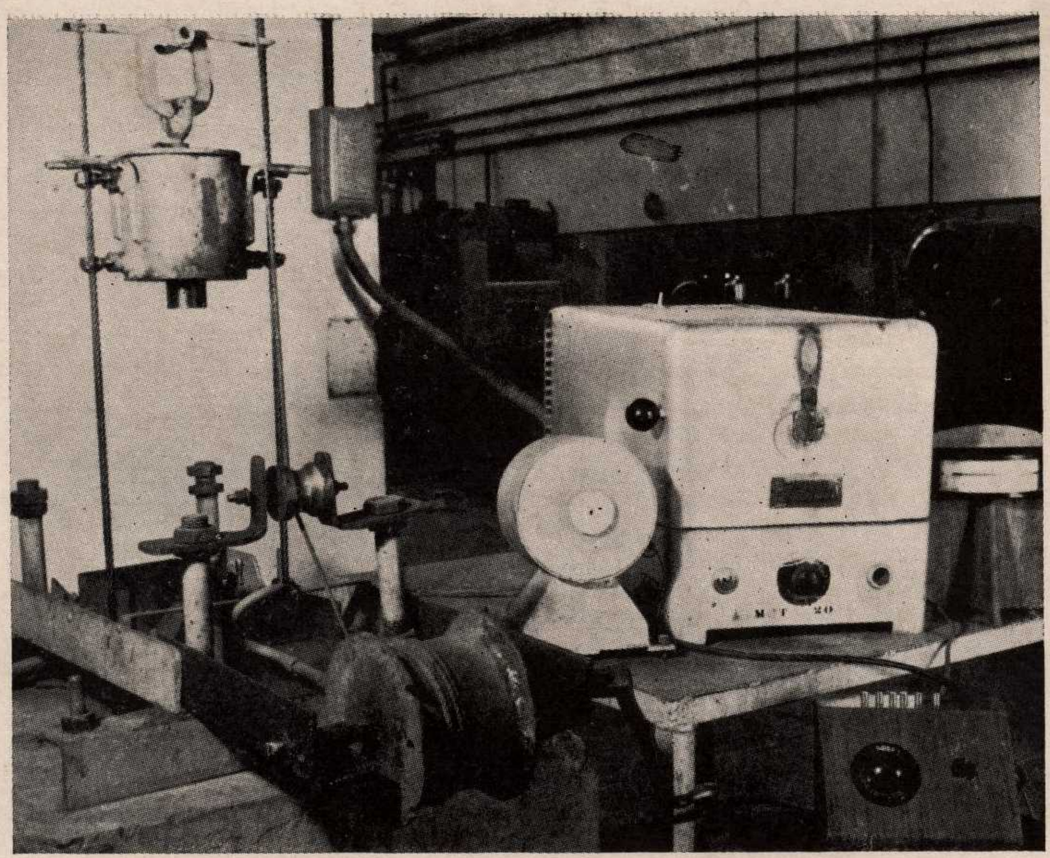


Fig. 35 — Drop Forging Hammer for studying the Hot Workability of Steel, designed and fabricated at the National Metallurgical Laboratory

were prepared, so that the diffusion of sulphides into it from the surface may be studied conveniently. These specimens are now under study at temperatures varying between 1000° and 1150°C. and period of heating ranging from 1 to 5 hours.

To study the effect of alloying elements on the overheating and burning properties of steel, six heats were prepared. First three of them were plain carbon steels with varying manganese content and the other three contained molybdenum with 1 per cent Mn. The compositions of these steels are given in Table 52.

Table 52 — Composition of Steels

Heat No.	C %	Mn %	Mo %	S %	P %	Si %
1	0·28	1·1	—	0·009	0·021	0·07
2	0·23	1·4	—	0·030	0·025	0·06
3	0·24	2·9	—	—	0·028	0·10
4	0·18	1·1	0·20	0·009	0·030	0·03
5	0·22	1·0	0·37	0·009	0·033	0·06
6	0·21	1·0	0·44	—	0·030	0·08

Each of these six steels were being forged at four different temperatures ranging from 1150° to 1300°C., and at each temperature there would be sequences of heating and forging. Thus from each heat 16 specimens would be obtained. These would then undergo mechanical testing and metallographic study.

58.0 Development of Clad Bearing Metals

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

The process of cladding aluminium on mild steel by rolling and the optimum conditions required for obtaining good bond strength was earlier worked out and reported. Further work was carried out to utilize the same technique for cladding aluminium-base bearing alloys on mild steel to be utilized as bimetallic bearings: (i) to develop a suitable aluminium-base bearing alloy possessing superior bearing properties and (ii) to clad the developed alloy with mild steel utilizing the above technique.

At the first instance attempts were made to develop a suitable aluminium-base bearing alloy of improved bearing properties and next to roll-clad it on mild steel as mentioned above. To develop soft bearing metal various heats were made and reported earlier. Further heats were made of aluminium-lead-antimony group and aluminium-lead-antimony-copper group for bearing over-lay application. Due care was taken while casting to obtain the uniform metallurgical structure throughout the ingot. In some cases repeated heats were made to standardize the casting technique. After metallographic examination requisite mechanical tests were carried out. Some of the test results are shown in Table 53 and compared with tin-base white metal, a standard bearing alloy used as over-lay application. Micro-structure of an experimental soft aluminium-base bearing alloy is given in Fig. 36.

Table 53 — Mechanical Properties of Heats

Heat No.	Young's modulus $K10^6$ lb./in.^{2}	Ultimate tensile strength in T.S.I.	Elongation %	Hardness in B.H.N.	Co-efficient of friction under lubrication
2	6·5	7·0	23·0	23·5	0·018
24	6·7	4·7	12·5	25·0	0·021-0·025
26	—	5·0	31·0	24·2	0·018-0·020
32	—	5·0	18·5	23·0	0·018
33	6·9	5·0	12·5	27·0	0·021
29	6·7	8·4	12·5	26·5	0·020
White metal	7·6	—	—	24·0	0·022

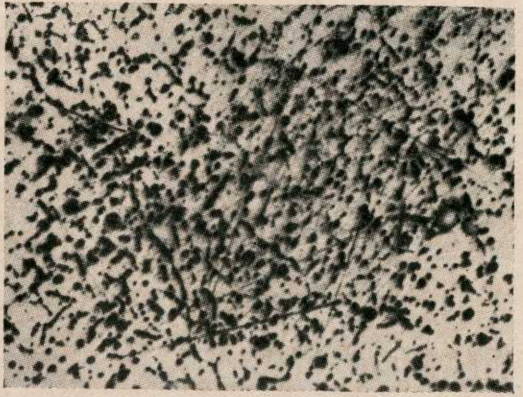


FIG. 36 — STRUCTURE OF AN EXPERIMENTAL SOFT ALUMINIUM-BASE BEARING ALLOY

Results so far obtained after various tests indicated that alloying of antimony and lead in small percentage with aluminium offer potential scope. Additions of copper and magnesium are helpful in achieving desired mechanical properties for varied bearing alloy application. A soft bearing alloy of the aluminium-lead-antimony group was also rolled and clad on mild steel to be utilized as sleeve bearing. After conducting few more experiments, the tests would be carried out initially by replacing conventional bearings in machines of non-critical service conditions and, finally, in machines of more demanding service conditions.

59.0 Production of Basic Refractories from Indigenous Resources

(i) *Development of Refractories from Almorah Magnesite*

This project was undertaken to assess the suitability of magnesite from Almorah region for the manufacture of basic refractories in view of the increasing demand of basic refractories due to fast expanding steel industry. Earlier work on the same magnesite gave encouraging results; so, the present work was taken up to evalute the suitability of Almorah magnesite for the manufacture of chrome-magnesite refractories.

The analysis of the Tulloc's chrome ore and the two magnesites used for the development of chrome-magnesite refractories are given in Table 54. Three compositions were made with 70 per cent chrome ore and 30 per cent magnesite, 60 per cent chrome ore and 40 per cent magnesite, and 15 per cent chrome ore and 85 per cent magnesite. In 70: 30 and 60: 40 compositions chrome ore was all coarse (−5 +52 B.S.S.) and magnesite all fine (−72 B.S.S.); but in 85 per cent magnesite composition, magnesite was graded suitably. The test samples made were fired at 1400°, 1500°, 1600° and 1700°C. The physical properties studied are given in Table 55. A few other properties like, refractoriness under load, iron oxide bursting, etc., are yet to be determined.

Table 54 — Analysis of Chrome and Magnesite Samples

%	Chrome ore	Almorah magnesite (raw)	Almorah magnesite (beneficiated)	Salem magnesite	Salem magnesite dead burnt 1650°C. with addition of iron oxide
L.O.I.	0·68	48·86	0·52	49·53	1·27
MgO	12·65	44·20	85·90	46·50	87·4
CaO	Traces	2·50	2·94	1·21	0·78
FeO	9·10	—	—	—	—
Al_2O_3	7·40	0·20	1·30	0·32	0·68
SiO_2	6·50	2·40	4·50	2·44	5·16
Fe_2O_3	7·99	2·30	4·47	0·16	4·20
Cr_2O_3	54·83	—	—	—	—
Alkalis	Traces	—	—	—	—
TiO_2	0·51	—	—	—	—
Moisture	—	—	Traces	—	0·18

Thermal spalling test of the samples was conducted at 1400°C. The samples were heated at 1400°C. in a down draft kiln for 10 minutes and were then cooled for 10 minutes in running water. The number of such heating and cooling cycles which the samples could stand before they were completely spalled was taken as the spalling index. As the test is not a standard one, imported chrome-magnesite bricks from U.K. (Stein KM) were used for comparison. While the imported bricks stood 2-3 cycles only, indigenous samples stood more cycles. It was also observed that the index of spalling was increasing with increasing firing temperatures and went up to 6-7 in case of D-30 specimens fired at 1700°C. Textural and mineralogical properties of the fired specimens were studied both in thin and polished sections. Both direct and silicate bonding between the chromite and periclase grains were observed to varying degrees depending on the composition and temperature of firing. But in almost all the cases the texture of the fired specimens was a bit open and there were hollows around the chrome grains indicating that the magnesia matrix possibly shrunk away from the chrome grains. Attempts are being made to study this phenomenon and thereby improve the texture of the fired specimens. Experiments are also being conducted to evalute the suitability of magnesite from Dewaldhar sector of Almorah region for the manufacture of basic bricks.

Table 55 — Physical Properties of the Chrome-magnesite Composition Fired at Different Temperatures

Composition	Sl. No.	Bulk density in g./cc.				Apparent porosity %				Cold crushing strength lb./in.^{2}				Linear change on firing				Specific gravities		
		1400°C.	1500°C.	1600°C.	1700°C.	1400°C.	1500°C.	1600°C.	1700°C.	1400°C.	1500°C.	1600°C.	1700°C.	1400°C.	1500°C.	1600°C.	1700°C.	1500°C.	1600°C.	1700°C.
Almora magnesite burned at 1650°C. and chrome ore	A-30	3·22	3·150	3·22	3·237	18·84	19·27	19·49	19·85	6378	2851	3027	4277	0·100 exp.	0·430 exp.	0·680 exp.	0·125 exp.	3·9280	4·012	4·083
	A-40	3·09	3·145	3·14	3·230	20·81	19·38	17·90	16·50	5666	2780	2637	3920	0·272 exp.	0·298 contr.	0·227 contr.	1·700 contr.	3·881	3·914	3·955
	A-85	2·77	3·000	3·16	3·213	23·13	14·04	11·58	8·40	6592	6521	7055	7340	0·166 contr.	3·382 contr.	3·868 contr.	4·760 contr.	3·567	3·648	3·658
Beneficiated almora magnesite and chrome ore	B-30	3·18	3·140	3·08	3·090	19·56	21·26	20·70	21·05	3849	2530	1924	1781	0·881 exp.	0·610 exp.	0·698 exp.	0·756 exp.	3·965	4·005	4·050
	B-40	3·07	3·040	3·03	3·090	22·83	22·75	21·73	21·11	4882	3100	2815	2066	0·878 exp.	0·6113 exp.	nil	0·262	3·859	3·959	3·904
	B-85	2·69	2·830	2·90	3·040	24·04	23·07	19·22	15·56	5346	5166	5273	5453	0·272 contr.	1·679 contr.	3·470 contr.	4·150 contr.	3·579	3·662	3·656
Dead burnt Salem magnesite with iron oxide addition and chrome ore	C-30	3·20	3·260	3·13	3·080	20·96	27·37	22·68	22·63	3599	2316	2993	2565	0·354 exp.	0·610 exp.	0·960 exp.	1·135 exp.	3·946	4·003	4·017
	C-40	3·09	3·020	3·03	3·050	22·09	22·51	23·42	22·31	3528	3386	3706	2673	0·529 exp.	0·611 exp.	0·663 exp.	0·785 exp.	3·917	3·923	3·953
	C-85	2·81	2·860	2·83	2·910	21·63	20·26	21·00	19·72	2886	3706	4633	3715	0·281 contr.	0·102 contr.	0·174 contr.	0·527 contr.	3·511	3·593	3·635
Salem magnesite dead burnt without iron oxide addition and chrome ore	D-30	3·19	3·270	3·14	3·220	19·23	19·82	19·70	18·52	4703	3991	3207	2316	0·309 exp.	0·419 exp.	0·533 exp.	0·785 exp.	3·949	3·955	4·008
	D-40	3·08	3·120	3·15	3·100	21·31	21·37	20·86	19·29	5273	4170	2743	2565	0·330 exp.	0·240 exp.	0·26 contr.	0·640 contr.	3·870	3·925	3·981
	D-85	2·75	2·810	2·91	3·150	25·72	23·20	19·35	11·38	5809	5310	3277	5417	0·724 contr.	0·787 contr.	1·14 contr.	2·070 contr.	3·610	3·570	3·646

(ii) *Investigation on Nattam Dolomite for studying its Suitability as Refractory for Neyveli-Salem Steel Project*

This investigation was to study the suitability of Nattam dolomite as steel plant refractory material for Neyveli-Salem Steel Project.

About 1 ton of dolomite was obtained. The sample was crushed in jaw crusher, roll crusher and ball mill and then Mix I and Mix II were prepared with the addition of 32 and 39 per cent Salem magnesite, respectively. The two mixes were fired to different temperatures from 1500°-1700°C. and the physical properties like bulk density, porosity and hydration tendency were studied. Besides these, sintering characteristics of raw dolomite lumps (1·5-2·5 cm.) under various firing conditions from 1500°-1700°C. were also studied. Table 56 indicates the difference in chemical analysis of dolomite from bag to bag.

Table 56 — Chemical Analysis of Dolomite

L.O.I. %	43·01	43·45	43·32	44·15	43·49	42·82	44·32
SiO_2%	3·27	2·80	2·65	2·18	2·66	3·52	1·70
Fe_2O_3E	0·41	0·42	0·37	0·53	0·32	0·34	0·34
Al_2O_3E	0·29	0·34	0·69	0·13	0·24	0·19	0·16
CaO%	31·77	31·42	31·42	30·52	30·52	30·52	30·52
MgO%	20·94	21·37	21·37	22·23	22·66	21·80	22·66
Moisture%	0·26	0·20	0·13	0·10	0·14	0·18	0·11

Table 57 indicates the bulk density, and porosity of Mix I and Mix II fired at different temperatures.

Table 57 — Bulk Density, and Porosity of Mix I and Mix II

Firing temperature	1500°C		1600°C		1700°C	
	B.D.	Porosity	B.D.	Porosity	B.D.	Porosity
Mix I	2·445	27·36	2·40	28·25	2·74	14·39
Mix II	2·320	30·54	2·41	27·80	—	—

Larger batches of Mix I and Mix II for tar-bonding and composition of complete stabilizing of dolomite are being prepared for further study.

60.0 Studies on Carbon Refractories

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 application in metallurgical and chemical industries.

During the period under review, work was carried out on (1) production of dense aggregate from Digboi raw petroleum coke to test and standardize the process variables, (2) characteristics of certain Indian petroleum cokes, and (3) testing carbon under actual electrolytic conditions to find out the suitability of dense-carbon-aggregates as cathode liner in aluminium reduction cell.

(i) *Study of Dense Aggregate*

The materials after sampling were passed through jaw and roll crushers, and pan mill to obtain powders of required particle sizes. Sieve analyses, packing densities, proximate analyses, etc., were determined by standard methods. Compositions were prepared using available binders in various proportions by weight at predetermined pressures. The green bodies were then fired in reducing atmosphere according to certain preset temperature schedule. Porosities, bulk densities and apparent specific gravities of the aggregates were determined after firing (Table 58).

Table 58 — Porosity, Bulk Density and Apparарent Specific Gravity of Aggregates

	App. porosity %	Bulk density g./cc.	App. specific gravity
Aggregate I	19·0	1·60	2·00
Aggregate II	18·0	1·63	2·01
Aggregate III	20·0	1·60	2·00
Aggregate IV	18·5	1·61	2·01
Aggregate V	17·0	1·65	2·00

(ii) *Characteristics of Certain Indian Petroleum Cokes*

For this purpose raw petroleum coke from Gauhati and Digboi were chosen. The aim of this work was to find out the changes in properties, if any, with the variation of calcination temperatures. The materials were crushed in jaw and roll crushers and graded to obtain -10 $+18$ mesh B.S.S. powders. They were then calcined in a tube furnace in N_2 atmosphere at 400° to 1300°C. at an interval of 100°C.

The following properties are being determined with these samples:

1. Loss in weight in nitrogen atmosphere.
2. True density.
3. Electrical resistivity.
4. D.T.A. in oxygen atmosphere.

(iii) *Testing of Carbon under Actual Electrolytic Condition*

For testing carbon under actual electrolytic condition, an apparatus was set up. The apparatus consisted of a kanthal wound pot furnace in which the bath temperature could go up to 1000°C. The electrolytic bath consisted of cryolite and alumina. The cylindrical cathode specimen measured about 2 in. in diameter and was 2 in. long. A $\frac{1}{2}$ in. diameter graphite rod was inserted in a hole in one end and cemented to it. Nitrogen atmosphere was to be maintained inside the furnace.

In connection with this investigation the following apparatuses were fabricated:

1. Giesler plastometer: This apparatus will be useful to measure the relative plastic behaviour of coal when heated under prescribed conditions in the absence of air (Fig. 37).
2. Electrical resistivity apparatus: With this apparatus electrical resistivity of carbon powders as well as blocks can be measured (Fig. 38).
3. Swelling apparatus: With this apparatus, the degree of swelling of tar and pitch can be determined when they are heated (Fig. 39).
4. A graphitization furnace which can go to a temperature of 3000°C., was constructed.

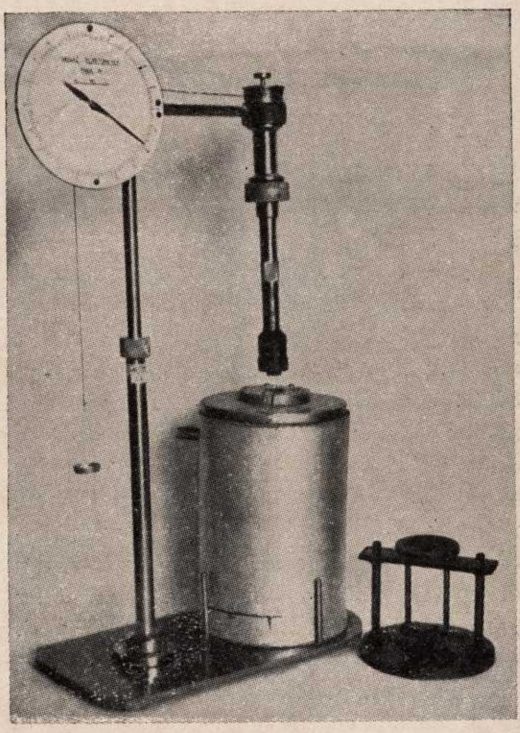


FIG. 37 — GIESLER PLASTOMETER TO MEASURE PLASTIC BEHAVIOUR OF COAL, FABRICATED AT THE NATIONAL METALLURGICAL LABORATORY

61.0 Study of Some Refractory Clays from Jammu & Kashmir

Large deposits of refractory clays with a high alumina content have been found associated with the bauxite deposits of Salal area of Rissi Tehsil in the Udhampur district of Jammu State and the resources are considered to be large. Eight samples of clays collected from different pits in this area were received from Director of Geology & Mining, Jammu & Kashmir Government, Jammu, for investigation of their suitability in the manufacture of fireclay refractories.

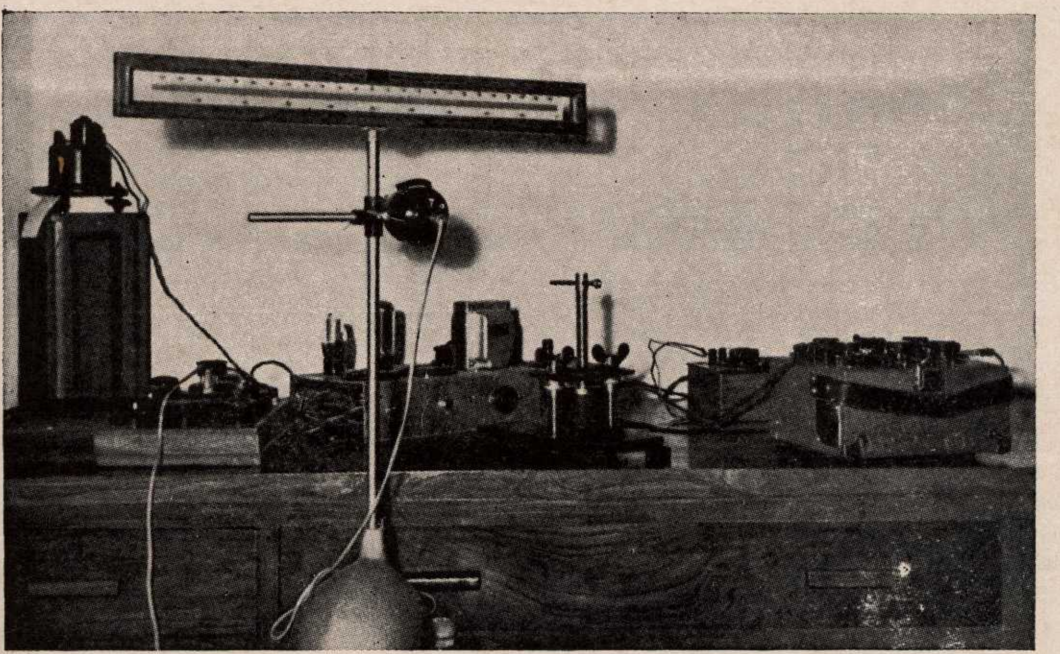


FIG. 38 — APPARATUS FOR MEASUREMENT OF ELECTRICAL RESISTIVITY OF CARBON POWDERS, DESIGNED AND FABRICATED AT THE NATIONAL METALLURGICAL LABORATORY

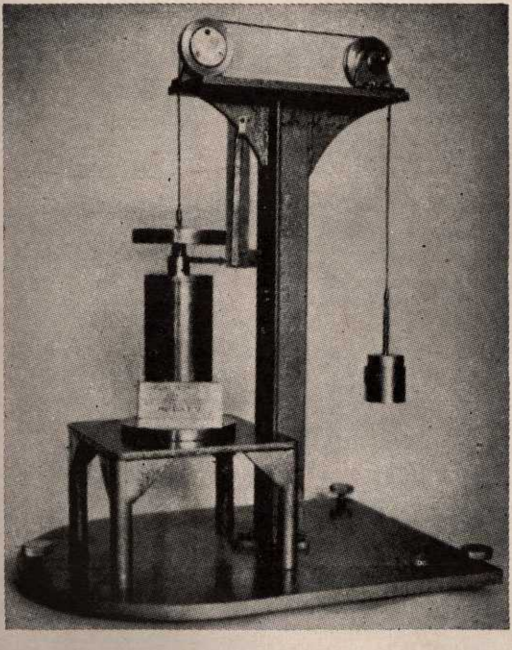


FIG. 39 — APPARATUS FOR DETERMINING THE SWELLING OF TAR AND PITCH, DESIGNED AND FABRICATED AT THE NATIONAL METALLURGICAL LABORATORY

Based on the data reported earlier on the physico-chemical and refractory properties as well as the mineralogical constitution of the clays, the work was taken up during the period under review on the development of superduty fireclay refractories of the high grog type.

To start with, the four clays were calcined at 1300°C. and test specimens were prepared from these grogs and clay C5 (bond clay) in various proportions. The test specimens were fired separately at 1350° and 1500°C. and the effect of firing temperature on their physical properties, namely apparent porosity, bulk density, cold compression strength, modulus of rupture, reheat shrinkage (volume) and permeability, was investigated. It is proposed to determine the refractoriness under load of the specimens before selecting the final composition suitable for the production of superduty fireclay refractories.

62.0 Plasticizing of Non-plastic Refractory Material

An important and major advance in refractory technology is that of producing artifacts from high-temperature refractory raw materials that 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. Of a lesser importance but of great commercial potential are materials, such as sillimanite, kyanite, etc. The aim of this project is to develop refractory specialities from non-plastic materials like sillimanite, kyanite and such other oxides without the addition of binders which are detrimental to their high-temperature properties.

In the earlier Report, the necessary plasticizing treatment and the slip casting method for sillimanite were reported. During the period under review, a coarse-bladed kyanite from Sringeri area in Mysore was investigated and grinding characteristics and optimum conditions for making castable kyanite slips were examined. Fig. 40 shows the results of particle size analysis of ball-milled kyanite with various weight ratios of charged material to grinding medium keeping grinding time (80 hours) and the weight ratio of material to be ground/water, constant. Fluidities of suspension with varying additions of electrolytes were determined using a Mariotte tube. These values are graphically shown in Fig. 41 and the effect of variation in water content of the slips is shown in Fig. 42. The data indicate that for a given fineness, kyanite needs an optimum water and electrolyte concentration to yield slips with good casting characteristics.

The casting rates of the slips prepared under various conditions were determined

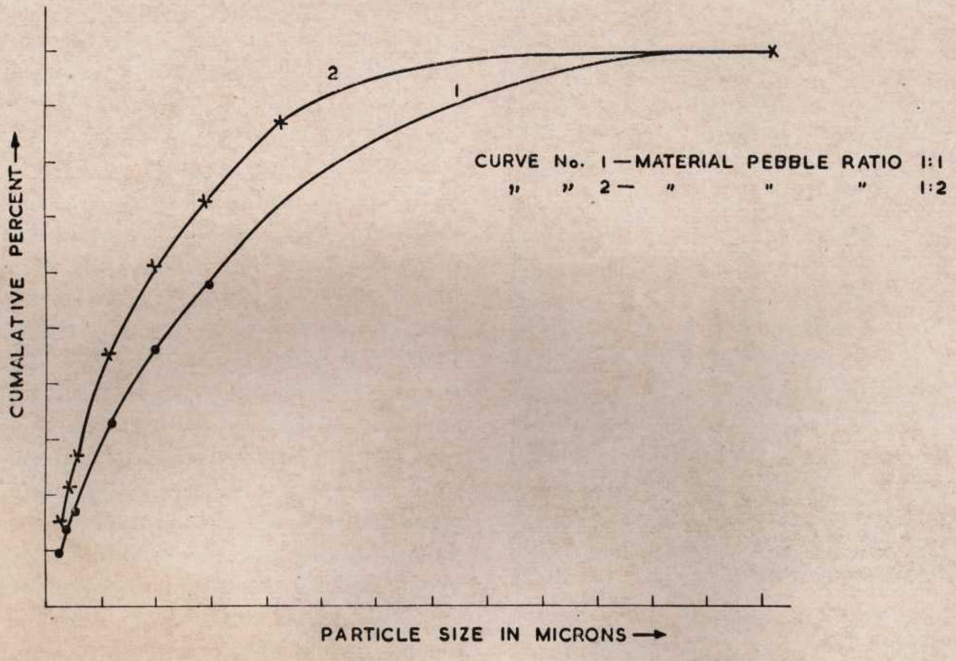


FIG. 40 — PARTICLE SIZE DISTRIBUTION OF BLADED KYANITE

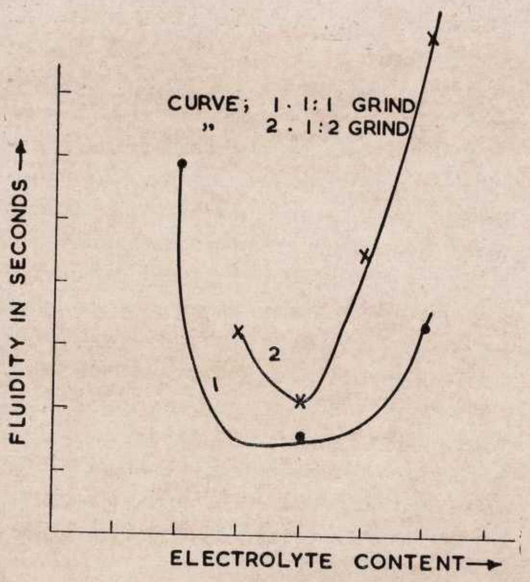


FIG. 41 — ACTION OF ELECTROLYTES ON FLUIDITY OF KYANITE SAMPLES

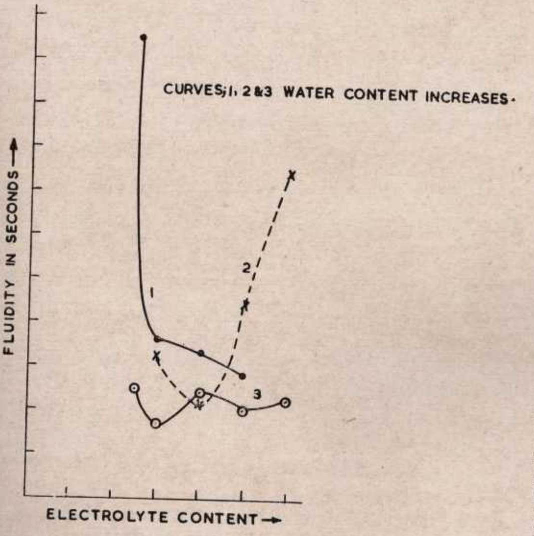


FIG. 42 — EFFECT OF WATER CONTENT OF THE KYANITE SLIPS ON ELECTROLYTE — FLUIDITY RELATIONSHIP

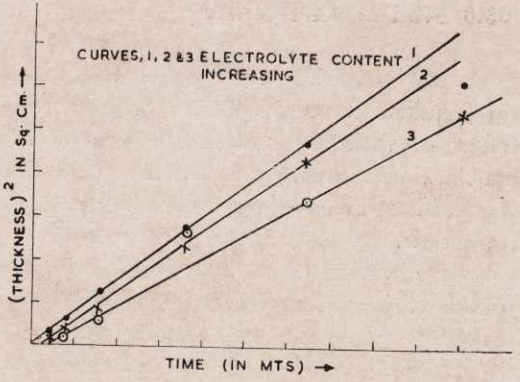


FIG. 43 — CASTING RATES OF KYANITE SLIPS

by the method of Dowall and Adcock. The data were plotted as graphs of thickness square versus time, which are found to be straight lines (Fig. 43). The graphs show that the increasing electrolyte in the kyanite slip, decreased the casting rate. The straight line relationship between L^2 and t (square of thickness of cast bed and time elapsed after starting the experiment) was reported for clay slips as well.

Based on the above results, some small crucibles and thermocouple sheaths were cast from the kyanite slips and fired at 1500°C. The samples had a porosity of 29 per cent and linear shrinkage (from mould to finish) of about 4·5 per cent. The firing range appeared to be narrow because on firing to 1600°C. the samples showed no porosity and started warping. It is, therefore, expected that a firing temperature of 1550°C. may be suitable. Fig. 44 shows the photograph of some slip-cast kyanite shapes.

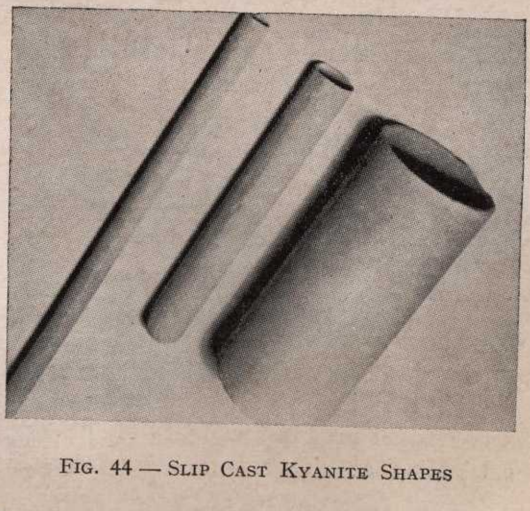


FIG. 44 — SLIP CAST KYANITE SHAPES

63.0 Studies on Kyanite

A raw material study on kyanites was undertaken with a view to find out the effect of various impurities present on the calcination properties, and to assess the possibilities of using them as refractory raw materials.

Three varieties of kyanites — a brown dense variety from Rajkharswan of Bihar called D grade kyanite, and the other two light blue in colour from Orissa — are being studied. The chemical analyses of these kyanites are given in Table 59.

Table 59 — Chemical Composition of the Kyanites (uncalcined)

Per cent	Brown variety (from Rajkharswan of Bihar)	From S. Lal Co. (P) Ltd	
		Sample No. 1	Sample No. 2
Moisture	0·06	—	—
Loss on ignition	0·66	0·87	0·44
Al_2O_3	56·10	58·04	51·96
SiO_2	38·40	38·00	44·24
Fe_2O_3	2·00	0·80	0·88
TiO_2	1·40	0·56	0·46
CaO	Trace	Trace	Trace
MgO	0·5	,,	,,

Samples were calcined at temperatures ranging from 1200° to 1600°C. at intervals of 100°C. in Remy furnace and the physical properties were determined. All kyanites except the brown variety became dull white after calcination at and above 1400°C. Brown variety retained a light pale yellow colour and became dull white on further calcination. A little shattering of the samples was observed, but less shattering was noted in the brown variety.

Physical properties like apparent porosity, bulk density and apparent specific gravity of the above kyanites were determined. A considerable increase in porosity was observed in all the kyanites up to 1400°C. but calcination at higher temperatures tended to bring it down. A fall in apparent specific gravity was observed up to 1400°C. and a little increment was noted on further calcination.

Thin sections of the calcined and uncalcined samples were studied under petrological microscope. Refractive indices were also determined by powder immersion method. Brown variety had twinned needle-like crystals while the other two were bladed type. Though they were colourless, some light yellow tints were seen in some sections. Iron oxides predominate as an impurity in brown variety, while rutile and fine corundum were also present. In samples 1 and 2 free silica and mica were the main associates, though a little corundum and iron oxides were present.

No mullitization was observed on calcination to 1200°C. in all the three kyanites. Samples from Orissa showed a marked strain in crystals and mica fused completely. Samples of brown variety calcined to 1300°C. showed no mullitization but a little strain was noted. Mullitization was seen in samples calcined to 1400°C. and completed at 1500°C. So, it can be said that complete mullitization might have taken place between 1350° and 1450°C. In the blue variety mullitization effects were observed below the above temperatures due to the fluxing action of the impurities. More glass formation was observed in samples No. 1 and 2 than in the brown variety. The studies are being continued.

64.0 Studies on Development of High-alumina Cements from Indigenous Raw Materials

(i) *Development of High-alumina Cement from Indigenous Raw Materials by the Sintering Techniques*

The work so far carried out in this laboratory on development of high-alumina cements was based on the melting process in which the raw materials were heated in a suitable furnace in the required proportions, and the molten cement, cast into blocks, was crushed and ground to give the final product. Alternatively, reactions between the raw materials can also be brought about in the solid state by sintering the charge under suitable conditions. It was with this view that studies were carried out on the development of high-alumina cements from Mysore and Shevaroy bauxites and Madras limestone with and without addition of mineralizers.

Among the factors that contribute to the success of cement-making by the sintering process are (1) chemical composition of the raw materials, particularly the Fe_2O_3 content, (2) fineness of the raw materials, (3) preparation of the batch, (4) temperature of sintering, and (5) soaking time. It was, therefore, decided to examine these factors carefully. Both the wet and dry milling of the raw materials were examined, and sintering was carried out at various temperatures between 1200° and 1400°C. and soaking times varying from 2 to 12 hours. The cementing properties of a number of cement batches were examined. Table 60 gives the properties of some cements developed by the sintering technique.

Table 60 — Properties of Some High-alumina Cements made by Sintering Technique

Cements	Free time test	Setting		Cold compression strength of 1:3 cement: sand; specimens cured in water for 72 hr
		Initial (min.)	Final (min.)	
A	nil	90	280	8600 lb./in.^{2}
B	nil	73	320	7886 lb./in.^{2}
C	nil	80	290	6180 lb./in.^{2}

(ii) *Trials with a Semi-pilot Melting Furnace for Large-scale Preparation of Molten Type High-alumina Cement*

In earlier investigation on the development of high-alumina cements, the cement batches were prepared by melting the raw materials in the desired proportions in a graphite crucible. As it would not be economical to use graphite crucible for melting cement on an industrial scale, a small reverberatory furnace was designed and built in the Laboratory for semi-pilot scale trials using coke oven gas as a fuel. The main objective of trials in this furnace was to collect sufficient data on the melting characteristics of large batches of high-alumina cement. Trials in this furnace are under way.

(iii) *Effect of Fineness of the High-alumina Cement on the Strength and Setting Properties*

With a view to understand the influence of the fineness of cement as controlled by the milling conditions on the setting time and strength of cement, a batch of cement from Shevroy ferruginous bauxite and Madras limestone was prepared. The solidified cement obtained from melting was initially crushed in a jaw crusher and roll crusher to pass through 18 mesh. The crushed cement was further dry-milled in a pot mill for different periods of grinding from 15 to 100 hours keeping the charge: ball ratio always constant. The fineness of the cements thus milled was determined on a Blaime's permeability apparatus. The initial and final setting times, using Gillmore Needle, and the cold compression strength of 1:3 cement: sand (crushed quartzite) mixes after curing for 72 hours in water were determined. The results are given in Table 61.

65.0 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, M/s. Hindustan Steel

Table 61 — Effect of Fineness on Properties of High-alumina Cement

Grinding time hr	Fineness cm.^{2}/g.	Strength lb./in.^{2}	Setting time	
			Initial min.	Final min.
15	2907	3250	172	390
18	3138	3920	182	442
20	3219	3920	285	450
25	3490	4030	350	450
30	3591	4100	365	450
35	3673	4370	420	450
40	3707	4930	245	285
50	3803	4750	248	291
63	3890	4360	110	293
75	4620	4100	120	290
100	4675	1480	88	295

Plant authorities of Durgapur Steel Project desired that investigation to develop these refractories from indigenous raw materials should be taken up to meet the pressing problem.

The aims of this project are two fold, e.g. (1) the comparison of some foreign nozzles with indigenous ones, and (2) effecting some changes in the fabrication process of nozzles of fireclay-grog composition which yield products similar in properties and performance to the foreign nozzles.

Following are the salient points of the results of the first part of the study:

(i) The imported nozzle and its insert are more or less free of alkali. Their alumina: silica ratios are comparable to that of Indian nozzles but the total flux contents of the latter are higher (10·98 per cent). The Indian nozzles invariably contain alkalis in varying proportions, the lowest figure being 0·70 per cent.

(ii) As a consequence of absence of alkalis in the imported nozzles, the glass formed can be expected to be more viscous. In the insert also due to the absence of alkalis it is only at 1500°C. that the glass formation reaches its maximum. The imported nozzle can be expected to possess greater pyro-plasticity near steel-pouring temperature than the indigenous nozzles. Presence of alkalis in nozzle composition may, therefore, be a feature to be avoided.

(iii) The imported nozzle has a low P.C.E. (cone 26) and the insert has a high P.C.E. (cone 31) as compared to the indigenous nozzles (cone 28-30).

(iv) The apparent porosity of the imported nozzle (15 per cent) is much lower than that of the Indian nozzles (23-24 per cent). The insert has a porosity of 19 per cent approached only by one Indian nozzle.

(v) Imported nozzles and insert show a volume expansion on reheating to 1600°C. whereas the indigenous nozzles show further contraction at the same temperature.

(vi) Refractoriness-under-load of imported nozzle is 1270°C. Ta, compared to 1400°C. Ta for the Indian nozzles.

(vii) Textures of these nozzles are shown in Fig. 45. The difference in textures between imported and indigenous nozzles is striking. Both the imported nozzle and insert are fine-grained and uniform in texture while all the Indian nozzles have coarse grains and uneven texture carrying air spaces and cracks.

Assuming that the imported nozzles proved satisfactory in service, the data given above appear to lend special significance to some of their textural and other physical characteristics. Their chemical composition is such that they can be pyro-plastic at steel-pouring temperatures and the uniformity of the textures as well as its fine-grained nature assure that this extends over the entire mass of the nozzle which attains that temperature. This would mean a leak-tight seat for the stopper, uniform erosion and resistance against thermal shocks. From this study, it appears that the fireclay composition for nozzles need not be very

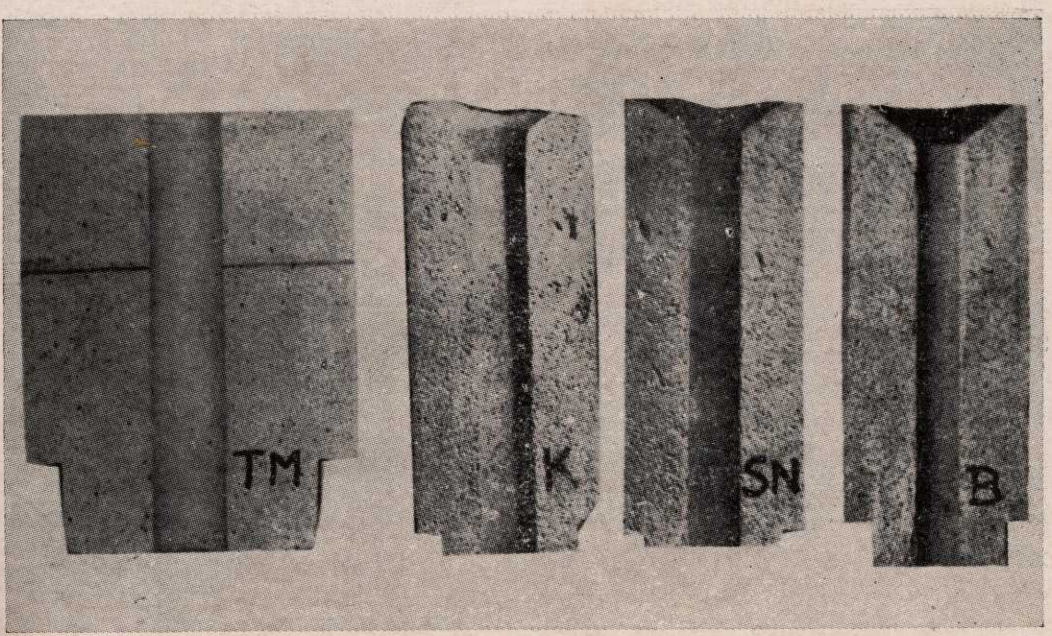


FIG. 45 — RELATIVE TEXTURES OF THE IMPORTED AND INDIGENOUS NOZZLES
TM — Imported Nozzle; K, SN and B — Indigenous Nozzles

refractory. It is, however, desirable that they should be, as far as possible, free from alkalis. If the grog grains and matrix are of the same chemical composition, then there would be no difference in their high-temperature characteristics. The fabrication process must be such as to yield a dense product of uniform texture.

Work is in progress on the second aspect of this problem, e.g. finding out the influence of forming variables in the manufacture of nozzles from fireclay-grog bodies.

66.0 Study on Refractory Plastics and Castables

During recent years, application of refractory castables and plastic mixes in industrial furnaces has gained considerable importance because these (i) provide joint-free furnace linings that present a solid monolithic front to slag and furnace gases; (ii) eliminate air infiltration resulting in lower fuel cost and increased efficiency; (iii) adaptable for any thickness or contour, eliminating the need for special refractory shapes; and (iv) reduce labour cost.

In order to evaluate the quality of the indigenous products, some samples of refractory plastics and castables were collected from different manufacturers of the country with the help of the Indian Standards Institution who are also, incidentally, interested in drawing up suitable specifications for these products. As a first step towards the development of indigenous castable and plastic refractories, it was considered necessary to carry out a systematic examination of some of the presently available castables and plastic refractories made in the country. The results on the tests carried out on plastic refractories were reported earlier. The results on tests carried ouf during the year on six samples of Indian castables claimed to be suitable for application at different temperatures ranging from 1200° to 1400°C. supplied by three different firms, are given in Table 62.

Table 62 — Properties of Indigenous Castables

	I	II	III	IV	V	VI
Chemical analysis %						
L.O.I.	2·96	0·96	1·28	3·28	0·32	0·26
SiO_2	9·60	28·80	17·00	52·68	56·48	30·48
Al_2O_3	53·94	47·40	69·45	33·93	38·48	61·96
Fe_2O_3	7·16	4·60	1·80	1·92	1·57	2·64
TiO_2	2·25	2·50	3·75	1·25	2·00	2·00
CaO	23·10	14·42	7·10	6·44	0·18	2·68
MgO	1·55	0·40	0·50	0·66	0·43	0·25
Alkalis	—	—	—	—	—	—
Sieve analysis (B.S.S.)						
5	—	—	—	—	2·55	1·77
−5 +25	40·0	52·5	52·5	47·66	51·70	53·99
−25 +100	20·0	12·1	5·4	13·05	10·33	20·55
−100	40·0	35·5	41·4	39·27	35·40	23·66
Workability index	10	11	12	14	12	10
Dry shrinkage	nil	nil	nil	nil	nil	nil
Permanent linear change at						
1200°C.	+0·001	nil	nil	−0·001	−0·003	nil
1300°C.	+0·021	−0·006	−0·003	fused	−0·015	+0·016
1400°C.	+0·028	+0·004	+0·007	fused	−0·013	+0·020
Modulus of rupture ($lb./in.^2$) at:						
110°C.	300	383	975	278	240	263
1100°C.	75	260	315	323	195	200
1200°C.	375	278	390	803	473	263
1300°C.	300	675	473	1710	720	338
1400°C.	325	1125	960	—	1515	450
1500°C.	825	1890	—	—	1740	1800
Apparent porosity % at:						
1100°C.	41·6	36·3	35·3	29·2	30·5	32·5
1200°C.	40·7	35·0	33·7	29·7	29·8	24·3
1300°C.	38·5	31·2	33·6	7·7	28·0	24·6
1400°C.	42·2	29·1	35·7	—	17·4	19·4
1500°C.	39·7	16·2	30·0	—	12·0	17·2
Bulk density g./cc. at:						
1100°C.	2·20	1·86	2·12	1·95	1·90	2·10
1200°C.	2·17	1·87	2·13	1·72	1·90	2·02
1300°C.	2·46	1·91	2·11	2·20	1·91	1·97
1400°C.	2·08	1·96	2·08	—	2·08	1·86
1500°C.	2·12	2·23	2·25	—	2·07	2·52

67.0 Standardization of Fireclay and Magnesite Stoppers and Nozzles for Steel Plants

At the instance of various steel plants in the country, the Refractories Committee of Indian Standards Institution has taken up the question of formulating Indian Standards on fireclay and magnesite stoppers and nozzles for use in steel plants. Before doing so, a detailed investigation is to be undertaken on both indigenous and imported stoppers and nozzles being used in various steel plants in the country. A detailed programme has also been chalked out by the Refractories Investigation Subcommittee, SMDC 18 : 3. The preliminary work to be carried out was entrusted to the National Metallurgical Laboratory and Central Glass & Ceramic Research Institute,

Table 63 — Chemical Analysis of Stopper and Nozzles

Per cent	578 HSL (Rourkela)	598 Nozzle Katni	600 Nozzle Orissa	599 Stopper Katni	601 Nozzle IB	602 Stopper SB	606 Stopper B7	607 Nozzle 4B	610 Stopper OR5 Orissa	611 Stopper 6B	612 Nozzle 1B	613 Stopper 5B	614 Nozzle 2B	615 Nozzle 3B	616 CP9 OR	617 Mag. Nozzle OR
Loss at 110°C	0·25	0·14	0·14	0·16	0·18	0·16	0·12	0·16	0·21	0·28	0·29	0·28	0·74	0·87	0·14	0·84
L.O.I.	0·58	0·24	0·24	0·24	0·44	0·42	0·50	0·34	0·56	1·22	0·69	0·69	1·12	1·22	0·40	1·02
Al_2O_3	56·66	51·16	33·96	51·32	31·92	34·88	32·48	37·48	35·04	30·78	36·10	35·80	32·78	43·72	58·86	2·07
SiO_2	37·56	37·28	58·12	35·88	58·88	56·42	55·88	52·28	58·58	60·94	59·08	57·82	60·30	42·84	36·10	7·60
Fe_2O_3	0·26	4·32	3·36	5·44	2·88	2·56	5·76	4·96	1·94	2·28	1·36	0·75	0·91	2·22	1·07	1·16
TiO_2	2·00	0·66	1·84	4·16	2·24	2·36	1·60	1·76	2·36	1·80	1·30	1·93	1·81	3·52	2·71	Traces
MgO	0·50	1·60	1·58	1·52	1·50	1·80	1·90	1·70	0·54	0·41	Traces	0·69	0·91	0·21	Trace	82·81
CaO	0·56	4·24	0·60	0·76	1·57	0·91	0·87	0·91	0·43	0·48	0·71	0·53	0·53	0·59	0·59	3·50
Na_2O	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
K_2O	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—

and both of the laboratories agreed to carry out the tests required to formulate the draft standard.

Scope

The tests to be carried out were as detailed below:

(i) Porosity as per IS specification.
(ii) P.C.E. as per IS specification.
(iii) Refractoriness underload as per IS specification.
(iv) Thermal spalling test on cut specimen or full size stoppers and nozzles at 1350°-1400°C., and cooling in a blast of air.
(v) Reheat shrinkage as per IS specification at 1400°C. for 5 hours.
(vi) Resistance to corrosion or erosion by Finger Test.
(vii) Size tolerance: (a) bore diameter, (b) outside diameter, and (c) joint curvature (seat of the stopper head).
(viii) Cold crushing strength as per IS specification.

Accordingly, a large number of samples of fireclay and magnesite stoppers and nozzles from different producers and consumers, as well as imported, were received for study. Chemical analyses of some of the nozzles and stoppers received are given in Table 63. Porosity test is given in Table 64. The reheat test was carried out at 1400°C. with a soaking of 5 hours.

Table 64 — Porosity Test of Stoppers and Nozzles

Sl. No.	Samples	Apparent porosity %
1.	Russian stopper	21·0
2.	B6	18·0
3.	B7	18·0
4.	Russian nozzle	17·0
5.	Mysore stopper	34·0
6.	Mysore nozzle	21·0
7.	BF N	12·0
8.	BF S	13·0
9.	Orissa nozzle	22·0
10.	Katni stopper	32·0

Results are given in Table 65. The dimensional measurements were taken on the stoppers and nozzles of Mysore Iron & Steel Ltd. The determination of other properties is in progress.

Table 65 — Results of Reheat Tests

Sl. No.	Samples	R.H. value
1.	Russian stopper 5/6 S (small)	+0·30
2.	1/6 Russian S (big)	+0·08
3.	5/6 S Russian stopper (big)	+0·15
4.	BN	+0·18
5.	BS	+0·25

68.0 A Comparative Study of the A.S.T.M. Thermal Conductivity and Blakeley & Cobb Apparatuses as Instruments for measuring the Thermal Conductivity of Refractories

In view of the need for a reliable apparatus for routine measurement of thermal conductivities both at the manufacturer's works as well as at the consumer's ends, it was considered worthwhile to make a study of both the A.S.T.M. and Blakeley & Cobb apparatuses and see how far the results obtained by them are comparable. Based

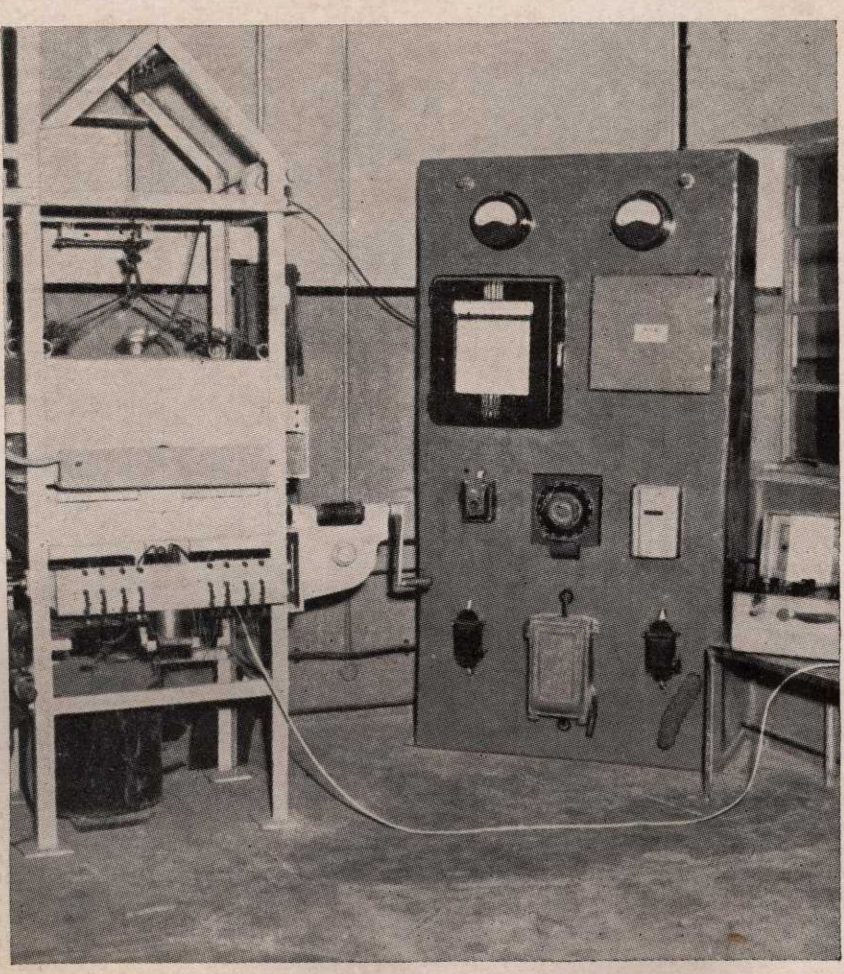


FIG. 46 — A.S.T.M. THERMAL CONDUCTIVITY APPARATUS, FABRICATED AT THE NATIONAL METALLURGICAL LABORATORY

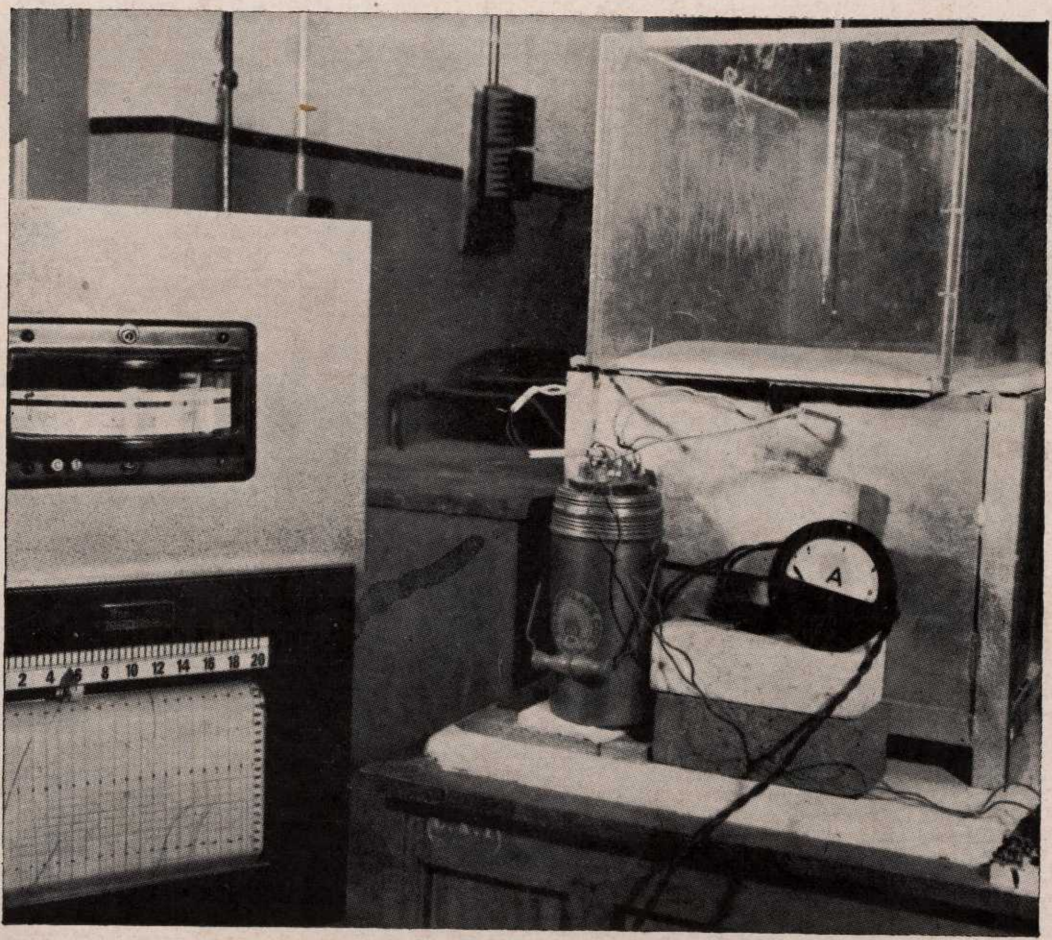


FIG. 47 — BLAKELEY & COBB THERMAL CONDUCTIVITY APPARATUS, FABRICATED AT THE NATIONAL METALLURGICAL LABORATORY

on this, recommendations could be made to Indian Standards Institution regarding the apparatus to be employed for routine thermal conductivity measurements.

Description of the Apparatus

(i) *A.S.T.M.*— This apparatus is based on the design and instructions prepared by M/s. Babcock Wilcox Company, New York (Fig. 46). Mainly, it consists of (1) a heating system composed of globar resistance elements that heats the top face of the sample, (2) a water system to maintain the head-of-water over the calorimeter at a constant level, (3) a calorimeter system adjacent to the bottom face of the sample in which water circulates at constant speed, and (4) a thermocouple system for determining temperatures of any desired locations. Here the amount of heat transmitted is being measured by the rise in temperature of the water flowing through the calorimeter under steady state conditions.

(ii) *Blakeley & Cobb Apparatus* — This apparatus is based on the original design by Blakeley & Cobb (Fig. 47). In this apparatus the specimen to be tested is kept over the heater and surrounded by asbestos packing and vermiculite insulation. The top face of the sample is covered by a blackened brass plate of 0·25 in. thickness. In the centre of this is embedded a circular disc of $1\frac{15}{16}$ in. dia. and $\frac{1}{4}$ in. thickness — the gap between them being rammed with asbestos fibre. Exposing this brass plate in the centre, the entire top face of the set up is covered by thin asbestos sheets. Finally a draught guard is provided which extends up to and encloses the thermometer bulb kept at a height of 14 in. from the blackened brass plate.

The heater temperature is controlled by a dimmerstat, ammeter and an 'on-off' pyrometric controller actuated by a chromel *vs* alumel thermocouple embedded in the centre of the hot face of the sample. Hot and cold face calorimeter temperatures are measured by means of a Honey Well Millivolt recorder and the room temperature is measured by thermometer reading up to 0·1°C.

Three sets of samples were investigated — one of them being a dense commercial sillimanite brick and the other two commercial insulation bricks. Physical properties and results of thermal conductivity tests are given in Tables 66 to 69.

Table 66 — Physical Properties of the Bricks

Specification	Apparent porosity %	Bulk density g./cc.	App. specific gravity	C.G.S. units permeability
Sillimanite	22·0	2·57	3·025	—
Unicast	77·0	0·64	2·720	0·41
Siliceous insulation	63·5	0·85	2·340	0·21

69.0 Determination of Physical Properties of IS : 6 and IS : 8 Firebricks

Samples of IS : 6 and IS : 8 quality firebricks from five sources selected by manufacturers are being examined with a view to suggest a basis for the revision of the relevant Indian Standards. The following properties were determined:

(i) Variations in dimensions.
(ii) Texture (qualitative).
(iii) Porosity, bulk density and apparent specific gravity.
(iv) Cold crushing strength (prism test).
(v) Modulus of rupture (small bars and full bricks).
(vi) Thermal spalling (prism test at 1000°C.).
(vii) Thermal expansion.
(viii) Solubility in hydroflouric acid.

The results are being analysed.

70.0 Development of Ramming Mixes for L.F. Induction Furnaces

This investigation was taken up with the object of developing suitable ramming mixes of high-alumina, siliceous and basic type for lining low-frequency induction furnaces as substitutes for imported mixes which are currently being used. During the period under review, a thorough examination of the physical properties of a foreign made high-alumina mix was made and several experimental mixes based on the indigenous raw materials, namely corrundum, alumina and refractory clays, were made. Physical properties of these mixes were determined. Trials are being made in the Laboratory furnaces with some of the best compositions from the experimental mixes.

71.0 Mineralogy of Indian Slags

A study of the constitution of metallurgical slags was taken up from the point of view of slag utilization and slag control in metallurgical practice.

In the earlier Report, studies on the mineralogy of blast furnace, open hearth and L.D. slags from Rourkela Steel Plant were reported. During the period under

Table 67 — Thermal Conductivity of the Sillimanite Brick at Various Temperatures

Hot face temp. °C.	A.S.T.M.				Blakeley & Cobb			
	Cold face temp.	Mean temp.	Mean thermal cond. (B.t.u.)	Spread of values	Cold face temp.	Mean temp.	Mean cond.	Spread of values
100	47	75	6·15	6·31- 5·99	63	82	4·92	5·30- 4·54
200	77	139	7·89	7·81- 7·98	120	190	7·06	7·06- 7·05
300	122	211	8·50	8·09- 8·92	190	245	8·50	8·70- 8·30
400	153	279	9·06	8·89- 9·22	237	318	9·13	8·94- 9·34
500	179	339	9·15	9·00- 9·29	280	390	9·31	8·83- 9·79
600	188	394	10·9	11·10-10·69	319	459	10·21	10·17-10·25

Table 68 — Thermal Conductivity of Unikast Brick

Hot face temp. °C.	A.S.T.M.				Blakeley & Cobb			
	Cold face temp.	Mean temp.	Thermal cond. (B.t.u.)	Spread of values	Cold face temp.	Mean temp.	Thermal cond. (B.t.u.)	Spread of values
100	29	65	1·34	1·41-1·26	39	70	1·25	1·28-1·21
200	37	120	1·52	1·41-1·62	60	130	1·36	1·37-1·34
300	41	171	1·50	1·55-1·46	79	189	1·47	1·48-1·45
400	45	223	1·70	1·76-1·64	96	247	1·76	1·74-1·78
500	52	277	2·01	1·97-2·06	117	308	1·95	1·93-1·97
600	65	333	2·47	2·31-2·63	145	373	2·04	2·03-2·04

Table 69 — Thermal Conductivity of Siliceous Insulation Brick

Hot face temp. °C.	A.S.T.M.				Blakeley & Cobb			
	Cold face temp.	Mean temp.	Thermal cond. (B.t.u.)	Spread of values	Cold face temp.	Mean temp.	Thermal cond. (B.t.u.)	Spread of values
100	30·0	65	1·03	1·09-0·97	46	73	1·37	1·13-1·60
200	46·0	123	1·45	1·53-1·36	76	138	1·80	1·82-1·78
300	51·0	175	2·48	2·30-2·65	108	204	2·03	1·95-2·12
400	62·0	231	2·56	2·57-2·54	140	270	2·55	2·43-2·67
500	69·5	285	2·68	2·67-2·69	166	333	2·55	2·58-2·54
600	83·0	343	2·76	2·72-2·81	201	400	2·78	2·63-2·93

review, slags from Tisco, Iisco, Durgapur and Bhadravati were received.

Mineralogical studies on Tisco slags by X-ray and petrographic method were completed. The blast furnace slags were predominantly composed of well-developed melilite crystals (Fig. 48). Other phases present were diopside, merwinite, oldhamite and tephroite. Occasionally spinel was also seen. Open hearth slags from Tisco were fully crystallized with colourless to yellowish brown radiating crystals closely interlocked with oxide phases. Nagelschmidtite, wustite and some tricalcium silicate were the predominant phases (Fig. 49). Other phases present in small amount were silico-carnotite and dicalcium ferrite.

The blast furnace slags from Iisco, also showed melilite as predominant phase. Other phases present were pyroxene (enstatite), monticellite, tephroite and oldhamite. The open hearth slag from Iisco were predominantly composed of oxide phase of wustite type (FeO, MnO, MgO), Beta-

FIG. 48 — POLISHED SECTION OF BLAST FURNACE SLAG FROM TISCO SHOWING LATH-SHAPED CRYSTALS OF MELILITE

FIG. 49 — POLISHED SECTION OF OPEN HEARTH SLAG FROM TISCO SHOWING THREE DIFFERENT PHASES — NAGELSCHMIDTITE, WUSTITE AND TRICALCIUM SILICATE

dicalcium siliate and some nagelschmidtite with small amount of dicalcium ferrite. These slags were poorly crystallized.

The blast furnace slags from Durgapur Steel Plant were well crystallized with melilite as the predominating phase. Other phases present were diopside, forsterite and some Mn-bearing phases. Glass was also present in considerable amount. The preliminary examination of Durgapur open-hearth slags indicates wustite and Beta-dicalcium silicate as the major phases. Further studies on other slags from Durgapur, Mysore Iron & Steel Works and Bhadravati are under progress.

72.0 Studies on Corrosion of Metals and Alloys

Atmospheric Corrosion

Studies on influence of atmospheric pollution, relative humidity, composition of materials and state of exposure on atmospheric corrosion of metals and alloys were continued both at Jamshedpur (industrial atmosphere) and Digha (marine atmosphere).

(i) *Monthly and Yearly Corrosion Rates of Mild Steel and Zinc* — Monthly atmospheric corrosion studies were continued on mild steel and zinc both at Jamshedpur and Digha. Role of atmospheric pollution and meteorological factors was studied at length. Corrosion rate of mild steel, along with the prevailing atmospheric conditions, is shown in Figs. 50 and 51.

The corrosivity of the atmosphere for all the months was found to be much higher at Digha when compared to that of Jamshedpur. The corrosion rate at Jamshedpur was dependent mainly on rain fall and to some extent on pollution, while at Digha the corrosion rate appeared to depend mainly on pollution. The maximum corrosion took place at Digha in those months when the pollution and average humidity conditions were high and there was light rain. Rainfall caused a marked decrease in salinity due to washing effect of rain and corresponding decrease in corrosion rate.

(ii) Long-term tests on corrosion resistance properties of copper, nickel, brass, zinc and monel metals are in progress. The above metals and alloys, including aluminium and aluminium alloys were also exposed at Marine Corrosion Research Station, Digha.

(iii) Further studies on properties of rust formed on mild steel and low-alloy steel, initiated in January 1961, were continued. The samples were taken out after an

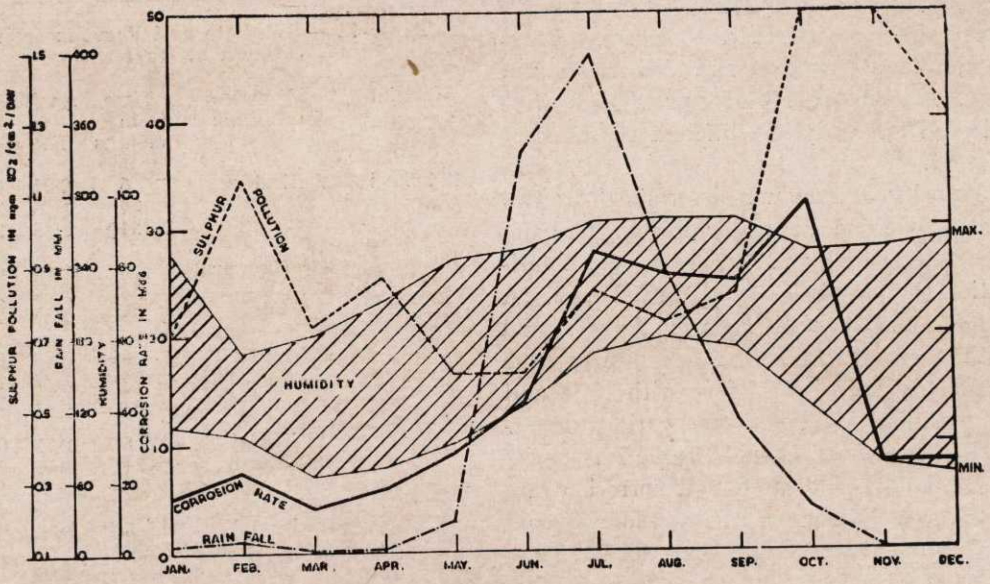


FIG. 50 — ATMOSPHERIC CORROSION DATA OF MILD STEEL AT JAMSHEDPUR

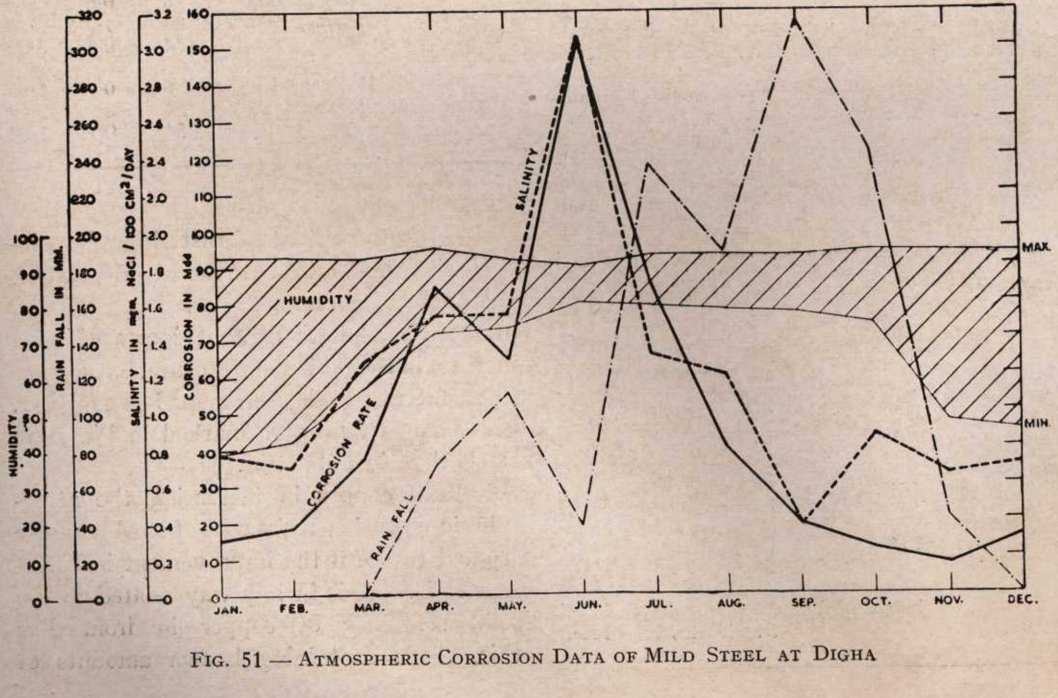


FIG. 51 — ATMOSPHERIC CORROSION DATA OF MILD STEEL AT DIGHA

exposure period of 4½ years, for studying the properties of rust, e.g. moisture, ferrous iron, sulphate content and also porosity of rust. Standard analytical procedures were adopted for analysing different constituents of rust.

Porosity of rust was determined by two methods — one was water drop method and the other was by measuring anodic polarization characteristics of the rusted steel. A regular and marked variation in potential with current was observed indicating the change in porosity of rust with time and composition of steel. The various observations made are shown in Table 70.

The studies indicated that corrosion rate decreased with time, as the rust films tended to be protective, specially more for low-alloy steels than mild steel. The effect of angle of exposure showed that samples exposed horizontally corroded most, while those exposed vertically the least. Rust collected from different samples was analysed for ferrous iron and sulphate content and the results are given in Table 71. Ferrous and sulphate content of rust showed variation with time, and was found to depend on the angle of the exposure and composition of steel. Sulphate content increased with increase in the period of exposure, but ferrous iron content remained more or less constant. This change was more marked in low-alloy steels than in mild steel.

Table 70 — Change in Potential of Rusted Sample with Applied Anodic Current

Material	Exposure angle	Exposure period in months	Weight of rust in g./dm.	Change in potential per 100 microamp.
Mild Steel	Vertical	24	1·55	70
		54	1·73	68
	45°	24	2·15	80
		54	2·28	93
	Horizontal	24	2·59	92
		54	3·52	115
Tisco	Vertical	24	1·86	88
		54	1·90	160
	45°	24	1·99	92
		54	1·86	96
	Horizontal	24	2·40	112·5
		54	2·78	137·5

Table 71 — Sulphate and Ferrous Iron Content of Rust

Material	Exposure condition	Time in months	Analysis % Fe^{+}	Analysis % SO_4^{-}
Mild Steel	Vertical	6	0·92	0·93
		24	1·33	2·01
		54	1·33	2·13
	45° (skyward)	6	1·14	0·69
		24	2·04	2·34
		54	1·80	1·80
	45° (groundward)	6	0·95	0·89
		24	1·17	1·93
		54	1·74	1·58
	Horizontal (skyward)	6	1·11	0·52
		24	2·66	2·18
		54	1·30	1·31
	Horizontal (groundward)	6	1·09	0·72
		24	0·87	1·75
		54	0·60	1·10
Low-alloy Steel	Vertical	6	0·39	1·00
		24	0·18	2·01
		54	0·81	1·86
	45° (skyward)	6	0·84	0·98
		24	0·11	1·85
		54	1·42	1·69
	45° (groundward)	6	1·06	1·17
		24	1·36	2·10
		54	0·63	1·39
	Horizontal (skyward)	6	0·61	0·84
		24	1·36	2·10
		54	0·63	3·09
	Horizontal (groundward)	6	0·78	1·00
		24	0·96	2·19
		54	0·15	1·88

Role of copper in increasing the atmospheric corrosion resistance of steel was also studied to see if the improvement in corrosion resistance is in any way related to the solid solubility of copper in iron. For this, steels containing known amounts of copper were exposed and the atmospheric corrosion rates were studied after taking out the sample at the end of 3 months. The results are given in Table 72. Even though it is difficult to arrive at any definite conclusion based on the studies of small duration, these short term tests have indicated that the amount of copper retained in solution may have an effect in improving the corrosion resistance. The tests are, therefore, being continued to find out the beneficial effect of copper during long term exposure tests.

(iv) Continuous recording of daily changes in atmospheric temperature and relative humidities are being done at both Jamshedpur and Digha. Data on rain fall and dew collection are also being kept. Sulphur pollution of the atmosphere at Jamshedpur was determined every month by exposing lead peroxide candle. The chloride pollution at Digha, on the other hand, was determined by exposing wet candles.

(v) *Atmospheric Corrosion of Metals during Day Time and Night Time* — To study the effect of variations in meteorological conditions, e.g. temperature, humidity, condensation, etc., during day and night time on atmospheric corrosion of metals, samples of mild steel and zinc were exposed to the atmosphere during day and night time. One set of samples was exposed to the atmosphere only between 6·00 a.m. to 6·00 p.m. of the day, and another set between 6·00 p.m. and 6·00 a.m. The results obtained are given in Table 73. The data obtained showed that corrosion rate was higher during night time, mainly because of higher humidities and condensation during night. Comparison of day and night exposure with continous exposure clearly showed that corrosion due to continuous exposure was much less compared to the total weight loss of day and night exposure. Detailed studies in this connection are under progress.

(vi) *Effect of Distance from Sea Coast on Corrosion Rate* — Corrosion rate is considerably affected by the proximity of the test site to sea coast. The difference in corrosion rate between the exposure site (50 ft) from sea, and the laboratory premises (520 ft) will be apparent from the data given in Table 74.

From the results it appeared that the effect of distance was more marked for mild steel than zinc. It is reasonable to assume that higher corrosion rates are due to higher salt content of the atmosphere near coast. Further studies on nature of the corrosion, with respect to the distance from sea coast, are under progress.

Table 72 — Weight Loss of Copper Containing Steel after Various Treatments

Sample No.	Copper content %	Treatment	Weight loss in g./dm.
MS	—	Quenched	1·298
B	0·05	Quenched	1·009
		200°C. for 72 hr	1·106
C	0·25	Quenched	0·993
		200°C. for 24 hr	1·004
		200°C. for 72 hr	1·108
		300°C. for 24 hr	1·188

Table 73 — Effect of Day and Night Exposure on Corrosion of Mild Steel and Zinc

Material	Month of exposure	Corrosion rate in m.d.d.		Continuous exposure
		Day	Night	
Mild Steel	February, 65	3·367	15·16	15·500
Mild Steel	March, 65	15·460	25·78	22·060
Mild Steel	April, 65	17·240	33·95	30·230
Mild Steel	May, 65	27·880	44·47	59·440
Zinc	February, 65	0·027	1·26	1·237
Zinc	March, 65	1·900	1·82	2·616
Zinc	April, 65	5·240	5·31	9·573
Zinc	May, 65	10·150	13·77	10·500

Table 74 — Effect of Proximity to Sea on Corrosion Rate

Material	Date of exposure	Period of exposure	Corrosion rate in m.d.d.	
			50 ft	500 ft
Mild Steel	1st May, 1965	1 month	178·14	75·03
Mild Steel	1st June, 1965	1 month	217·65	73·16
Zinc	1st May, 1965	1 month	16·53	11·30
Zinc	1st June, 1965	1 month	17·32	15·80

(vii) *Studies on Chromate Passivation of Aluminium and Copper*— Studies on chromate passivation of aluminium and copper were continued. In addition to the tests conducted earlier on treated sample, the effect of temperature increase on chromate-treated samples was studied. The studies indicated that heating of the films at temperature of 50°, 100° and 200°C. for 1 hour deteriorated the protective properties of the chromate films, as confirmed by salt spray test.

72.1 Fuel Ash Corrosion and its Prevention

The extensive use of low-grade coals and oils in power plants and aviation has increased the importance of study of fuel ash corrosion in recent years. The problem was taken up to investigate the mechanism of fuel ash corrosion and to reduce this type of attack by (i) developing new alloy compositions, and (ii) giving suitable protective coating over the alloy-surfaces and by adding neutralizing additives to the fuels.

The active agents for the fuel ash corrosion are vanadium salts, sodium sulphate, sodium chloride, etc. As a first step, the corrosion resistance of high-alloy steels was evaluated in mixtures containing various amounts of V_2O_5 and Na_2SO_4. An apparatus set up for these studies based on that proposed by de S Brasanas and Grant comprising a furnace, a gas reservoir and a movable head was used for measuring the amount of oxygen absorbed at definite intervals up to 5 hours.

The studies on the corrosion resistance properties of 18/8 stainless steel, nickel-free stainless steel developed by the NML, and inconel were continued at different temperatures using different ratios of V_2O_5 and Na_2SO_4. A series of tests were completed which gave some interesting data as regards the mechanism of attack and the resisting capacity of the alloys.

It has been reported that corrosion first increases with the addition of Na_2SO_4 to V_2O_5 reaching maximum at 10 per cent Na_2SO_4 and then decreases with further increase in the Na_2SO_4 content of the mixture. In the present studies this maximum was observed at 820°C. in case of 18/8 Cr/Ni and nickel-free stainless steels. No such increase in corrosion rate with Na_2SO_4 addition was observed at 870° and 950°C. The maximum attack with the mixture containing 10 per cent Na_2SO_4 was also noted in the case of inconel at 820° and 870°C. These studies show that temperature also plays an important role on the effect of variation of Na_2SO_4 content on corrosion rate. With increase in temperature the corrosion rate of 18/8 steel and inconel increased. The total attack and the rate of attack was very small up to 780°C. but increased rapidly with temperature above 800°C. At still higher temperatures the rate fell off as not much difference in corrosion rate between those at 870° and 950°C. was observed. The attack on nickel-free stainless steel, on the other hand, showed a maximum with increase in temperature, the value depending on the mixture composition.

Another interesting observation was the sudden increase in the rate of attack at at 820°C. of 18/8 steel by the mixture containing 10 per cent Na_2SO_4 after $1\frac{1}{2}$ hours when the salt mixture was placed in the boat along with the specimen and then pushed into the heating zone after the temperature had reached a steady state. Experiments are in progress to explain this behaviour.

72.2 Stress Corrosion Cracking of Homogeneous Alloys

The problem of stress corrosion cracking of homogeneous alloys was undertaken with a view to throw some light on the fundamentals of the process in the context of the various existing theories. A number of copper alloys, viz. α-brass, Cu-Mn, Cu-Sn and Cu-Al are under investigation.

Brass and Cu-Mn alloys cracked readily in a solution containing Cu in the form of $CuSO_4,5H_2O$ and NH_3 as $(NH_4)_2SO_4$. Cu-Sn and Cu-Al alloys did not show any initiation of cracking in a total experimental period of 3 days, but they did fail in the ammonia atmosphere. All these experiments had been carried out using wire-specimens under tensile loads applied by means of a lever. Effect of increasing the Mn content results in changing the cracking mode from intergranular to transgranular in the vicinity of 16 per cent (atomic) Mn. This also corresponds to the minima in the cracking time *vs* per cent Mn plot. Annealed Cu-Sn and Cu-Al alloys show transgranular mode of cracking.

Effect of cold work and prior strain on the stress corrosion behaviour of brass and Cu-Mn alloys was studied. Increase in susceptibility to cracking with an increase in the cold work, attainment of a maximum and then decrease in the susceptibility were observed in case of brass and Cu-Mn alloy of 17·6 per cent Mn content. They were in the vicinity of 28 and 18 per cent deformation, respectively. Cu-Mn alloys of lower Mn content had shown an increase in cracking time with increasing deformation. Maximum susceptibility of brass was associated with an inter- to transgranular transition of cracking. Prior straining was seen to induce resistance of some order to stress-corrosion of brass in the region of 5-10 per cent strain. Cu-Mn alloys were apparently without any effect; a 25 per cent Mn alloy behaved somewhat opposite to brass thus opposing Forty's hypothesis for effect of prior strain on alloys showing transgranular failure. Effect of solution *p*H was studied. Cu-Mn alloys of different Mn content gave maximum susceptibility in the different *p*H ranges. Effect of grain size on the stress corrosion behaviour is under study. Electrochemical studies and studies with thin films constitute further work in this line.

72.3 Oxidation of Copper and Copper Alloys

This investigation was undertaken with a view to measure the oxidation-kinetics of copper and copper alloys, and thereby to develop the oxidation-resistance characteristics of copper and copper alloys.

The apparatus for the studies of oxidation rates of copper and copper alloys by adsorption method was designed and fabricated in the Laboratory. A preliminary experiment was carried out for standardization of this apparatus by taking an annealed and commercially pure copper wire specimen of 0·1 cm. dia. and 15 cm. length. The rate of oxidation was measured at a constant temperature and atmospheric pressure by measuring the amount of commercial oxygen consumed at different time intervals. It was observed that the results obtained were in agreement with the parabolic-rate law of oxidation. Further work is under way to design and set up an apparatus for the analysis of thin films on copper and copper alloys by Coulometric Method.

72.4 Anodic Polarization and Passivation of Metals and Alloys

This project was taken up with a view to determine the critical current density of metals and alloys for imparting passivity. The determination of critical current density of 18/8 stainless steel and 8·01 per cent chromium-iron, solution-treated at 1000° and 950°C., respectively, and tempered at different temperatures in argon gas atmosphere, was carried out in 1·28 N sulphuric acid and 3 per cent sodium sulphate solution, respectively.

The specimens, tempered at different temperatures varying from 300° to 800°C.

after solution treatment, were pickled and washed with deaerated distilled water. The cell was then filled with deaerated electrolyte (3 per cent sodium sulphate solution for chromium iron and 1·28 NH_2SO_4 for 18/8 stainless steel) without bringing the sample in contact with air, and potential with respect to calomel electrode was measured after passing different current densities through the specimen and a platinum electrode which was acting as cathode. The change of potential with time was noted till the potential came to a constant value. The graphs were plotted I *vs* 1/t according to the equation $I-I_c=K$ where I is applied current density, I_c critical current density, t is the time and K represents coulamb/cm.^{2} to achieve the passive stage. Deaeration of solution and distilled water was carried out passing the pure nitrogen 6 to 8 hours. The measurement of critical current density for 8·01 per cent chromium iron and 18/8 stainless steel is under progress.

For chromium-iron it was observed that critical current density for passivity increased with increase of tempering temperature from 400° to 800°C.

73.0 Gases in Metals

Samples of ferrous and non-ferrous metals and alloys were received from different research projects in the Laboratory for the determination of gases in them by vacuum fusion method and by non-reactive carrier gas method. Samples were also received from different firms, laboratories and institutes for the determination of gases in metals and alloys as a regular routine work. The vacuum fusion apparatus designed and fabricated is providing satisfactory and reproducible results. The new electrochemical method of determination of nitrogen in solid solution in steel developed in NML which has been reported earlier is being applied to various steels and alloys. The method was found dependable and reproducible results were obtained. Analytical data are given in Table 75.

Table 75 — Estimation of Nitrogen in Solid Solution

Sample	N_2 in solid soln. by electrolytic method %	Total nitrogen by vacuum fusion method	Nitride nitrogen by electrolytic method	N_2 in solid soln. by difference of total N_2 and nitride N_2
Nitrided steel (0·85% Mn)	0·0080	0·036	0·0270	0·0090
Nitrided steel (0·90% Mn)	0·0070	0·029	0·0220	0·0070
Nitrided steel (1·1% Mn)	0·0084	0·038	0·0288	0·0092
Low-alloy steel (0·62% Cr)	0·0020	0·013	0·0110	0·0020
Medium manganese rail steel	0·0022	0·009	0·0066	0·0024

73.1 Nature of Hydrogen in Steel

To know the actual mechanism of hydrogen embrittlement, the knowledge of the state of hydrogen in which it exists in steel is of utmost importance. According to some authors, hydrogen which is dissolved in the metal, affects ductile properties of steel, while others hold the view that this effect is produced by hydrogen which is transformed during plastic deformation from dissolved to the molecular state. Some are also of the opinion that hydrogen is present in iron alloys in the form of negatively or positively charged ions which are also responsible for lowering the ductility. Yavoiskii and Batalin observed that when a direct current is passed through molten steel, an enrichment in hydrogen content occurs at the cathode. Yavoiskii and Chernega studied the migration of hydrogen in different steels in solid state under the influence of an electric field at elevated temperature. They found some enrichment of hydrogen at the cathode, but they could not reproduce the result. V. I. Lokomskii showed in his experiments that hydrogen was present in iron alloys in the form of positively and negatively charged ions. Hydrogen values obtained by him at the anode and cathode ends after direct current did not differ appreciably for taking any conclusion. In order to make a systematic study to find out whether hydrogen could remain as positive or negative ions and flow under the influence of direct current, the following experiment was conducted.

About a metre long M.S. wire of 3 mm. dia. was normalized and the surface was cleaned by emery paper (00). The wire was charged cathodically in a 10 per cent sulphuric acid-bath at a current density of 0·1 amp./cm.^{2} for 1½ hr. Two grams of sodium sulphide were added per litre of the solution as a promoter of hydrogen pick-up. After saturation with hydrogen the wire was kept at room temperature for 48 hours to allow sufficient time to diffuse hydrogen from the surface. After this, the loss of hydrogen became negligible. Two pieces from both the ends were cut to find the initial hydrogen at two ends. A direct current of 15 amperes was passed through this wire for 4 hours. During the passage of direct current the sample got heated causing some hydrogen to diffuse out of the sample To minimize this loss the wire was kept under water while the current was passed. Test pieces were cut and analysed from anode, cathode and from the middle portion for hydrogen in the vacuum heating hydrogen determination apparatus. Results are given in Table 76.

From the experimental data it is evident that in all cases an increase in the concentration of hydrogen takes place at the cathode. This increase, no doubt, is due to the migration of proton towards the cathode. But there is no clear indication of a corresponding increase of hydrogen in the anode end. Further work is in progress.

74.0 Spectrographic, Polarographic and other Analytical Work

(i) *Spectrographic Analysis of Copper-beryllium Alloys*

Work was taken up to develop a suitable spectrographic method of analysis of beryllium, cobalt and nickel in beryllium copper alloy.

Table 76 — Migration of Hydrogen in Solid M.S. Wire Under the Flow of Current

Period of passage of D.C. hr	H_2 content in region of the cathode (cc. per 100 g.)			H_2 content of the middle portion of the wire after passing D.C. (cc. per 100 g.)	H_2 content in region of the cathode (cc. per 100 g.)		
	Before D.C. was passed	After D.C. was passed	Change in H_2 content		Before D.C. was passed	After D.C. was passed	Change in H_2 content
4	7·44	6·04	−1·40	6·15	7·39	9·85	+2·46
4	4·71	4·62	−0·09	3·60	3·35	4·22	+0·87
4	1·54	1·74	+0·20	—	1·31	1·61	+0·30
4	1·56	3·13	+1·57	1·30	1·83	3·24	+1·41
4	12·40	10·59	−1·81	13·14	12·85	13·48	+0·63
4	11·47	13·44	+1·97	11·20	10·78	12·47	+1·69
4	18·49	20·28	+1·79	18·21	18·00	26·07	+8·07
4	5·70	4·49	−1·21	4·46	5·28	5·56	+0·28
4	7·67	8·02	+0·35	8·71	8·07	9·73	+1·66

Copper-1·8 per cent beryllium, with a small quantity of nickel or cobalt, and copper with 2·6 per cent cobalt and 0·4 per cent beryllium have assumed industrial importance. Accurate estimation of the alloying elements like beryllium, cobalt and nickel in this alloy is important. Wet chemical method for determination of these elements is time-consuming, whereas spectrographic method can be very well adopted for simultaneous determination, accuracy and speed. Hilger medium quartz spectrograph was employed and excitation was obtained by high-voltage condensed spark. Synthetic standards were prepared for the working curves for Be, Cu and Ni. Samples also were taken into solutions. Few drops of the solution were dried on spec. pure graphite electrode 10 mm. dia. which served as lower electrode. The upper electrode was a pointed carbon rod. A number of experiments were conducted under various conditions of exposure, excitation source, etc., and the results obtained were encouraging. Further experiments are being carried out to determine the reproducibility and accuracy of the method.

(ii) *Determination of Cr_2O_3 and V_2O_5 in Ilmenite*

Indian ilmenite contains minute amounts of Cr_2O_3 and V_2O_5 which are difficult to determine accurately by conventional methods. Accordingly, the following method was developed for rapid determination of Cr_2O_3 and V_2O_5 in ilmenite. The results obtained by this method compared favourably to those obtained by other methods.

Two grams of finely ground sample was fused with 10 g. of sodium peroxide in an iron crucible till decomposition was completed. The crucible was cooled and placed in a 400 ml. beaker covered with an watch glass. About 200 ml. of hot water was then added. When the vigorous action ceased, the beaker was heated to boiling and boiled for 5 minutes. The crucible was then taken out from the beaker rinsing with a small amount of hot water. At this point a pink or green colour may form due to manganese. A pinch of sodium peroxide was added to destroy the colour and the solution again boiled for five minutes to destroy the peroxide. After cooling, the solution along with the precipitate was transferred to a 250 ml. measuring flask, diluted to the mark, mixed thoroughly, and allowed to settle. The supernatant liquid was then filtered through a retentive paper previously washed with 4 per cent NaOH solution, rejecting first portion of the filtrate.

For V_2O_5 the yellow colour of phospho-vanado-tungstic acid was measured in the spectrophotometer as follows:

Two 50 ml. aliquots of the filtrate were taken in two beakers and acidified to litmus with H_2SO_4 (1 : 8), adding 1 ml. excess. Then they were transferred to two 100 ml. measuring flasks. To one flask 0·3 ml. phosphoric acid and 2 ml. of filtered 5 per cent aqueous sodium tungstate was added, diluted to volume and mixed. The absorption was then measured at 400 mμ. To measure the absorption due to background colour, 0·3 ml. phosphoric acid was added to the other flask, diluted to volume and mixed. The absorption was then measured at 400 mμ and deducted from the previous figures.

For Cr_2O_3 the absorption due to the yellow colour of the chromate ion in the original alkaline filtrate was measured directly at 370 mμ in the spectrophotometer. The results are given in Table 77.

(iii) *Determination of Calcium in the Presence of Large Excess of Magnesium*

This investigation was taken up for developing rapid method of analysis of calcium in magnesite. The procedure developed consists of making magnesium by tartaric acid in strongly alkaline medium in the presence of small amount of hydroxylamine hydrochloride, estimating the calcium after

Table 77 — Analysis of Ilmenites

Sample No.	TiO_2 %	FeO %	Fe_2O_3 %	Cr_2O_3 %	V_2O_5 %	SiO_2 %	Al_2O_3 %	MgO %	CaO %
1	48·92	32·91	14·32	0·072	0·21	0·48	1·10	1·00	0·39
2	52·50	9·57	26·04	0·15	0·23	—	—	—	—
3	52·92	26·49	18·33	0·087	0·18	0·98	0·73	0·14	Trace
4	52·15	26·77	17·11	0·084	0·17	—	—	—	—
5	50·06	29·79	18·87	0·096	0·21	1·24	—	—	—
6	53·96	25·23	17·59	0·09	0·04	1·23	1·47	Trace	Trace

precipitating it with oxalate solution. The oxalate so precipitated was estimated by N/10 $KMnO_4$ solution. The masking of magnesium under different conditions was studied. It was found that 0·5 g. of tartaric acid followed by 0·5 to 1 g. of hydroxylamine hydrochloride at *p*H 11-12 was sufficient for masking 50 mg. of magnesium. Some of the findings are given in Table 78.

(iv) *Analysis of Ore, Minerals, Metals, Alloys and Other Products, Employing X-ray Fluorescence*

X-ray fluorescence analysis was adopted to expedite analyses of routine tests of research divisions of National Metallurgical Laboratory as well as for specialized analytical problems. The equipment used for this purpose was the Automatic X-ray

Table 78 — Results of Chemical Analysis

Calcium taken mg.	Magnesium taken mg.	Ratio	Calcium found	Method used	Number of pptn
10·0	100	1 : 10	9·96 10·00	Oxalate	Double pptn
10·0	200	1 : 20	9·92 10·00	do	Triple pptn
10·0	300	1 : 30	10·04 9·96	do	do
10·0	400	1 : 40	9·68 9·80	do	do
10·0	500	1 : 50	9·88 10·00	do	Quarterly pptn
10·0	100	1 : 10	9·80 10·00	Tartaric acid	Single pptn
10·0	200	1 : 20	9·56 10·00	do	do
10·0	250	1 : 25	9·41 9·60	do	do
10·0	400	1 : 40	9·41 9·50	do	Double pptn

Calcium in Almorah Magnesite Determined by Different Methods

Calcium determined %	Methods used
1·13	Tartaric acid method
1·16	Sulphate method
4·15	Tartaric acid method
4·20	Sulphate method

Fluorescence Spectrograph 'Autrometer'. The analyses obtained with the instrument were highly satisfactory both from the point of view of the rapidity of the process and its accuracy which was found to be as good as or better than analyses carried out using conventional analytical techniques. With the equipment available, it was possible to analyse all the elements ranging from magnesium (At. No. 12) and upwards in the periodic table. Calibration curves were prepared using some of the test products of known chemical analysis. In some cases it was necessary to make synthetic standards because products of known chemical analyses were not available for comparison purposes.

Calibration curves and synthetic standards are being prepared for estimation of trace amounts of Ge and Ga in flue dusts and fly ash samples, Mg and Al analyses in Mg-Al alloys, and P_2O_5 in apatite.

(v) *Differential Thermal Analysis*

A comprehensive investigation was taken up on the qualitative and quantitative estimation of mineralogical constituents of the Indian iron ores by a four-channel automatic differential thermal analysis apparatus 'Deltatherm'. Thermograms of clay and bentonite samples from various localities in the country were prepared. By high-intensity magnetic separations and water elutriation it had been possible to prepare high-purity concentrates of some of the iron ore constituents. These will be subsequently used for preparing D.T.A. curves for quantitative estimations.

75.0 Preparation of Plating Salts

The electroplating industry in India currently employs imported plating salts, polishing compositions and plating equipment. Due to strict import restrictions, these proprietory plating salts are not available in the market and the 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 was also investigated from indigenous sources.

The common electroplating salts mostly used by light engineering industries for decorative and protective finish have been formulated using indigenous raw materials and chemicals made in India. In arriving at the composition of the plating salts, due consideration was given to the chemical and electrochemical characteristics of the plating electrolytes derived from the salt such as conductivity, cathodic and anodic polarizations, throwing power, current efficiency, etc., as well as to the physical and chemical properties of the plate obtained from these, such as decorative value, reflectivity, colour, hardness, abrasion resistance, corrosion resistance, etc. Thus, the composition of alkaline copper plating salt, dull nickel plating salt, alkaline cadmium plating were established and standardized and their performance were tested and compared in continuous production plating practice. All these formulated salts have proved satisfactory in performance as reported by consumers in light engineering industry. In the formulation of dull nickel plating solutions, replacing $NiCl_2$ by NaCl, the performance of cast nickel anode was found better than the costly depolarized nickel anodes. Thereby, it entails less capital cost towards plating salt and nickel anodes. It is, therefore, advocated for commercial plating practice.

The process for bright nickel plating which requires no buffing after plating had been established with organic reagents containing a suitable brightener, viz. naphthalene sulphuric acid, a leveller like 'Coumarin' and an anti-stress reagent 'Saccharine' together with a sodium salt of weak organic acid. The reagents were added in minute traces in right proportions in nickel plating solution and bright and mirror finish of nickel under the rigid control of *p*H and temperature was obtained on the properly polished and degreased cathode surface. The suitability of such bright platings on production practice was tried on continuous plating performance on much bigger scale in plating vats in light engineering industries.

Behaviour and performance of different types of Nickel anodes, viz. cast, rolled, electrolytic and depolarized, have been investigated in nickel plating solution. The controlled amount of impurities such as iron, manganese, copper, sulphur, phosphorus, carbon, silicon and oxygen, present in nickel anodes has been found to affect overall reactivity of nickel or improved the performance in the plating solution. It is interesting to note that oxygen and sulphur in nickel anodes have significant role in the depolarization of nickel and increases the reactivity of nickel and inhibit passivity.

A process for crack-free bright chromium plating from self-regulating high-speed bath has been established. The ordinary conventional chromic acid-sulphuric acid-bath for chromium plating does not throw well and has a low current efficiency. Self-regulating high-speed chromium plating bath as developed contains, besides chromic acid, $SrSO_4$ and K_2SiF_4 in a suitable proportion. A certain portion of chromic acid in trivalent state in the plating solution by reduction with tartaric acid is to be maintained to get bright chromium deposits free from cracks from the said bath. Systematic studies to ascertain the conditions for the desired objectives have been completed and the process is under trial for its commercial feasibility.

75.1 Imparting Black Colour to 'Bidri' Wares

At the instance of All-India Handicraft Board, New Delhi, a process was developed for imparting black colour by chemical immersion process to zinc wares containing 4 per cent Cu and having the composition similar to 'Bidri' wares. The colour at present is usually imparted by an indigenous way and in vogue for long with a particular type of clay, well known to the craftsmen, and available in and around 'Bidar', a small district town in Mysore state.

The chemical substitute developed by NML contains a mixture of a few common chemicals, viz. $NiSO_4,7H_2O$, NaCNS, $ZnSO_4$ and $(NH_4)SO_4$. The solution imparts a black colour to zinc surface when immersed in the solution after a certain duration of immersion. All-India Handicraft Board had been furnished with the full details of the process and a craftsman from this industry had also been trained in the process to utilize the same in place of the conventional method.

76.0 Treatment of Zinc Wastes

During galvanizing considerable quantities of waste products are obtained in the form of zinc dross, ashes, skimmings and blowings which do not directly find any application. Generally in galvanizing of tubes about 25 per cent zinc is lost as zinc dross, about 20 per cent is lost as blowings, about 12 per cent is lost as skimmings, and 16 per cent as ashes. The investigation aims at the recovery of the metallic values from the various products for further use in the galvanizing plant.

(i) *Zinc Dross*

Distillation studies were carried out in the 6 in. dia. mild steel vacuum pot at different temperatures for various periods with a charge of 6 kg. of zinc dross per batch. The results are shown in Table 79.

The results indicated that the rate of distillation of zinc increased with increasing temperature. A maximum zinc recovery of 98·3 per cent was attained by distillation of the dross either for 4 hours at 600°C. or for 3 hours at 650°C. It is seen from Table 79 that zinc recovery increases with increasing distillation temperature and period.

Based on the laboratory scale experiments, a semi-industrial vacuum distillation unit, comprising a mild steel retort and dust trap, was designed and fabricated at National Metallurgical Laboratory. The cylindrical portion of the shell was made of a 10 mm. thick plate. The bottom plate was 25 mm. thick. The shell was reinforced with channels and strips welded on the outside of the shell to provide strength and avoid buckling of the plate at the operating temperature and pressure. The pot was 48 in. deep and 24 in. in dia. A water-cooled ribbed type condenser, 24 in. long, was fabricated. A dust trap was provided in the vacuum line to eliminate the possibility of any fine dust entering the oil-sealed rotary pump. A thermo-couple well was provided from the top of the condenser to measure the charge temperature. The furnace was constructed of fire brick and arrangement was made to heat the pot from the bottom.

Table 79 — Results of Distillation Tests at Different Temperatures for Various Periods with a Charge of 6 kg. Zinc Dross

Expt. No.	Temperature of distillation	Duration of distillation	Metallic zinc recovery %	Remarks
1	550	3	77·6	
2	550	4	87·9	
3	600	2	77·6	
4	600	3	86·2	
5	600	4	98·3	
6	650	1	51·7	
7	650	2	86·2	Radiation baffle was used
8	650	2	86·2	
9	650	3	98·3	
10	650	4	98·3	

A few experiments were carried out by distilling 300 kg. of charge per batch for 5 to 9 hours between 500° to 550°C. at a pressure ranging between 0·3 to 0·5 mm. of mercury. A maximum of 192 kg. of distilled zinc was collected on distillation of the dross at 550°C. for 9 hours.

Further work is in progress to investigate the effect of different variable factors and improved recoveries as obtained on a 6 kg. batch.

(ii) *Zinc Blowings*

Melting of zinc blowings by addition into a molten pool of zinc was not successful and further melting trials were carried out with flux addition. Six kilograms of blowings were mixed with 5 per cent by weight of flux containing different proportions of ammonium chloride and zinc chloride. The mixed charge was heated to a temperature slightly above the melting point of zinc with vigorous stirring from 2-3 minutes. On stirring the zinc particles were found to coalesce and collect at the bottom in the molten pool. The results of experiments carried out with and without flux addition are shown in Table 80.

It was observed that either NH_4Cl or $ZnCl_2$ or a blend containing both can be used effectively as flux for melting zinc blowings, and above 80 per cent of zinc could be recovered irrespective of the flux composition. In view of the hygroscopic nature of zinc chloride and its relatively high cost, subsequent trials were made with ammonium chloride alone as flux. Six kilograms of blowings were mixed thoroughly with different amounts of ammonium chloride and melted. The results are shown in Table 81.

Table 80 — Recovery of Zinc from Zinc Blowings by Melting under a Flux

Expt. No.	Flux composition		Zinc recovery %
	$ZnCl_2$ %	NH_4Cl %	
7	0	0	45
8	0	100	78
9	10	90	84
10	20	80	84
11	30	70	81
12	40	60	83
13	50	50	78
14	60	40	79
15	70	30	81
16	80	20	83
17	90	10	78
18	100	0	78

Table 81 — Effect of increasing Flux Additions on Recovery of Zinc

Expt. No.	Wt of NH_4Cl %	Zinc recovery %
19	0	45
20	2	55
21	5	78
22	8	79
23	10	83
24	12	92
25	15	94
26	20	94

Table 82 — Zinc Blowings Melting Trials Without Flux and with Varied Amounts of NH_4Cl

	Wt of NH_4Cl in the mix. %	Metallic zinc recovery %
+30 mesh	nil	88.0
	5	90.0
	10	90.0
	12	90.0
	15	90.0
−30 +170 mesh	nil	53·5
	5	78.0
	10	86.0
	12	86.0
	15	88.0
	20	88.0
−170 mesh	nil	0.0
	7	80·0
	10	85·4
	12	86·3
	15	90·0
	20	90·0

It was observed that zinc recovery improved with increasing flux additions up to 12 per cent. Beyond 12 per cent the recoveries increase appreciably and the operation also becomes easy as a molten slag which floats on the top of the molten metal, is formed. Vigorous stirring is also not required as the charge is in a fluid condition. The product obtained after melting the blowings with flux contained Fe, 0·28; Pb, 0·31 and Cd, 0·04 per cent as impurities.

Zinc blowings were divided into three fractions, viz. +30, −30 +170 and −170 mesh B.S.S., and subjected to melting trials by stirring with and without flux addition for evaluation of flux requirements (Table 82).

It was observed that increased additions of NH_4Cl did not have much effect on zinc recovery on coarser fractions of zinc blowings and coarser +30 mesh fractions can be melted without any flux. It was, however, not possible to recover zinc from −170 mesh fractions without flux addition.

77.0 Utilization of Basic Slag as Soil Conditioner

Work on utilization of basic slag as soil conditioner was taken up with the objective of determining its suitability as a cheap liming material for agricultural purposes. Basic slag was found to give encouraging results being a source of micro-nutrients such as boron, manganese, molybdenum, copper, zinc, cobalt, tungsten, titanium, etc., besides P_2O_5.

During the period under review, field trials were conducted with slag samples from Bhilai blast furnace, Bhilai open-hearth, Rourkela open-hearth, Rourkela L.D. and NML Low-shaft Furnace crushed to −100 mesh along with standard liming material, i.e. calcite and dolomite, for use as solid conditioner in agricultural crop such as groundnut, maize and gram at the Agricultural Research Institute, Ranchi. The comparative statement of liming effect

Table 83 — Comparison of Basic Slags with Standard Liming Material (As Lime Carrier)

In term of yield data, md./acre

Lime carrier	Composition %		Wheat		Ground-nut 65-66	Maize 65-66	Gram 65-66
	CaO	P	64-65	65-66			
Without lime carrier	—	—	20·4	Threshing not completed	24·0	23·4	7·7
Lime (calcitic or dolomitic)	33·0	—	18·9	—	28·5	29·2	17·1
Bhilai blast furnace	29·7	1·59	24·0	—	27·7	28·5	21·0
Bhilai open hearth	37·0	Trace	21·0	—	31·6	24·1	15·0
Rourkela open hearth	36·7	0·9	26·0	—	27·9	25·7	19·4
Rourkela L.D.	37·0	0·86	24·7	—	30·0	27·7	16·7
NML (low shaft furnace slag)	33·0	0·027	18·8	—	27·2	25·3	15·9

produced by different slag samples and the standard liming material calculated on the basis of yield of crops obtained at different field trials is given in Table 83.

From the above comparative statement it was found that both Rourkela and Bhilai slag samples are more effective in increasing the *p*H of soil. Field trials of liming effect of slag have already been completed on four different crops in one season. In order to establish the slag as a better liming material, field trials need to be repeated in two more seasons on each crop in the same field, work on which is under way.

77.1 Utilization of Blast Furnace Slag as Rail Road Ballast

This investigation is also a part of broad-based research and development programme of utilization of blast furnace slag as rail road ballast which was initiated at the instance of Ministry of Railways. Detailed tests were conducted on the TISCO blast furnace slag ballast. A comparative study of the results obtained with that of the ballast, as well as results of standard ballast used in the rail-track, is given in Table 84.

Table 84 — Comparative Study of Blast Furnace Ballast with Standard Ballast

	Abrasion test %	Crushing test %	Unit wt lb./cu. ft	Sp. gr.	Absorption by wt %
Standard	12·51-26·52	15·82-25·23	98·106 app. 104	2·34-2·96	4-2·2
B.F. Slag	37·98	11·3	95·3	2·65	2·25

The comparative study indicates that the blast furnace slag can replace granite, limestone, quartz, etc., which are at present being used for rail-track ballast. Moreover, the rough angular pieces provide an interlocking, stable, non-shifting road bed which is essential for rail tracks particularly on curves. As slag does not contain organic matters, it can withstand severe weathering conditions. The above test results indicate the possibility of utilizing the Indian blast furnace slag as rail-road ballast. A few more confirmatory tests are however, required to be done with other slag samples from different iron and steel plants of India.

78.0 Utilization of Emery Flour

This investigation was taken up at the instance of M/s. Shevroy Bauxite Product Co. Ltd, Yeracud, to examine the suitability of emery flour, a waste product in the manufacture of synthetic emery flour bauxite for industrial usage. A 50 kg. sample of the emery flour was received for investigation. The emery flour was dark steel grey to black powder. A cone quartered sample of the emery flour was subjected to chemical and sieve analysis.

Experiments conducted indicated the distinct possibility of utilizing emery flour for (i) making sharpening sticks and blocks for polishing razor blades, etc., (ii) manufacture of non-slip tiles, and (iii) as a feed-back to the cupola in the manufacture of synthetic emery flour bauxite.

PILOT PLANTS

79.0 Low-shaft Furnace Project

With the installation of the fuel oil/ naphtha injection system and the incorporation of pipeline oxygen supply to the Low-shaft Furnace Pilot Plant, facilities for studying the fundamental and operational aspects of iron smelting with blast additives became available. In the absence of any reference in technical literature on the direct injection of highly volatile, explosive liquid naphtha into the hearth of an iron smelting furnace, the adventurous investigation was initiated with the object of utilizing surplus naphtha from the oil refineries and improvement in iron smelting efficiency by reduction in coke rate and increase in productivity.

Programme of Research and Development Work

Phase I

(i) Iron ore 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.

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

(iii) Iron ores from Chapra, Antribeharipur in Mahindergarh, Punjab, with nut coke or non-coking coals.

(iv) Iron ores from Anantpur, Warangal, etc., with non-coking coals (and low-temperature carbonized coke made thereof) from Kothagudem, Yellandu, etc., in Andhra Pradesh.

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

(vi) Iron ores from Nathare-ki-Pal near Udaipur and Morinja (Chomu-Samond) near Jaipur, Rajasthan, initially with nut coke and then with high-temperature carbonized Palana lignite (as and when it becomes available) [vide *Phase III*(i)].

Phase II

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

Phase III

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

(ii) Utilization of Salem magnetite after its beneficiation and agglomeration by pelletizing or briquetting in conjunction with carbonized lignite briquettes.

Phase IV

Research and development work on oxygen-enrichment of air blast, direct injection of naphtha and furnace oil, 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 ferromanganese with or without oxygen injection in the Low-shaft Furnace Pilot Plant.

Extensive investigations under *Phase I*, (i) to (vi), and *Phase II* were completed, and several feasibility Project Reports were issued. A comprehensive feasibility Project Report for the establishment of a Foundry Pig Iron Plant employing low-temperature carbonized coke with details of plant facilities, capital investment and economics of iron production in an industrial low-shaft blast furnace was prepared. Necessary assistance was rendered to the Government of Punjab for their Pig Iron Project in connection with the Project Report submitted earlier.

In the year under review, extensive smelting trials were conducted with injection of either fuel oil or naphtha with simultaneous enrichment of the blast with oxygen (*Phase IV*). The investigation undertaken during the period under review can be broadly classified into the following campaigns:

Thirtieth Campaign (Continued from earlier work)

Smelting trials were conducted with iron ore fines (B. Patnaik, Orissa), low-temperature carbonized coke (C.F.R.I.) and blended fluxes with the object of studying the effect of (a) oxygen enrichment of the blast without any hydrocarbon injection, and (b) progressive increase in oxygen enrichment of the blast at constant rate of oil injection on the smelting characteristics.

Preliminary Trials with Naphtha Injection

In order to familiarize the operational crew with the handling of naphtha, its distribution, metering and injection system, a preliminary smelting trial was conducted employing iron ore fines, low-temperature carbonized coke and blended fluxes.

Thirty-first Campaign (Series A)

In this series, the low-temperature carbonized coke (C.F.R.I) employed in the previous preliminary trial with naphtha injection was replaced with nut coke to evaluate the effect of the nature of the fuel on the smelting behaviour.

Thirty-first Campaign (Series B)

After the preliminary trials with naphtha injection was concluded without any operational difficulties, a smelting trial was conducted with iron ore fines, low-temperature carbonized coke and blended fluxes with injection of naphtha and simultaneous enrichment of the blast with oxygen with the objective of fully assessing the effect of these blast additions on the smelting characteristics.

Thirty-second Campaign

For the purpose of utilization of hitherto unused Dabok limestone from Rajasthan for iron smelting purposes, a trial was conducted with iron ore fines (Orissa Minerals), nut coke, fluxes of known smelting characteristics and the Dabok limestone.

Thirty-third Campaign (Series A)

With a view to utilize tertiary Assam coal for iron smelting, the suitability of a coke made from a blended coal-mix containing 20 per cent Assam coal therein was investigated.

Thirty-third Campaign (Series B)

Smelting trials were conducted with high-grade iron ore from Deulgaon (Maharashtra), low-temperature carbonized coke made from the Wardha Valley coals (R.R.L.) and limestone from Rajur, Maharashtra, with the object of utilization of the non-coking coals of Wardha Valley for iron smelting.

Details of Furnace Campaigns

Thirtieth Campaign (Continued from earlier work)

In this campaign the burden consisted of iron ore fines (B. Patnaik, Orissa) low-temperature carbonized coke made from non-coking coals (C.F.R.I.) and blended fluxes. The object of this exploratory campaign was primarily to familiarize the operational crew with the handling of the naphtha injection system, as due to the highly explosive character of naphtha and absence of any reference of its direct injection into the iron smelting furnace was considered as extremely hazardous. The fuel oil/naphtha distribution and metering of naphtha in the control room is shown in Fig. 52. The naphtha or oil injection lance is made out of concentric stainless steel tubes. Atomization of either fuel oil

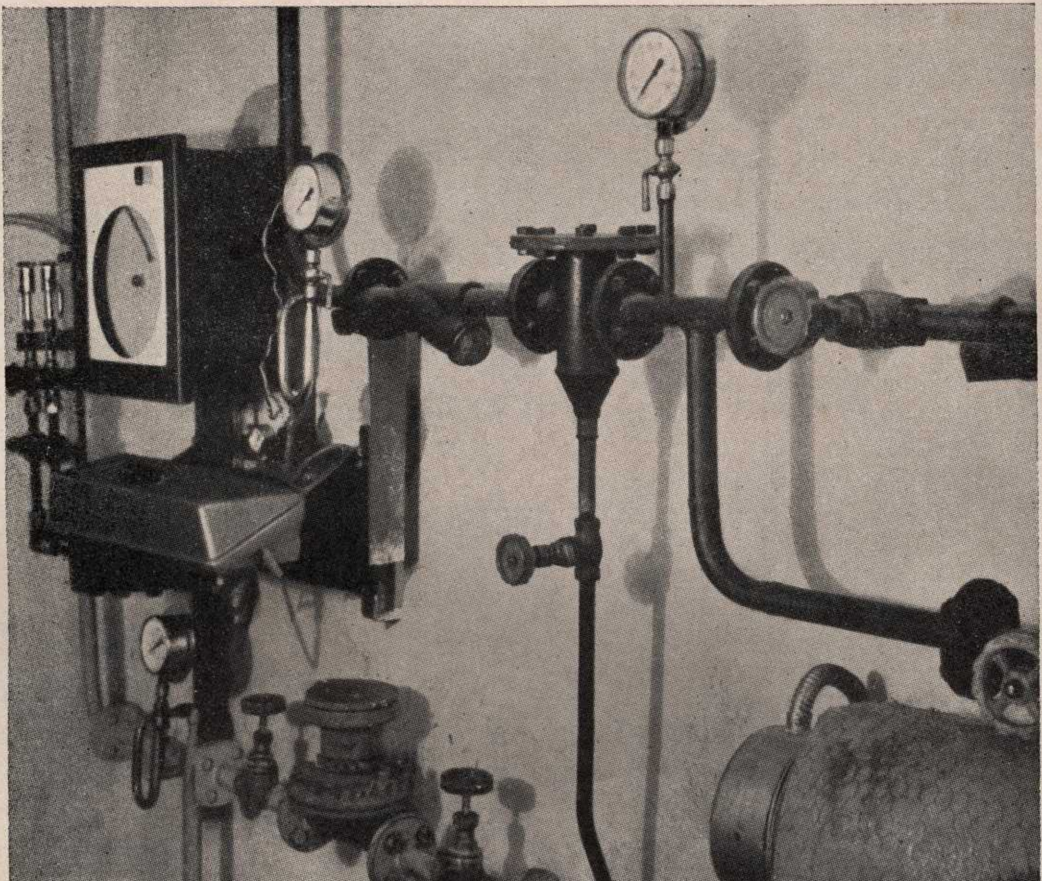


FIG. 52 — NAPHTHA-OIL DISTRIBUTION AND NAPHTHA METERING UNIT OF LOW-SHAFT FURNACE PILOT PLANT

or naphtha is effected by compressed air. Due to the fluctuations in the operational conditions, the data are not given.

Thirty-first Campaign (Series A)

In order to ascertain the effect of the value of fuel employed for smelting on the operational characteristics with naphtha and oxygen additions, nut coke was employed instead of low-temperature carbonized coke used in the previous exploratory trial.

Table 85 — Chemical Analysis of Raw Materials

1. Proximate analysis of nut coke:

F.C. %	V.M. %	Mn %	Ash %	S %
74·70	25·0	0·20	22·90	0·50

2. Analysis of coke ash:

CaO %	SiO_2 %	MgO %	Al_2O_3 %	Fe %	P %
3·8	52·08	2·12	33·0	6·0	0·69

3. Analysis of iron ore (Orissa mineral):

Fe %	SiO_2 %	Al_2O_3 %	S %	P %
64·14	3·28	4·57	0·01	0·02

4. Analysis of Limestone (Madras):

CaO %	MgO %	SiO_2 %	Al_2O_3 %	S %	Fe %
54·1	1·0	0·88	1·2	0·027	—

5. Analysis of Dolomite (Andhra):

CaO %	MgO %	SiO_2 %	Al_2O_3 %	S %	Fe %
32·20	25·0	0·3	0·56	0·55	N.D.

The physical and chemical characteristics of fine-grained iron ore, nut coke and blended fluxes employed for smelting in this campaign are recorded in Tables 85 and 86.

The furnace was blown in under the following conditions:

Tuyere diameter, mm.	75
Hot-blast temperature, °C	475-500
Hot-blast volume Nm.^{3}/hr	2400-2600
Hot-blast, pressure mm.WG	1600-1900
Top gas temperature, °C	450-480

The operational results in the base period and simultaneous injection of naphtha with oxygen are recorded in Table 87.

Table 87 — Operational Results with Naphtha Injection and Enrichment of the Blast with Oxygen

Data on			Base period	Naphtha injection with 2% oxygen enrichment
Daily production, tonne			5·86	6·33
Increase in production %			—	8·00
Naphtha injection rate kg./tonne of pig iron			—	38·00
Dust rate, % of raw material			3·90	4·06
Analysis:				
	Pig iron	Si%	3·80	3·00
		S%	0·06	0·08
	Slag	CaO%	38·00	35·00
		SiO_2%	32·00	33·00
		FeO%	1·50	2·50
	Gas	CO%	26·00	25·80
		CO_2%	5·60	5·80
		CH_4%	2·30	4·10

Table 86 — Screen Analysis of Raw Materials

Raw Material %	−50·8 mm.	−50·8 +25·4 mm.	−25·4 +12·7 mm.	−12·7 +6·35 mm.	−6·35 +13·175 mm.	−3·175 mm.
Nut Coke	—	53·00	30·00	5·48	2·98	8·54
Orissa mineral ore	—	—	5·95	46·50	39·00	8·55
Andhra limestone	—	7·35	62·00	12·25	7·50	10·90
Madras limestone	21·27	30·00	18·23	7·91	8·07	9·66

In the absence of oxygen injection, evidences of chilling of the smelting zone were noticed. Apart from other benefits, the increase in production became evident.

Thirty-first Campaign (Series B)

In order to examine the technological implications of naphtha injection with simultaneous enrichment of the blast with oxygen, a preliminary campaign was conducted with a burden composed of iron ore fines, low-temperature carbonized coke and blended fluxes. The chemical analysis of iron, ore, fluxed and low-temperature carbonized coke are given in Table 88.

The furnace was operated under the following conditions:

Tuyere diameter, mm.	75
Hot-blast temperature, °C	450-500
Hot-blast volume, Nm.^{3}/hr	2600-2800
Hot-blast pressure, mm.WG	1600-1900
Top gas temperature °C,	400-480

The operational results of each steady state period of smelting are summarized in Table 89 in which the data for consistent operation without wide fluctuations in chemical analysis of pig iron and slag were considered. In the base period the blast additives like naphtha and oxygen were not employed. The injection of naphtha with simultaneous enrichment of the air blast with oxygen was noticed to increase the productivity with appreciable decrease in coke rate. Although significant increase in production was noticed, further investigation was considered necessary.

Table 88 — Chemical Analysis of Raw Materials

1. Chemical analysis of iron ore (Orissa):

Fe %	SiO_2 %	Al_2O_3 %	S %	P %
59·92- 64·50	3·20- 6·34	4·10- 5·20	0·01- 0·29	0·02

2. Chemical analysis of fluxes:

	CaO %	SiO_2 %	Al_2O_3 %	MgO %
i) Limestone (Madras)	45·31	0·88	1·23	1·01
ii) Dolomite (Assam/W. Bengal	31·30	0·63	0·40	20·70

3. Chemical Analysis of low-temperature carbonized coke (C.F.R.I.):

Moisture %	V.M. %	F.C. %	Ash %	S %
5·80	4·60	61·60	26·00	0·23

4. Ash analysis of low-temperature carbonized coke (C.F.R.I.):

SiO_2 %	Al_2O_3 %	CaO %	MgO %	Fe %	P_2O_5 %	S %
56·31	22·00	3·45	2·20	7·61	1·83	0·27

Thirty-second Campaign

In order to assess the possibilities of hitherto unused Dabok limestone from Rajasthan for iron smelting, the physical and dissociation characteristics was thoroughly studied followed by an extensive iron smelting trial. The chemical and screen analyses of raw materials are given in Tables 90 and 91.

The smelting was conducted under the following operational conditions:

Tuyere diamter, mm.	75
Average blast volume, Nm.^{3}/hr	2400-2600
Average blast pressure, mm.WG	1700-1900
Average hot blast temp. °C.	380-450
Average top gas temp. °C.	360-400

As the behaviour of Dabok limestone as a flux for iron smelting was hitherto unknown, the smelting operation was brought to a reasonably steady-state condition employing raw materials earlier investigated for iron smelting, in which the previously used limestone was progressively replaced in stages of 25, 50 and 75 per cent till the flux exclusively consisted of Dabok limestone.

Table 89 — Operational Results with Naphtha Injection and Enrichment of the Blast with Oxygen

Data on		Base period without naphtha or O_2	Naphtha injection	
			Enrichment of blast with 1% O_2	Enrichment of blast with 2·5% O_2
Daily production tonnes.		6·85	7·94	8·08
Increase in production%			15·9	17·7
Naphtha injection. Rate kg./tonne of pig iron		—	33·0	33·0
Analysis:				
Pig Iron	Si%	2·5-3·5	2·6-3·75	2·5-3·0
	S%	0·05-0·08	0·05-0·07	0·05-0·07
Slag	CaO%	34	35	36
	SiO_2%	33	32	32
	FeO%	1·5	1·3	1·2
Top Gas	CO%	23-34	25·26	24-25
	CO_2%	3·5-4·0	4·7-5·0	4·8-5·6
	H_2%	0·1	0·2-0·6	0·2-0·7
	CH_4%	3·0-3·5	3·5-3·7	3·5-3·75

Table 90 — Chemical Analysis of Raw Material

1. Analysis of iron ore (Orissa minerals):

Fe%	SiO_2%	Al_2O_3%	S%	P%
64·14	3·28	4·57	0·01	0·02

2. Proximate analysis of nut-coke (air-dried basis):

F.C.%	V.M.%	Ash%	H_2%	S%
74·4	2·5	22·9	0·2	0·50

3. Analysis of coke ash:

CaO%	SiO_2%	MgO%	Al_2O_3%	Fe%	P%
3·8	52·08	2·12	33·0	6·0	0·69

4. Analysis of fluxes:

	CaO%	MgO%	SiO_2%	Al_2O_3%	S%
Limestone (Madras)	54·31	1·00	0·88	1·20	0·027
Limestone (Bisra)	44·80	3·57	6·96	1·60	Tr.
Dabok (Rajasthan)	54·80	2·00	1·24	1·22	Tr.

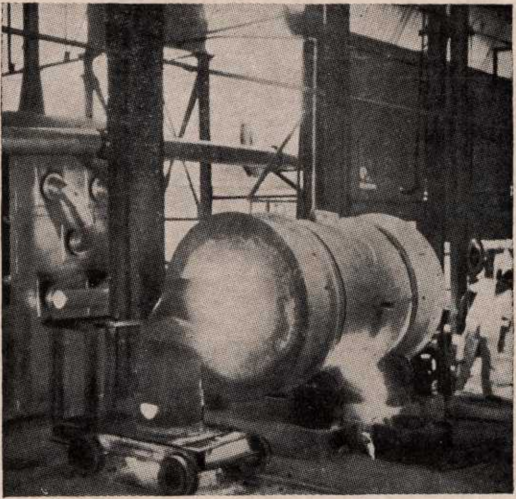


FIG. 53 — OIL-FIRED ROTARY FURNACE USED AS MIXER FOR SIDE-BLOWN CONVERTER

The amount of flux addition was adjusted to maintain the identical basicity degree (CaO/SiO_2) of the slag in the different stages.

The pig iron produced analysed:

C %	Si %	S %	P %	Mn %
2·0-3·0	3·25-4·5	0·0·7-0·10	0·18-0·29	0·15-0·32

The slag volume was 1·15 tonnes/tonne of pig iron produced and it analysed:

CaO %	SiO_2 %	Al_2O_3 %	MnO %	FeO %
35·37	32·36	18·23	3·5-5·5	1·2-2·9

The top gas analysed:

CO %	CO_2 %	CH_4 %	H_2 %
26·27	3·6-4·2	3·8-4·1	0-0·8

Based on its chemical analysis, physical properties, dissociation characteristics and smelting trials, the Dabok limestone was found to be satisfactory for iron smelting in low-shaft furnace.

Thirty-third Campaign (Series A)

With a view to utilize tertiary Assam coal containing high sulphur contents of 3-4 per cent but low ash contents of about 8 per cent and possessing fairly good coking properties, smelting trial was conducted to assess the suitability of a coke made out of a blend containing 20 per cent Assam coal for iron smelting purposes. It was understood that the coke was made in an industrial coke oven plant employing 20 per cent Assam coal in the blend, the balance being normal coking coal. The chemical and screen analyses of raw materials emoloyed are given in Tables 92 and 93.

Table 91 — Screen Analyses of Raw Materials

Raw materials	+50 mm. %	−50+25 mm. %	−25+12 mm. %	−12+6 mm. %	−6+3 mm. %	−3 mm. %
Iron ore fines	—	—	4·52	42·28	38·84	13·66
Nut coke	—	54·90	28·83	4·59	3·25	8·43
Limestone (Madras)	20·17	37·11	18·00	7·80	6·39	10·53
Limestone (Bisra)	—	—	2·60	8·00	32·90	56·50
Limestone (Dabok)	—	28·00	23·80	14·40	9·00	24·80

Table 92 — Chemical Analyses of Blended Coke and Low-temperature Carbonized Coke, Iron Ore and Flux

1. Proximate analysis of Assam coke (air-dried basis):

F.C. %	Volatile matter %	Moisture %	Ash %	S %
77·46	2·04	1·06	19·44	1·20

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

F.C. %	Volatile matter %	Moisture %	Ash %	S %
61·5	4·1	8·5	25·9	0·23

Analyses of coke ash:

	CaO %	SiO_2 %	MgO %	Al_2O_3 %	Fe %	P %
3. Assam coke:	4·76	50·88	0·80	27·60	10·08	0·28

4. Low-temperature carbonized coke made out of totally non-coking coals (C.F.R.I.):

CaO %	SiO_2 %	MgO %	Al_2O_3 %	Fe %	P %
5·68	53·8	1·88	28·0	4·6	0·80

5. Analysis of iron ore:

	Fe %	SiO_2 %	Al_2O_3 %	CaO %	MgO %	S %	P %
Iron ore (Orissa mineral)	64·14	3·28	4·57	Trace	Trace	0·01	0·02

Chemical analyses of fluxes:

	CaO %	SiO_2 %	MgO %	Al_2O_3 %	S %
6. Limestone (Madras)	54·31	0·88	1·0	1·20	0·027
7. Dolomite (Assam)	32·20	0·30	25·0	0·56	0·055

Table 93 — Screen Analyses of Raw Materials

Location	+50·8 mm. %	−50·8 +25·4 mm. %	−25·4 +12·7 mm. %	−12·7 +6·35 mm. %	−6·35 +3·175 mm. %	−3·175 mm. %
Assam coke	Nil	71·00	21·25	1·90	1·75	4·10
Low-temperature carbonized coke (C.F.R.I.)	10·3	48·20	37·70	2·70	0·50	0·60
Iron ore (Orissa mineral)	—	—	4·75	46·25	41·60	5·40
Limestone (Madras)	—	45·25	24·10	10·25	8·00	12·40
Limestone (Andhra)	—	17·50	61·50	10·75	4·75	5·50

The sulphur content of the blended coke was 1·2 per cent in comparison to 0·4-0·6 per cent in normal blast furnace coke employed in India. The exclusive replacement of the normal coke by the blended coke will raise the sulphur input by 140 per cent.

The furnace was blown under the following operational conditions:

Tuyere diameter, mm.	75
Average blast volume, Nm.^{3}/hr	2800-3000
Average blast pressure, mm.WG	1600-2000
Average hot-blast temp., °C	400-460
Average top gas temp., °C	450-500

As the characteristics of the blended coke for iron smelting were unknown, smelting in the low-shaft furnace was brought to a reasonably regular condition by employing iron ore fines, fluxes and low-temperature carbonized coke, smelting characteristics of which had earlier been investigated in the low-shaft furnace. The low-temperature carbonized coke was replaced in three stages till the fuel burden exclusively consisted of the blended coke. The progressive replacement was done on the basis of appropriate burden adjustments and was effected without any serious operational irregularity. It may, however, be mentioned that due to the availability of a very small quantity of coke blended with Assam coal for undertaking the trial, effects of various operational variables, particularly, the basicity of the slag and different silicon contents attainable on partition of sulphur could not be fully investigated. The calculated lime basicity degree of the slag was maintained at 1·2, but the analytical basicity degree was lower. The operational conditions and the analyses of pig iron, slag and top gas are given in Table 94.

The physical characteristics of the blended coke appeared to be satisfactory for iron smelting. In view of high sulphur input it was obvious that partition of sulphur between the hot metal and the slag would have to be considerably improved for confining the sulphur contents within the specified limits by raising the basicity degree and increasing the volume, or else, external desulphurization of hot metal would be necessary. Due to the availability of limited amount of blended coke, the effects of the different variables on the sulphur contents of the pig iron could not be comprehensively investigated and full-scale trials are warranted to assess the possibilities of utilization of Assam coal for iron smelting in normal blast furnaces.

Thirty-third Campaign (Series B)

With a view to ascertain the possibilities of establishment of a regional iron works based on the exploitation of raw materials available in the state, the Government of Maharashtra had sent iron ore, limestone and low-temperature carbonized coke made from the Wardha Valley coals for conducting smelting trials.

Table 94 — Operational Data, Chemical Analysis of Pig Iron, Slag and Top Gas

Data on	25% Assam coke 75% L.T.C. coke (CFRI)	7% Assam coke 30% L.T.C. coke (CFRI)	100% Assam coke
Daily production/ tonne	6·80	6·50	10·20
Fuel rate tonnes/ tonne of pig iron	1·85	1·80	1·65
Hot blast temperature °C.	405	405	440
Hot blast volume Nm^3/hr	2850	2800	3200
Hot blast pressure mm.WG	1600	1660	2000
Top gas temperature °C.	440	500	550
Slag volume/tonne of pig iron	1·41	1·24	1·02
Flue dust %	6·50	5·80	4·60
Average analysis of pig iron:			
C%	2·70	2·560	2·63
Si%	3·70	4·000	3·60
S%	0·07	0·083	0·13
P%	0·18	0·170	0·35
Mn%	0·25	0·350	0·18
Average analysis of slag:			
CaO%	35·2	35·90	36·40
SiO_2%	34·8	33·90	34·00
FeO%	3·9	3·50	1·30
Al_2O_3%	20·5	19·30	22·30
MgO%	5·2	4·90	3·80
S%	0·8	0·76	1·40
Average analysis of top gas:			
CO%	24·90	24·60	25·20
CO_2%	3·30	3·10	3·95
CH_4%	4·06	4·20	3·90
CO:CO_2 ratio	7·50	7·90	6·40

The physical characteristics of these raw-materials were studied. The chemical and screen analyses of raw materials used in one group comprising a part of the investigation are given in Tables 95 and 96.

The smelting trial was conducted under the following operational conditions:

Tuyere diameter, mm.	75
Average blast volume, $Nm.^3$/hr	2800-3000
Average blast pressure, mm.WG	1700-2100
Average hot blast temp., °C	410-450
Average top gas temp., °C	430-475

Table 95 — Chemical Analyses of Raw Materials employed

1. Proximate analyses of low-temperature carbonized coke made from the Wardha Valley coals of Maharashtra (RRL):

Moisture%	Ash%	Volatile%	F.C.%	S%
0·75	31·74	13·09	54·42	0·36

2. Analysis of ash of low-temperature carbonized coke (Wardha Valley coal):

SiO_2%	MgO%	Al_2O_3%	CaO%	Fe_2O_3%	P%
50·1	Trace	31·02	9·47	8·78	0·14

3. Analysis of iron ore (Deulgaon, Maharashtra):

Fe%	SiO_2%	Al_2O_3%	S%	P%	TiO_2 %
64·4	3·72	2·0	0·089	N.D.	0·10

4. Analysis of limestone (Rajur, Maharashtra):

CaO%	MgO%	SiO_2%	Al_2O_3%	Fe%	S%
48·03	2·74	5·56	1·16	0·20	Trace

Table 96 — Screen Analysis of Raw Materials employed

Raw materials	+50 mm. %	−50 +25 mm. %	−25 +12 mm. %	−12 +6 mm. %	−6 +3 mm. %	−3 mm %
Kolsit (Maharashtra)	10·87	48·30	31·37	4·25	0·50	4·70
Iron ore (Deulgaon)	nil	29·25	90·00	14·50	3·95	2·50
Limestone (Rajur)	nil	7·50	74·87	13·00	2·25	2·37

As the smelting behaviour of Deulgaon iron ore, low-temperature carbonized coke made from the Wardha Valley coals and Rajur limestone was hitherto unknown, the smelting operation was initiated with raw materials of known characteristics. The iron ore in this burden was progressively replaced with Deulgaon iron ore in the first stage. After a reasonable duration of smelting operation, the limestone was progressively substituted by the Rajur limestone, in the second phase. In the final phase, the nut coke was progressively replaced in stages of 25, 50 and 75 per cent till the fuel in the burden consisted exclusively of low-temperature carbonized coke (R.R.L.) made from the Wardha Valley coals of Maharashtra. In view of gradual and progressive replacement of nut coke by the low-temperature carbonized coke, the transition was effected without any major operational irregularities in the descent of the burden, chilling of the hearth and wide swings in the chemical analysis of slag and pig iron.

The addition of flux was based on a calculated basicity degree of slag CaO/SiO_2 of the slag in the range of 1·0 to 1·1. Although lower basicity degree of the slag will not allow optimum partition of sulphur in the slag, the minimum flux consumption leads to economical operation. During the extended trials, the effect of the operational variables, particle size of the raw materials and basicity degree of the slag on the fuel rate, productivity, desulphurization and dust generation can be assessed. The smelting with the low-temperature carbonized coke was characterized by regular descent of the burden and the normal smelting features in the experimental furnace. The analyses of pig iron, slag and top gas are given in Table 97.

Table 97 — Analyses of Pig Iron, Slag and Top Gas

1. Average analysis of pig iron:

C %	Si %	S %	Mn %
2·54	3·7	0·07	0·15

2. Average analysis of slag:

CaO %	SiO_2 %	FeO %	Al_2O_3 %	MgO %
32-35	31-33	10-2·9	17-24	4-5

3. Average top gas analysis:

CO %	CO_2 %	CH_4 %	H_2 %
25·0	4·25	3·7	0·45

79.1 Investigation on Briquetting

Briquetting of Iron Ore Fines

The mechanized mining of iron ore, upgrading of the metallic value by beneficiation, utilization of metallurgical wastes such as flue-dust, necessitate agglomeration treatments by briquetting, pelletizing and sintering. Amongst these processes, the hot-briquetting does not depend on the limited particle size tolerance of the feed. The hot briquetting needs preheating of the ore feed to high temperature followed by briquetting at high pressure. In view of the limitations of either of these prerequisites at the Low-shaft Furnace Plant, attempts were made for briquetting iron ore fines with small addition of coking coal, with the expectation that the plasticity and the fusion of the coal particles at moderately high temperature would function as a binder. The chemical analyses of iron ore and coking coals are recorded in Table 98.

For briquetting, the mix containing the iron ore fines and different additions of 5, 10, 15 and 20 per cent coking coal of −0·2 mm. size was heated to a temperature of 400°-425°C. and fed to a roller briquetting machine.

Table 98 — Chemical Analysis of Raw Materials

1. Analysis of iron ore (Orissa minerals):

Fe %	SiO_2 %	Al_2O_3 %	S %	P %
64·14	3·28	4·57	0·01	0·02

2. Proximate analysis of coking coals:

	H_2O %	V.M. %	F.C. %	Ash %	Caking index (BSS)
i) Kargali (Bokaro washed coking coal)	1·2	30·0	56·6	12·2	24
ii) Dishergarh (New Sitalpur)	1·5	36·3	49·3	12·9	20

Assessment of physical properties of the briquettes by the shatter test and crushing strength showed that the room temperature strength was unsatisfactory due to low preheating temperature and inadequate pressure of the available roller briquetting machine. Further work will be conducted after preheating the mix to a temperature of about 1100°C.

79.2 Experimental Baby Blast Furnace for Studying the Effect of Nature of Metallic Burden and Injection of Auxiliary Fuels on the Smelting Efficiency

The experimental miniature blast furnace was designed and fabricated with the object of studying the effect of nature of the metallic feed such as sinters, pellets and lumpy ore, injection of oxygen, injection of various auxiliary fuels, etc., on the fuel rate.

After the determination of the coke rate at the hot-blast temperature of 200°C., the recuperator was modified. With a hot-blast temperature of 300°C. the coke rate was ascertained using iron ore fines (Orissa Mineral). The enrichment of the blast with 4 per cent oxygen reduced the fuel rate significantly and increased the daily production substantially. Further increase of oxygen enrichment to 8 per cent led to operational difficulties; particularly hot-spots that developed around the tuyere region and the tuyeres, which were not water-cooled, became red hot. Although the fluidity of the metal and slag were satisfactory, the absorption of carbon was low. Further work envisages the effect of nature of metallic burden and auxiliary fuel injection on the coke rate.

80.0 Malleabilization Characteristics of Low-shaft Furnace Pig Iron

It was mentioned earlier that smelting of pig iron with low-temperature carbonized coke made from low-phosphorus non-coking coals yielded pig iron with low phosphorus contents instead of 0·3 to 0·4 per cent in pig iron produced in iron blast furnace. In view of this, the malleabilization characteristics of pig iron produced in the experimental low-shaft furnace were studied. The effect of additions of boron and aluminium of reducing the malleabilization cycle were investigated. Attempts were made to ascertain the completion of malleabilization by dilatation technique.

81.0 Pneumatic Method of Steel-making in Basic-lined side-blown Converter

The object is to produce acceptable grade of steel from Indian pig iron containing about 1·0-1·2 per cent Si and 0·30-0·40 per cent P in a half-tonne basic-lined side-blown converter, designed and fabricated in the Laboratory. Numerous trials have demonstrated the possibility of conversion of hot metal analysing C, 3·3 to 3·6; Si, 0·9-1·4; P, 0·3 to 0·4 per cent to low-carbon steel conforming to the specification for sulphur and phosphorus contents. As the conversion was accomplished without consuming electrical power or commercially pure oxygen at high pressure, the process can be adopted in steel foundries or small scale regional steel works.

In order to improve the economics of the process, the factors affecting the yield of steel ingot from the hot metal were thoroughly investigated. The basicity degree of the slag was adjusted to reduce the FeO contents and also to minimize the slopping. The blowing time and the angle of tilt of the vessel during the blow were also suitably adjusted. The typical results of a few selected trials are recorded in Table 99.

In view of the efficacy of the phosphorus removal due to injection of powdered lime in the stream of oxygen in basic oxygen steel-making processes, a mechanism to

Table 99 — Typical Experimental Results of Some of the Blows

Sl. No.	Approx. wt of hot metal blows	Temp. of hot metal	Time of blow min.	Chemical Analysis, per cent											Add. to the converted kg.			
					C	Si	P	S	Mn	CaO	SiO_2	MgO	P_2O_5	$FeO+Fe_2O_3$	Lime	Fe-Si	Fe-Mn	Yield
49	500	1200	20	H B	3·28 0·054	1·36 0·14	0·31 0·072	0·16 0·06	0·74 0·08	17·45 11·37	15·60 16·08	13·75 18·36	2·1 0·99	46·66 44·94	60	4	4	80
50	500	1200	25	HM BM	3·55 1·21	1·07 0·04	0·21 0·04	0·069 0·6	0·51 Trace	— 22·54 14·02	— 36·4 15·6	— 0·58 0·54	— 2·2 1·95	— 14·73 45·2	60	4	4	75
51	400	1180	15	HM BM	3·34 0·09	1·21 0·16	0·4 0·16	0·07 0·09	0·62 Trace	— 13·9	— 29·52	— 15·80	— 2·10	— 33·80	30	4	4	73
52	400	1220	20	HM BM	3·4 0·054	1·21 0·7	0·4 0·02	0·07 0·087	0·62 Trace	— —	— —	— —	— —	— —	40	4	4	80
53	550	1200	20	HM BM	3·6 1·6	0·9 0·35	0·38 0·13	0·07 0·065	0·65 0·06	—	—	—	—	—	50	2	4	75
										After 5 min 18·5	22·4	28·20	0·84	3·00 24·10				
										After 15 min 24·5	21·6	24·80	1·6	2·6 19·4				
										After 20 min 23·1	22·5	24·60	2·3	4·25 18·40				
										22·5	21·2	27·20	1·8	2·48 19·85				
54	450	1810	18	HM BM	3·01 0·41	3·5 0·15	0·34 0·25	0·097 0·11	0·24 0·24	— 21·07	— 24·36	— 24·4	— 3·3	— 15·9	40	3	4	78

entrain lime powder in the air blast was designed and is under fabrication. Further investigations on the process control, absorption of steel scrap, quality of the flux on dephosphorization are under way.

82.0 Investigation on Carbon Saturation in Normal Cupola Practice

Pig iron containing low sulphur and phosphorus cannot be produced in normal blast furnace practice under Indian raw material conditions. Such pig iron is required for making special castings. The addition of steel scrap in the normal cupola practice lowers the carbon content. The absorption of carbon during the melting of steel scrap in acid- and basic-lined cupolas have been reported earlier.

In this part of the investigation, different types of solid carbonaceous ingredients were introduced with or without injection of oxygen in the extended metal well of the cupola. Metal was tapped at regular intervals, a typical analysis is recorded in Table 100.

83.0 Scrap Oxygen Steel Making in L.D. Converter

With a view to utilize pig iron and steel scrap readily available in foundries for making steel, extensive investigation on the scrap-oxygen steel-making were conducted. In this process, solid pig iron and steel scrap were converted into steel by top blowing with oxygen in the presence of a cheap combustible material in the cold charge. In order to reduce the requirements of solid fuel and phosphorus input through it, the minimum amount of fuel was ascertained. The effect of the nature of the flux, and addition of millscale or iron ore on the dephosphorization were studied. The addition of ferro-manganese and other blowing conditions on the removal of sulphur were investigated. The progressive removal of C, Si, P and S with the blowing time was studied. The technique of oxygen lancing was altered to reduce the blowing time. Experimental results of some typical blows are given in Table 101. Further work on the size of the scrap, ratio of steel to pig iron scrap on the process, dephosphorization and desulphurization under

Table 100 — Some Typical Results on Carbon Saturation

Name of the sample	Holding in time	Type of fuel used in the extended metal well	Chemical analysis of the tapped metal									
			Without O_2					With O_2				
			C	P	Si	Mn	S	C	P	Si	Mn	S
$1R_1$	0	X	4·00	0·23	0·28	0·24	0·05	1·44	0·20	1·21	0·14	0·13
$1R_2$	5	X	4·05	0·28	2·20	0·47	0·04	1·98	0·38	2·34	0·25	0·10
$1R_3$	15	X	4·23	0·23	0·84	0·39	0·05	2·43	0·45	3·15	0·30	0·15
$1R_4$	30	X	4·77	0·26	0·67	0·45	0·09	2·25	0·39	3·36	0·33	0·16
$2R_1$	0	Y	2·75	—	—	—	—	2·65	0·22	2·97	0·24	0·12
$2R_2$	5	Y	2·76	—	—	—	—	2·16	0·24	1·98	0·21	—
$2R_3$	15	Y	2·16	—	—	—	—	2·70	0·17	2·00	0·21	0·12
$2R_4$	30	Y	2·48	—	—	—	—	2·34	0·17	1·20	0·17	—
$3R_1$	0	Z	—	—	—	—	—	1·224	0·287	0·027	0·123	—
$3R_2$	5	Z	—	—	—	—	—	1·780	0·275	1·401	0·130	0·174
$3R_3$	15	Z	1·78	0·271	1·998	0·195	—	—	—	—	—	—
$3R_4$	30	Z	2·25	0·303	1·756	0·195	—	—	—	—	—	—

different operational conditions are under way.

Table 101 — Experimental Results of Some of the Blows

Experiment No.	Analysis of scrap %					Analysis blown metal %					Slag bosicity (CaO: SiO_2 ratio	Scrap to ingot yield %	Blowing time in min.	Oxygen consumed in Nm^3 100 kg. scrap	Nature of flux
	C	Si	S	P	Mn	C	Si	S	P	Mn					
1.	2·6	3·7	0·05	0·30	0·55	0·05	0·10	0·05	0·06	0·15	1·6	88	23	16	Burnt line
2.	3·15	3·5	0·09	0·47	0·32	0·12	0·04	0·07	0·032	0·19	1·8	90	25	18	Burnt line briquetted
3.	3·0	3·0	0·06	0·45	0·45	0·14	0·07	0·11	0·10	0·07	1·4	85	25	16	Limestone concentrated pellets
4.	3·2	3·2	0·08	0·49	0·30	0·045	0·56	0·074	0·06	0·10	1·6	85	28	22	Limestone
5.	2·8	3·8	0·09	0·43	0·45	0·12	0·16	0·09	0·09	0·09	1·5	85	24	16	Limestone concentrated pellets

84.0 Study on Sulphide Capacities of Slag

Iron smelting in low-shaft furnace employing low-grade fuel leads to high sulphur input and, therefore, the partition of sulphur between the metal and slag is of importance. Hence the capacity of $CaO\text{-}Al_2O_3\text{-}SiO_2$ slags to absorb sulphur was investigated in the Laboratory.

Basic slag is known to absorb sulphur from a mixed gas containing $CO\text{-}CO_2\text{-}SO_2$ by the reactions:

$$CaO+SO_2+3CO = CaS+3CO_2 \qquad \ldots(1)$$
$$CaO+SO_2+CO_2 = CaSO_4+CO \qquad \ldots(2)$$

It can be described in terms of oxygen and sulphur:

$$CaO+\tfrac{1}{2}S_2 = CaS+\tfrac{1}{2}O_2 \qquad \ldots(3)$$
$$CaO+\tfrac{1}{2}S_2 = \tfrac{3}{2}O_2+CaSO_4 \qquad \ldots(4)$$

Under reducing atmosphere, sulphide formation takes place, the equilibrium constant, K of the reaction is:

$$K = \frac{{}^{a}CaS.PO_2^{\frac{1}{2}}}{{}^{a}CaO.PS_2^{\frac{1}{2}}} \qquad \ldots(5)$$

where sulphurizing power, $A = \dfrac{PS_2^{\frac{1}{2}}}{PO_2^{\frac{1}{2}}}$

$$= \frac{\text{wt percent S} \times PO_2^{\frac{1}{2}}}{{}^{a}CaO \times PS_2^{\frac{1}{2}}} \qquad \ldots(6)$$

Sulphide capacity, CS

$$= \text{wt percent of S}\,\frac{PO_2^{\frac{1}{2}}}{PS_2^{\frac{1}{2}}}$$

$$= K\,{}^{a}CaO$$

The activity of CaS in a slag can be considered as percent wt of S.

The experimental set up consists of a platinum-10 per cent rhodium high-temperature furnace with a number of gas purification trains, flowmeters, and gas measuring burettes as shown in Figs. 54 and 55.

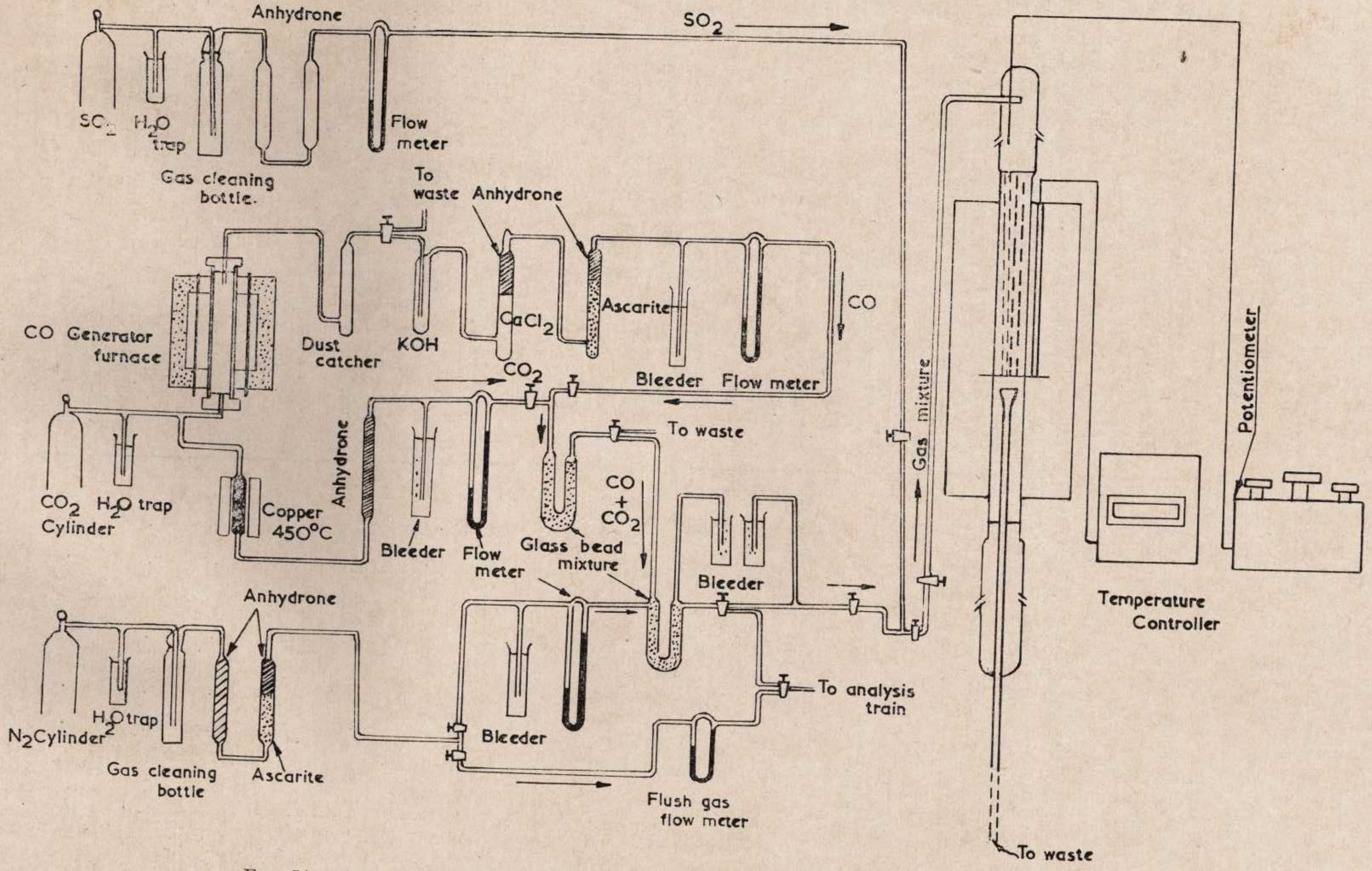


FIG. 54 — SCHEMATIC FLOW DIAGRAM FOR THE STUDY OF SULPHIDE CAPACITIES OF SLAGS

A platinum cup containing slag sample is supported in an alumina block for placing the sample at the constant temperature zone of the tube furnace. The general arrangement of the apparatus is shown in Fig. 55. The 1 m. long sillimanite tube connected at both ends with standard glass joints, served as the reaction tube. The furnace is brought to the required temperature and flushed with nitrogen. The slag sample was pushed slowly into the centre of the furnace. After passing nitrogen for a further period of 10-15 minutes, the appropriate gas mixture was passed through the furnace. At the completion of the experiment, the end of the reaction tube is opened with the mixed gases still flowing, and the sample was quickly drawn outside thereby solidifying it.

For the preparation of synthetic slags, chemical reagents are used. Small quantity 0·2-0·4 g. of the synthetic slag sample was taken and the rate of sulphur transfer of those melts were determined.

Gas mixture of controlled composition were prepared by taking the appropriate gases from the respective cylinders and mixing them in metered streams. CO_2 and SO_2 were available from cylinders with negligible amounts of oxygen and nitrogen as impurities. CO was obtained by passing dry CO_2 over powdered electrode graphite

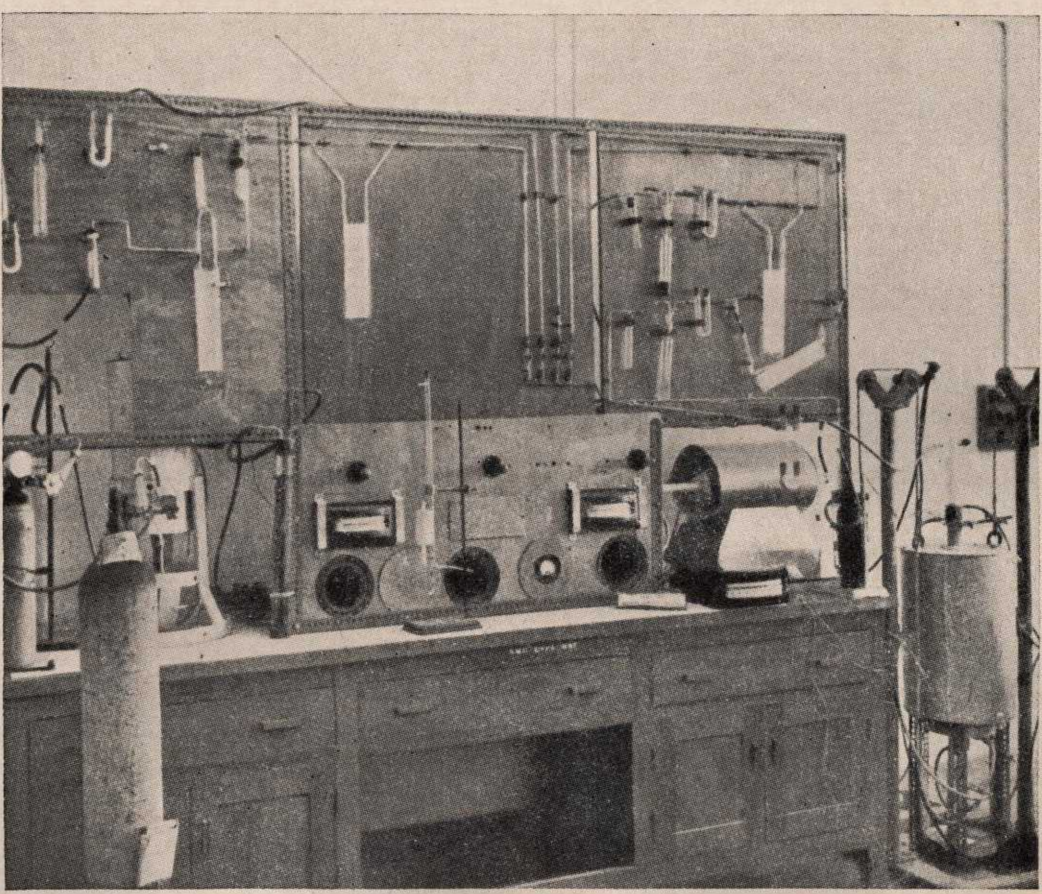


FIG. 55 — SET UP FOR THE STUDY ON SULPHIDE CAPACITIES OF SLAGS

at 1100°-1150°C. Soda asbestos was used to remove unchanged CO_2. The dried gases were passed through the capillary manometer and constant flow during an experimental run was maintained. The flowmeters were duly calibrated by conventional methods. The amount of CO-CO_2-SO_2 per cent in the gas mixture were controlled to a predetermined value.

It is desired to study the ratios of sulphur and oxygen transfer from gas to CaO-SiO_2-Al_2O_3 slags of different compositions. Subsequently MnO, Al_2O_3 additions to the slag will be investigated.

85.0 Reactivity of Solid Fuels employed for Iron Smelting in Low-shaft Furnace

Reactivity of fuel has a marked effect on the reducing reactions in the stack of a blast furnace. In the Low-shaft Furnace the limited stack height requires fuel of optimum reactivity.

The general principle underlying the various empirical methods available for the determination of the reactivity of coke ultimately depends upon the measurement of the corresponding specific reaction rate of coke sample with some oxidizing agent, such as carbon dioxide, steam or air. The method for the determination of reactivity towards CO_2 involves the passing of CO_2 under specific conditions through a bed of coke and comparative reactivity values are obtained by analysing the exit gases for CO. The different methods available for the determination of reactivity of coke to oxygen have been differently designated as 'Combustibility', 'Ignition point' and the 'Critical-air-blast (C.A.B.)'.

Reactivity to CO_2

The method adopted has been described in literature. It is measured by the volume of coke of specified size maintained at a constant temperature. Fig. 56 shows the apparatus used. The sample was heated to 950°C. in a current of nitrogen. Pure CO_2, 100 ml., was passed over the sample at a rate of 5 ml./min. and 150 ml. was used for flushing the system. The gases resulting from the balance 50 ml. of CO_2 were collected in a nitrometer over NaOH solution to absorb the unconverted CO_2 gas. The volume of CO is taken as reactivity towards CO_2.

Critical-air-blast

The critical-air-blast is a measure of the reactivity of the fuel to air. It is the minimum rate of air-blast which will maintain combustion of closely graded fuel in an ignited bed of specified dimensions. The more reactive the fuel, the lower is its C.A.B. value. The method adopted is that of IS Specification.

A bed of graded coke was ignited electrically in a standard combustion chamber, with dry air passing through the bed at a rate of 0·0042 M^3/min. When the coke was fully ignited the air rate was reduced to a preselected value for 20 minutes and then raised to the original value to attempt resuscitation. The procedure was repeated with fresh portions of the coke with other rates of air flow until the critical value was found.

The fuel used for the determination of C.A.B. graded to pass a 1·18 mm. IS Sieve and to be retained on a 600-micron IS sieve which was dried in a shallow tray in an air oven at 105°-110°C., and re-sieved on a 600-micron IS sieve immediately before use.

Air was supplied from a compressor and was dried by means of a drying tower containing silica gel. The flow rate was measnred by an inclined capillary flow gange. Fig. 57 gives the details of the C.A.B. apparatius.

Studies will be carried ou tus. inferior grade fuels used for iron smelting in Low-shaft Furnace to determine their reactivity and assess the effect of furnace operation.

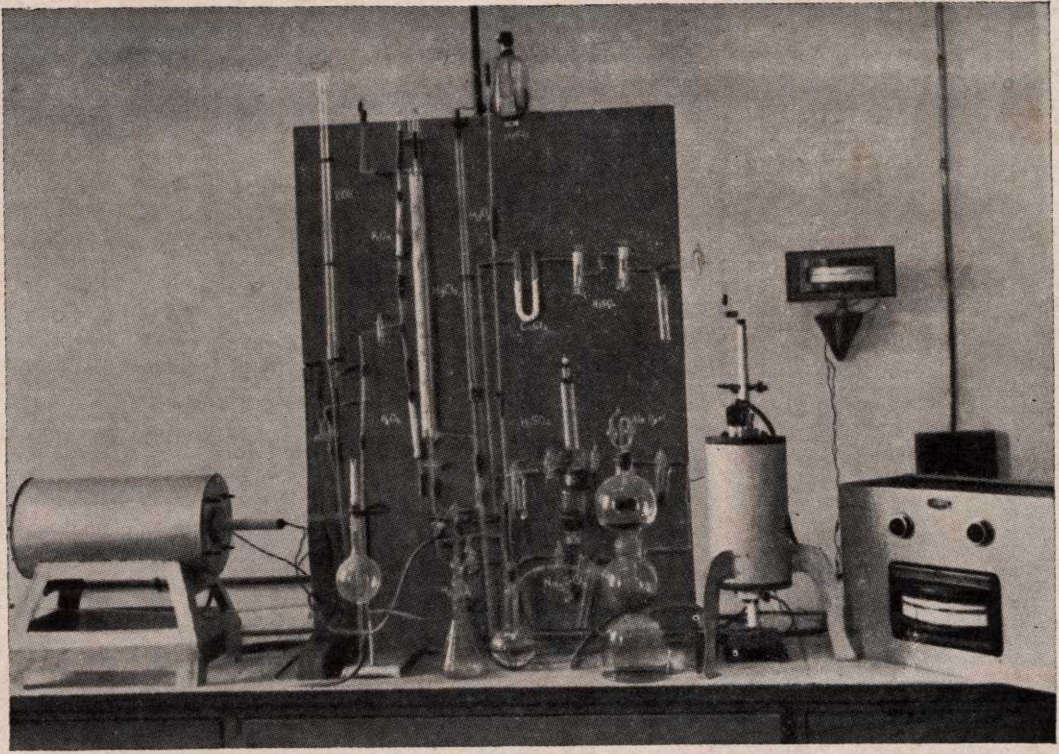


FIG. 56 — SET UP FOR STUDY ON REACTIVITY OF COKE TO CO_2

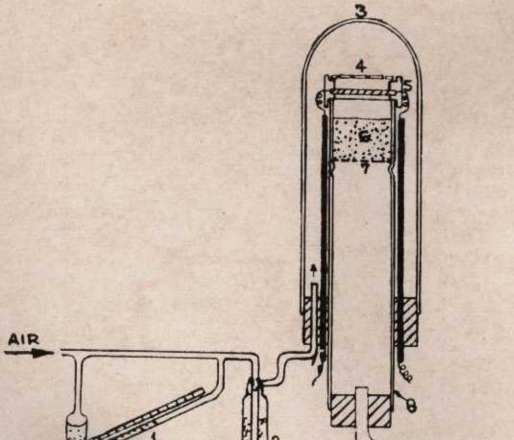


FIG. 57 — LINE DIAGRAM OF THE APPARATUS FOR DETERMINATION OF CRITICAL-AIR-BLAST

86.0 Production of Pig Iron from Run-of-mine Iron Ore, Limestone and Coke Breeze in a Rotary Furnace

In order to utilize metallurgical wastes such as the run-of mine iron ore fines, limestone dust and the huge quantity of coke breeze produced during carbonization of coal, investigations were continued for the production of molten pig iron in an acid-lined rotary furnace. As the CO liberated in the chemical reaction is burnt to CO inside the vessel by oxygen lancing, the chemical heat of the fuel is fully utilized and no air blast and its heating are necessary. In the year under review smelting character of different iron ores were taken up with coke breeze.

The chemical analyses of the raw material employed are reported in Table 102. The sieve analyses of the raw material are given in Table 103.

As reported earlier, the furnace was initially heated up by oil burner and a pool of molten metal was obtained by melting pig iron. Then the charge consisting of iron ore, coke breeze and limestone dust was fed intermittantly. The average analysis of the metal obtained was as follows:

Pig Iron:	per cent
C	2·4 to 3·2
Si	0·1 „ 0·4
P	0·28 „ 0·32

87.0 Appraisal of Raw Materials for Iron-Making

(i) *Iron Ore — Physical and Chemical Characteristics*

In view of limited time of descent of raw materials in the Low-shaft Furnace technique of iron smelting, the determination of reducibility and decrepitation characteristics of iron ore, and the dissociation behaviour of limestones are of immense technological significance.

Results of physical tests, reducibility and decrepitation characteristics of Deulgaon,

Table 102 — Chemical Analyses of the Raw Material employed for Smelting

Raw material:	Fe %	Mn %	SiO_2 %	Al_2O_3 %	CaO %	MgO %	P %
Iron ore (O)	64·14	—	3·28	4·59	—	—	0·02
Iron ore (B)	63·2	—	2·88	5·6	—	—	0·05
Iron ore (Bo)	57·00	—	5·00	6·50	0·10	0·20	0·05
Manganese Ore (D)	5·6	50·7	11·2	10·30	—	—	—
Limestone (M)	—	—	0·88	1·2	54·1	1·0	—
Limestone (A)	0·47	—	0·3	0·56	32·20	25·07	—

	Fe %	VM %	Moisture %	Ash %	S %
Coke breeze:	69·4	2·5	0·4	27·2	0·5

	CaO %	SiO_2 %	MgO %	Al_2O_3 %	Fe %	P %
Analysis of coke ash:	3·87	53·31	2·10	33·01	6·02	0·69

Table 103 — Sieve Analyses of all the Raw Materials employed for Smelting

Raw Material:	+50·8 mm. %	−50·8 +25·4 mm. %	−25·4 +12·7 mm. %	−12·7 +6·35 mm. %	−6·35 +3·17 mm. %	3·17 mm. & below %
Iron ore (O)	—	—	1·32	18·96	26·12	53·60
Iron ore (B)	—	—	2·01	20·60	28·71	48·68
Iron ore (O)	—	—	3·16	19·67	30·10	47·07
Manganese Ore (D)	—	—	18·25	58·34	15·78	7·63
Limestone (M)	—	1·68	58·72	30·50	3·73	5·37
Limestone (A)	—	2·79	59·81	29·38	4·84	3·18

Mechanical grading of coke breeze:

+7 mesh sieve	−7 +16 mesh sieve	−16 +30 mesh sieve	−30 +60 mesh sieve	−60 +100 mesh sieve	−200 mesh & below
8·60	21·66	30·14	15·27	10·67	13·66

Table 104 — Results of Shatter and Tumbler Tests, Reducibility and Decrepitation Characteristics of Some Iron Ores

Iron ore	State	Reducibility Test				Shatter strength size $-50{\cdot}08$ $+25{\cdot}4$ min. on 12·7 mm. size	Abrasion strength	Decrepitation temperature	Structural characteristics
		Size $-12{\cdot}7$ $+6{\cdot}4$ mm. at 900°C. 800/min. redn %	Time in min.	At 800°C. H_2 gas flow redn %	Time in min.				
Mineral	Orissa	91·7	32·0	83·5	42·0	89·5	71·0	410/415	Mostly laminated
Meghatubura	Orissa	64·6	75·0	64·6	90·0	94·17	74·4	405/435	Massive
Taldih	Orissa	79·6	90·0	71·5	115·0	95·7	72·0	490/500	Massive
Gua	Bihar	70·1	85·0	66·4	110·0	95·43	78·3	440/460	Massive
Bolani	Orissa	81·7	80·0	70·0	90·0	85·16	78·57	500	Massive
Deulgaon	Maharashtra	55·0	60·0	52·6	40·0	98·7	90·0	410/440	Massive

Taldih, Meghatuburu, Gua and Bolani ores are recorded in Table 104. The shatter strength varied for 89·5-98·7 per cent on +12·5 mm. fraction. The decrepitation temperature was around 400°-500°C.

The producibility of these ores were compared with an iron ore (Orissa Mineral, Orissa), which was employed as a standard in view of its high reducibility. All these ores had inferior reducibility.

(ii) *Limestone — Dissociation Characteristics*

During the descent of the burden in the shaft of the iron smelting furnace, it is subjected to progressively higher temperature. The dissociation of limestone to CaO and CO_2 is endothermic and needs a large quantum of heat, and it should preferably be completed before the limestone descends to the higher temperature smelting zone and hearth of the furnace. The dependence of CO_2 evolution on temperature and time, and degree of calcination of the limestone are important in iron smelting.

The dissociation characteristics were studied by heating the sample at a predetermined rate of 5°C./min. which, to a certain extent, simulated the progressive increase in temperature during its descent in furnace stack. The rate of evolution of CO_2 with respect to time was ascertained by inserting the sample in a furnace heated to a temperature of 600°C. The particle size was maintained at −24·4 +19 mm. for all samples.

88.0 Electrolytic Manganese Metal Pilot Plant

During the year under review, two ores, having the composition given below, were used:

	I	II
	%	%
Mn	35	27
Fe	21·7	3·0
Al_2O_3	7·4	0·55
SiO_2	7·3	43·2
P_2O_5	0·15	—
(S)	0·03	—

The ores were ground to the size −35 to +72 mesh, reduced in rotary kiln and leached with spent liquor containing manganese sulphate, ammonium sulphate and sulphuric acid. Recovery of manganese as manganese sulphate solution was 80 to 90 per cent. It was also noticed that high iron content in the ore inhibited the settling of the insolubles in the leach liquor. High silica content of the ore apparently did not affect the settling rate. As a result, the overall recovery of manganese by leaching ore II was in no way inferior to the richer ore.

Experiments on washing of the undissolved residue showed that unless washing was thorough and wash water carefully preserved, loss of manganese sulphate and ammonium sulphate was quite considerable. Some improvements in maintaining a stable *p*H value in the catholyte were effected by stopping leakages of anolyte into the cathode compartment. Cases of uneven deposits on the cathode surfaces were minimized by improved spacer blocks for the cathodes which securely held the cathodes equidistant from the adjacent anode surfaces. Several experiments were carried out to determine the optimum period for which a particular cathode could be used continuously for metal deposition in the cell currently in use. Thirty-six hours deposition cycle for a cathode was found to be satisfactory as regards current efficiency, stability of the operative conditions of the cell and character of deposit.

Out of the total quantity of manganese produced in the pilot plant about 1·5 tonnes of the metal was utilized for research activities in NML and about 250 kg. of the metal was sold to a firm.

88.1 Electrolytic Manganese Dioxide Pilot Plant

The two manganese ores whose compositions are given in Clause No. 88.0, were tested for their leaching characteristics making concentrated solutions of manganese sulphate for manganese dioxide cells. In this case also the insoluble residue in the leach liquor from the ore containing high iron did not settle easily. Recovery of manganese from both the ores varied between 80 and 90 per cent. The manganese dioxide cell was fitted partly with carbon anodes and partly with graphite anodes with a view to make a comparative study of their behaviour as anode. On prolonged operation of the cell at 93°-96°C. both types of anodes were found affected at the air electrolyte inter-face, and some of them broke under the load of the deposited manganese dioxide. Current efficiency of deposition was around 90 per cent. The composition of the manganese dioxide produced is given below:

	per cent
MnO_2	91·11
Acid insolubles	0·12
SO_4	1·65
Fe	0·13
Fb	Trace
Moisture	6·99 (by diff.)

The manganese dioxide cells were also run with lead anodes. During the period under review, 500 kg. of electrolytic manganese dioxide were produced and sold to a firm for production of dry cells.

89.0 Pilot Plant for Hot-dip Aluminizing of Steel

During the year under review, the pilot plant was extensively operated to produce different types of aluminized products and also to give practical demonstration of the technique developed to a large number of licensees. A brief account of them are furnished below:

M/s. Usha Martin Black (Wire Ropes) Ltd, Ranchi

Two hundred kilograms of aluminized steel wire coated at NML pilot plant was supplied for trial and development of wire ropes to M/s. Usha Martin Black (Wire Ropes) Ltd, Ranchi. The party felt that coating obtained from the commercial aluminium-bath used had inadequate ductility. In order, therefore, to improve the wrapping properties, silicon additions were made to the aluminizing bath to decrease the thickness of alloy layer and its micro-hardness. One hundred and fourty-four kilograms of 14 and 16 SWG m.s. wires coated from Al-2 per cent Si-bath were supplied and these passed the wrapping tests.

M/s. Special Steels Ltd, Bombay

One hundred and eighty kilograms of aluminized steel wire of two types were supplied to M/s. Special Steels Ltd, Bombay. First type was a 0·62 per cent C high-tensile wire with heavy coatings to meet IS : 398-1961 or B.S. 443-1964 and U.T.S. and torsion values. Second type of low-carbon steel had to meet property requirements as per IS : 280-1964, i.e. U.T.S., elongation and torsions. This firm has been trying to arrange import of equipment from a West German firm as indigenous supply is not available so far.

M/s. Fort William Co. Ltd, Hooghly

One hundred and thirteen kilograms of four types of wire were aluminized for M/s. Fort William Co. Ltd, Konnagar, Hooghly. Type 1 was 0·098 in., 75/80 TSI UTS, 26 Torsion, wire for drawing to 0·035 in., 100/110 TSI UTS, 32 Torsions wire. Type 2 was 0·092 in., 0·7 to 0·75 per cent C, 37 Torsion, wire for drawing to 0.032 in., 100/110 TSI UTS, 32 Torsions. Type 3 was 0·082 in., 0·25-0·3 per cent C, 106 Torsions, 19 per cent Elongation wire. Type 3 and 4 were to be aluminized so as to get 24/32 TSI, UTS, 12 per cent Elongation, 40 Torsions. Coating weights asked for were equivalent to those of B.S.S. 443-1961. Weight of aluminium coatings to get the same thickness as zinc coatings was about 2·5 times less. Type 3 and 4 could be aluminized to meet all requirements. To get desired results for Types 1 and 2, party was advised to send 0·048 and 0·040 in., 100/110 TSI UTS.

Barbed Wire

M/s. Assam Steel & Wire Products, Tinsukia, and Shiva Wire & Steel Products Ltd, Calcutta, are interested in manufacture of barbed wire. The cut edges in barbed wire are likely to show localized rust on exposure which though unsightly will not be harmful as it does not spread. Field trials of aluminized steel wire at Visakhapatnam have already established that it lasts three times longer than galvanized wire in the worst saline corrosive atmosphere and would thus be still more lasting in dry, humid, industrial sulphurus atmospheres. Aluminium coating obtained from baths with silicon addition were ductile and could withstand wrapping, twisting and stranding.

Hot-dip Aluminizing of ACSR Core Wire

ASTM B341-63 T lays down the following chemical composition for ACSR core wire:

	per cent
Carbon	0·50-0·95
Manganese	0·50-1·30
P max.	0·040
S max.	0·050
Si	0·10-0·30

It also specifies the following mechanical properties for the aluminized wire for this application.

Nominal dia. in.	Stress at 1% extension min. psi	U.T.S. min. psi	Elongation on 10 in G.L, min. %
0·05-0·19	170,000- 135,000	185,000- 165,000	3·5-5·0

The minimum weight of coating is in the range of 0·23-0·38 oz/ft^2 for these wires.

The wires should also pass the wrap test. Extensive trials were carried out to aluminize steel wires to meet the above specifications. Effect of variation of diffusion parameters, i.e. temperature, time of dipping and composition of bath, were studied and the optimum conditions to achieve desired results worked out. With the material sent by M/s. Tensile Steel Ltd, Baroda (C, 0·63; Mn, 0·61; P, 0·034; S, 0·023 and Si, 0·21), the specification obtained was as follows:

- (i) 173,543 psi UTS with 4·37 per cent elongation for 0·119 in. dia. in the 'as aluminized' condition; or
- (ii) 184,484 psi UTS with 2·19 per cent elongation for 0·116 in. in the drawn condition against the specification of 180,000 psi UTS with 4 per cent elongation required vide ASTM B341-63T for this dia. range of wire.

It is, therefore, felt that further improvements could be made by:

- (i) Choosing high tensile steel wire of such composition as would withstand aluminizing conditions, i.e. in the higher carbon range nearer to 0·95 per cent, and similarly having Mn also towards the higher side.
- (ii) Starting with a wire of such mechanical properties as would give an end-product within specification, knowing the change in properties which occurs during aluminizing, could be achieved by drawing practice or a suitable heat treatment, i.e. proper air- or lead-bath patenting.
- (iii) Alternatively, while formulating IS specification, the property requirements could be somewhat modified as there is always sufficient margin in design considerations.

P & T Department

For field trials at different sites in India on telephone and telegraph lines, 350 kg. of aluminized steel wire has been supplied to the P & T Department.

The General Manager, P & T Workshops, Calcutta, has since applied for license to NRDC for commercial exploitation of NML process initially for pole-line hardware at Alipore, Calcutta, and Jabalpur (M.P.) A detailed questionnaire was answered to enable P & T Department to prepare a Project Report. P & T Department also sponsored a small-scale trial at the works of one of NML licensees for aluminizing of Hamilton Poles.

Railway Electrification Hardware

Two hundred and twenty-six assorted angles and welded hardware items thereof were aluminized for Railway Electrification Project, S.E. Rly, for field trials which are currently in progress. Accelerated salt spray corrosion tests in comparison with galvanized materials showed that aluminized hardware will have several times the life of galvanized hardware.

High-tensile Transmission Towers

One hundred and thirth-eight aluminized angles were supplied to M/s. Kamani Engineering Corporation for the construction of a test tower at their Jaipur site. Some of the angles were quenched immediately after aluminizing to obtain a matt surface suitable for immediate painting without having to wait for natural weathering.

Bucket Aluminizing

One hundred bucket samples were aluminized for the Bucket Manufacturers' Association, Calcutta, and 50 for Uttar Bihar Veapar, Muzaffarpur. Practical demonstration of bucket aluminizing was given to our licensees.

Auto-mufflers

M/s. Premier Automobiles Ltd, Bombay, have been conducting extensive field trials with aluminized argon arc welded Fiat 1100 mufflers supplied by NML.

Button Chaplets

Tinned button chaplets are at present employed in foundries for supporting cores in moulds. Sample chaplets were aluminized for G.S. Syndicate Calcutta. Obtaining a smooth coating in aluminized chaplets as in tinned ones was not possible. Smoothness and surface appeal should not, however, be the only criteria in changeover with possibilities of saving foreign exchange spent on import of tin.

Sheet Aluminizing

Three hundred and sixty mild steel panels, 6 × 4 in., were supplied to Metals Research Committee, Jamshedpur, for atmospheric corrosion field tests at 12 different sites in India. Two to three thin coatings from commercial aluminium and silicon containing baths were given. These will be exposed as such, as well as, after various schemes of painting.

90.0 Refractories Pilot Plant

Forsterite Project

Work done on this project has indicated that it is possible to process serpentine to make forsterite refractories by a one-stage process provided serpentine powders are properly graded and blends of high-packing density are used. Magnesite additions in such bodies should preferably contain certain proportions of light calcined magnesia. Fabrication pressure of the order of 10,000-14,000 psi appears to be optimum for these mixes. The results confirm that on a large scale the two-stage process developed earlier also yield good compacts of satisfactory properties although the process is expensive both in the capital outlay as well as in process costs. On the basis of the above process, a supply of 6-7 tons of forsterite blocks, hand- as well as air-rammed, was made for one of the public sector steel plants to test the products under actual service conditions. These bricks were placed in open-hearth checkers under actual service conditions along with other imported bricks. Comparative properties of NML forsterite blocks with that of imported ones as reported before and after use, are as follows:

Number of heats ... 194
Average temperature ... Maximum 1350°C.
Minimum 850°C.

	Foreign 1	Foreign 2	NML
1. Porosity %	24·20	22·95	30·80
2. Bulk density g./cc.	2·54	2·73	2·37
3. Specific gravity	3·36	3·54	3·38
4. P.L.C. at 1550°C. (2 hours)	0·127	0·275	0·04
5. Cold crushing strength kg./cm.^{2}	291	365	348
6. Hydration (steaming for 12 hours):			
(a) Cold crushing strength (kg./cm.^{2}) before hydration	291	270	279
(b) Cold crushing strength (kg./cm.^{2}) after hydration	311	260	277
7. Tests on used bricks: F2 — 194 heats			
(a) Porosity	27·50	—	33·20
(b) Cold crushing strength (kg./cm.^{2})	114	—	114

Above data shows that NML forsterite bricks showed fairly satisfactory performance in the checkers of the open-hearth furnace. They are comparable to the imported bricks which were placed along with them in the open hearth checker.

Trials with Mysore Dunite

Part II of this work was to assess the suitability of various basic rocks for the production of forsterite refractories. For this purpose dunite rock from Mysore was obtained for experimental purposes.

Dead burnt magnesite from Salem area was used. Sulphite lye of specific gravity (as received) 1·55 g./cc. was employed. After preliminary studies on the dunite rock from Mysore, 3-4 tons of dunite rock (5-6 in. lumps) were crushed in jaw and roll crushers to pass through 7 mesh B.S.S. Dunite rock was sampled after crushing, and chemical analysis and sieve fractions were determined. One hundred parts of dunite, was incorporated with suitable quantity of dead burnt MgO, 6 parts of sulphite lye and 4 parts of water. Simpson Muller Mixer was used for mixing purposes. Mixing time was 20 minutes. Mixing was considered complete when moisture distribution throughout the mass was uniform. Blocks of size 15 × 4½ × 3 in. were hand-rammed, dried in tunnel dryer and fired at 1600°C. in D.D. Kiln with 5 hours soaking.

Using jaw crusher for primary crushing and roll crusher for secondary crushing, and passing the over-size again through the roll crusher, the sieve fractions of the rock are given in Table 105 and chemical analysis in Table 106.

Table 107 shows physical properties of the forsterite blocks made from dunite and fired at 1600°C. These blocks were taken from various parts of the Down Draft Kiln after firing and represented as follows:

F — Bricks from front part of the kiln
M — Bricks from middle part of the kiln
B — Bricks from back part of the kiln

Table 105 — Sieve Analysis

Sieve B.S.S.		Sieve fraction %
	+14	1·0
−14	+25	29·9
−25	+52	26·8
−52	+72	9·8
−72	+100	11·2
−100		21·3
		100·0

Table 106 — Chemical Analysis of Mysore Dunite

	Per cent
SiO_2	31·5
Al_2O_3	0·17
Fe_2O_3	17·9
Cr_2O_3	10·9
MgO	26·1
CaO	1·3
Alkalis	N.D.
Loss on ignition	10·9
TiO_2	N.D.

Table 107 — Physical Properties of the Blocks fired at 1600°C.

Sample No.	F	M	B
Porosity (average) %	44·9	44·3	45·3
Bulk density g./cc. (average)	1·99	2·00	1·98

Table 108 — Sieve Analysis of Forsterite Grog

Sieve No.		Sieve analysis %
	+ 7 B.S.S.	5·5
−7	+ 14	18·5
−14	+ 25	21·1
−25	+ 52	18·6
−52	+ 72	7·1
−72	+100	8·9
−100		20·3
		100·0

These blocks were crushed through jaw and roll crusher to make grog to pass 5 mesh B.S.S. Sieve analysis of the gorg is given in Table 108.

Packing density of the forsterite grog and ball mill fines is given in Table 109.

As can be seen from the tables, the maximum packing density of 2·52 g./cc. was obtained during packing studies. Forsterite blocks were made with the above mix using 5 per cent sulphite lye and 4 per cent water. These blocks were air-rammed at a pressure of 40 psi and fired at 1600°C. with 5 hours soaking. Physical properties of these blocks are shown in Table 110. Fig. 59 is a photograph of Down Draft Kiln loaded with forsterite blocks after firing at

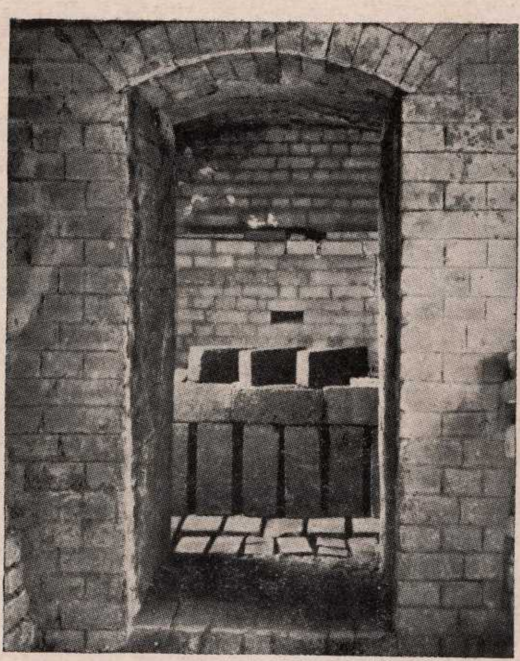


FIG. 58 — DOWN DRAFT KILN, DESIGNED AND FABRICATED AT THE NATIONAL METALLURGICAL LABORATORY, IS LOADED WITH FORSTERITE BLOCKS AFTER FIRING AT 1600°C.

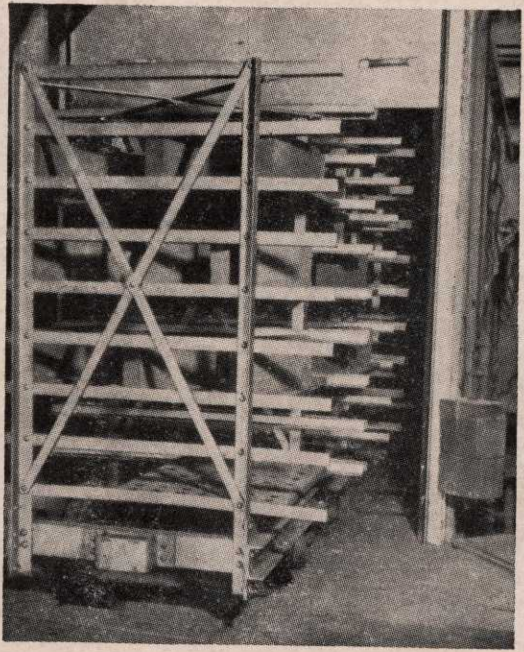


FIG. 59 — FORSTERITE BLOCKS DRIED IN TUNNEL DRYER, DESIGNED AND FABRICATED AT THE NATIONAL METALLURGICAL LABORATORY, BEFORE LOADING IN DOWN DRAFT KILN

Table 109 — Packing Density of Forsterite Grog and Ball Mill Fines

Sl. No.	Forsterite grog −5 mesh	Ballmill fines −60 mesh	Packing density g./cc.
1	90	10	2·353
2	85	15	2·406
3	80	20	2·443
4	75	25	2·490
5	70	30	2·525

Table 110 — Physical Properties of Forsterite Blocks (Two-stage Process) fired at 1600°C.

Sample No.	Porosity, %	Bulk density g./cc.	Modulus of rupture psi
1	31·9	2·37	480
2	32·0	2·38	445
3	33·0	2·35	518
4	31·2	2·40	522
5	31·4	2·40	562
6	31·4	2·38	481

1600°C. Fig. 59 shows forsterite blocks dried in tunnel dryer before loading in Down Draft Kiln.

91.0 Pilot Plant for Production of Synthetic Cryolite

A pilot plant for the production of 50 kg. of cryolite per day by fluoboric acid process is being set up in the NML.

The building for the pilot plant is ready. The specifications and drawings of the machinery were prepared in the Laboratory and the purchase of the equipments is in progress. The installation of the structural part of the plant was completed. A 220 V electric main was installed. The steam line drawings were prepared according to the Indian Boiler Rules Specifications for the extension of the steam line from the existing electrode boiler to the Cryolite Pilot Plant.

RESEARCH GROUPS

DURING the period under review, the various Research and Development Groups maintained satisfactory progress in their specialized fields of metallurgical discipline. The scope of these Research and Development Groups is fairly wide and flexible to suit the pattern of specific objectives and requirements of the industry. These groups also prove useful in the training of graduate and trade apprentices that are recruited for a two to three years' training programme at the National Metallurgical Laboratory.

High Temperature Creep Research Group

High Temperature Creep Research Group has recently been formed at the National Metallurgical Laboratory to provide the industry with the basic data and information on the creep and rupture properties of the conventional high-temperature alloy for design purposes and to promote research and development work on high-temperature creep-resistant alloys based on maximum effective use of indigenous alloying elements.

Extensive work was done at the NML on Cr-Mn-N austenitic steel developed as substitute for 18 Cr 8 Ni stainless steel. Based on this Cr-Mn-N austenitic steel, further work was initiated to evolve suitable composition and heat treatment to obtain optimum high-temperature creep strength and rupture ductility in these alloys. To study the effect of different amounts of C and N on the high-temperature creep properties, steels with a range of C varying from 0·08 to 0·47 and N varying from 0·50 to 0·32 with corresponding C : N ratio varying from 0·16 to 1·47 were taken. The steels were obtained as 4 in.^{2} ingots, cast from heats made in a 0·8 ton arc furnace. These were hot-forged from 1180°C. to $1\frac{1}{2}$ in.^{2} bars — finishing temperature ranging 900°-950°C. Creep test specimens were machined from these as forged bars. In a few cases a prior treatment at 750°C. for a couple of hours had to be given for better machinability. The specimens were solution-treated at a range of temperature varying from 1050° to 1200°C. The high temperature creep tests were conducted at 650°C. and at three stress levels, viz. 15, 20 and 25 kg./mm.^{2}.

A set of creep curves, drawn for one of the steels with lower C and higher N content and tested at 650°C., and a range of stress are given in Fig. 60. From the nature of the curves it is seen that the tertiary stage of creep starts from quite early part of the creep life of the material. This of course is associated with good rupture ductility in the material. The effect of solution-treatment temperature on the creep properties and a steel with higher C and lower N content has been shown in Fig. 61. In this curve creep rupture time, min. creep rate and rupture elongation are plotted against the solution-treatment temperature. The test temperature and stress were kept same in all cases, viz. 650°C. and 15 kg. per mm.^{2}, respectively. It is evident from these curves that solution-treatment temperature has a strong influence on the creep properties especially rupture ductility. The optimum solution temperature range appears to be from 1050° to 1100°C.

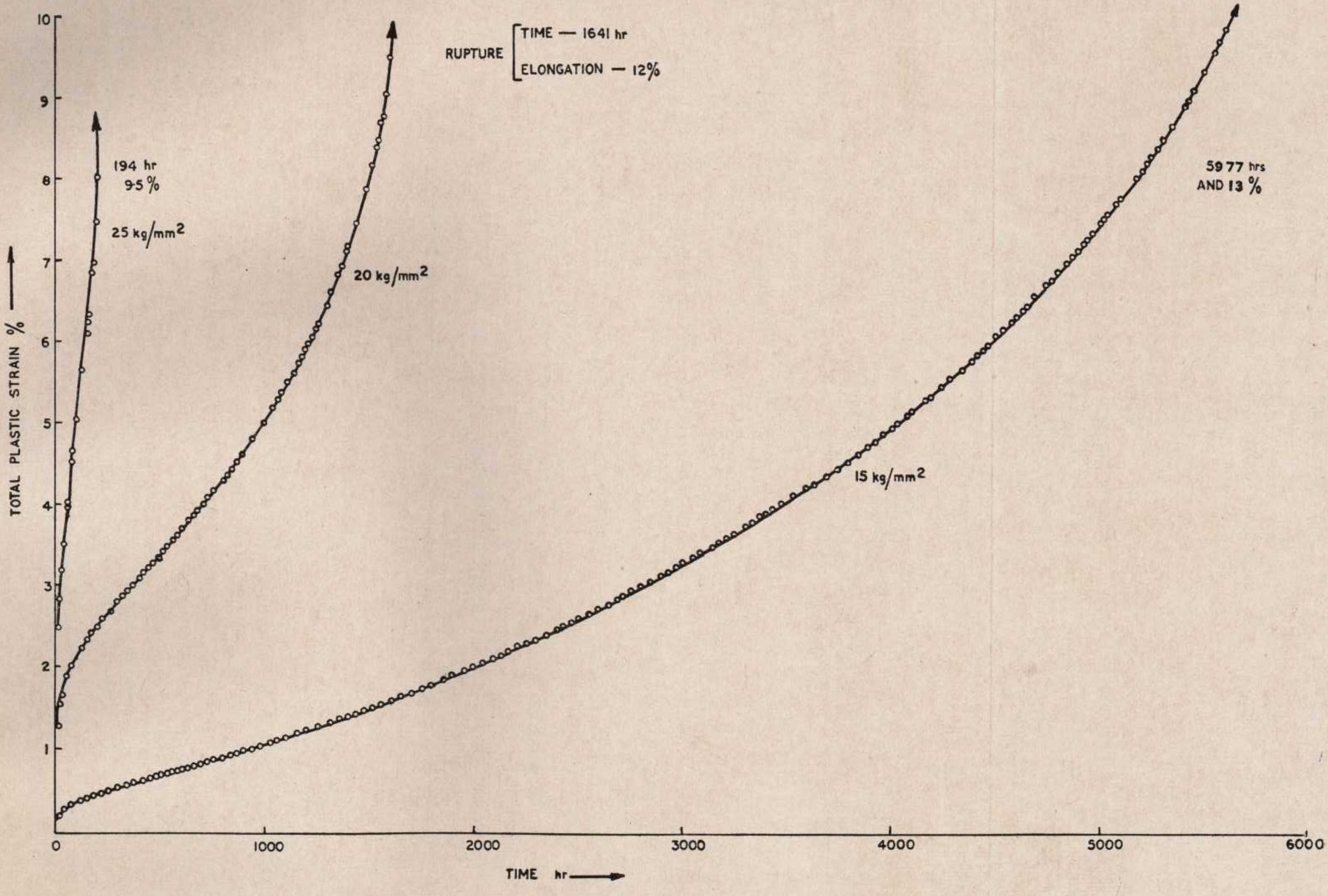


Fig. 60 — A Set of Creep Curves of Low-carbon Austenitic Steel at 650°C. at Various Stress Levels

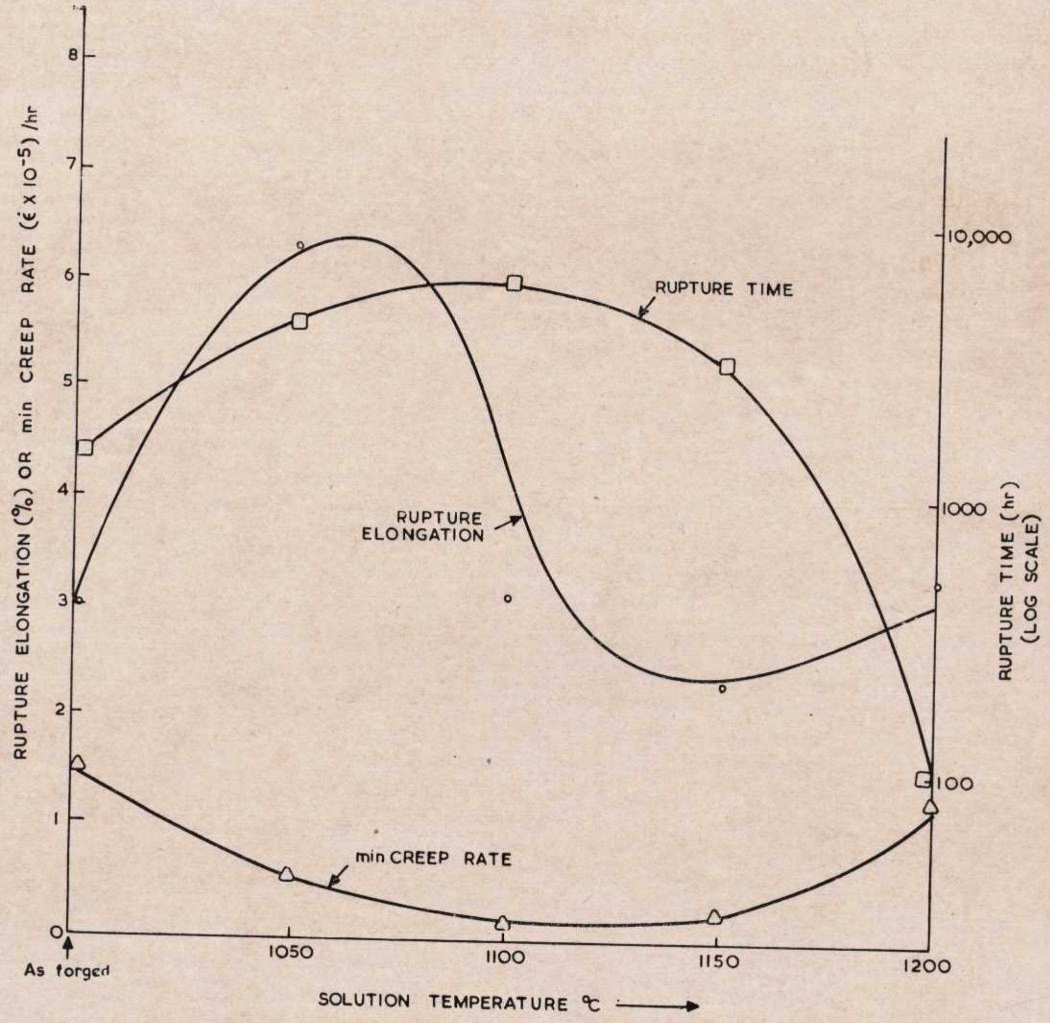


FIG. 61 — EFFECT OF SOLUTION TEMPERATURE ON CREEP PROPERTIES AT 650°C./15 KG. PER MM.^{2}.

Effect of relative proportions of C and N on the high-temperature creep rate was studied. The minimum creep rates, examined against C : N ratio were obtained during test at 650°C. and 15 kg./mm.^{2} stress. As C : N ratio increases, minimum creep rate decreases first at a relatively faster rate and finally the rate slows down. At above about 1:1 ratio of C:N there is not much reduction in the min. creep rate. From the microstructure (Fig. 62) of the creep tested specimen from steel with lower carbon and higher nitrogen it is evident that the mode of precipitation is strongly stress-dependent and the precipitates grow preferentially, in a direction transverse to that of loading. Steels with higher N and lower C contents were observed to be more prone to this type of influence. Further work is in progress to study the creep behaviour of this group of

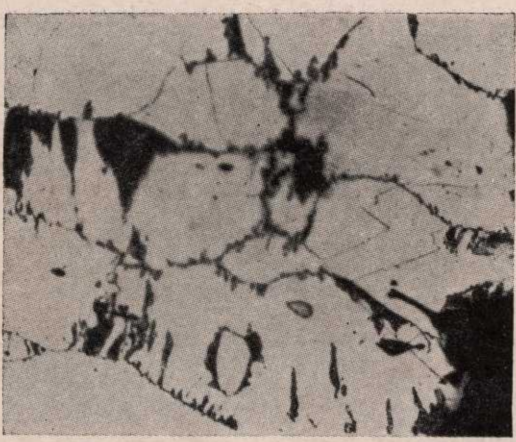


FIG. 62 — PRECIPITATES DURING CREEP AGING GROW PARALLEL TO THE FRACTURE FACING TRANSVERSE TO DIRECTON OF STRESSING

steels at still higher temperatures and a range of stress to provide sufficient creep data for extrapolation purposes.

Electrical Furnace Development Group

The object of the Group is to evolve suitable refractory cement compositions for use in electric furnaces and also for making suitable designs of furnaces for specific research projects.

During the period under review the Electrical Furnace Development Group conducted design and fabrication of various types of furnaces. Of these, wire wound tube furnaces were found to be of great demand for specific research projects in the Laboratory. Several heating elements of various length and diameter were tried to achieve the required temperature. For temperature up to 1200°C., nickel-chromium (nichrome) and iron-chromium-aluminium (Kanthal) wires were used as heating elements. It was observed that both the wires have a long life at 1100°C. The latter is serviceable at a higher temperature but has low specific resistance. In some cases, the nichrome or kanthal heating elements were wound internally to make tubeless furnaces. By this way the use of alumina tubes, which are at present in short supply in this country, was kept to minimum. For temperatures up to 1500°C., platinum wire was used as heating element. To achieve a still higher temperature, platinum alloyed with 10 to 20 per cent rhodium wire has been used. Platinum and platinum-rhodium heating elements are used in air and deteriorate slowly at the maximum working temperature by recrystallization and volatilization of the element. Furnaces made with 0·8 mm. diameter platinum 20 per cent rhodium wire wound on a porous alumina tube and covered with a thin layer of 98 per cent pure alumina cement gave satisfactory results at a temperature of 1650°C. A number of such furnaces are in use in the Laboratory.

Radio-isotope Research Group

A Radio-isotope Research Laboratory has been started in the NML with a view to use radioactive isotopes as tools in metallurgical research problems such as grain boundary and surface segregation of metals in alloy systems, friction, lubrication and wear studies, zone melting of metals, corrosion of metals, electro-deposition, mineral ore dressing, radio-activation analysis for determination of the minute quantity of elements (10^{-8} to 10^{-12} g.), etc. Radio-active isotopes will also be used for industrial studies like study of processes in blast furnaces and ancillary metallurgical equipment, studies on the refining and casting of steel, measurement of wear of blast furnace linings, production control using thickness gauges, etc. Necessary specimens for radio-tracer studies in the Radio-isotope Laboratory would be irradiated in the atomic reactor piles available with the Atomic Energy Establishment, Trombay. With the above objectives in view, the setting up of the Radio-isotope Laboratory made considerable progress.

Following electronic instruments, accessories and materials were set up for the Radio-isotope Laboratory.

Electronic Instruments

1. Automatic sample changer (Type PW 4001/00).
2. 2·5 kV. supply/preamplifier (Type PW 4022/00).
3. Preset timer (Type ET 450 A).
4. Quenching unit (Type PU 630).
5. Gamma dose attenuation calculator (Type G 1650).

Accessories

1. Universal lead castle suitable for almost all types of G.M. tubes, scintillation and proportional counters.
2. K-600 Kompak with base frame assembly and cell.
3. Stainless steel planchets (Type P 1850).
4. Shelves for samples or absorbers.
5. Support frames for geiger counters.
6. Standard rack for gamma ray spectrometer.
7. Lead discs (Type PW 4123/00).
8. Sample slides assembly (Type 4122/00).
9. A set of 25 stainless steel sample pans (Type PW 4135/00).
10. Lead rings forming the central portion of the lead castle (Type PW 4123/00).
11. Polythene storage bins.

Materials

1. Rabialac plastic emulsion paint.
2. Barico Anticorrosive paint.
3. Linoleum sheets (Type P-30, plain red).
4. Adhesive for laying of Linoleum sheets.

The percentage of U_3O_8 equivalent in the active ore samples received from the Ore Dressing Division of the Laboratory was determined using the standard samples from Atomic Energy Establishment, Trombay, with the Geiger counting set up. Two Beta-Gamma Boxes required for the tracer studies in metallurgical research problems, were fabricated in the Laboratory. The fabrication of the necessary gloves, glove boxes, fume hoods and isotope safe for the storage of active specimens is in progress.

Arrangements for the disposal of the high level active liquid and solid wastes, low level active liquid and solid wastes and low level active wastes which would result from the tracer studies and for the film badge service required for the workers in the Radio-isotope Laboratory, had been made in consulation with the Atomic Energy Establishment, Trombay. A plant room for the ventilation system required for the Radio-isotope Laboratory had been constructed. Installation of the proper ventilation plant is nearing completion. The installation of an air-conditioning unit for the conditioned air required for the non-active area and the counting room, the painting of the walls of the Radio-isotope Laboratory with high finish plastic emulsion paint and the laying out of linoleum sheets on the floor of the active area of the Radio-isotope Laboratory, are in progress. Figs. 63 and 64 show the automatic sample changer and fabricated Beta-Gamma Boxes.

Standard Metals Development Group

Preparation of Standard Cast Iron Sample

For the preparation of standard samples of cast iron it was felt necessary to carry out investigation for finding out the distribution of the constituents in cast iron millings. The ingot of cast iron for this purpose was cast and after removing about 5 mm. all round the surface, millings were obtained in a lathe. The millings were

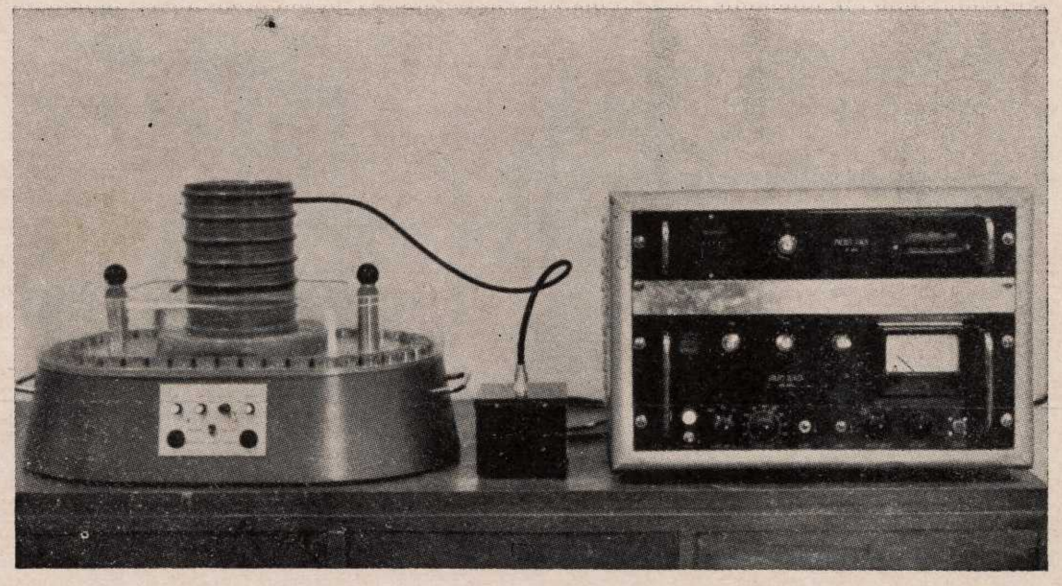


FIG. 63 — AUTOMATIC SAMPLE CHANGER FOR RADIO-ISOTOPE STUDY

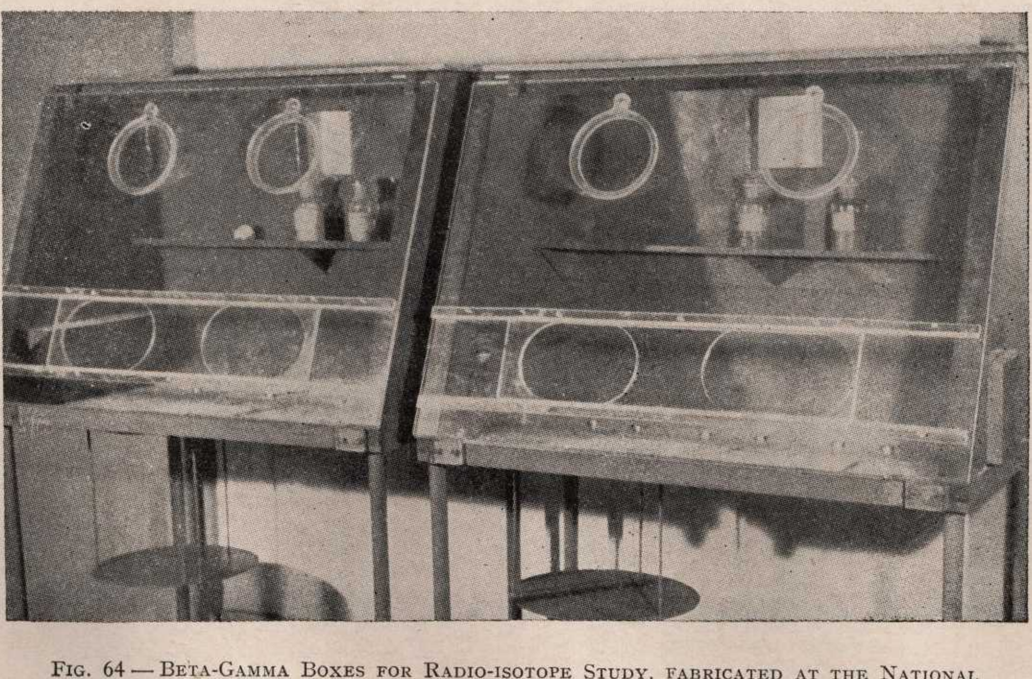


FIG. 64 — BETA-GAMMA BOXES FOR RADIO-ISOTOPE STUDY, FABRICATED AT THE NATIONAL METALLURGICAL LABORATORY

sieved in a 10 mesh sieve and the +10 mesh fraction was crushed till all of them passed through the sieve. The combined millings were thoroughly mixed and sieved successively through 18, 25, 44, 72 and 100 mesh sieves to obtain six fractions (f_1-f_6), viz. −10+18, −18+25, −25+44, −44+72, −72+100 and −100. Representative samples of the different sieve fractions were analysed for C, Si, S, P and Mn content to find out the variation in distribution of these constituents. The results obtained are given in Figs. 65 and 66. From the results it was found that while S, P and Mn were quite evenly distributed in the first five fractions, C and Si showed marked variation in their distribution. In the case of carbon, it was observed that with increase in the fineness of the millings, the carbon content decreased and in the last fraction increased abruptly. The increase in carbon content in the last fraction is to be expected as all the graphite has accumulated there. In the case of silicon, the distribution was nearly constant for the first three fractions and showed an increase in the last three. This distribution of C and Si may be due to surface effect for linear relationship between the surface area, which is proportional to the square of the mean radius of the aperture of the sieves, and carbon content of the sieve fractions has been observed in the fractions f_2 to f_5 (Fig. 67). Further work on the subject is in progress.

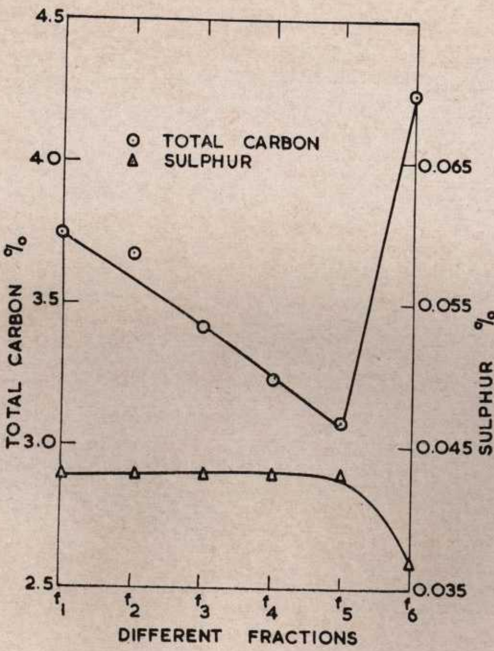


FIG. 65 — VARIATION OF TOTAL CARBON AND SULPHUR AS OBSERVED IN THE ANALYSIS OF DIFFERENT SIEVE FRACTIONS OF CAST IRON SAMPLE

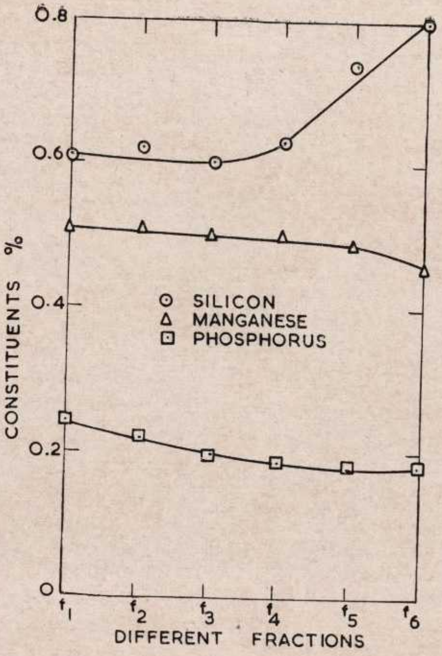


FIG. 66 — VARIATION OF SILICON, MANGANESE AND PHOSPHORUS AS OBSERVED IN THE ANALYSIS OF DIFFERENT SIEVE FRACTIONS OF CAST IRON SAMPLE

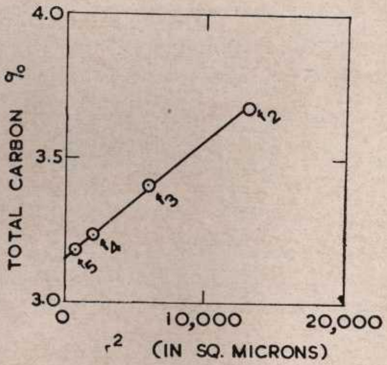


FIG. 67 — RELATIONSHIP BETWEEN SURFACE AREA AND CARBON CONTENT OF SIEVE FRACTIONS

During the year under review, the following standard samples were prepared:

Alloy Cast Iron

About 10 kg. of alloy cast iron was prepared from a casting made in the Laboratory. Proper millings were obtained after scraping out ¼ in. all round the surface of the ingot. Sieving, sampling and bottling were done. The analyses are given below:

T.C.	3·20 per cent
Si	1·66 ,, ,,
Mn	0·80 ,, ,,
Cr	0·58 ,, ,,
S	0·004 ,, ,,
P	0·243 ,, ,,

Standard Steel Sample (0·4 *per cent* C)

About 5 kg. of standard steel sample was prepared from a 2 in. dia. rod. Proper size drillings were obtained from the rod after removing ¼ in. all round the surface. It was sieved through −16 mesh sieve. The (−16) mesh fraction was taken as the proper size. About 9 kg. of (+16 mesh) fraction was being crushed to obtain −16 mesh fraction.

Standard Manganese Ore

About 17 kg. high-grade manganese ore was prepared from the ore collected from the Ore Dressing Division of the Laboratory. It was crushed to 10 mesh, and ball-milled till the whole of it passed through 100 mesh sieve. The sampling and bottling of the ore was done and the analysis was in progress. The results of the constituents determined is given below:

SiO_2	1·52 per cent
MnO_2	84·0 ,, ,,
Mn	56·0 ,, ,,
BaO	0·83 ,, ,,
CaO	trace
MgO	,,
Fe_2O_3	2·80 per cent
TiO_2	trace

Standard Iron Ore (*Blast furnace grade*)

About 30 kg. of iron ore collected from Mineral Beneficiation Pilot Plant of the Laboratory was first crushed to 10 mesh in a crusher and then to 100 mesh in a ball mill by running it for 32 hours. Whole of the sample was sieved through 100 mesh sieve. The +100 mesh fraction was crushed again in mortar to obtain −100 mesh product. It was then properly sampled and bottled. Different laboratories have already been approached to get the sample analysed by them.

Supply of Standard Samples

During this year standard samples were supplied to the following firms on payment:

(a) 200 g. of cast iron to M/s. Addison & Co. Madras.
(b) 100 g. of cast iron to M/s. Metal Box Co., Calcutta.
(c) 100 g. of cast iron to M/s. Arc Electrode, Madras.
(d) 100 g. of cast iron to M/s. R.M. Engineering Works, Ahmedabad.
(e) 200 g. of cast iron to M/s. Sakthi Pipe, Madras.
(f) 100 g. of cast iron to M/s. K.C.P. Ltd, Madras.
(g) 1000 g. steel sample to Hindustan Steel, Durgapur.
(h) 100 g. steel sample to M/s. Sakthi Pipe, Madras.
(i) 100 g. steel sample to M/s. K.C.P. Ltd, Madras.
(j) 90 g. steel sample to M/s. Indian Telephone Industries, Bangalore.
(k) 100 g. of brass sample to M/s. Metal Box, Calcutta.

SUMMARY OF RESEARCH THESES BASED ON WHICH DOCTORATE DEGREES HAVE BEEN AWARDED TO SCIENTISTS OF NATIONAL METALLURGICAL LABORATORY

(i) *Low Nickel and Nickel Substantive-free Stainless Steel*

Although the development of fully austenite Cr-Mn-N stainless steels has been reported, no data are available to show the effects of the manganese content on the properties of such substitute stainless steels. This subject has been studied in detail and the optimum manganese content necessary to yield a stable 17 per cent chromium was established. The beneficial effects of small additions of nickel and copper on the work hardening of these steels on cold working were also examined. As the manganese content was lowered, the tendency of steel to undergo martensitic transformation was noticed and conditions were then investigated which could produce low-nickel steels comparable in mechanical properties to the 17-7 chromium-nickel precipitation hardening stainless steels. The equation

$$0{\cdot}12(\%\ \mathrm{Cr} - 13{\cdot}8) = (\%\ \mathrm{C} + \%\ \mathrm{N}) + 0{\cdot}05\%\ \mathrm{Ni}$$

derived from phase data has been shown to be related to the quantities of ferrite-forming element chromium with the austenite forming elements carbon, nitrogen, nickel and copper, to give the phase relationship at 1050°C. It has further been shown that the excess of the ferrite-forming element over austenite-forming elements can be quantitively related to the amount of ferrite present in the steel at 1050°C. by the equation

$$\%\ \text{delta-ferrite} = 95[0{\cdot}12(\mathrm{Cr} - 13{\cdot}8)] - [(\%\ \mathrm{C} + \%\ \mathrm{N}) + 0{\cdot}05\%\ \mathrm{Ni}]$$

The mechanical properties of steels containing varying amounts of manganese were determined. The coefficient of thermal expansion for a steel containing 12 per cent manganese was determined and found to be of the same order as 18-8 Cr-Ni steel. The deep drawing properties of several steels were determined in Mohr and Federhaff and in Swift cupping testing machines. The beneficial influence of nickel on deep drawing properties has been reported. The corrosion resistance of several steels, with varying manganese contents, was determined in 65 per cent boiling nitric acid and it has been shown that both the manganese content and the presence of delta-ferrite in small quantities have no significant effect on corrosion rate. The effect on hardness after cold working delta-ferrite-free steels containing varying amounts of manganese showed that as manganese decreased from 12 to 6 per cent, hardness increased for the same amount to cold working. The effect of the quantity of delta-ferrite in reducing hardness obtained after cold working is shown for 8 per cent manganese steels. It is further shown that, though ferrite decreases the hardness after cold working 6 per cent manganese steels, no clear relationship exists between the amount of delta-ferrite and the hardness obtained in cold working as in the case of 8 per cent manganese steels. The effect of nickel and copper on decreasing the hardness on cold working and on conferring resistance to tempering after cold working was noticed. The superiority of 8 per cent manganese steel over 6 per cent manganese steel, considering all the mechanical properties, in cold-rolled condition has been shown. It was noticed that no significant increase in strength occurred on liquid air quenching solution-treated steels, though some increase occurred in 30 per cent cold-reduced steels after this treatment. This shows that sub-zero treatment to increase the strength of this type of steels in annealed condition will not be effective. On tempering the 30 per cent cold-reduced and liquid air-quenched specimens of 6 per cent manganese steels the elongation was found to drop to 0 per cent at 500°C. On aging solution-treated steels containing, 6 per cent manganese at 750°C. for 14 hours, the steels were found to have excellent properties. For this treatment to be effective, about 6 per cent ferrite and 1 per cent nickel were found to be essential. The isothermal transformation curve for a 12 per cent manganese steel was determined and the pearlite-like structure obtained was noted. Welding trials and sensitization tests showed that steels having more than 0·03 per cent carbon are subject to weld decay, but those having carbon content of this order or less were not subject to this defect. Metallographic study of the various structures obtained during the course of this investigation has also been made. X-ray diffraction study of two 18 per cent manganese steels after solution treatment and 30 per cent cold working was carried out. In similar conditions, 18-8 Cr-Ni steels were also tested for purposes of comparison. It has been shown that while 18-8 Cr-Ni steel did partially transform to martensite on cold working, no such transformation was observed in the Cr-Mn-N stainless steels.

(ii) *Ion-exchange of Clays with Special Reference to the Effect of Exchangeable Cations on Thermal Behaviour of Kaolinite*

This work has covered the study of the effect of exchangeable cations on thermal changes of kaolinite based on differential thermal analysis. Exchangeable cations significantly influence the low-temperature properties of clays, viz. plasticity, workability, viscosity of clay slips and drying behaviour of clay-water bodies, but little work has been reported on the thermal behaviour of clays. Three kaolinites of varying crystallinity and base exchange capacity were chosen and the effects of some monovalent and divalent cations such as Na^+, K^+, Li^+, Ca^{++}, Mg^{++} and Ba^{++} were studied on their thermal behaviour. The thermal reactions of the three kaolinites were followed up to 1350°C. by differential thermal analysis; changes in various reaction peaks were corroborated, wherever necessary, with thermogravimetric and X-ray diffraction data.

Of the three kaolinites examined, Tranvancore China clay was relatively most well crystallized and ball clay was a b/3 disordered type kaolinite. Rajmahal ranked intermediate in crystallinity. The three kaolinites varied in crystalline impurities and the mineralogical composition of the clays was calculated by Norm method keeping in view the X-ray data on some crystalline impurities. In the *p*H-neutralization study of the hydrogen forms, Travancore China clay exhibited two inflexion points whereas others showed only one point; such difference in behaviour has been examined. The 'bec' of the clays was determined by three different methods with a view to compare different techniques.

There was no appreciable change in the peak temperatures of the dehydroxylation reaction with ion saturated, but the cations affected the peak shapes. Hydrogen gave the highest amplitude, area and slope ratio of the peak and barium gave the lowest values for these factors. Even though no rigid order existed between the various cations, closer examination revealed that Na^+, especially of monovalent cations, tended towards hydrogen and divalent cations appeared to group together. The changes in the peak shapes were considered to be due to differences in rate of dehydroxylation.

The exothermic peak in the region 950°-1000°C. was greatly affected by various cation, Na^+ and Li^+ decreasing peak intensity and H^+ and divalent cations in general increasing it. The reaction peak was considered to be due to rapid formation of spinel phase within a temperature range; the spinel phase was considered to be identical with the one reported by Brindley and coworkers in view of the similarity of its crystal size and the nature of its development from metakaolin as revealed by electron microscopic studies. The change in peak intensity with the various cations was considered to be due to particle flocculation or deflocculation brought about by cations in the original clay. With a view to explain the changes in peak intensity with ion saturated, relative proportions of spinel and mullite phases in the various samples heated at 1000°C. for 20 hours were assessed semiquantitatively in a manner similar to that of Baker and coworkers. It was considered that the variation in relative proportion of spinel and mullite phases in accordance with peak intensity was coincidental and may not have any direct bearing on peak intensity.

A single exothermic peak was observed above 1000°C. in Rajmahal and ball clays and no peak was observed in the case of Travancore China clay. The peak was considered to be due to rapid formation of mullite on the presumption that kaolinites which gave rise to rapid formation of mullite would exhibit exothermic peaks above 1000°C. whereas the peak may not be detected if there was progressive development of mullite without rapid formation within a particular temperature range. The peak was not due to cristobalite formation since there was absolutely no change in the peak shape of Na^+ and K^+ forms of ball clay even though the phase was completely undetected by X-rays in these cationic forms.

A detailed study of the effect of cations on high-temperature phases in ball clay revealed that the cations had no mineralizing effect on mullite formation. The cristobalite phase was very much affected by cations and the variation in intensity of the characteristic lines of cristobalite in X-ray diffractograms was considered to be due to the degree of disorder brought by the cations on cristobalite lattice. Perhaps, differences in the degree of disorder caused by the various cations may lead to changes in the X-ray intensity reflected by disordered cristobalite. 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^+ forms, and hence complete non-appearance of cristobalite phase, cannot be ruled out.

(iii) *Studies on the Phase Transformation in Alloys by X-ray Diffraction and Other Techniques*

These studies relate to mechanism and kinetics of precipitation reactions involving phase transformations in alloy steels when subjected to various heat-treatments and/or deformation under severe stresses.

Detailed investigation carried out on the transformation characteristics of carbides in low chromium steels, based on X-ray structural analyses and metallographic examination of the isothermally treated steel samples, revealed the following features.

It has been established that Fe_3C is the initial carbide formed during isothermal transformation at all temperatures ranging from 250° to 700°C., which has been shown to transform to Cr_7C_3 and then to $Cr_{23}C_6$; the reaction continues even after the completion of the decomposition of austenite. The type of the final carbide formed has been shown to be the function of both chromium content and chromium : carbon ratio in the steels. On the basis of changes observed in the lattice parameters and chemical composition of carbides, it has been shown that transformation of Fe_3C to Cr_7C_3 or Cr_7C_3 to $Cr_{23}C_6$ is by general enrichment of Fe_3C or Cr_7C_3 lattice by chromium. The maximum chromium content in Fe_3C has been shown to be nearly 27 per cent and that of Cr_7C_3 to be nearly 65 per cent.

The other parts of the studies relate to the effects of subcritical aging and cold working on the stability of austenitic phase and/or formation of other phases in chromium-manganese-nitrogen-carbon steels. It has been established that as in 18 per cent chromium and 8 per cent nickel austenitic stainless steels, austenite to ferrite transformation takes place during cold working in chromium-manganese-nitrogen-carbon steels. The crystal structure and the lattice parameter of the precipitated phase have been examined and were found to be the same as those of ferrite. During the subcritical aging treatment, Cr_2N and $Cr_{23}C_6$ types of precipitates were identified, depending on the temperature of aging, and carbon and nitrogen contents of the steel studied. The relation between C:N ratio in steels and the different equilibrium precipitates (i.e. Cr_2N or $Cr_{23}C_6$ or both) at different temperatures of aging has been determined. Intersolubility of various alloying elements (i.e. Fe and Mn) in the Cr_2N type of precipitate with time and temperature of aging have been determined by X-ray spectrochemical analysis. The amounts of these various alloying elements in the precipitate have been shown to be a function of time of aging at any particular temperature. The activation energy of the formation of Cr_2N type of precipitate in chromium-manganese-nitrogen steels during aging has also been determined.

(iv) *Studies on Age-hardening*

Studies were carried on aging of industrially important aluminium-copper and iron-nickel-aluminium permanent magnet alloys.

It has been shown that natural aging of aluminium-copper alloys is conditioned by the concentration of quenched-in vacancies rather than quenching stresses or dislocation channels as postulated by other workers. The greater the concentration of the vacancies retained during quenching, the greater will be the acceleration and extent of first stage of aging in which Guinier-Preston zones develop. Any process which reduces the number of quenched-in vacancies will also retard zone formation and, therefore, hardening of the alloy. On the basis of these investigations, it has been possible to rationalize the effect of quenching medium, specimen size, interrupted quench and plastic deformation on the aging and formation of Guinier-Preston zones. Increasing specimen size, quenching medium of poor heat-abstracting capacity and interrupted quench will invariably lead to slow rate of aging as most of the vacancies are likely to be lost during slow cooling of the specimens. Slow rate of aging after cold deformation has been explained on the basis that extra dislocations produced during deformation act as skins for vacancies. The vacancy hypothesis has further been able to clear many controversies connected with dissipation of hardness acquired during aging, on reversion treatment. It has been shown that such dissipation or softening takes place in two stages. The first stage of softening is associated with evaporation of G.P. zones through trapped vacancies and takes place between 150° and 170°C. The second stage of softening is due to dispersal of dislocation loops formed from quenched-in vacancies and takes place between 180° and 200°C.

The investigation on iron-nickel-aluminium alloys showed that such alloys also decompose and harden by the same mechanism as aluminium-copper alloys. First iron atoms separate out from the matrix in the form of clusters or zones, which subsequently give rise to intermediate and stable phases. Some evidence of presence of clusters and intermediate aligned phases is provided by the electron-microscopy of these alloys carried out at the Laboratory. It has also been shown by X-ray diffraction studies that the solid solution of iron-nickel-aluminium alloys decomposes into B.C.C. stable phases in a way iron does through formation of ϒ-iron at intermediate temperatures. Hardening and coercive face changes of the alloys as a function of cooling rates, and tempering temperature and time were also studied and explained on the basis that precipitation of critical size is the dominant factor for obtaining good magnetic properties.

(v) *Phase Transformation in Electrolytic Alloys with Special Reference to Copper-Cadmium Alloys deposited at Constant Cathode Potetial*

A comparison of the X-ray powder structures of the thermal and electrolytic copper-cadmium alloys deposited at constant cathode potential over a range of 1·27 to 85·6 per cent copper by weight was made. The phase-boundary limits for the existence of electrolytic alloy phases at ordinary temperatures do not correspond to the thermal equilibrium diagram. The existence of the metastable electrolytic alloy phases in the 'as deposited' state and their subsequent transformation to the stable structures have been explained in the light of the co-deposition of hydrogen into the alloy composition and the rearrangement of the discharged atoms at the cathode from the amorphous unordered state to the stable or metastable phases. These rearrangements are influenced by the cathode potential and are facilitated by the disappearance of the primarily codeposited hydrogen thus leaving vacant gaps in the lattice. The formation of the electrolytic metastable phase is considered in the light of the excess energy associated with the unordered atoms. For each electrolytic phase there is a tolerance limit where it can exist beyond its equilibrium range, if not disturbed otherwise, and this observation has some practical implications.

(vi) *Chemical and Metallurgical Factors affecting Failure of Metals and Alloys under combined Action of Stress and Corrosion*

All metal systems contain certain alloy composition ranges where they are susceptible to stress corrosion cracking under proper environmental conditions. This type of failure occurs when alloys are subjected to the simultaneous action of static tensile stresses and of an electrochemical corrosion process. The cracks developed are propagated in a direction perpendicular to the acting stress and are intergranular or transgranular and sometimes both.

In this work, the objective has been to study the effect of variations in type and amount of alloying elements, viz. zinc, manganese and aluminium, added to copper on stress corrosion cracking susceptibility of resulting alloys. Effects of variations in corrosive conditions, anodic and cathodic polarization, cold working and annealing on cracking time and mode of cracking have been determined for different homogeneous copper alloys. Role of electrochemical factors and stacking fault parameter of alloy systems on the mechanism of cracking has been investigated.

Survey of literature shows that electrochemical factors controlling corrosion of copper alloys have not been studied very extensively. This is due to the fact that in ammonia solution the cracking takes place much more easily in the gaseous phase than in the aqueous phase. Recent studies by Mattsson on the stress corrosion cracking of Cu-Zn alloy containing 37·2 per cent zinc in ammoniacal copper sulphate solution of varying *p*H values between 3·9 and 11·2 have initiated a more elegant approach to the underlying made.

In this investigation more detailed studies on effect of change in *p*H and composition of solution, effect of cold working, annealing, etc., on cracking susceptibility of Cu-36 per cent Zn and Cu-(2-6) per cent Mn alloys have been made.

It has been shown in this investigation that with increase in the amount of copper present in Mattsson's solution, the cracking susceptibility increases. It has been further established that the state in which copper is present in solution, i.e. as Cu or $Cu(NH_3)^{2+}4$, and the case of formation of oxide layer are important in controlling the stress corrosion cracking susceptibility. In the maximum susceptible range of *p*H, copper is present predominantly as $Cu(NH_3)^{3+}4$ and is also associated with the formation of a sufficient amount of cuprous oxide on the surface.

It has also been shown that cracking can be prevented by cathodic polarization. Anodic polarization has been found to increase slightly the cracking susceptibility for Cu-Zn alloy. On the contrary in the case of Cu-Mn alloys anodic polarization at lower current densities increased the cracking time which, however, became constant with further increase in the current density.

Cold working and annealing have been found to have a marked influence on cracking. For Cu-36 Zn alloy, it has been shown that with increase in degree of cold drawing, the cracking time first decreases and then increases with further increase in degree of cold drawing. The minimum in the percentage reduction versus cracking time curve has been shown to lie between 30 and 35 per cent reduction. In Cu(2-6) per cent Mn alloys, however, no such minima were observed at reductions above 16 per cent, which was the range of study in this investigation.

In Cu-Zn alloys, transition from inter- to transgranular cracking occurs above 18-20 per cent zinc and when plastically deformed. In Cu-Mn and Cu-Al alloys also similar (dependence of composition) transition has been shown to occur between 6 and 10 per cent and at 5 per cent, respectively.

The results obtained have been explained in the light of reactivity of structural sites, stress concentration at these sites and stacking fault energy that defines the mode of deformation and creation of barriers in a lattice. Mode of cracking has thus been shown to be dependent on the stacking fault parameter of the alloys studied; alloy composition having lower parameter failed intergranularly and those having higher parameter failed transgranularly.

(vii) *High-temperature Phase Equilibria in Some Refractory Mineral System involving CaO-MgO-Al_2O_3-SiO_2*

Phase equilibria in the quarternary system monocalcium monoaluminate (CA) calcium dialuminate (CA_2), gehlenite and spinel were investigated both in the solid state and above the solidus. The crystalline phases CA-CA_3-gehlenite and spinel form a condensed quarternary sub-system within limiting quarternary system CaO-MgO-Al_3O_3-SiO_2. X-ray analysis of the heated mixtures of these crystalline phases did not indicate any solid solution effects. No new quarternary or ternary phases were encountered in this system either below the solidus or in equilibrium with liquids. Of the six limiting binary systems of this quarternary system, five binary systems such as CA-CA_2, CA_2-gehlenite, CA_2-spinel, gehlenite-spinel and gehlenite-CA showed true binary relationships. The system CA-spinel is not truly binary as CA_2 exists in equilibrium at the liquidus temperature in a portion of the system. The systems CA-CA_2-gehlenite and CA_2-gehlemite-spinel showed true ternary relationships with an eutectic located within the respective composition triangles. In the system CA-CA_2-spinel, the invariant point lies outside the composition triangle. It is a reaction point. The system CA-gehlenite-spinel does not show true ternary relationsips as CA_2 crystallizes as a primary phase in certain portions of the join. Two piercing points are located in this join. Two univariant curves traverse the composition tetrahedron. Along one such curve, CA_2, gehlenite and spinel are in equilibrium with the liquids. Along the other univariant curve, CA, CA_2 and gehlenite are in equilibrium with liquids. The quarternary invariant point lies outside the composition tetrahedron. Its composition is not known, but the temperature of the invariant point is 1475° ±3°C. as indicated by the crystallization of melts in the system CA-gehlenite-spinel.

(viii) *Transformation Characteristics of Alloy Steel*

The Thesis embodies results of a systematic investigation on the effects of addition of nickel, chromium and manganese, either singly or in combination at 0·34 to 0·36 per cent C steel on the isothermal as well as continuous cooling transformation characteristics of alloy steels. A correlation between the isothermally transformed micro-structures and the mechanical properties of a number of alloy steels was established. A study of the cause of the irregularities observed in the intermediate transformation range and a comparison of the mechanical properties of isothermally transformed, and quenched and tempered products were also carried out. In addition, continuous cooling transformation diagrams of a number of alloy steels as well as their detailed microstructural characteristics at different cooling rates were studied. Moreover, continuous cooling transformation diagrams in respect of some alloy steels were derived from their isothermal transformation diagrams.

The experimental techniques employed covered both the dilatometric and metallographic methods, hardness and microhardness measurements, and determination of tensile strength, yield strength, reduction in area and percentage elongation. Detailed microstructural analyses were also carried out with electron microscope, using formvar replicas.

The study had established that in most of the alloy steels investigated, pearlite and bainite reactions overlap in respect of time and temperature in the intermediate transformation range between 450°-550°C. In this range an acicular type of ferrite, designated as 'X' constituent by Devenport, was precipitated. The quantity of this constituent was found to be maximum in chromium steel and minimum in nickel steel. The work had further established that hardness and strength values of isothermally transformed products increase with the decrease of transformation temperature both in the bainite and pearlite range. In the intermediate range, however, each steel behaved differently. In the case of nickel steel, the hardness increased with decrease of transformation temperature; in the nickel-chromium and manganese steels, the hardness failed to increase; whereas in chromium and manganese-chromium steels the hardness decreased considerably showing irregularity in the hardness curve — the extent of irreugularity being maximum in chromium steels. The cause and extent of the irregularities were investigated at higher carbon level, keeping the amount of chromium constant.

The mechanical properties of isothermally transformed structures were compared with those of tempered martensite of comparable strength and hardness. It was observed that tempered martensite exhibited consistently superior properties than the isothermally transformed structures except lower bainite. Continuous-cooling-transformation characteristics of a number of alloy steel were investigated. Oil cooling and air cooling curves of appropriate size bars were determined to test the reliability of equivalent relationship proposed by

Nayer and Steven. It was observed that there was an overall satisfactory agreement between the two sets of curves.

Effect of sub-zero treatment of the continuously cooled specimens was also investigated and it was observed that on treatment at −120° and −180°C. there was an appreciable increase in hardness in chromium, manganese-chromium and nickel-chromium-molybdenum steels at cooling rates, at which considerable amounts of acicular and proeutectoid ferrite or upper bainite had been precipitated. It was also observed that when the specimen was cooled at a rate faster than the critical cooling rate and quenched in water below the Ms temperature, the martensite formed was simultaneously tempered. The effect of alloying elements and cooling rates on such self-tempering was also investigated. C.C.T. diagrams were derived from T.T.T. diagrams on the basis of Schel's theory. It was observed that the calculated ' beginning of transformation ' temperatures were in general higher than those determined experimentally. Progress of transformation was also calculated at 50 and 99 per cent transformation and these were found to be generally lower than that experimentally determined.

ENGINEERING SECTIONS

Design and Mechanical Engineering

Some Major Noteworthy Design and Fabrication Projects completed and in Progress

(i) *Strip Aluminizing Prototype Plant*

Considerable progress was made in the fabrication of many plant items of equipment such as dross shields, uncoilers, drag bridles, pinch rolls and jockey rolls. Work has also commenced on the integrated pretreatment line structure.

The strip aluminizing furnace design was finalized (Fig. 68 and 69) after modifications to accommodate a premelting rotary furnace being procured indigenously. This rotary furnace will supply molten aluminium to the dip-coating furnace thus making it possible to eliminate the necessity of utilizing a cast iron holding pot. A refractory lined pot furnace to hold the molten aluminium will be utilized instead. Plans for an alternate arrangement using a salt-bath type of melting furnace are also being drawn up for evaluation trials.

(ii) *Wire Ausforming Unit*

Detailed design work on this unit has almost been completed. Specifications for equipment to be procured indigenously are being drawn up and finalized for procurement purposes.

(iii) *Zinc Recovery Plant*

Detailed equipment design of a full-scale plant for recovery of zinc from galvanizing wastes continues (Fig. 70) based upon the Engineering Project Report prepared earlier.

(iv) *Tube Aluminizing Prototype Plant*

The overall preliminary design of a prototype tube aluminizing plant was completed. After this is finalized, detailed design of each plant section and item of equipment will be taken up. An actual scaled down working model unit is proposed to be built for mechanical evaluation trials as a preliminary to the setting up of an operational pilot unit.

(v) *Synthetic Cryolite Pilot Plant*

Plant and equipment for this project was selected and orders placed. Erection of equipment and commissioning trials will be taken up on receipt of the equipment (Fig. 71).

(vi) *Fluidized Bed Reactor*

The design of a bench scale Fluidized Bed Reactor was taken up and is nearing completion. This bench-scale reactor will be utilized for iron ore reduction trials.

(vii) *Baby Cupola*

The design of an air-cooled Baby Cupola with a capacity of 500 kg./hr. is under way which will be fabricated for installation in the NML Foundry.

(viii) 1500 *kg. Side-blown Converter*

Following the successful operation of the 500 kg. Side-blown Basic Converter, designed

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g. *Side-blown Converter*

successful operation of the
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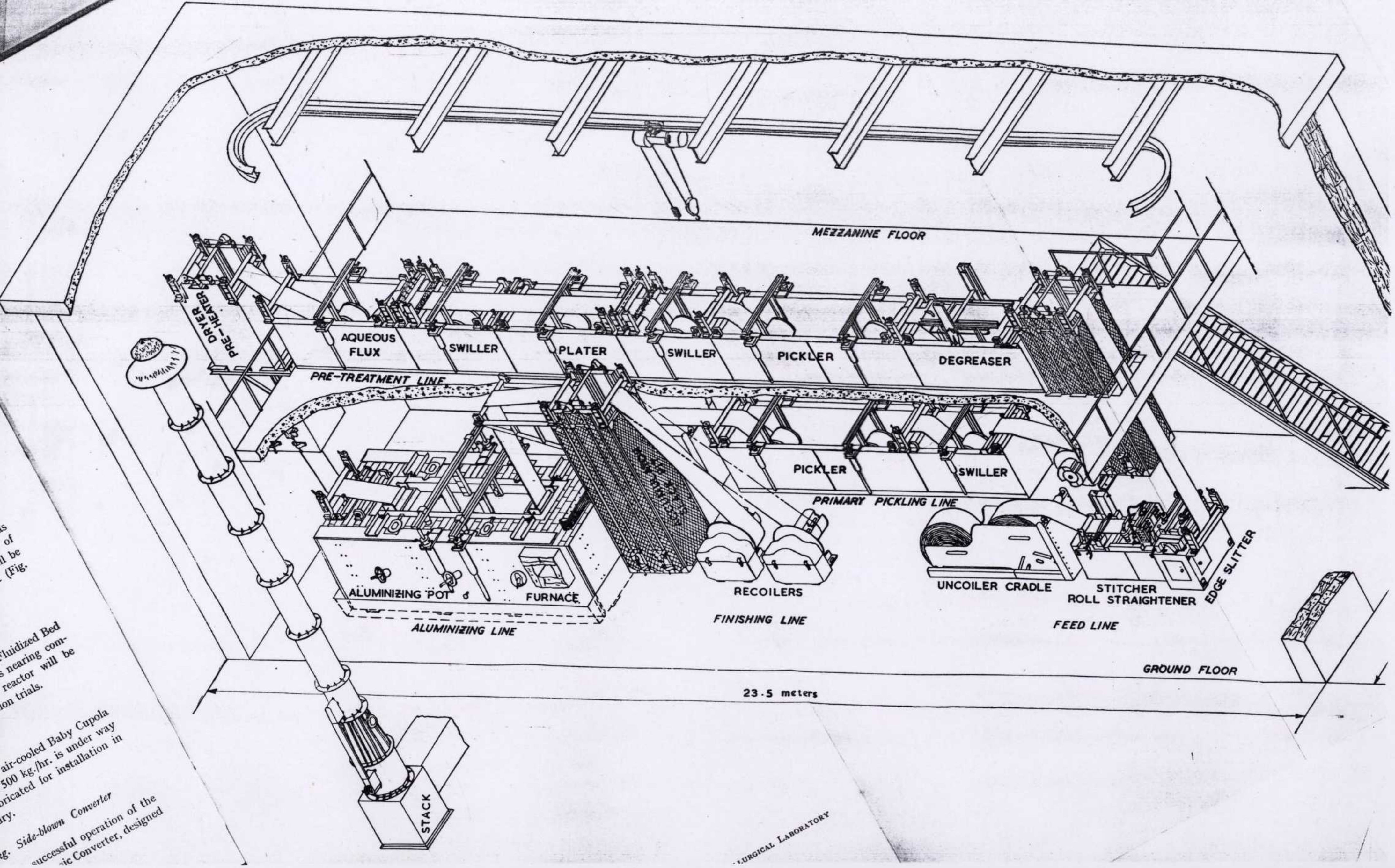


and fabricated in the Laboratory, work was initiated to finalize the design of a 150 kg. Side-blown Basic Steel-making Converter for fabrication and installation in the Steel-making Section at the Low-shaft Furnace Project site.

(ix) *Graphite Crucibles Turn-key Industrial Plants*

Work has been initiated for the complete turn-key projecting of industrial-scale commercial plants producing 600 to 1000 tonnes of carbon-bonded and clay-bonded graphite crucibles for prospective clients.

(x) *Cost Data Correlations*

Cost data correlation was prepared and published. Data collection continues to cover further indigenous items of equipment and industrial products.

(xi) *Development Work*

(a) The development of Hot-wire Anemometers for measurement and control of flow of fluids was initiated in collaboration with the Electronics Section. It is proposed to develop a wide variety of these flow measuring Anemometers for different ranges and flow materials for eventual indigenous commercial exploitation.

(b) The development of specialized novel items of equipment such as Variable Tension Looping Pits for the Strip Aluminizing Prototype Plant continuous.

Research Instruments and Equipment Designed and Fabricated

1. Giesler Plastometer for plasticity measurements of clays, etc.
2. High-temperature Graphitization Furnace for temperatures up to 3000°C.
3. An improved Zinc Vacuum Distillation Prototype Retort with a water-cooled finned type condenser.
4. Peel Strength Apparatus for evaluation of adhesive bonds in plastic-coated steels.
5. D.T.A. Specimen Holder.
6. Perspex Extrusion Press Model for visual evaluation of the flow of extruded materials.
7. Soil Sampling Tube for corrosion investigations.
8. Photo Electric Cell Housing.
9. Wire Uncoiler for wire drawing lines.
10. Swelling Determination Apparatus for evaluation of clays, etc.
11. Thermal Conductivity Apparatus.
12. Rotary Seal for Vertical Reduction Kiln.
13. Transformer Regulator for Underload Furnace.

Research Equipment Design and Fabrication Under Way

1. Electrode Lifting Mechanisms for the submerged arc ferro-alloy and electrolytic magnesium projects.
2. Vacuum Sampling Accessory for sampling metallic distillate vapours.
3. Vacuum Distillation Pot for magnesium powder.
4. Fume Hoods for Radioactive Isotopes (Tracer) Section of NML.
5. Perspex Model of a countercurrent extraction system.

Research Equipment for which the Design and Fabrication are projected and Under Planning

1. Glove Boxes for the Radioactive Isotopes Tracer Section.
2. Circular and Rectangular X-ray Film Punches (normally imported).
3. Vertical Travel Tubular Furnaces.
4. Rotary Furnace for slag and hot abrasion tests.

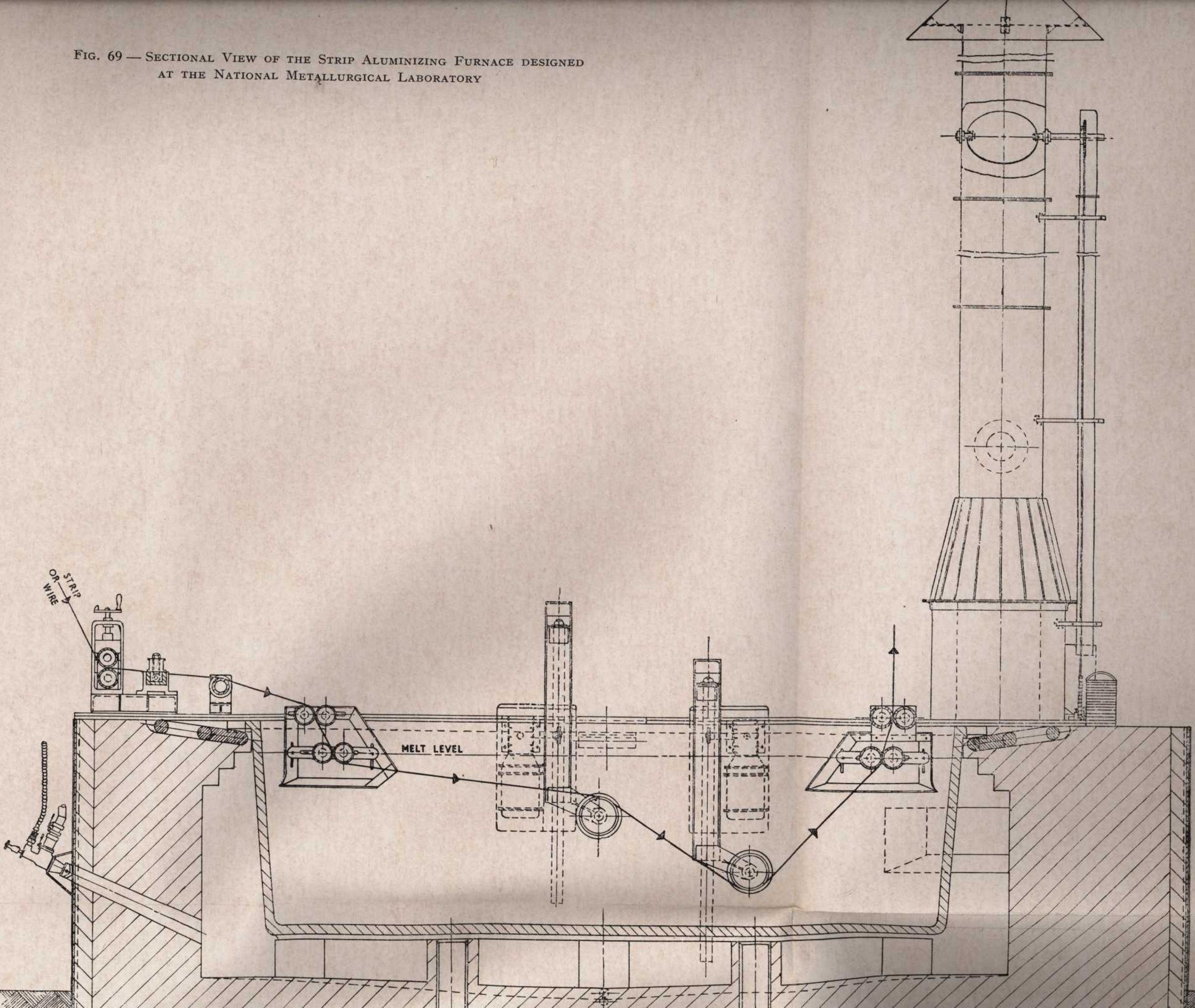


Fig. 69 — Sectional View of the Strip Aluminizing Furnace designed at the National Metallurgical Laboratory

(iii) *Transistorized Portable R.C. Oscillator*

It is used for frequency up to 3 kc/s. It is a common emitter amplifier, based by a voltage divider. It is used as a sinewave power source for measurements. The frequency stability of this oscillator is found satisfactory. It is constructed by arranging the phase shift components to form a wein network. The advantage of this network is that the attenuation factor is only 3 at the frequency which gives zero phase shift. It is a two-stage oscillator using two OC 70/71 transistors. Both stages are d.c. stabilized. A potentiometer of 1 Kohm provides a convenient means of adjusting the waveform for amplitude and distortion, and of compensating for changes in ambient temperature. The wave form and the short-term temperature stability is fairly good. Supply voltages from 6 to 9 V may be used provided the potentiometer is suitably adjusted. The output is taken from the collector.

(iv) *Thermister Thermometer*

An inexpensive, accurate and miniaturized thermister thermometer was developed. With 4 thermisters of 220 ohms each in series, the sensitivity is about 1°C./0·01 ma. A variable resistor is used for selection of temperature range. For protection the thermistor is enclosed in a glass envelope and sealed in paraffin. A potentiometer is used for sensitivity control. It works on the principle of a wheatstone's bridge one arm of which consists of thermisters. It can be used to measure temperatures from $-10°$to $+150°$C. with high accuracy. A battery of 1·5 volts is used for its operation.

(v) *Instrumentation of Mineral Beneficiation Pilot Plant*

The following instruments were installed at the Mineral Beneficiation Pilot Plant site (Fig. 72).

1. Six Bristol Flow Recorders for producer gas plant.
2. Two Bristol Temperature Recorders for sinter plant.
3. Beckman Industrial *p*H Meter with Recorder.
4. Oxygen Analyser with Recorder.
5. Beckman Gas Chromatograph with Recorder.

Several major maintenance jobs were completed during the year under review. These include repair and maintenance of AEI Electron Microscope EM6, Differential Thermal Analysis Apparatus, Electronic Control for Albert Mann Rolling Mill and various types of Temperature Controllers and Recorders.

Electrical Engineering

(i) *High Temperature Graphitizing Furnace*

The work on the design and development of special high-temperature furnaces to meet the specialized research needs in National Metallurgical Laboratory was taken up. In the course of this work, a High-Temperature Graphite Resistance Tube Furnace and its power transformer were designed and fabricated in the Laboratory as reported earlier, with all indigenous components, except the graphite electrode (out of which resister is made). During the period under review, the work chiefly related to the study of power and temperature characteristics of the furnace. The power input necessary to maintain equilibrium condition in the furnace at various temperatures was determined and is shown in Fig. 73. It was found that 10·9 watts per cm.^{2} was necessary to raise the temperature up to 3000°C. This data is useful for designing other bigger or smaller furnaces. Operational characteristic for one cycle of heating and cooling was determined which showed that the temperature of the furnace could be rapidly raised up to 3000°C.

(ii) *Platinum-rhodium Furnace*

One furnace to attain a temperature of 1400°C. was designed and fabricated. Platinum-10 per cent rhodium wire of 0·5 mm. diameter was used to wind the tube of 88·9 mm. dia. and 38·1 mm. length. One auxiliary winding, internally wound with kanthal 'A' wire was used for this furnace to reduce the watts per cm.^{2} on main winding of platinum-rhodium and thus to increase the life of costly element. Fig. 74 shows the failure of one Platinum 10 per cent Rhodium furnace without auxiliary winding. This furnace has given unsatisfactory service.

It was found by experiment that the diameter of the auxiliary winding for above-mentioned furnace should be 178 mm. in order to keep the winding in the isotherm of 1150°C. which is quite safe for Kanthal 'A' wire. It was also observed that the temperature of the furnace could be stabilized at 1400°C. with a surface loading of 4·2 watts per cm.^{2} on platinum-rhodium winding if the auxiliary winding is 'on' along with the main winding.

(iii) *Silicon Carbide Furnaces*

Two furnaces, each 20 kW, using silicon carbide rods as heating elements, were designed and fabricated to suit the special shape and geometry for particular research projects. These furnaces can attain a temperature of 1400°C. Their construction is rugged and cost of fabrication is considerably economical.

(iv) *Design and Installation of Power Distribution System for Pilot Plants*

Power distribution system and lighting for Cryolite Pilot Plant and Strip Aluminizing Pilot Plant were designed and electrical layout was prepared. Specifications of materials were detailed and their purchase is under progress.

Design, preparation of layout and installation of several new electrical equipment were completed during the year. These equipment mostly consisted of high-temperature semi-commercial resistance furnaces, electrical motors, and controllers for various machine tools and testing units.

(v) *Import Substitution of Parts*

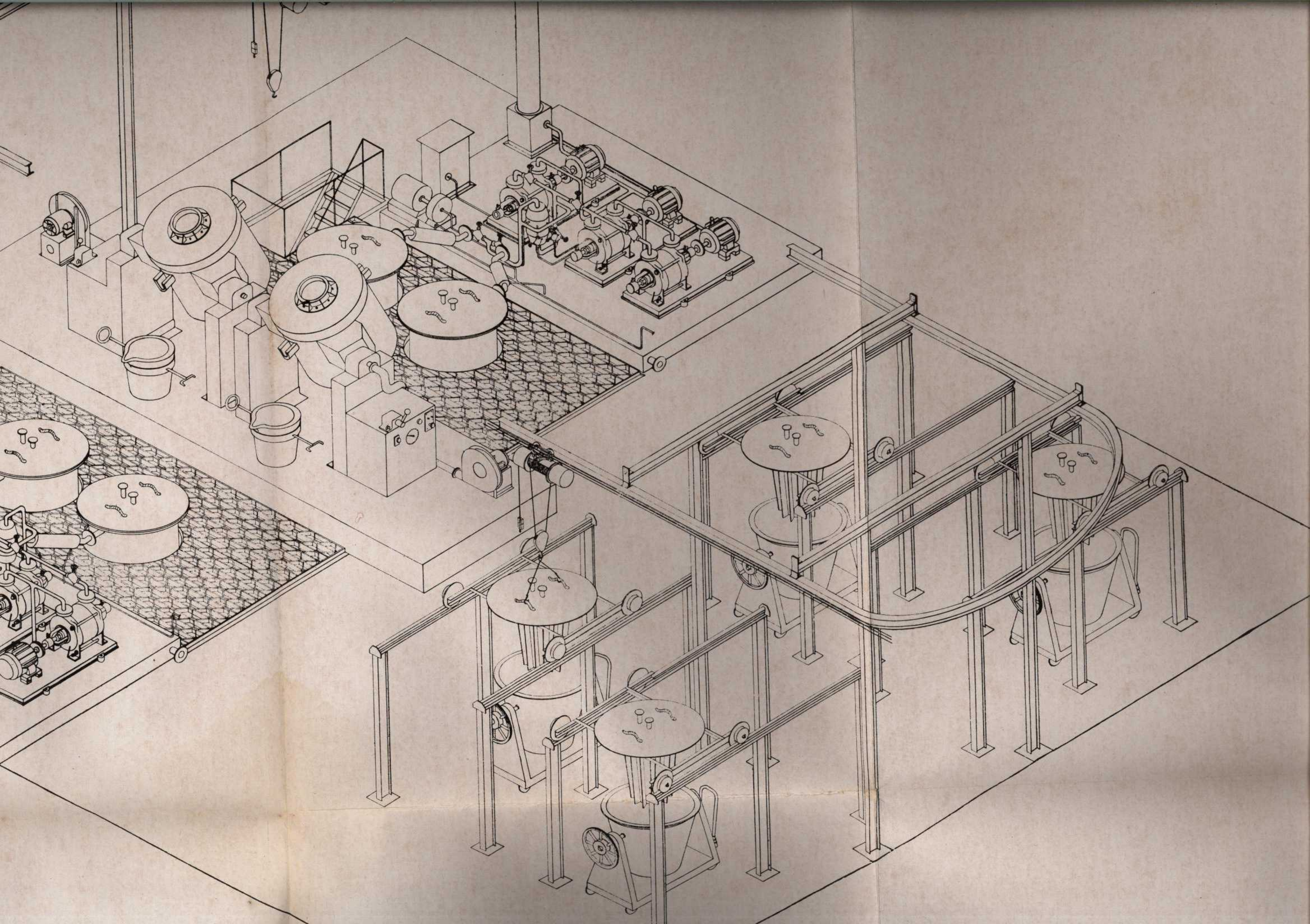
Every attempt was made to repair or fabricate the imported parts of electrical equipments. A few major fabricating or repairing jobs are described here.

Shaft seal assembly of 65 ton capacity air conditioning machine started leaking. Some parts of this assembly were worn out. These parts were fabricated out of die steel. After proper heat treatment and fine setting, it is giving satisfactory service.

High frequency generator for the high-frequency melting unit was not able to give sufficient power to melt the desired materials. Generator is driven by the squirrel cage motor. Rotor of the motor developed some fault. Short circuiting ring of the rotor bars of the motor had cracked and thus some of the bars were out of circuit. This was causing reduction in speed of the motor and thus decreasing the power output of the generator. Since rotors of both generator and motor have a common shaft, it was not possible to replace the motor by any other indigenous one. As such, repairing of the motor was taken up and by brazing the ring with great precautions and thereafter balancing the rotor, the motor was found giving very satisfactory service.

Bus bar trunking pieces for 6·6 kV. Rey Roll switch gear were designed and fabricated in order to complete the extension of power line to pilot plant.

One fractional H.P. servo-motor was successfully repaired by designing suitable fixtures to facilitate its repair.



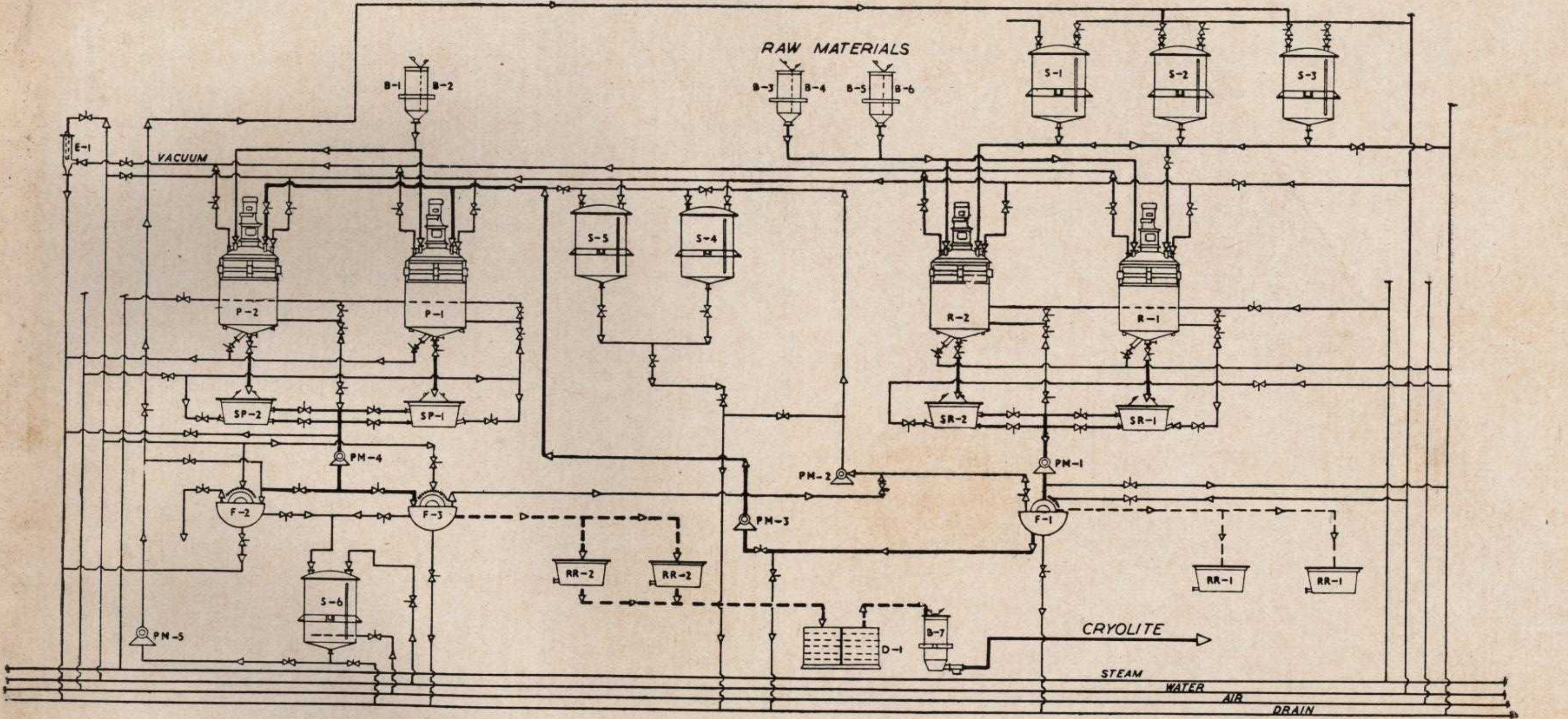


FIG. 71 — LAYOUT OF THE SYNTHETIC CRYOLITE PILOT PLANT DESIGNED AT THE NATIONAL METALLURGICAL LABORATORY

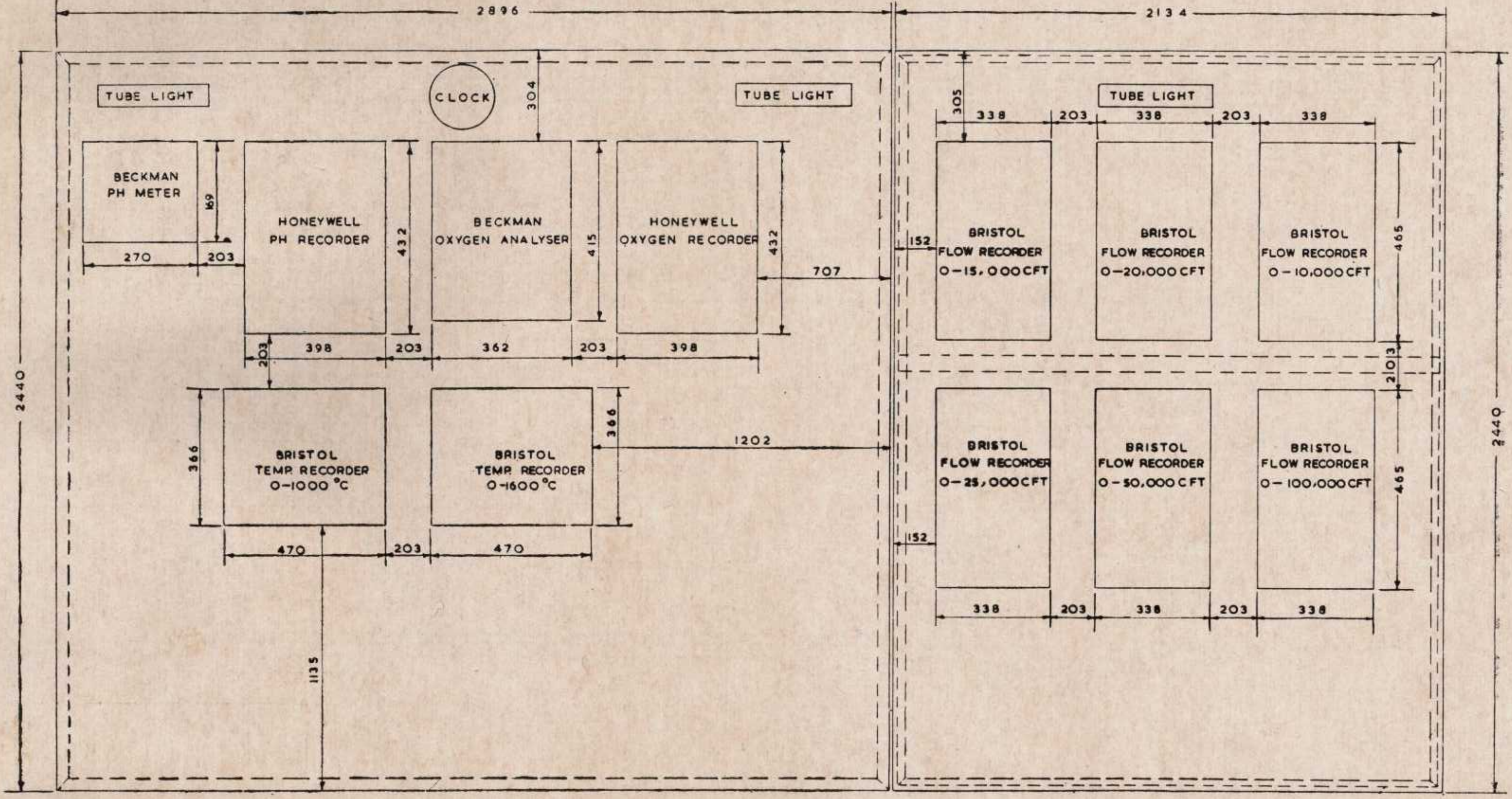


Fig. 72 — Control Instruments Panel for Mineral Beneficiation Pilot Plant

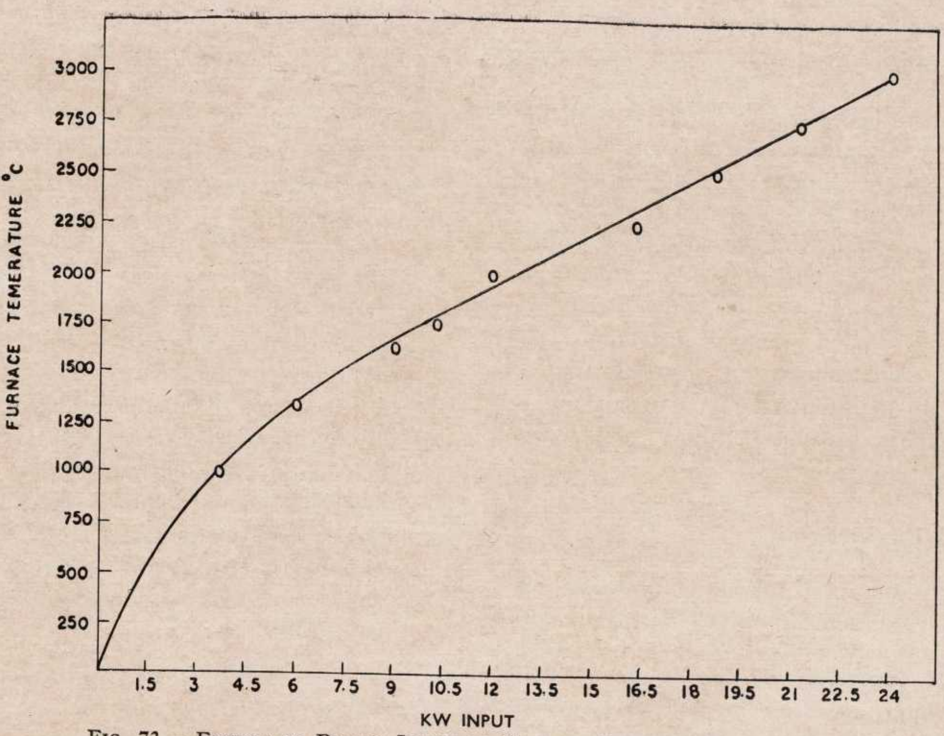


FIG. 73 — EFFECT OF POWER INPUT AT VARIOUS FURNACE TEMPERATURES

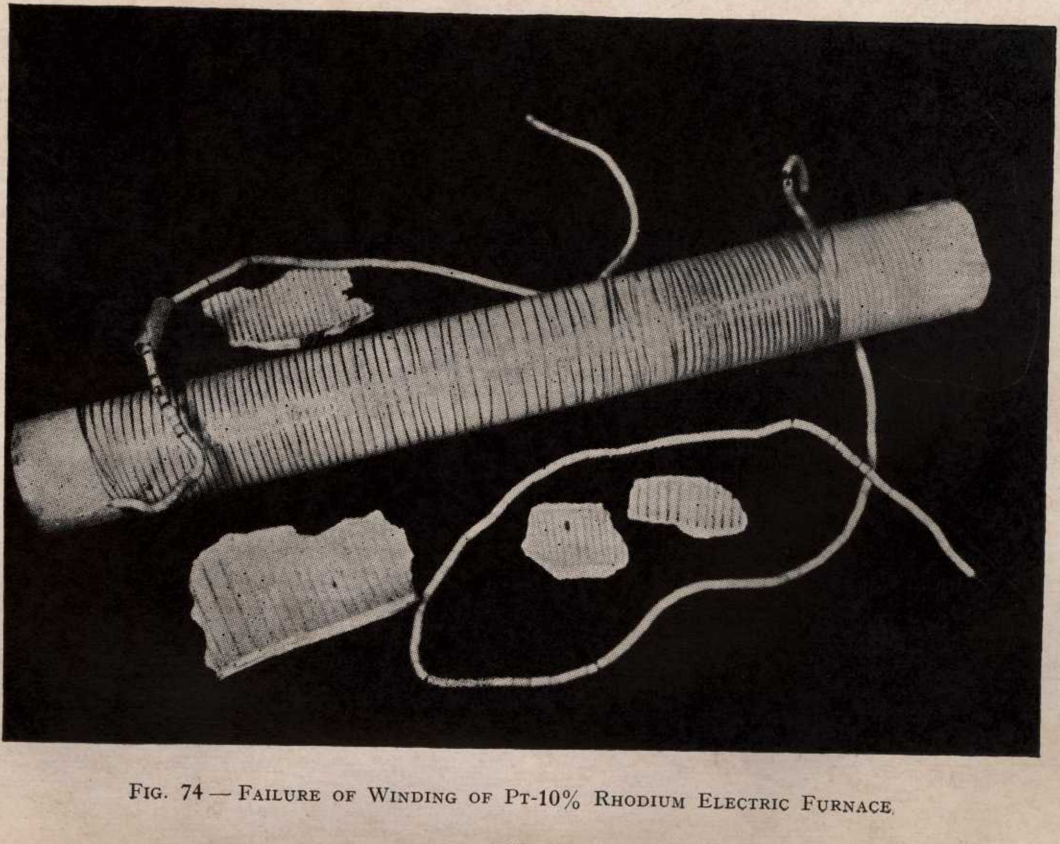


FIG. 74 — FAILURE OF WINDING OF PT-10% RHODIUM ELECTRIC FURNACE

(vi) *Planned Preventive Maintenance Scheme*

It has been greatly felt that planned preventive maintenance of electrical equipment in the Laboratory is very essential for their efficient economic operation. To secure a proper allocation of time, it is desired to prepare a schedule, setting out the frequency of inspections and overhauling of each group of machines, and to devise some means of ensuring that the regular maintenance is carried out. A systematic scheme is under consideration in this direction.

Civil Engineering

The following major civil works were completed and in progress during the period under review.

(i) *Completed*

1. Modification of Standard Sample Room.
2. False ceiling at first floor of Ore Dressing Bay.
3. Construction of Cabin for Air-conditioning Unit at Rolling Mill.
4. Flooring to the Eastern side of Guest House.
5. Installation of Furnaces attached to Centrifuge Room.
6. Construction of Precision Elect. Room at constant temperature in western lighting area.
7. Construction of Co-operative Stores.
8. Modification to Pilot Plant Motor Garage for installation of Ball Mill for Ext. Met. Division.
9. Construction of Smithy Shop.
10. Modification to Cryolite Pilot Plant.
11. Replacement of old overhead gas line in NML.

(ii) *In Progress*

1. Construction of Strip Aluminizing Plant.

INDUSTRIAL LIAISON, OPERATIONAL RESEARCH, INFORMATION AND EXTENSION SERVICES

LIAISON, Information & Extension Services of the National Metallurgical Laboratory continued to play an important role in the field of dissemination of research results, consultancy work, and commercial exploitation of the techniques and processes developed in the Laboratory. A brief account is furnished below.

Consultancy Work

National Metallurgical Laboratory has continued to maintain a high tempo of consultancy work. Consultancy services extended comprised the beneficiation and agglomeration studies of the raw materials, smelting studies, testing of indigenous materials for use as refractory purposes in iron and steel plants, etc. During the period under review, NML had done beneficiation and sintering studies on mixed laminated as well as massive and laminated iron ores from Rajhara Mines for Bhilai Steel Plant, beneficiation and sintering studies on Meghatuburu iron ore for NMDC, beneficiation and sintering studies on three more iron ore samples from Dalli Mines for Bhilai Steel Plant, and beneficiation and pelletization of iron ore fines from Redi mines for Government of Maharashtra. Equal importance was also given to the work relating to preparation of raw materials of non-ferrous industries, such as flotation studies on samples of Khetri and Kolihan copper ore from Rajasthan and a sample of Rakha copper ore from Bihar, and the Reports prepared on the findings of the studies had already been furnished to NMDC. Pilot plant smelting trials were conducted with low-temperature carbonized coke made from Talcher non-coking coal in Low-shaft Furnace for iron smelting. Report prepared on the studies conducted on reducibility of Kiriburu iron ore had been sent to Bokaro Steel Ltd. Smelting trials had also been conducted in Low-shaft Furnace Pilot Plant to study the suitability of using Dabok limestone for iron ore smelting referred to by M/s. Kamani Industrial Development Corporation.

Regarding the development of refractory materials, studies were completed on clays from Jammu & Kashmir and the Report prepared incorporating suggestions for the suitability of using clays as refractory material was sent to Government of Jammu & Kashmir. Based on the comparative studies completed on high-temperature coke oven coal tar pitch as a binder in the manufacture of carbon refractories, a Report had been sent to Bhilai Steel Plant who had sent their pitch samples for study. Similar testing was completed to determine the use of pyrophillite sample as refractory material sent by the M/s. Assam Sillimanite Co. Investigations referred to by many other private organizations were also taken up and reports submitted to them.

In the field of granting industrial licences, National Metallurgical Laboratory's views were obtained. All the cases were critically examined, and where indigenous technical ' know-how ' was available, it was recommended. During the period under review, 106 cases were reviewed.

Collaborative Research and Development Work

The National Metallurgical Laboratory is collaborating with the Commonwealth Committee on Mineral Processing as well as International Working Group on Bonding Clays and conducted a number of investigations. Besides, the NML is collaborating with the Indian Standards Institution in the formulation and rationalization of Standards, and had conducted and undertaken a number of investigations apart from comments furnished on the various draft specifications. For the Ministry of Defence, work was conducted on top priority basis. During the period under review, about 1·8 metric ton of magnesium powder received from ordnance factory was reconditioned and sent to the defence establishments. Nearly 200 kg. of Mg-Mn alloy powder received from the ordnance factory was also reconditioned. As per Defence Specification 1642 kg. of ferro-aluminium were manufactured and supplied. National Metallurgical Laboratory had also supplied 50 kg. of chromium metal as per Defence Specification.

Industrial Surveys and Collection of Statistical, Economic and Market Data

An exhaustive survey of the foundry industries in India was made and all pertinent data relating to production of each unit, type of casting, specialized equipment available, sand processing system, different products manufactured, etc., were collected. Besides, statistics relating to latest production and consumption figures of different types of raw materials, ferrous and non-ferrous metals and alloys of India and other countries of the World were collected and maintained on Card Index System. Scanning of technical news of the industry in the home and overseas press and research developments was done.

Technical Aid to Industry

Over three hundred enquiries on different subjects pertaining to process details, raw materials, quality production, etc., were attended to during the period under review. Nearly 80 per cent of the total enquiries originated from private industries and individuals. Detailed technical notes were furnished wherever necessary. Over hundred short-term investigations and specification tests were conducted of which about 64 per cent related to private sector and 36 to public sector organizations.

Practical Demonstration

Practical demonstration of the following NML-developed processes were arranged to impart the technical ' know-how ' as well as the actual plant operation to the licensees.

(i) Hot-dip aluminizing of ferrous materials.
(ii) Production of electrolytic copper powder.
(iii) Production of ceramic magnets.
(iv) Production of flux for submerged arc welding.

Training

Under the Scientific and Technical Co-operation Agreement between the Governments of India and the United Arab Republic, two scientists from United Arab Republic are undergoing advanced research training in the National Metallurgical Laboratory in different specialized metallurgical subjects for two years. Besides, many technical personnel from Government and industrial organizations, and students from technical institutes were given training in heat treatment, mechanical testing, spectrographical analysis, refractories, ore-dressing techniques, etc. All-India Handicraft Board deputed a technical person for training in the colouring techniques of ' Bidri ' ware developed in the Laboratory.

Operational Research

(i) *Study of Slag with the Help of O.R. Techniques*

The work on the study of slags for their utility as slag cement with the help of mathematical programming was completed. A model had been developed for determining the maximum permissible limits of MgO and MnO contents in the slag as well as its utility as slag cement. The technique of Linear Programming is being utilized to achieve the optimum composition of slag (determined from the model) at the least cost.

(ii) *Statistical Analysis of Data Pertaining to Foundry Industry with the Help of an Electronic Digital Computer*

The planning of card designs for transferring the data on foundry industry on to punch cards, preparation of codes, coding procedure and tabulation formats were completed. The data from computer tabulations thus obtained were subjected to statistical analysis for incorporating the same in the Foundry Directory.

Colloquia

A number of colloquia were held during the period. The research staff spoke on different research and technical themes. Lectures were arranged from distinguished scientists from home and abroad.

NATIONAL METALLURGICAL LABORATORY FOUNDRY STATIONS AND MARINE CORROSION RESEARCH STATION

Foundry Stations

The second NML Foundry Station set up by the National Metallurgical Laboratory at the Industrial Estate, Guindy, Madras, was declared open on 14th December 1965 by Shri R. Venkataraman, Minister for Industries, Government of Madras, in the midst of over 500 foundry industrialists from all over Madras State and other parts of India.

Sir Jehangir Ghandy presiding over the function said that he was happy to see that the NML is today acting as a nucleus for many of the long-range and short-term foundry projects. Long-range fundamental work has also been done for making the best use of foundry raw materials and to evolve national standards. He hoped that with the setting up of the foundry stations at important centres in the country, foundries would be able to utilize their services.

Shri R. Venkataraman, Minister for Industries, Government of Madras, while inaugurating the Foundry Station, said that the establishment of this station was not only the manifestation of the magnificient growth of the NML but also the realization of a long-felt need of Madras State. It is a creditable achievement of the NML that during the short period since its inception, it has been able to do very valuable research work in various fields of foundry to grow rapidly on sound lines. By undertaking comprehensive investigations on moulding sands and bonding clays and by evolving national standards for quality castings, this Laboratory has become the nucleus of the foundry industry in India. He hoped that the industrialists would fully realize the value of this station and utilize the research results and technical facilities offered by this Foundry Station and improve the quality of their products to keep pace with the demands.

Dr. B. R. Nijhawan, Director, National Metallurgical Laboratory, in his welcome address, expressed his deep debt of gratitude to Sir Jehangir Ghandy, thanked the distinguished guests, foundry executives and foundrymen for their full co-operation and also expressed his gratitude to Shri M. Bhaktavasalam, Chief Minister of Madras, for the keen interest shown by him and to Shri R. Venkataraman for participating in the function. Dr. Nijhawan said that the Foundry Station at Madras has been provided with modern equipments for testing of foundry sands and bonding clays for routine control of sand mixtures. A chemical laboratory attached to the station will undertake chemical analysis of sands, metals and alloys and will give the foundry industry necessary technical assistance and operational guidance. The service facilities will further be expanded to enable the station to undertake metallography and mechanical testing of metals and alloys and the station will also be equipped with necessary melting units for undertaking experimental work on problems relating to metal melting.

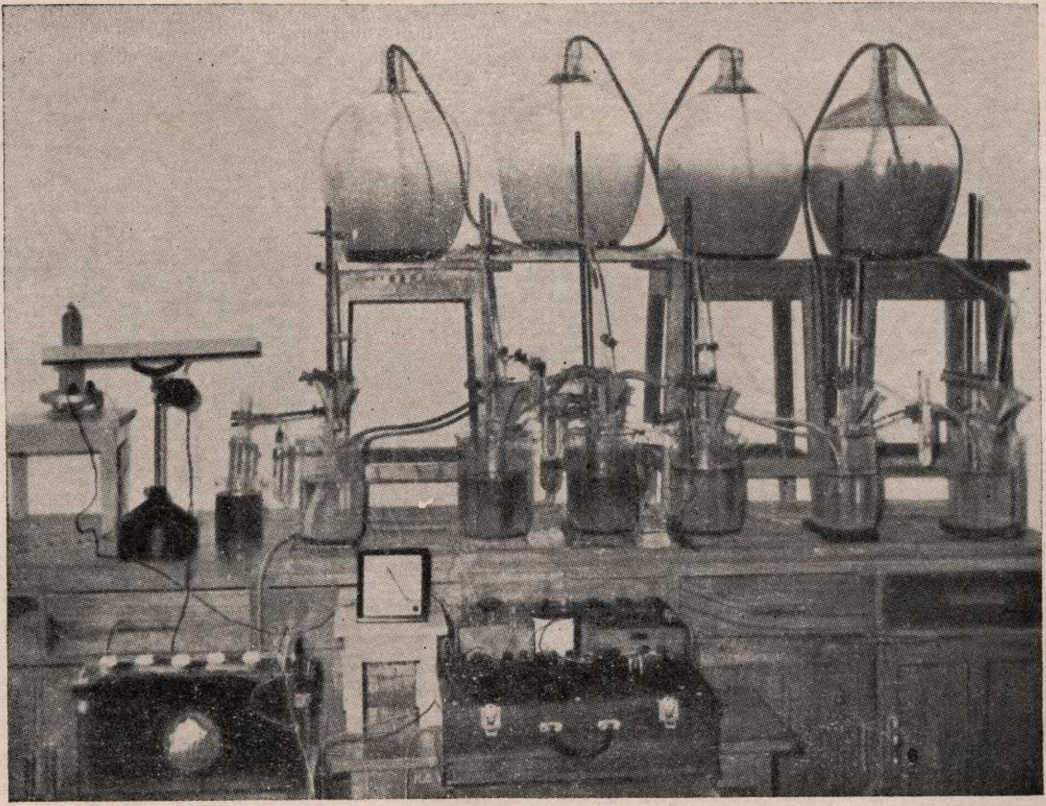


FIG. 75 — EXPERIMENTAL SET-UP FOR THE DEVELOPMENT OF SACRIFICIAL ANODES FOR CATHODIC PROTECTION

The first NML Foundry Station which was earlier opened at Batala has been engaged in providing testing facilities and technical assistance to the large number of foundries in and around Batala. Considerable progress was made for the establishment of other Foundry Stations at Howrah and Ahmedabad. In fact the Foundry Station at Howrah has now well-equipped sand testing and chemical analysis laboratories and is already offering testing facilities to the various foundries in the region. All the equipment, apparatus, etc., for the NML Foundry Station at Ahmedabad has already been received and steps are under way to set up the laboratory.

Action is also under way to set up the fifth Foundry Station at Agra. All the National Metallurgical Laboratory Foundry Stations will be equipped with adequate facilities for testing of foundry sands and bonding clays for scientific control of sand mixtures, as well as to find out their suitability for foundry moulding purposes. Stations will also be equipped to undertake chemical analysis of both ferrous and non-ferrous metals, moulding sands and other foundry raw materials. In the second phase, the National Metallurgical Laboratory Foundry Stations will expand the service facilities to include metallographic examination and mechanical testing of metals and alloys. It is also proposed to equip the NML Foundry Stations with melting units for undertaking experimental work on problems relating to metal melting.

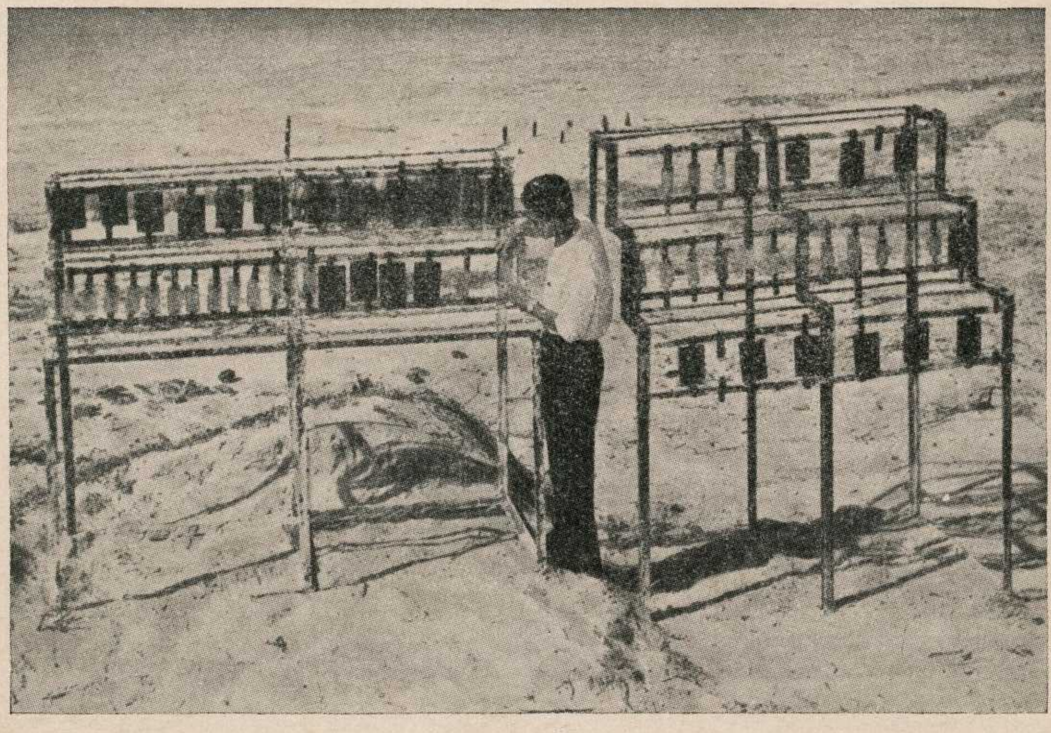


FIG. 76 — ATMOSPHERIC EXPOSURE OF METAL SAMPLES ON SEA BEACH AT DIGHA

Each NML Foundry Station will have a small library which will be gradually expanded to provide facilities for technical reference on diverse fields of foundry technology.

Programme of Work of National Metallurgical Laboratory Foundry Stations

The programme of work at the National Metallurgical Laboratory Foundry Stations will cover the following scope:

(i) Periodical testing of foundry sands, bonding clays used in foundries, and raw materials like pig iron, limestone, coke, etc., used for melting, as well as finished products.

(ii) To tender technical advice regarding selection and application of indigenous foundry sands and other moulding materials, including selection of metal charges for the production of various types of castings.

(iii) To undertake ad-hoc investigations on the problems faced by individual foundries and to suggest suitable remedial measures thereof.

(iv) To conduct a regional survey of the moulding materials available in those regions and to conduct a complete study of the chemical and physical properties of the moulding materials.

(v) To conduct periodical refresher courses to the staff sponsored by the foundries.

(vi) Standardization of melting and moulding techniques in under-developed foundries.

(vii) To advise the operational staff on modern foundry technology and to issue technical bulletins periodically.

(viii) To carry out research and development work, standardization and simplification of moulding, melting and other technological procedures to achieve quality as well as productivity.

Marine Corrosion Research Station, Digha

Work is in full swing at the Marine Corrosion Research Station at Digha. Long-range and short-term research projects were initiated. The corrosion of various types of metals and alloys under marine atmosphere condition at Digha, sea-water immersion corrosion, corrosion during day and night time, etc., are some of the projects under study which have been reported in the appropriate section of this Report.

LIBRARY, DOCUMENTATION, TRANSLATION AND REPROGRAPHIC SERVICES

Library and Documentation Services

The continued growth of the Library necessitated replanning of the space as also a programme of more drastic weeding out. The services rendered by the Library were maintained at a high standard. The documentation slips had now all been interfiled. A system of photocopying and microfilming was put on a sound footing, so that all interlending is now made with the help of such copies. The administration of the Library was further rationalized so that in spite of increasing annual additions of books and periodicals routine services are maintained at the usual level.

Translation Service

Articles in French and German of direct interest to research work projects under way at the National Metallurgical Laboratory were translated into English. Oral translations were also rendered to give the research workers a quick appraisal of the results contained in the technical publications with a view to enable him to precisely define the portion of the text of specific interest to him. Additionally other routine translation work was also attended efficiently. About 300 technical papers published in French and German were translated during the period under review, each paper being of an average of 4000 words.

Reprographic Service

Photostat, reflex prints and microfilms were made of the scientific and technical papers of interest to the research workers and were widely circulated. Documentary films were taken of important technical functions of the Laboratory as well as outstanding processes and techniques developed. Coloured transparencies and microphotographs, X-ray photographs, etc., are taken of the various research and investigation projects.

INTERNATIONAL SYMPOSIA

WITH a view to cataloguing and focussing attention on known families of substitute alloys in relation to others offering potential scope for research and development, a Symposium on 'Metallurgy of Substitute Ferrous and Non-ferrous Alloys' was organized by the National Metallurgical Laboratory from 27th to 30th April, 1966; thereby providing an international forum to discuss the metallurgy of substitute ferrous and non-ferrous alloys and to exchange technical 'know-how', examine inter-related problems and facilitate further research on this subject.

The Symposium drew a large gathering and more than 200 delegates from all over the World including top ranking scientists

FIG. 77 — DR. B. R. NIJHAWAN, DIRECTOR, NATIONAL METALLURGICAL LABORATORY, WELCOMING THE DELEGATES TO THE SYMPOSIUM

FIG. 78 — SHRI T. N. SINGH, UNION MINISTER FOR IRON & STEEL, GOVERNMENT OF INDIA, DELIVERING THE INAUGURAL ADDRESS AT THE SYMPOSIUM

and technologists participated in its deliberations.

The following 36 technical papers were presented and discussed in seven technical sessions.

1. Scope of research and development work on substitute alloys at the National Metallurgical Laboratory.
2. Metal matrix composite materials.
3. Economic utilization, recovery and substitution of some strategic non-ferrous metals.
4. A study of copper consumption in some industrial countries and its relevance to India.
5. Indigenous substitution of imported materials on Indian Railways.
6. Electrical Conductivity of Substitute Aluminium Alloys.
7. The influence of aluminium and iron additions on copper-manganese-zinc alloys.
8. Studies on the properties of some copper-manganese alloys.
9. Development of substitute magnetic materials.
10. Research and development work on substitute electrical resistance alloys for heating elements.
11. Substitute solders.

FIG. 79 — SIR JEHANGIR GHANDY, CHAIRMAN, EXECUTIVE COUNCIL, NATIONAL METALLURGICAL LABORATORY, DELIVERING THE PRESIDENTIAL ADDRESS AT THE SYMPOSIUM

12. Low-carbon high-chromium alloy without low-carbon ferro-chrome.
13. Study of some carburizing and hardenable steel with and without nickel and molybdenum for automotive industries.
14. Some precipitation reactions in substitute Cr-Mn-N austenitic stainless steels.
15. Research and development work on substitute nickel-free austenitic stainless steel.
16. The applications of electrolytic manganese as a substitute ferro-alloy.
17. The recent developments of aluminium conductors in France in public distribution and industrial plants.
18. Aluminium foil for electrical windings.
19. Some aspects of redesigning in aluminium.
20. ACAR Conductors — A techno-economic evaluation.
21. Substitute alloys as bearing materials.
22. Critical raw-materials and substitutes in the light of India's self-sufficiency.
23. Improved mild steels for structural purposes.
24. Thermo-mechanical ausforming technique for producing substitute ultra high strength steels.
25. Development of low-tungsten substitute tool steels.
26. Development of substitute high-temperature creep resistant alloys.

27. Conservation of non-ferrous metals by protective treatments and substitution by aluminium.
28. Substitution of non-indigenous non-ferrous metals and alloys.
29. Substitution of galvanizing by aluminizing of steels.
30. Economic uses and substitution of non-ferrous metals and alloys.
31. Scope of aluminium-base bearing alloys.
32. Properties of some substitute bearing alloys.
33. The role of selenium and tellurium in ferrous metals.
34. Substitution of scarce metals with special reference to standardization in India.
35. Physical metallurgy of substitution of alloys.
36. Theoretical considerations in the substitutions of some common elements in alloy steels.

The Symposium was inaugurated by Shri T. N. Singh, Union Minister for Iron & Steel. Sir Jehangir Ghandy, Chairman, Executive Council, National Metallurgical Laboratory, presided at the function.

Dr. B. R. Nijhawan, Director, National Metallurgical Laboratory, in welcoming the distinguished delegates from India and abroad, said that such international Symposia offer stimulation of thought and provide an ideal forum for exchange of technical ' know-how ' and ' know-why ' of diverse aspects of the subject.

Continuing Dr. Nijhawan said, The National Metallurgical Laboratory has been called upon to handle various research and development projects vital to the growth of mineral metallurgical industries and to provide solutions to the numerous problems facing the industries. The present emergency has highlighted the research and development work of the National Metallurgical Laboratory covering ' Substitution

FIG. 80 — A VIEW OF THE DELEGATES AT THE SYMPOSIUM

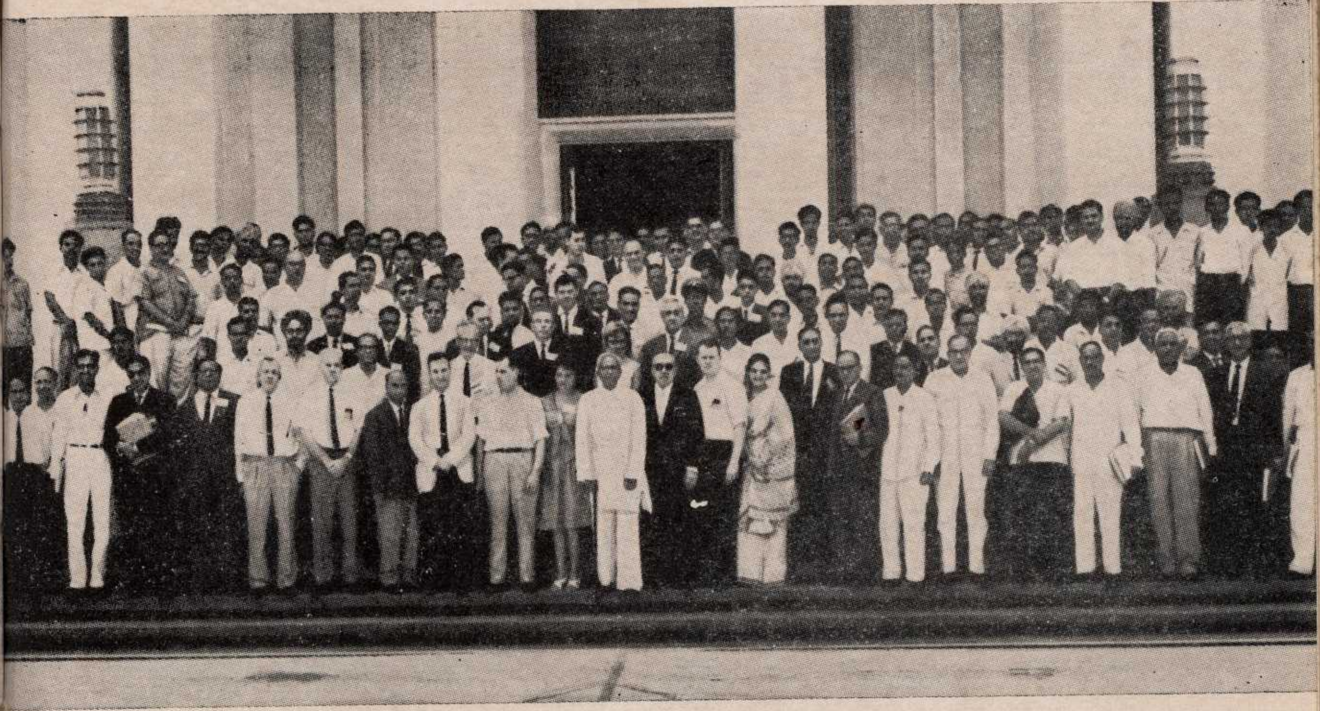


Fig. 81 — Group Photograph of Some of the Delegates who participated in the Symposium

of ferrous and non-ferrous alloys' on which the National Metallurgical Laboratory had started active work when there was indeed no ' emergency ' in the country. In India, with chronic foreign exchange shortages in the midst of ambitious consecutive Five-Year Plans, the need for substitution as short-term measure and development of substitute alloys in the long-range research effort call for sustained work on the subject on which the National Metallurgical Laboratory has been actively engaged upon. Some major research and applied themes on substitute alloys and alloy steels at the National Metallurgical Laboratory were related to aluminizing of steel, development of substitute stainless steels, electrical resistance alloys, substitute manganese bearing brasses, indigenous tool and die steels, iron-aluminium alloys, aluminium-based substitute bearing alloys for diverse end products to exacting specifications and practical needs, etc. Such pioneering work carried out at the National Metallurgical Laboratory has been well-rewarding and presents interesting industrial potentialities, placing high premium on research ingenuity and applied technology.

Sir Jehangir Ghandy, Chairman, Executive Council, National Metallurgical Laboratory, stated that in view of the acute shortage of foreign exchange in the country, there has arisen immediate need for substitute metals and alloys as a short-term measure. Added to this is the necessity of development of substitute alloys calling for long-range research and sustained work. The National Metallurgical Laboratory which has been engaged in this field almost since its inception, Sir Jehangir said, has undertaken major research and applied themes on substitute alloys relating to the development of nickel-free austenitic stainless steels, nickel and cobalt-free electrical resistance alloys, coinage alloys without nickel and copper, manganese-bearing brasses, tool and die steels, iron-aluminium alloys, aluminium-based alloys for the production of a wide range of end products to exacting specifications. Substitute processing techniques, such as hot-dip aluminizing of steel and related surface treatments have also been developed. The results of these pioneering investigations have been rewarding and offer great industrial potentialities, he added. On behalf of the Executive Council of the National Metallurgical Laboratory, Sir Jehangir thanked Dr. B. R. Nijhawan, Director of the Laboratory, and his colleagues for their excellent work devotedly pursued at the National Metallurgical Laboratory.

Sir Jehangir also took the opportunity to mention that Dr. B. R. Nijhawan was conferred last year the Hony. Membership of the Japanese Iron and Steel Institution and the Gold Medal of the Institution. Incidentally, Dr. Nijhawan is the first Indian to receive this internationally coveted and distinguished honour. Sir Jehangir also

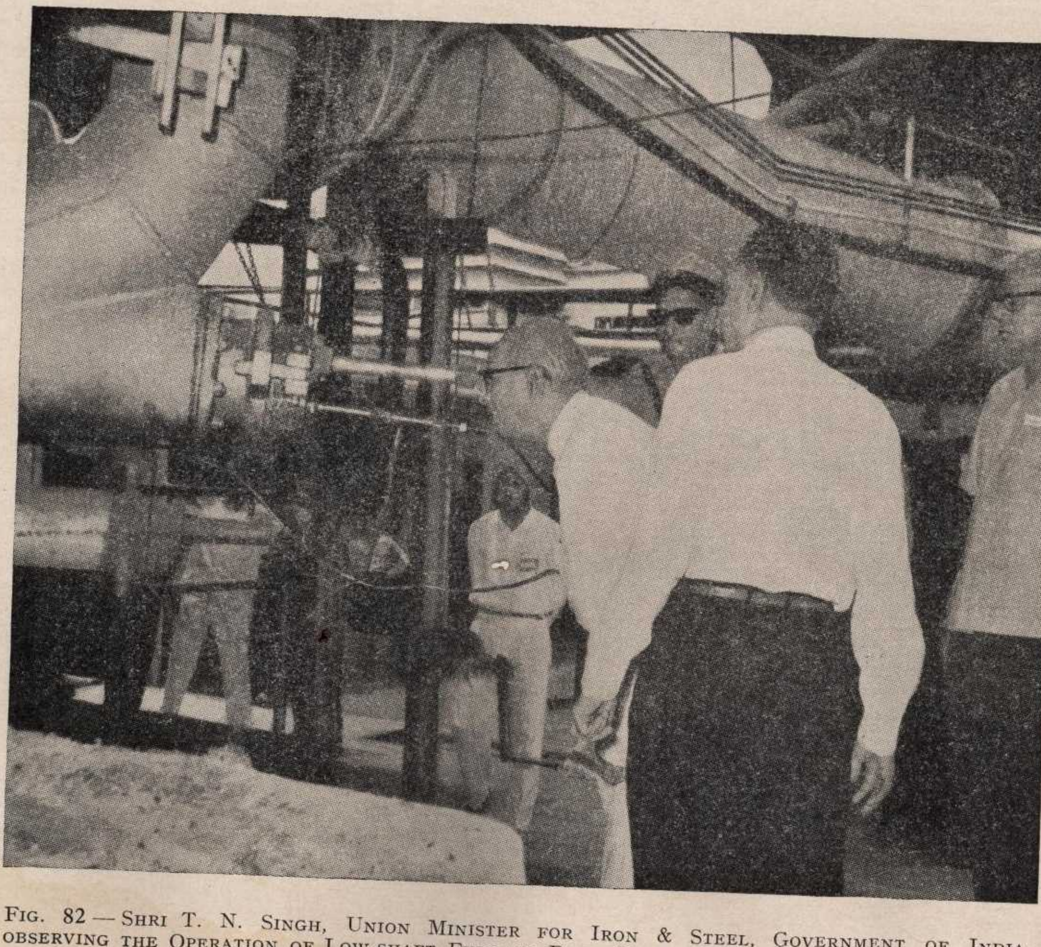


FIG. 82 — SHRI T. N. SINGH, UNION MINISTER FOR IRON & STEEL, GOVERNMENT OF INDIA, OBSERVING THE OPERATION OF LOW-SHAFT FURNACE PILOT PLANT, DURING HIS VISIT TO NATIONAL METALLURGICAL LABORATORY

mentioned that the coveted Bhatnagar Memorial Award has been made to Dr. B. R. Nijhawan by the Indian Council of Scientific & Industrial Research for his outstanding contribution in Engineering Sciences last year.

Shri T. N. Singh, Union Minister for Iron & Steel, while inaugurating the Symposium stressed the importance and necessity of development of substitute alloys in the background of meagre deposits of some strategic metals and the emergent situation arising out of aggression on the border of the country. He paid a warm tribute to Dr. B. R. Nijhawan and his colleagues for the excellent researches underway at the National Metallurgical Laboratory on the development of substitute alloys many of which have significant potential industrial applications. He also advocated the substitution of galvanizing by aluminizing and attached highest importance to aluminizing process developed by the National Metallurgical Laboratory and expressed his intention to have a whole section of aluminizing plant in one of the Public Sector steel plants in India. Shri Singh also appreciated the valuable assistance which the Steel Industry in India has constantly received from the National Metallurgical Laboratory. The National Metallurgical Laboratory, he said, has done highly creditable work in undertaking these researches and pilot plant investigations in finding solutions of many problems including the preparation of raw materials for the projected Bokaro Steel Plant.

Concluding Shri Singh said that in the context of the present foreign exchange shortage, the importance of the NML Symposium cannot be over-emphasised. The papers to be presented and discussions to follow will contribute to the development of processes and techniques which will help production of substitute ferrous and non-ferrous alloys under Indian conditions and with Indian processing facilities. He congratulated Dr. Nijhawan and his colleagues for organizing the International Symposium at a most opportune time.

Shri N. N. Wanchoo, I.C.S., Secretary, Ministry of Iron & Steel, Government of India, addressing the Delegates to the Symposium said that the National Metallurgical Laboratory is well known for arranging excellent international symopsia of topical interest to the metallurgical industries. This Symposium is yet another instance of their enterprises. It is very encouraging that leading Indian scientists, as well as distinguished men of science from abroad, are participating in the Symposium. Continuing, Shri Wanchoo highly commended the work of the National Metallurgical Laboratory in the field of development of substitute alloys and stressed that even if complete substitution of strategic metals is not possible, partial substitution will also help a great deal. He greatly valued the valuable assistance rendered by the National Metallurgical Laboratory to Indian Iron & Steel complexes in solving their multitude problems of both short-term and long-range importance.

Many of the overseas scientists and delegates who took part in the discussion expressed the view that the Symposium was extremely useful and the subjects discussed were indeed of a high scientific standard. Mr. Jacomet; Chief Engineer of the Aluminium Francais said that technical exchanges in the various scientific disciplines with France should be intensified and this would be of great mutual benefit to India and France. He complimented Dr. B. R. Nijhawan, Director, National Metallurgical Laboratory, and his colleagues for the exceedingly good work done in the various branches of metallurgy and particularly in the field of substitute alloys. Mr. M. J. Smith, Director, International Copper Development Council, said that he was extremely impressed with the research work under way at the National Metallurgical Laboratory. The development work on substitute alloys, particularly the work done on aluminium alloy bearings, Mr. Smith said, should not only be of importance to India but also to other countries where a constant search is always made in the development of new alloys. Lt. W. F. Stuhrke, 1st Lt. USAF, of Air Force Materials Laboratory, Ohio, U.S.A., fully shared the sentiments expressed by his colleagues from U.K. and France, and said that substitution problems in the United States are engaging the attention of the materials engineers. Referring to the problem carried out at the National Metallurgical Laboratory, Lt. Stuhrke said that the results achieved in the various problems undertaken by NML are highly praiseworthy, and in particular, he considered the development work done on nickel-free stainless steel as a work of international importance.

Dr. B. R. Nijhawan, Director, National Metallurgical Laboratory, in his concluding address, gave a résumé of the technical papers presented and the discussions that followed. He sincerely thanked the distinguished delegates from home and overseas, and authors of the technical papers. He also heartily thanked the organizers of the Symposium.

The delegates were conducted round the various research divisions of the National Metallurgical Laboratory and the different Pilot Plants such as the Low-shaft Furnace Pilot Plant, Mineral Beneficiation Pilot

FIG. 83 — DR. B. R. NIJHAWAN, DIRECTOR, NATIONAL METALLURGICAL LABORATORY, EXPLAINING TO LATE DR. H. J. BHABHA, F.R.S., SOME OF THE PRODUCTS AND PROCESSES DEVELOPED AT THE NATIONAL METALLURGICAL LABORATORY

Plant and other pilot plants of the Laboratory during the Symposium.

The Symposium Supplement

On the inaugural day of the Symposium, special supplements were brought out in a masterly fashion by *Economic Times* and *Financial Express*. The supplements contained leading papers from eminent scientists and technologists. *Economic Times* took a special interest in the Symposium and excellent coverages of day-to-day proceedings were made. Excellent coverage was also done by other leading news papers like *Hindustan Standard, Amrita Bazar, Patrika, Statesman, Hindu, Financial Express,* etc. of the inaugural function and technical sessions of the Symposium.

PUBLICATIONS

NML Technical Journal

The *NML Technical Journal*, a quarterly publication, has stepped into the eighth year of its publication. The journal has established a wide exchange programme with many Indian and foreign technical journals, and nearly 220 journals and other publications are obtained on ' free exchange basis '. The number of Indian and foreign subscribers are increasing gradually. The papers published in *NML Technical Journal* are regularly abstracted by leading international abstracting services such as *Chemical Abstracts*, *Journal of Iron & Steel Institute*, U.K., and American Society of Metals the contents of the Annual Report of the National Metallurgical Laboratory are likewise abstracted by leading abstracting establishments.

Papers Published

During the period under review 99 scientific papers were published and presented as per Appendix I.

Research and Investigation Reports

During the period under review 79 research and investigation reports were prepared. The details are furnished in Appendix II.

Brochures

A brochure on NML Foundry Station, Madras, was brought out on the Inauguration of the Foundry Station. A new brochure on Mineral Beneficiation Pilot Plant of the National Metallurgical Laboratory was also printed.

GENERAL

Plan and site for the Semi-commercial Magnesium Plant, having a production capacity of 250 tonnes/year, were finalized. A plot of land covering an area of 7 acres has been given by M/s. Tata Iron & Steel Co. for its construction. Design and blue prints of the Plant were made in collaboration with Central Design & Engineering Unit of Council of Scientific & Industrial Research and the Plant is expected to be erected before the scheduled time. The cost of the plant will be about Rs. 67 lakhs.

Major construction work of the various extension units of the National Metallurgical Laboratory were all completed. The NML Foundry Station at Madras was declared open and those at Howrah and Ahmedabad were almost ready. Work is in full swing in the Foundry Stations at Batala and Madras. A number of long- and short-term research projects were initiated at the Marine Corrosion Research Station at Digha.

The Laboratory has continued to design and fabricate many of the special items of equipment needed for the research and pilot plant work, apart from the regular check up and maintenance of the various specialized equipments and apparatus.

The staff of the Laboratory are working in the various committees of Indian Standards Institution and special committees set up by the Government of India and State Governments. The Laboratory played an active part in the Foundry Pig Iron Panel constituted by the Government of India to study the requirements of pig iron to the various foundries and to make recommendations as to the type and quality.

Purchase and Stores Sections of the National Metallurgical Laboratory kept up their unceasing activity for procurement of capital equipment, raw materials and consumable stores and equipment, etc. The administrative work of the Laboratory was efficiently maintained and the heavy budgetary accounts of the Laboratory were also handled with efficiency and speed. Details of National Metallurgical Laboratory budget expenditure are furnished in this Report. Co-operative Banking Society of the National Metallurgical Laboratory continued its good work throughout the year. The Co-operative Stores is doing an exceedingly good job in the supply of rationed and other foodstuffs, stationery articles, etc., to the staff members.

The National Metallurgical Laboratory Team participated in the Shanti Swarup Bhatnagar Memorial Tournament held at Hyderabad and was runners up in the volley-ball tournament.

Award

Dr. B. R. Nijhawan, Director, National Metallurgical Laboratory, has been nominated as the first Vice-chairman of the Indian Section of the American Institute of Mining, Metallurgical and Petroleum Engineers, Inc. (U.S.A.). Dr. B. R. Nijhawan has also been nominated Honorary Member of Societe Francaise de Metallurgie, France. Besides Dr. B. R. Nijhawan has been elected as the President of Indian Institute of Metals.

Shri M. R. K. Rao, Scientist, has been awarded the Ph.D. degree from Banaras Hindu University.

Safety First and First Aid Section

There are two first aid posts of the Laboratory — one at the main Laboratory premises and the other in the Low-haft Furnace Project and the Mineral Beneficiation Pilot Plant area. A large number of minor injury cases were treated in these first aid posts. Two mojor injury cases were hospitalized. There was no fatal injury during the year.

The first aid posts also administrated medicines for minor physical ailments. The safety in the Laboratory was strictly supervised, regular safety classes were held and checking was made on the safety procedures. A ‘ No Accident Campaign ’ was organized. Film shows on safety procedures to be followed while working in the shop floor were also arranged.

PATENTS

Patents	*I.P. No.*	*Title*
Filed	102483	An improved method for the recovery of metallic values from waste metallic fines (12.11.1965)
	102481	A method for recovering volatile metals from their waste metallic fines (12.11.1965)
Accepted	96661	A novel method of separating iron from ilmenite and its application in upgrading of ferruginous ores (11.5.1966)
	94769	An improved device for the continuous hot-dip coating of metallic strip and wire (15.1.1966)
	94768	An improved device for the isolation of dross in molten baths during continuous hot-dip processing of strip or wire (22.4.1966)
Sealed	82191	An improved jacketed electrolytic cell for the electro-deposition of metals and metallic oxides in general and manganese dioxide in particular (10.5.1966)

Processes Ready for Commercial Exploitation

The following processes developed at NML were ready for commercial exploitation, out of which items 1 to 21 have already been released.

Sl. No.	*Title*
1.	Production of carbon-free ferro-alloys by alumino-thermic reactions.
2.	Production of iron powder for autogenous cutting.
3.	Production of liquid gold.
4.	An improved process for production of electrolytic manganese metal (I.P.No. 49355).
5.	An improved process for production of Mn salts from manganese ores and its application for regeneration of spent elect. $MnSO_4$ baths (I.P.No. 48499).
6.	An improved process for electrolytic production of high-purity manganese dioxide (I.P.No. 47982).
7.	Hot-dip aluminizing of ferrous materials (I.P.Nos. 55289, 57938 and 65230).
8.	Production of improved graphite crucibles (carbon-bonded) (I.P.No. 58869).
9.	Production of improved graphite crucibles (clay-bonded) (I.P.No. 62352)

Sl. No.	*Title*
10.	Production of flux for submerged arc-welding (I.P.No. 51524).
11.	Electroplating of metals on aluminium or its alloys (I.P.No. 51524).
12.	Chemical polishing of aluminium (I.P.No. 47401).
13.	Metallization of non-conductors (I.P.No. 45579).
14.	Brass plating from non-cyanide bath (I.P.No. 45565).
15.	Production of dense carbon aggregate suitable for being used as base material for carbon products in general and soderberg paste in particular (I.P.Nos. 62938 & 65696).
16.	Production of ceramic magnets.
17.	Production of electrical resistance alloys for heating elements.
18.	Production of alnico type permanent magnets.
19.	Technology of production of thermostatic bimetals.
20.	Production of electrolytic copper powder (I.P.No. 76997).
21.	Production of modified aluminium alloys containing silicon (I.P.No. 76415).
22.	Production of high-purity sponge iron.
23.	Improvements in or relating to magnesite refractories (I.P.No. 83652).
24.	Production of mullite refractories from kyanite (I.P.No. 58553).
25.	Production of chemically bonded metal-clad or unclad basic refractories (I.P. No. 65610).
26.	Production of completely stabilized dolomite refractories (I.P.No. 61981).
27.	Production of improved magnesium silicate refractories (I.P.No. 57884).
28.	Production of refractory compositions containing non-refractory chrome ores and refractory products (I.P.No. 68174).
29.	Production of vanadium pentoxide and ferro-vanadium from vanadium bearing titaniferrous magnetities.
30.	Production of metal powders for atomization of metals.

BUDGET FIGURES FOR THE NATIONAL METALLURGICAL LABORATORY FOR THE YEAR 1965-66

		(*Figures in lakh of Rupees*) (*One lakh* = 10^5 *rupees*)
RECURRING		
P-1	Pay of Officers	8·682
P-2	Pay of Establishment	10·456
P-3	Allowances	7·332
P-4	Contingencies	6·455
P-6	Maintenance	0·684
P-7	Chemicals, Apparatus and Equipment for Research	10·279
F.S.S.—	Fellowships and Scholarships	0·605
	Scientist Pool	0·172
		44·665
CAPITAL		
P-5 1.	Works	0·431
2.	Services	1·087
3.	Apparatus and Equipment	3·641
4.	Miscellaneous	0·659
		5·818
PILOT PLANT		
PP-1 (i)	Equipment and Installation	4·094
(ii)	Building and Services	0·938
PP-2	Provision for Staff	6·497
PP-3	Purchase of Raw Materials	0·248
PP-4	Miscellaneous and Contingencies	3·740
		15·517
LOW-SHAFT FURNACE PROJECT		
PP-1 (i)	Equipment and Installation	0·888
(ii)	Building and Services	0·014
PP-2	Provision for Staff	3·514
PP-3	Purchase of Raw Materials	2·122
PP-4	Miscellaneous and Contingencies	5·670
		12·208
MAGNESIUM PLANT		
PP-1 (i)	Equipment and Installation	—
(ii)	Building and Services	2·010
PP-2	Provision for Staff	—
PP-3	Purchase of Raw Materials	—
PP-4	Miscellaneous and Contingencies	0·025
		2·035
CONSTRUCTION OF STAFF QUARTERS		4·503
DEFENCE PROJECTS		0·212
	GRAND TOTAL	84·958

APPENDIX I

SCIENTIFIC PAPERS PUBLISHED AND PRESENTED

1. Symposium on Micro-Metallurgy — The Role of Minute Additions to Ferrous and Non-ferrous Metals and Alloys — A Review — P. K. Gupta, *NML Technical Journal*, VII (2), (4-9), 1965.
2. Consideration of Surface Area and Crystallinity of Kaolinite as Factors in its Base Exchange Capacity — P. Prabhakaram, *NML Technical Journal*, VII (2), (10-14), 1965.
3. Development of Superduty Silica Bricks with Indigenous Raw Materials — Gurbux Singh Minhas & H. V. Bhaskar Rao, *NML Technical Journal*, VII (2), (15-19), 1965.
4. Some Observations on the Effect of Heat Treatment on Stress Corrosion Cracking of Hardenable Steel — A. K. Lahiri, T. Banerjee & B. R. Nijhawan, *NML Technical Journal*, VII (2), (20-24), 1965.
5. Determination of Nitrogen in Solid Solution in Steel by Electro-chemical Method — A. N. Chatterjee, N. G. Banerjee & T. Banerjee, *NML Technical Journal*, VII (3), (4-6), 1965.
6. Some Laboratory-scale Trials on Electro-slag Melting — R. D. Gupta, P. K. Gupte & B. R. Nijhawan, *NML Technical Journal*, VII (3), (7-14), 1965.
7. Recovery of Zinc and Lead from the Slime obtained from Electrolytic Zinc Plant — Narinder Singh & Mgr. Inz. Jan Sosin, *NML Technical Journal*, VII (3), (15-20), 1965.
8. Studies on the Preparation of Cryolite by Fluoboric Acid Process — Gurdial Singh & B. C. Kar, *NML Technical Journal*, VII (3), (21-24), 1965.
9. Perminvar Effects in Some Alnico Magnets — Ved Prakash, C. R. Tewari & B. R. Nijhawan, *NML Technical Journal*, VII (3), (25-26), 1965.
10. Studies on Mechanical Properties of Refractories at High Temperatures — T. V. Prasad, *NML Technical Journal*, VII (3), (27), 1965.
11. Scope of Research and Development Work on Substitute Alloys at the National Metallurgical Laboratory — B. R. Nijhawan, *NML Technical Journal*, VIII (1), (56-79), 1966.
12. A New Method of Modification of Aluminium-silicon Alloys — S. S. Bhatnagar, G. G. Nair, P. K. Gupte & B. R. Nijhawan, *Proceedings of* 31*st International Foundry Congress*, Amsterdam, Sept. 1964.
13. Phase Transformation in Electrodeposited Copper-cadmium Alloys — P. L. Ahuja & T. Banerjee, *Journal of the Institute of Metals*, Vol. 94, pp. 200-203, 1966.
14. Pneumatic Steel-making in Basic-lined Side-blown Converter with Phosphoric Indian Pig Iron — B. R. Nijhawan, A. B. Chatterjea & J. Mohan, *Blast Furnace and Steel Plant*, 54 (4), 324-336, 1966.

15. Micro-metallurgy of Abnormality in Steel — B. R. Nijhawan, A. B. Chatterjea & S. S. Bhatnagar, *Journal of Iron and Steel Institute*, 204 (8), 821-830, 1966.

16. Reversion in Al-3·8 per cent Cu Alloys: The Effect of Specimen Size — Interrupted Quench and Plastic Deformation — Ved Prakash & B. R. Nijhawan, *Journal of Indian Institute of Metals*, Vol. 94, pp. 180-185, 1966.

17. Indian Metallurgist and the Present Emergency — B. R. Nijhawan (Presidential Address, Indian Institute of Metals), *Transactions of Indian Institute of Metals*, Vol. 19, March 1966.

18. Transformation Characteristics of Carbides in Low-alloy Steels — J. K. Mukherjee & B. R. Nijhawan, *Transactions of Indian Institute of Metals*, Vol. 18, Dec. 1965.

The following papers were published in *Transactions of the Indian Ceramic Society* (Item Nos. 19-24).

19. A Composite Study of Some Methods of Compaction of Mixes of Dry Press Consistency — H. P. S. Murthy, M. C. Kundra & H. V. Bhaskar Rao, XXIV (2), 35-44, 1965.

20. A Comparative Study of Hydraulic Press and Pneumatic Hammer as Brick Forming Machines — H. P. S. Murthy, M. C. Kundra & H. V. Bhaskar Rao, XXIV (2), 45-54, 1965.

21. Comparative Studies on a High Temperature Coke Oven Coal Tar Pitch as a Binder in the Manufacture of Carbon Refractories and Compacts — H. P. S. Murthy & Bimal Chatterjee, XXIV (4), 122-126, 1965.

22. Pilot Plant Studies on the Production of Forsterite Refractories, Part I: Study of Process Variables — H. P. S. Murthy, M. C. Kundra, P. C. Sen, M. R. K. Rao & H. V. Bhaskar Rao, XXV (1), 1-11, 1966.

23. Development of Superduty Silica Bricks with Indigenous Raw Materials — Gurbux Singh Minhas & H. V. Bhaskar Rao — XXV (1), 25-32, 1966.

24. Investigations on Some Refractory Clays from Jammu & Kashmir — N. S. Sahota, T. V. Prasad & H. V. Bhaskar Rao — XXV (2), 49-60, 1966.

25. Electrodeposition of Iron-nickel-chromium Alloys — S. K. Ray, *Research & Industry*, 10 (12), 367-368, 1965.

26. Present Position of Electroplating in India and Suggested Ways and Means to Improve It.— S. K. Ray, *Chemical Age of India*, 16 (9), 729-732, 1965.

27. Blast Furnace Operation with Oxygen Enriched Blast — A. B. Chatterjea, *Indian Industries Annual*, 1965.

28. Scope of Research in Micro-metallurgy at the National Metallurgical Laboratory — B. R. Nijhawan, *Mid-Year Special Number of Eastern Metals Review*, 1965.

29. Alloy and Special Steel Industry in India — B. R. Nijhawan, *Science & Culture*, April 1965.

30. Powder Metallurgy — B. R. Nijhawan, *Science Reporter*, May 1965.

31. Role of Research in Iron and Steel Industry — B. R. Nijhawan, *Economic Times Supplement*, July 1965.

32. The Economics of Coastal Plant — B. R. Nijhawan, *Economic Times Supplement*, July 1965.

33. Growth of Foundry Industry and Research in India — B. R. Nijhawan, *Special Number on Foundry and Castings Industries of the Economic Times*, Oct. 4, 1965.

34. Production of Ferro-alloys with Oxygenized Blast — B. R. Nijhawan, *Amrita Bazar Patrika, Supplement*, Nov. 13, 1965.

35. Hot-blast Cupola — K. C. Choudhury, B. R. Nijhawan & P. K. Gupte, *Eastern Metals Review, Annual Number*, Feb. 1966.

36. Fluidity Testing Methods for Foundry Alloys — B. R. Nijhawan & G. N. Rao, *Indian Journal of Engineering, Foundry Number*, 1965.

37. Scope for Port-based Iron and Steel Works in India — B. R. Nijhawan, *World Trade, Annual Number*, 1965.

38. Economic Productivity in Iron & Steel Making — B. R. Nijhawan, *Industrial and Engineering Annual*, 1965.

39. Increased Iron Production through Raw Materials' Preparation and Technological Improvements, *Economic Times*, April 1966.

40. Iron-making in Small Blast Furnace — B. R. Nijhawan, *Monograph published by Indian Institute of Metals.*

41. Steel-making in Side-blown Converter with Phosphoric Indian Pig Iron — B. R. Nijhawan, A. B. Chatterjea & Jatinder Mohan — *Tisco Technical Journal*, 13 (2), 72-82, 1966.

42. Micro-metallurgy of Austenitic Grain Size Control of Steel — A. B. Chatterjea & B. R. Nijhawan, *Economic Times Supplement*, March 28, 1966.

43. Low-shaft Furnace Smelting of Low-grade Iron Ore with Lignite Coke — A. B. Chatterjea, *Iron & Steel Engineering* 6 (1), 5-13, 1965.

44. Pig Iron Production in a Low-shaft Furnace — A. B. Chatterjea, *Minerals & Industries*, 11 (4), 3-12, 1965.

45. Pneumatic Steel-making in Basic-lined Side-blown Converter — B. R. Nijhawan, A. B. Chatterjea & J. Mohan, *Economic Times Supplement*, Nov. 14, 1965.

46. Scope of Research and Development Work on Substitute Alloys at National Metallurgical Laboratory — B. R. Nijhawan, *Economic Times Supplement*, April 27, 1966 & *Financial Express Supplement*, April 26, 1966.

47. Copper-manganese Alloys and Their Properties— A. K. Lahiri, K. P. Mukherjee & T. Banerjee, *Economic Times Supplement*, April 27, 1966.

48. Electrolytic Manganese as Substitute Alloys — B. C. Kar & T. Banerjee, *Financial Express Supplement*, April 26, 1966.

49. A Note on ' Stress Corrosion Cracking of Copper-manganese Alloys ' — A. K. Lahiri & T. Banerjee, *Corrosion Science*, Vol. 5, p. 731, 1965.

50. Letter to the Editor ' Stacking Fault Densities of Copper-manganese Alloys ' — A. K. Lahiri & T. Banerjee, *British Journal of Applied Physics*, Vol. 16, p. 217, 1965.

The following papers were presented at the 19th Annual Technical Meeting of the Indian Institute of Metals (Item Nos. 51-64).

51. Curie Temperature of Iron Alloys: Part I — A. N. Sinha & L. J. Balasundaram.
52. Curie Temperature of Iron Alloys: Part II — A. N. Sinha & L. J. Baladundaram.
53. Temper Brittleness — N. K. Das & L. J. Balasundaram.
54. Structure and Magnetic Properties of Some Alnico Magnet Alloys: Effect of Cooling Rate and Tempering Treatment — Ved Prakash, C. R. Tewari & B. R. Nijhawan.
55. Structure of Liquid Al-Cu Alloys — Manjit Singh & Rajendra Kumar.
56. Thermodynamic Properties of Pb-Sn Alloys — Gangotri Misra & Rajendra Kumar.
57. Structure and Crystallization of Metallic Liquids — Rajendra Kumar.
58. Some Aspects of Phase Transformation in Fe-Mn Alloys — S. P. Mukherjee & Rajendra Kumar.
59. Studies on Properties of Ternary Copper Alloys containing Manganese—A. K. Lahiri, K. P. Mukherjee & T. Banerjee.
60. Ausforming: A Thermo-mechanical Technique for the Development of Tougher High Strength Steels — J. J. Irani & J. K. Mukherjee.
61. Decrepitation and Physical Properties of Some Indian Iron Ores — K. N. Gupta, J. S. Padan & A. B. Chatterjea.
62. Some Observations on Ausforming of High Speed Tool Steel — R. K. Dubey, P. K. Gupte & B. R. Nijhawan.
63. Studies on the Recovery of Nickel from Low-grade Siliceous Ore — S. Samanta, R. N. Misra & P. P. Bhatnagar.
64. Some Observations on the Direct Reduction of Iron Ore by Petroleum Product (Naphtha) — B. K. Sexena, A. N. Kapoor & B. R. Nijhawan.

The following papers were presented at the 30th Annual Session of the Indian Ceramic Society (Item Nos. 65-68).

65. Some Observations on the Slip Casting of Kyanite Bodies — A. V. Subramanyan & H. P. S. Murthy.
66. Influence of Process Variables on Products made by Soft Mud Pressing — A. B. Subramanyan & H. P. S. Murthy.
67. Development of Chrome-magnesite Refractories utilizing Indian Chrome Ore — P. C. Sen & M. R. K. Rao.
68. Some Empirical Methods of Evaluating the Suitability of Raw Dolomite for Use in Steel Melting Furnaces — P. C. Sen.

The following papers were presented at the Symposium on ‘ Metallurgy of Substitute Ferrous and Non-ferrous Alloys ’ organized by NML (Item Nos. 69-87).

69. Scope of Research and Development Work on Substitute Alloys at the National Metallurgical Laboratory — B. R. Nijhawan.

70. Economic Utilization, Recovery and Substitution of Some Strategic Non-ferrous Metals — M. Subramanian, R. N. Misra, P. P. Bhatnagar & B. R. Nijhawan.

71. Electrical Conductivity of Substitute Aluminium Alloys — Rajendra Kumar & Manjit Singh.

72. Studies on the Properties of Some Copper-manganese Alloys — A. K. Lahiri, K. P. Mukherjee & T. Banerjee.

73. Development of Substitute Magnetic Materials — Ved Prakash.

74. Research and Development Work on Substitute Electrical Resistance Alloys for Heating Elements — R. Choubey, S. Choudhuri, B. N. Das & B. R. Nijhawan.

75. Some Precipitation Reactions in Substitute Cr-Mn-N Austenitic Stainless Steels — J. K. Mukherjee & B. R. Nijhawan.

76. Research and Development Work on Substitute Nickel-free Austenitic Stainless Steel — B. R. Nijhawan, P. K. Gupte, S. S. Bhatnagar, B. K. Guha & S. S. Dhanjal.

77. The Applications of Electrolytic Manganese as a Substitute Ferro-alloy — B. C. Kar & T. Banerjee.

78. Improved Mild Steels for Structural Purposes — R. Chattopadhyay, B. K. Guha, S. S. Bhatnagar & B. R. Nijhawan.

79. Thermo-mechanical Ausforming Technique for Producing Substitute Ultra High Strength Steels — J. K. Mukherjee.

80. Development of Low Tungsten Substitute Tool Steels — R. K. Dubey, A. K. Das, P. K. Gupte & B. R. Nijhawan.

81. Development of Substitute High Temperature Creep Resistant Alloys — R. Choubey, B. R. Nijhawan, P. K. Gupte, B. N. Das, S. S. Bhatnagar & K. Prasad.

82. Substitution of Non-indigenous Non-ferrous Metals and Alloys — B. R. Nijhawan & C. Sharma.

83. Substitution of Galvanizing by Aluminizing of Steels — S. M. Arora, A. N. Kapoor, P. K. Gupte & B. R. Nijhawan.

84. Economic Uses and Substitution of Non-ferrous Metals and Alloys — B. N. Das.

85. Scope of Aluminium-base Bearing Alloys — B. N. Das & S. K. Banerjee.

86. Physical Metallurgy of Substitution of Alloys — Rajendra Kumar & Ved Prakash.

87. Theoretical Considerations in the Substitutions of Some Common Elements in Alloy Steels — P. Tiwari, S. S. Bhatnagar & B. R. Nijhawan.

88. Electrodeposition of 18: 8 Stainless Steel — S. K. Ray & T. Banerjee, Presented at the 53rd Session of Indian Science Congress (Chemistry Section).

89. A Study of Indian Foundry Bentonite Clays — A. T. Chirayath, J. Mohan, P. K. Gupte & B. R. Nijhawan — Presented at the 32nd International Foundry Congress held at Warsaw.

90. Studies on the Mechanism of Compaction of Ceramic Mixes of Dry Press Consistency — H. P. S. Murthy, M. C. Kundra & H. V. Bhaskar Rao — Eighth Conference of the Silicate Industry at Budapest.

91. Production of Dense Carbon Aggregate from Low and High Volatile Carbonaceous

Substances — Studies on High Volatile Ligneous Coals of Assam — H. P. S. Murthy & Bimal Chatterjee — VII Biennial Conference on Carbon, Cleveland, Ohio.

92. Pneumatic Steel-making in Basic-lined Side-blown Converter with Phosphoric Indian Pig Iron — B. R. Nijhawan, A. B. Chatterjea & J. Mohan — Presented at the Symposium on ' Evaluation of Steel Making Process ' organized by Indian Institute of Metals on 3rd National Metallurgists' Day.

93. Studies on the Recovery of Zinc from Zinc Dross — M. Subramanian, V. S. Sampath, P. P. Bhatnagar & B. R. Nijhawan — Presented at the Seminar on ' Zinc Wastes & Their Utilization ', organized by Indian Lead Zinc Information Centre.

94. Effect of Polar Organic Compounds on Metal Dissolution and Hydrogen Absorption During Pickling — A. K. Lahiri, H. Vaidyanathan & T. Banerjee — Second European Symposium on Corrosion Inhibition.

95. Strategic Non-ferrous Minerals and Metals — B. R. Nijhawan & C. Sharma — Presented at the ' Get-together on Research & Industry ', New Delhi.

96. Self-sufficiency in Indigenous Metals and Alloys — V. Muthukrishnan, K. N. Srivastava, R. M. Krishnan & B. R. Nijhawan — Presented at the ' Get-together on Research & Industry ', New Delhi.

97. Hand Sorting and Mechanical Picking — B. R. Nijhawan — Presented at the U.N. Seminar on ' Ore Concentration in Water-short Areas ' in New York, Feb. 1966.

98. Some Aspects of Iron and Steel Research in the United Kingdom — B. R. Nijhawan — Presented at the Meeting of Indian Institute of Metals, Calcutta, June 1965.

99. On the Nature of Fe-Al and Fe-Si Bonds in Fe_3Al and Fe_3Si Superlattices — L. J. Balasundaram — Presented at the Symposium on ' Nuclear Physics and Solid State Physics ' organized by the Tata Institute of Fundamental Research, Bombay.

APPENDIX II

SCIENTIFIC INVESTIGATIONS COMPLETED AND REPORTS PREPARED

1. Moulding Characteristics of Hardwar River Sand Supplied by M/s. B.H.E. Ltd, Hardwar — R. C. Arora, V. S. Bhandary & P. K. Gupte (I.R. 322/65).
2. Pilot Plant Studies on the Beneficiation and Sintering Studies on a Mixed Iron Ore Sample from Kondekasa Block of Dalli Pahar Deposits for Bhilai Steel Plant, Hindustan Steel Ltd — M. V. Ranganathan, B. L. Sengupta, G. S. R. K. Rao, G. P. Mathur & P. I. A. Narayanan (I.R. 323/65).
3. Reducibility of Specular Hematite Iron Ore Samples of Nathara-Ki-Pal Deposits, Rajasthan — M. Subramanian, S. B. Mathur & P. P. Bhatnagar (I.R. 324/65).
4. Pilot Plant Studies on (1) Beneficiation of an Iron Ore Sample from the Kiriburu Mines of National Mineral Development Corporation, (2) Production of Additional —10 mm. Fines, and (3) Production of High Basicity Sinters for the Bokaro Steel Plant — R. K. Kunwar, A. Peravadhanulu, N. Chakravarty, G. S. R. K. Rao, G. P. Mathur & P. I. A. Narayanan (I.R. 325/65).
5. Moulding Characteristics of Godavari River Sand No. 1 — S. K. Sinha Babu, V. S. Bhandary & P. K. Gupte (I.R. 326/65).
6. Moulding Characteristics of Pachpahar Sand — S. K. Sinha Babu, V. S. Bhandary & P. K. Gupte (I.R. 327/65).
7. Moulding Characteristics of Kandleru River Sand — R. C. Arora, V. S. Bhandary & P. K. Gupte (I.R. 328/65).
8. Moulding Characteristics of Zircon Sand — K. S. Vijayanarayanan, V. S. Bhandary & P. K. Gupte (I.R. 329/65).
9. Moulding Characteristics of Manduri Glass Sand — P. R. Sastry, V. S. Bhandary & P. K. Gupte (I.R. 330/65).
10. Reduction of Silica Content in a Magnesite Sample from Salem, Madras State — S. K. Banerjee & P.I.A. Narayanan (I.R. 331/65).
11. Beneficiation of a Phosphate Rock Sample from Singhbhum District, Bihar — P. V. Raman & P. I. A. Narayanan (I.R. 332/65).
12. Grindability Tests on a Fluorspar Sample from Rajasthan — S. K. Dhar, S. K. Banerjee & P. I. A. Narayanan (I.R. 333/65).
13. Flotation Studies on a Copper Ore Sample from Ghatiwalli Adit, Khetri, Rajasthan received from National Mineral Development Corporation. Ltd — R. Ganesh, A. E. J. Gallagher & P. I. A. Narayanan (I.R. 334/65).
14. Reducibility of Kiriburu Iron Ores and Sinters — M. Subramanian, S. B. Mathur & P. P. Bhatnagar (I.R. 335/65).

15. Production of High-carbon Ferro-chrome using Talcher Low-temperature Carbonization Coke as the Reductant in the Pilot Submerged Arc Smelting Furnace — M. Subramanian, V. S. Sampath, M. P. Menon, P. P. Bhatnagar & B. R. Nijhawan (I.R. 336/65).

16. Beneficiation and Pelletization Studies on Iron Ore Fines from Redi Mines, Ratnagiri District, Maharashtra — P. K. Sinha, P. K. Verma & P. I. A. Narayanan (I.R. 337/65).

17. Beneficiation and Sintering Studies on Laminated Iron Ore from Dalli Mines of Bhilai Steel Plant of Hindustan Steel Ltd — P. V. Raman, N. Chakravarty, G. S. R. K. Rao, G. P. Mathur & P. I. A. Narayanan (I.R. 338/65).

18. Investigation of Some Refractory Clays from Jammu & Kashmir — N. S. Sahota, T. V. Prasad & H. V. Bhaskar Rao (I.R. 339/65).

19. Comparative Studies on a High-temperature Coke Oven-Coal Tar Pitch as a Binder in the Manufacture of Carbon Refractories and Compacts — H. P. S. Murthy & Bimal Chatterjee (I.R. 340/65).

20. Investigation on MP33 Bentonite Supplied by M/s. Ambica Minechem Industries, Bhavnagar, Gujarat — R. C. Arora, V. S. Bhandary & P. K. Gupte (I.R. 341/65).

21. Investigation on PHG Bentonite Supplied by M/s. Ambica Minechem Industries, Bhavnagar, Gujarat — R. C. Arora, V. S. Bhandary & P. K. Gupte (I.R. 342/65).

22. Beneficiation and Sintering Studies on Massive Iron Ore Samples from Dalli Mines of Bhilai Steel Plant of Hindustan Steel Ltd — R. K. Kunwar, M. V. Ranganathan, B. L. Sen Gupta, N. Chakravarty, G. S. Ramakrishna Rao, G. P. Mathur & P. I. A. Narayanan (I.R. 343/65).

23. Production of Sized Fraction from a Quartz Sand Sample from Ennore District, Madras State — N. Chakravarty, G. S. Ramakrishna Rao, G. P. Mathur & P. I. A. Narayanan (I.R. 344/65).

24. Moulding Characteristics of Bhandhara Sand — P. R. Sastry, V. S. Bhandary & P. K. Gupte (I.R. 345/65).

25. Moulding Characteristics of Bhainsahi Glass Sand (Upper Unit) — S. K. Sinha Babu, V. S. Bhandary & P. K. Gupte (I.R. 346/65).

26. Moulding Characteristics of Bhainsahi Glass Sand (Lower Unit) White — S. K. Sinha Babu, V. S. Bhandary & P. K. Gupte (I.R. 347/65).

27. Beneficiation and Sintering Studies on a Compact Laminated Iron Ore Sample from Dalli Mines of Bhilai Steel Plant of Hindustan Steel Ltd — P. V. Raman, N. Chakravarty, G. S. R. K. Rao, G. P. Mathur & P. I. A. Narayanan (I.R. 348/65).

28. Production of Sized Sand for Foundry and Glass making from Quartzite Sands from Chaibasa, Singhbhum District, Bihar — S. K. Dhar, S. K. Banerjee & P. I. A. Narayanan (I.R. 349/65).

29. Flotation Studies on a Copper Ore Sample from Kolihan, Rajasthan — S. K. Banerjee & P. I. A. Narayanan (I.R. 350/65).

30. Utilization of Dabok Limestone for Iron Smelting -- A. B. Chatterjee & B. R. Nijhawan (I.R. 351/65).

31. Appraisal of Some Indian Iron Ores — K. N. Gupta, J. S. Padan, A. B. Chatterjea (I.R. 352/65).

32. Flotation Studies on a Copper Ore Sample from Rakha Mines, Bihar, of National Mineral Development Corporation Ltd — K. N. Rakshit, P. V. Raman & P. I. A. Narayanan (I.R. 353/66).

33. Pilot Plant Studies on Beneficiation and Sintering of a Sample of Hydroxide Ore from Dalli Mines of Hindustan Steel Ltd — R. K. Kunwar, B. L. Sengupta, N. Chakravorty, G. S. R. K. Rao, G. P. Mathur & P. I. A. Narayanan (I.R. 354/66).

34. Investigation on 'BS — Bentonite' Received from M/s. Hargovindas Shivlal & Co., Bombay — Amitava Das, V. S. Bhandary & P. K. Gupte (I.R. 355/66).

35. Investigation on 'BR-Bentonite' from Gujarat — Amitava Das, V. S. Bhandary & P. K. Gupte (I.R. 356/66).

36. Investigation on the Use of Pyrophyllite as a Refractory Material — P. C. Sen (I.R. 357/66).

37. Beneficiation Studies on a Copper Ore Sample from Shaft No. 3, Khetri, Rajasthan, of the National Mineral Development Corporation Ltd — R. Ganesh, A. E. J. Gallagher & P. I. A. Narayanan (I.R. 358/66).

38. Development of Chrome-magnesite Refractories from Indian Chrome Ores — P. C. Sen & M. Ramakrishna Rao (I.R. 359/66).

39. Moulding Characteristics of Amegonder Sand received from Directorate of Geology & Mining, Lucknow, U.P. — R. C. Arora, V. S. Bhandary & P. K. Gupte (I.R. 360/66).

40. Moulding Characteristics of Maharashtra Silica Sand (MSS-6) — R. R. Dash, V. S. Bhandary & P. K. Gupte (I.R. 361/66).

41. Moulding Characteristics of Bhainsahi Glass Sand (Lower Unit) Yellow — S. K. Sinha Babu, V. S. Bhandary & P. K. Gupte (I.R. 362/66).

42. Beneficiation and Sintering Studies on a Sample of Iron Ore from Meghataburu Mines of National Mineral Development Corporation Ltd. — M. V. Ranganathan, N. Chakravarty, B. L. Sengupta, G. S. Ramakrishna Rao, G. P. Mathur & P. I. A. Narayanan (I.R. 363/66).

43. Reduction of Silica Content of a Magnesite Sample from Salem, Madras State, employing Crushing and Screening Methods — M. V. Ranganathan, G. P. Mathur & P. I. A. Narayanan (I.R. 364/66).

44. Utilization of Low-temperature carbonized Coke made from Talcher Non-coking Coal for Iron smelting — A. B. Chatterjee & B. R. Nijhawan (I.R. 365/66).

45. Pilot Plant Studies on Beneficiation and Sintering of a Mixed Laminated Iron Ore Sample from Rajhara Mines of Bhilai Steel Plant, Hindustan Steel Ltd — M. V. Ranganathan, B. L. Sengupta, N. Chakravarty, G. S. Ramakrishna Rao, G. P. Mathur & P. I. A. Narayanan (I.R. 366/66).

46. Electrolytic Recovery of Tin from Tinplate Scrap — B. N. Singh & H. K. Chakravarti (R.R. 195/65).

47. A Study of Indian Foundry Bentonite Clays — A. T. Chirayath, Jatinder Mohan, P. K. Gupte & B. R. Nijhawan (R.R. 196/65).

48. Studies on the Mechanism of Compaction of Ceramic Mixes of Dry Press Consistency — H. P. S. Murthy, M. C. Kundra & H. V. Bhaskar Rao (R.R. 197/65).

49. Formulation of Electroplating Salts, Part I — Alkaline Copper-plating Salt — S. K. Ray (R.R. 198/65).

50. Formulation of Electroplating Salts, Part II — Dull Nickel-plating Salt — S. K. Ray (R.R. 199/65).

51. Formulation of Electroplating Salts, Part III — Improvement on Dull Nickel-plating Salt — A. N. Chatterjee & S. K. Ray (R.R. 200/65).

52. Expansion Characteristics of Indigenous Sands — V. S. Bhandary, P. K. Gupte & B. R. Nijhawan (R.R. 201/65).

53. Particle Size Determination of Indigenous Clays and Their Properties — T. A. Beck, V. S. Bhandary & P. K. Gupte (R.R. 202/65).

54. Dephosphorization of Indian Pig Iron in Basic-lined Cupola — R. K. Dubey, C. A. Naresh Rao, S. S. Bhatnagar & P. K. Gupte (R.R. 203/65).

55. Utilization of T.T.T. Curves for Heat Treatment of Alloy Steel Castings — R. K. Dubey, P. K. Gupte & B. R. Nijhawan (R.R. 204/65).

56. Reversion in Aluminium-3·8 per cent Copper Alloy — The Effect of Specimen Size, Interrupted Quench and Plastic Deformation — Ved Prakash & B. R. Nijhawan (R.R. 205/65).

57. Structure and Magnetic Properties of Some Lead-Barium Ferrites — S. K. Bose & Ved Prakash (R.R. 206/65).

58. The Effect of Interrupted Quench and Specimen Size on the Natural Aging of an Aluminium-3·8 per cent Copper Alloy — Ved Prakash (R.R. 207/65).

59. Extraction Characteristics of Alumina from Mysore Bauxite — M. S. Mahanty, R. N. Misra & P. P. Bhatnagar (R.R. 208/65).

60. Determination of Nitrogen in Solid Solution in Steel by Electrochemical Method — A. N. Chatterjee, N. G. Banerjee & T. Banerjee (R.R. 209/65).

61. Some Laboratory-scale Trials on Electro-slag Melting — R. D. Gupta, P. K. Gupte & B. R. Nijhawan (R.R. 210/65).

62. Effect of Polar Organic Compounds on Metal Dissolution and Hydrogen Absorption During Pickling — A. K. Lahiri, H. Vaidyanathan & T. Banerjee (R.R. 211/65).

63. Production of Dense Carbon Aggregate from Low and High Volatile Carbonaceous Substances — Studies on High Volatile Ligneous Coals of Assam.— H. P. S. Murthy & Bimal Chatterjee (R.R. 212/65).

64. Pilot Plant Studies on the Production of Forsterite Refractories, Part I — Study of Process Variables — H. P. S. Murthy, M. C. Kundra, P. C. Sen, M. R. K. Rao & H. V. Bhaskar Rao (R.R. 213/65).

65. Occurrence and Refractory Properties of Rajasthan Magnesite — P. C. Sen, M. Rama Krishna Rao, Ashimesh Dutt & H. V. Bhaskar Rao (R.R. 214/65).

66. Some Aspects of Cation Exchange of Kaolinite — P. Prabhakaram (R.R. 215/65).

67. Structure and Magnetic Properties of Some Alnico Magnet Alloys — Effects of Cooling Rate and Tempering Treatment — Ved Prakash, C. R. Tewary & B. R. Nijhawan (R.R. 216/65).

68. Studies on the Recovery of Zinc from Zinc Dross — M. Subramanian, V. S. Sampath, P. P. Bhatnagar & B. R. Nijhawan (R.R. 217/65).

69. Electrodeposition of 18: 8 Stainless Steel — S. K. Ray & T. Banerjee (R.R. 218/65).

70. Perminvar Effects in Some Alnico Magnets — Ved Prakash, C. R. Tewary & B. R. Nijhawan (R.R. 219/65).

71. Studies on the Recovery of Nickel from Low-grade Siliceous Ore — (Miss) S. Samanta, R. N. Misra & P. P. Bhatnagar (R.R. 220/65).

72. Study on Temper Brittleness — N. K. Das & L. J. Balasundaram (R.R. 221/65).

73. Curie Temperatures of Iron-aluminium & Iron-silicon Alloys-II — A. N. Sinha & L. J. Balasundaram (R.R. 222/65).

74. Some Observations on Ausforming of High Speed Tool Steel — R. K. Dubey, A. K. Das, P. K. Gupte & B. R. Nijhawan (R.R. 233/65).

75. Studies on Properties of Ternary Copper Alloys containing Manganese — A. K. Lahiri, K. P. Mukherjee & T. Banerjee (R.R. 224/65).

76. Study on the Suitability of Cellulose Materials as Core Binders — T. A. Beck, V. S. Bhandary & P. K. Gupte (R.R. 225/66).

77. Some Observations on the Direct Reduction of Iron Ore by Petroleum Product (Naphtha) — B. K. Saxena, A. N. Kapoor & B. R. Nijhawan (R.R. 226/66).

78. Role of Different Variables affecting Atmospheric Corrosion of Steel — S. Rao Addanki, K. P. Mukherjee, A. K. Lahiri & T. Banerjee (R.R. 227/66).

79. Stress Corrosion Cracking of Homogeneous Copper Alloys — A. K. Lahiri & T. Banerjee (R.R. 228/66).

APPENDIX III

MAJOR SPONSORED PROJECTS AT THE NATIONAL METALLURGICAL LABORATORY

Sl. No.	*Title*	*Sponsor*
1.	Flotation Studies on a Copper Ore Sample from Kolihan, Rajasthan.	National Mineral Development Corporation Ltd
2.	Floation Studies on a Copper Ore Sample from Rakha Mines, Bihar, of N.M.D.C. Ltd.	National Mineral Development Corporation Ltd
3.	Beneficiation Studies on a Copper Ore Sample from Shaft No. 3, Khetri, Rajasthan, of N.M.D.C. Ltd.	National Mineral Development Corporation Ltd
4.	Beneficiation and Sintering Studies on a Sample of Iron Ore from Meghataburu Mines of N.M.D.C. Ltd.	National Mineral Development Corporation Ltd
5.	Flotation Studies on a Copper Ore Sample from Ghatiwalli Adit, Khetri, Rajasthan, received from N.M.D.C. Ltd.	National Mineral Development Corporation Ltd
6.	Reducibility of Kiriburu Iron Ores and Sinters.	Bokaro Steel Ltd
7.	Beneficiation and Sintering Studies on Laminated Iron Ore from Dalli Mines of Bhilai Steel Plant of HSL.	Bhilai Steel Plant
8.	Beneficiation and Sintering Studies on Massive Iron Ore Samples from Dalli Mines of Bhilai Steel Plant of HSL.	Bhilai Steel Plant
9.	Beneficiation and Sintering Studies on a Compact Laminated Iron Ore Sample from Dalli Mines of Bhilai Steel Plant of HSL.	Bhilai Steel Plant
10.	Pilot Plant Studies on Beneficiation and Sintering of a Sample of Hydroxide Ore from Dalli Mines of HSL.	Bhilai Steel Plant
11.	Beneficiation and Pelletization Studies on Iron Ore Fines from Redi Mines, Ratnagiri District, Maharashtra.	Government of Maharashtra
12.	Utilization of Dabok Limestone for Iron Smelting.	Kamani Engineering Corporation
13.	Utilization of Low-temperature Carbonized Coke made from Talcher Non-coking Coal for Iron Smelting.	Industrial Development Corporation, Orissa
14.	Reduction of Silica Content in a Magnesite Sample from Salem, Madras State.	Salem Magnesite Pvt. Ltd