ANNUAL REPORT

1966-1967

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

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INTRODUCTION

THE progress of research and development work at the National Metallurgical Laboratory has been steady and rewarding. Besides consolidation of research activities in diverse fields, the research and development projects have been further streamlined to suit the immediate requirement of the country's needs. During the period under review, metallurgical industries both in Public and Private Sectors continued to call upon heavily the assistance of the National Metallurgical Laboratory in the diversified subjects of metallurgical research pertaining to raw material problems, quality production, process control, etc. Steady progress has been maintained on the various pilot plants of the laboratory, e.g. Mineral Beneficiation Pilot Plant, Low-shaft Furnace Pilot Plant, Ferro-alloy Pilot Plant, Electrolytic Manganese Pilot Plant, Hot-dip Aluminizing Pilot Plant, etc.

Beneficiation and sintering studies on iron ore samples, limestone, fluorspar, apatite, pyrite, gold ore, etc., were conducted on behalf of Hindustan Steel Ltd., National Mineral Development Corporation, Gujarat Mineral Development Corporation, State Governments and other industrial concerns. Systematic investigations were conducted on the extraction of nickel from low grade nickel-bearing ores from different regions of the country. The pioneering and challenging investigation on the direct injection of liquid naphtha into the hearth of Low Shaft Furnace with simultaneous enrichment of the blast with oxygen were successfully conducted.

Work on behalf of Defence Establishments was conducted on top priority basis. The National Metallurgical Laboratory has so far reconditioned about 9 tons of magnesium powder and supplied to various Ordnance Establishments.

The long range research projects on development of substitute alloys and products have yielded encouraging results. The scope of work in the field of non-ferrous metals has been further enlarged and close contact is maintained in this connection with Non-ferrous Metal Manufacturers' Association. Graphite crucible based on NML developed technique is now produced on commercial scale by the licensee firm.

Foundry Stations established so far, e.g. those at Batala, Madras and Howrah, are working in full swing. The Foundry Station at Ahmedabad is almost complete. A number of long and short term projects are under way at the Marine Corrosion Research Station at Digha.

Progress on fundamental projects has been pursued with equal vigour and during the period one scientist has been awarded a doctorate degree on the basis of the thesis submitted by him. The design and fabrication of the many of the pilot plant equipment and apparatus are progressing satisfactorily.

During the period under review, this Laboratory had the privilege of collaborating with the Institute of Indian Foundrymen in organizing the 33rd International Foundry Congress for the first time in any country of east of Suez.

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

Patents Filed

1. Indian Patent No. 108583 — Chemically bonded forsterite refractories

and method of their preparation" (26-12-1966).

2. Indian Patent No. 108081 — Refractory ramming, plastering and patching mixes consisting of kyanite and method of making and using the same (12-11-1966).
3. Indian Patent No. 107982 — Stable castable suspensions of non-plastic alumino-silicate materials and methods of making the same (16-11-66).
4. Indian Patent No. 106906 — An improved method for removal of iron and upgrading ferruginous chrome ores and other low grade ferruginous ores (3-9-1966).
5. Indian Patent No. 105895 — A process for the conversion of molten iron of various compositions to different grades of steel by employing air for oxidizing (25-6-1966).

Patents Accepted

1. Indian Patent No. 96661 — A novel method of separating iron from ilmenite and its application in the upgrading of ferruginous ores (11-5-1966).
2. Indian Patent No. 96017 — Production of high purity iron (12-7-1966).
3. Indian Patent No. 94768 — An improved device for isolation of dross in molten metallic baths during continuous hot-dip processing of strip or wire (22-4-1966).
4. Indian Patent No. 91134 — Improvement in or relating to a precision temperature controller for use with electrical resistance furnaces up to 1600°C. (15-6-1966).

Patents Sealed

1. Indian Patent No. 96661 — A novel method of separating iron from ilmenite and its application in the upgrading of ferruginous ores (11-5-1966).
2. Indian Patent No. 94769 — An improved device for the continuous hot-dip coating of metallic strip and wire (22-6-1966).
3. Indian Patent No. 94767 — An improved C.I. Pot for melting and holding of non-ferrous metals in general and aluminium and zinc in particular (10-11-1966).
4. Indian Patent No. 82191 — An improved jacketted electrolytic cell for the electrodeposition of metals and metallic oxides in general and MnO_2 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 magnets.
7. Hot-dip aluminizing of ferrous materials.
8. Refractory compositions — comprising graphite and silicon carbide (carbon-bounded 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 alumino-thermic reactions.
11. Production of iron powder for autogenous cutting.

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

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

RESEARCH PROJECTS

1.0 Pilot Plant Studies on Beneficiation and Sintering on a Sample of Iron Ore from Meghataburu Mines of National Mineral Development Corporation

At the instance of National Mineral Development Corporation, extensive beneficiation and sintering studies were conducted on pilot plant scale on a 100-tonnes sample of iron ore from Meghataburu mines with a view to supplying the ore to the Bokaro Steel Plant. The sample assayed: Fe 61·87, SiO_2 2·08, Al_2O_3 4·00 and loss on ignition (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 $+3/8$ in. ($+9·5$ mm.) size free from adherent ore fines for direct charging into the blast furnace or for export purposes and a $-3/8$ in. ($-9·5$ mm.) slime-free classifier sand product for sintering. The results obtained from both wet screening test 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 63·60 per cent Fe, 1·2 per cent SiO_2 and 3·3 per cent Al_2O_3. 32·3 per cent of the total SiO_2 and 18·1 per cent of the total Al_2O_3 should be rejected in slime weighing 6·7 per cent. Though the insoluble content of the $-3/8$ in. ($-9·5$ mm.) classifier sand obtained after washing was not considered high, it was subjected to jigging. The combined jig concentrate assayed 62·50 per cent Fe, 2·33 per cent SiO_2 and 2·90 per cent Al_2O_3 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 $-3/8$ in. ($-9·5$ 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 super-fluxed 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.

The results of beneficiation tests is given in Fig. 1. The flowsheet employed for treating this sample 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 rate vis-a-vis prepared and beneficiated ore burdens from incidental much desired benefits of uniform smelting operation and slag volumes. Table 1 gives the economic evaluation of beneficiation of iron ore assessed on the following basis which has uniformly been kept for Indian iron ores.

(i) The cost of raw ore as mined is taken at Rs. 5·00 per tonne. Transport charges for ore are not included in this.

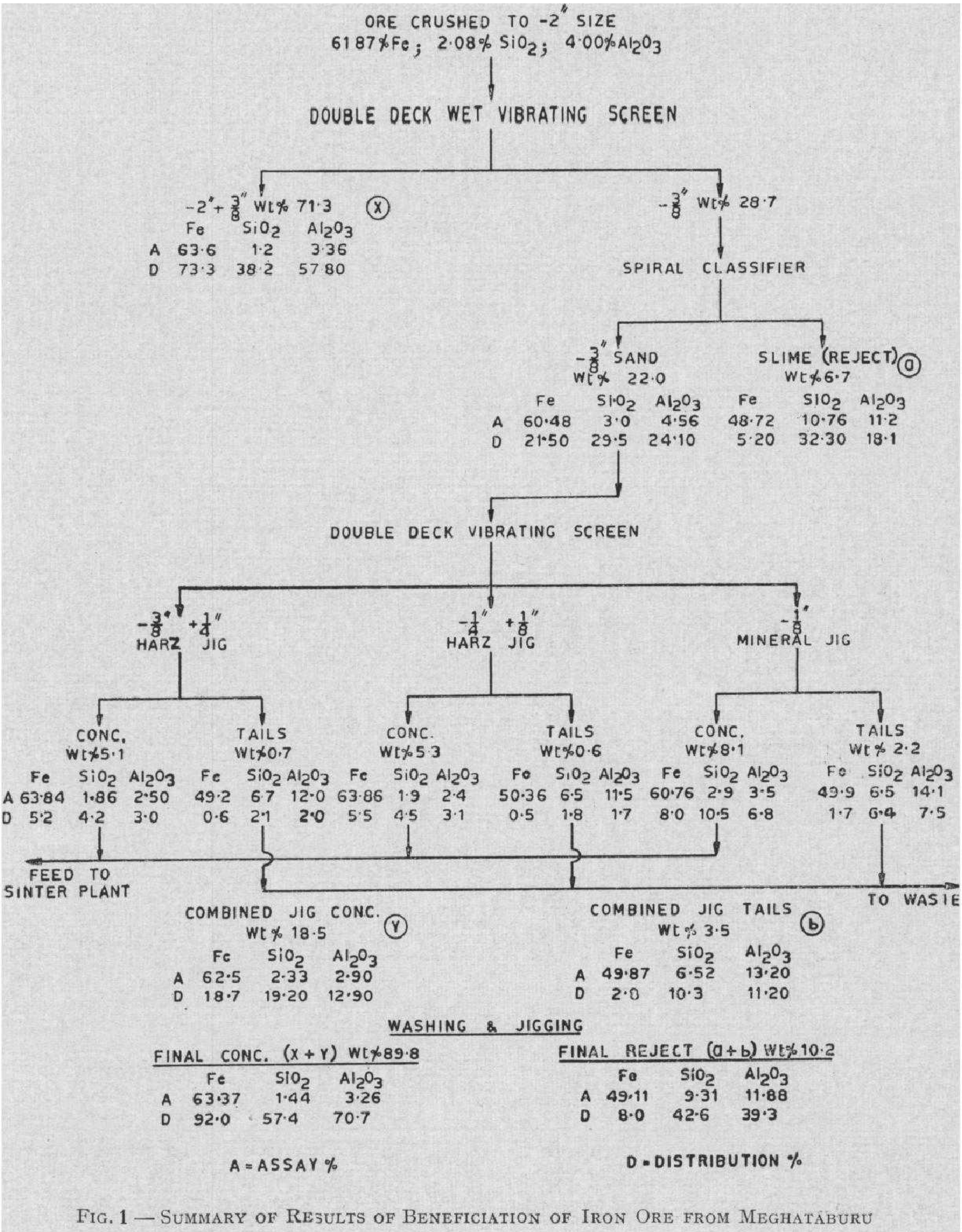


FIG. 1 — SUMMARY OF RESULTS OF BENEFICIATION OF IRON ORE FROM MEGHATABURU

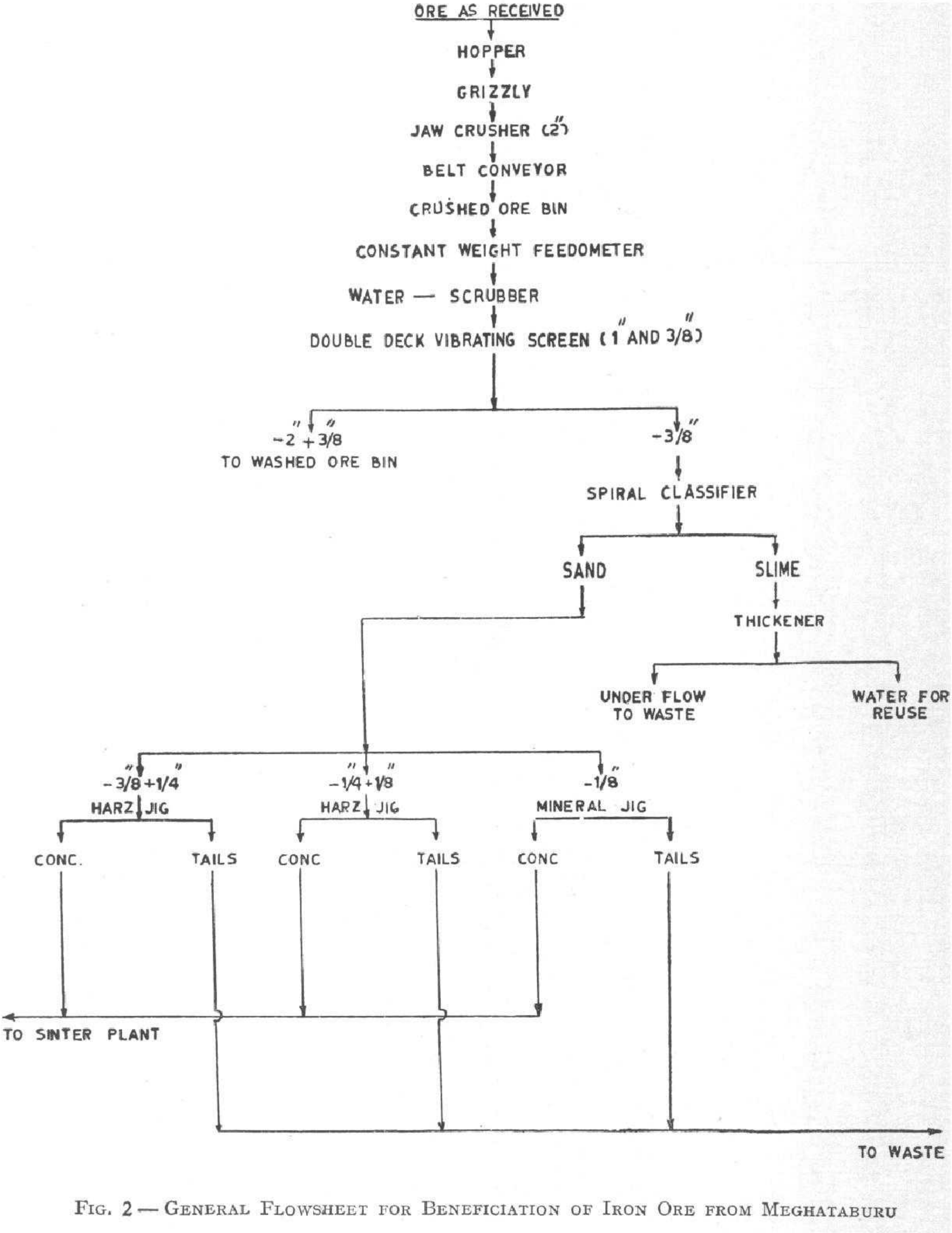


FIG. 2 — GENERAL FLOWSHEET FOR BENEFICIATION OF IRON ORE FROM MEGHATABURU

Table 1 — Economic Evaluation of Beneficiation of Meghataburu Iron Ore

	Crushing to 2″, dry screening and utilization of $-\frac{3}{8}''$ fines			Crushing to 2″, dry screening and rejection of $-\frac{3}{8}''$ fines			Crushing to 2″, scrubbing and wet screening and dewatering of $-\frac{3}{8}''$ fines			Crushing to 2″, scrubbing and wet screening and beneficiation of $-\frac{3}{8}''$ fines only		
	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, %	61·87	2·08	4·00	62·5	1·68	3·80	62·87	1·62	3·64	63·37	1·44	3·26
Yield tonnes/day	14,000 (100%)			11,242 (8·03%)			13,062 (93·3%)			12,572 (89·8%)		
Capital cost of ore treatment plant	Rs. 2·50 crores			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. 1·113			Rs. 1·287			Rs. 1·792		
(b) Operating cost	Rs. 0·180			Rs. 0·224			Rs. 0·259			Rs. 0·362		
(c) Cost of raw ore as mined	Rs. 5·000			Rs. 6·227			Rs. 5·359			Rs. 5·568		
Total cost per tonne of prepared ore	Rs. 6·074			Rs. 7·564			Rs. 6·905			Rs. 7·222		
Tonne of ore required for tonne of pig iron	1·557			1·541			1·532			1·520		
Total cost of ore required per tonne of pig iron	Rs. 9·458			Rs. 11·650			Rs. 10·580			Rs. 11·730		
Difference in alumina per cent	—			0·20			0·36			0·74		
Expected saving in production cost per tonne of pig iron in blast furnace iron smelting due to decrease in alumina by beneficiation	—			Rs. 1·00			Rs. 1·80			Rs. 3·70		

(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 coke rate and flux rate would decrease by 40 kg. 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. 5·00 after allowing for depreciation, etc.

1.1 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 one massive ore) were received from Rajhara mines of Bhilai Steel Plant for detailed beneficiation and sintering studies. 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 62·2 per cent Fe, 3·65 per cent SiO_2, 3·47 per cent Al_2O_3 and 4·5 per cent L.O.I. Petrological examination of the sample showed that hematite was the chief iron-bearing mineral followed by minor amounts of goethite and magnetite.

Screenability tests with varying moisture contents in the ore indicated that screening efficiency would be minimum, when the moisture content of the ore was 10 per cent. Dry screening test performed after crushing the sample to −2 in. (−50·8 mm.) size showed that the +3/8 in. (+9·5 mm.) lumps constituting 46·6 per cent by weight, assayed 63·8 per cent Fe, 1·4 per cent SiO_2 and 2·51 per cent Al_2O_3. Washing test (scrubbing followed by wet screening) carried out with the sample at −2 in. (−50·8 mm.) size indicated that a clean sized, washed product free from adhered ore fines and −3/8 in. (−9·5 mm.) classifier sand product free from undesirable slime could be obtained. The results showed that (1) the improvement in the Fe grade of washed lumps was 2 per cent, and (2) the rejection of slica and alumina through slime was 51·7 and 34·2 per cent respectively, for a corresponding Fe loss of 15·1 per cent. The −3/8 in. (−9·5 mm.) classifier sand was almost slime-free, was of a better grade than 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 silica and alumina content could be brought down by jigging. Jigging of the above product yielded a combined jig concentrate constituting 26·9 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 63·65 per cent Fe 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 −3/8 in. (−9·5 mm.) washed sand for sinter production.

Sintering characteristics of −3/8 in. (−9·5 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 also possessed a high degree of oxidation when compared with those made by the conventional sintering process.

The summary of the results of washing and jigging are given in Fig. 3. Based on these results, a general flow sheet adopted is given in Fig. 4.

The overall economics of beneficiation is given in Table 2.

1.2 Pilot Plant Beneficiation and Sintering Studies on 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 from Rajhara mines of Bhilai Steel Plant. The samples (i) and (ii) were mixed thoroughly in the proportion of

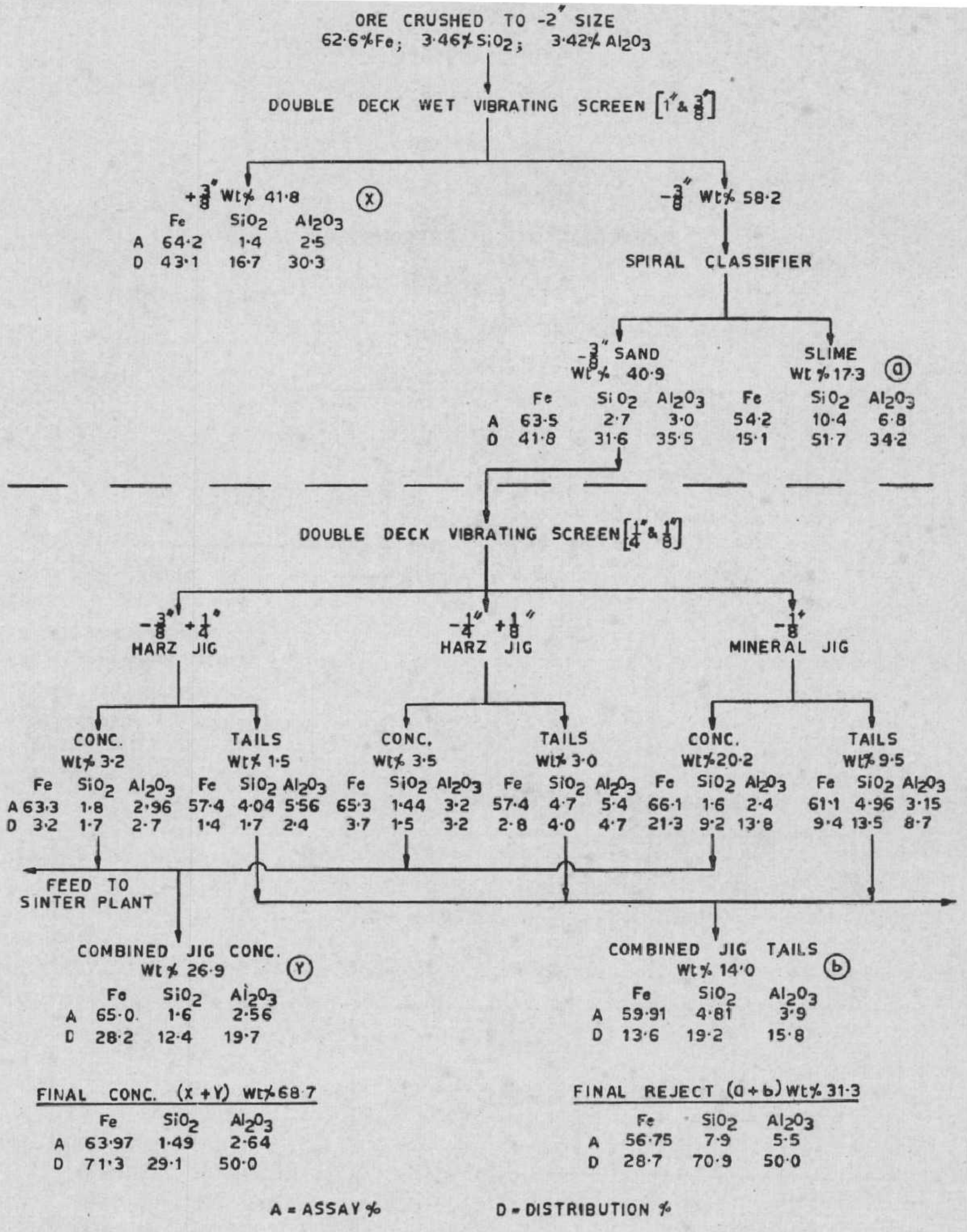


FIG. 3 — SUMMARY OF RESULTS OF WASHING & JIGGING OF RAJHARA MIXED LAMINATED IRON ORE (AT −2″ SIZE)

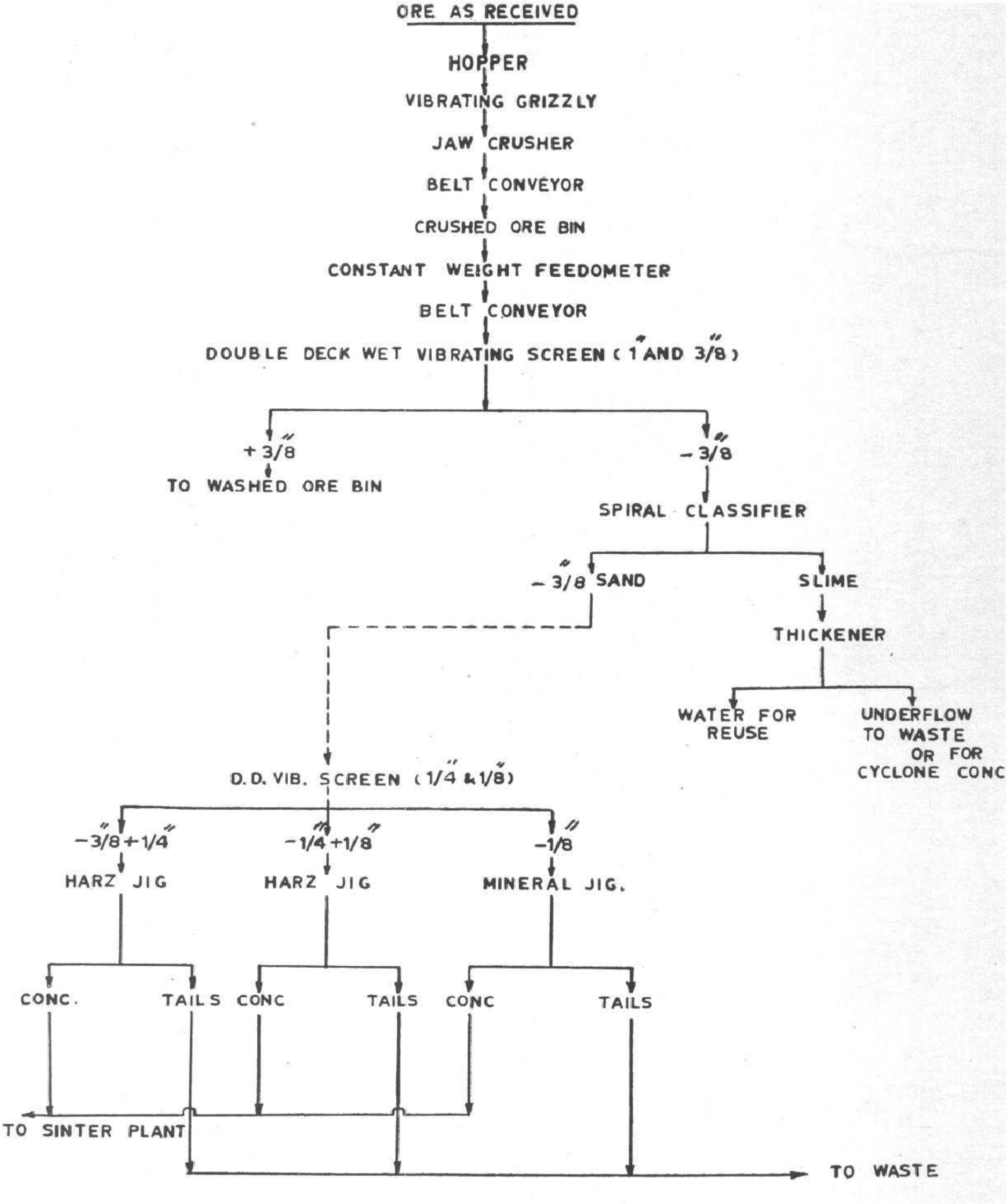


FIG. 4 — FLOWSHEET EMPLOYED IN NML FOR THE BENEFICIATION OF RAJHARA LAMINATED IRON ORE

Table 2 — Economic Evaluation of Beneficiation of Mixed Laminated Iron Ore from Rajhara Mines

	Crushing to −2″, dry screening and utilization of −$\frac{3}{8}$″ fines			Crushing to −2″, dry screening and rejection of −$\frac{3}{8}$″ fines			Crushing to −2″, washing and dewatering of −$\frac{3}{8}$″ fines			Crushing to −2″, washing and beneficiation of −$\frac{3}{8}$″ 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 ores, %	62·6	3·46	3·42	63·8	1·4	2·51	63·84	2·04	2·75	63·97	1·49	2·64
Yield tonnes/day	14,000 (100%)			6,524 (46·6%)			11,578 (86·7%)			9,618 (68·7%)		
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 beneficiated ore												
(a) Capital cost	Rs. 0·894			Rs. 1·918			Rs. 1·453			Rs. 2·341		
(b) Operating cost	Rs. 0·180			Rs. 0·387			Rs. 0·292			Rs. 0·471		
(c) Cost of raw ore as mined	Rs. 5·000			Rs. 10·730			Rs. 6·045			Rs. 7·278		
Total cost per tonne of prepared ore	Rs. 6·074			Rs. 13·035			Rs. 7·790			Rs. 10·090		
Tonne of ore required per tonne of pig iron	1·539			1·510			1·509			1·507		
Total cost of the ore required per tonne of pig iron	Rs. 9·348			Rs. 19·682			Rs. 11·75			Rs. 15·20		
Difference in Alumina per cent	—			0·91			0·67			0·78		
Expected saving in production cost per tonne of pig iron in blast furnace iron smelting due to decrease in alumina by beneficiation	—			4·55			3·35			3·90		

9:1 and the results of pilot plant beneficiation and sintering studies carried out with the mixed laminated ore has been furnished in item No. 1.1.

The second series of tests was carried out on a 40 tonnes mixed sample by mixing the massive iron ore sample (iii), with the mixed laminated iron ore in the proportion of 45:55. The mixed sample of massive and laminated ore was of good grade assaying Fe 65·6, SiO_2 2·1, Al_2O_3 2·51 and L.O.I. 2·7 per cent. Petrological examination of the sample showed that hematite was the chief iron bearing mineral and was accompanied by smaller amounts of goethite and minor amounts of magnetite. The chief contaminant in the ore was laterite and highly aluminous clayey matter followed by small amounts of quartz, etc.

Screenability tests showed that screenability was least efficient at 5·0 per cent moisture. Dry screening and scrubbing followed by wet screening (washing) of the sample after crushing to 2 in. (50·8 mm.) yielded +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 difficulties. 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. (1) The +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. (2) 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 −3/8 in. (−9·5 mm.) washed classifier sand was almost slime-free and was of a better grade than the dry screened −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 unnecessary. The rejection of slime by washing was 10·6 per cent by weight. The slime assayed 54·04 per cent Fe and contained a high percentage of insolubles (10 per cent SiO_2+8·06 per cent Al_2O_3) 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 respect to original and assayed 64·95 per cent Fe and 5·16 per cent insolubles with a Fe recovery of 7 per cent with respect to original. 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 −3/8 in. (−9·5 mm.) washed classifier sand but without 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 strong unfluxed sinters. Good quality fluxed sinters up to a basicity of 2·4 could be produced by suitable additions of limestone. The results further showed that solid fuel, e.g. coke, could be substituted by gaseous fuel (producer gas) up to 30 per cent of the total amount of coke needed for proper sintering. The sinters thus produced were of good quality and also possessed a high degree of oxidation when compared with those produced 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 and within tolerable limit.

The summary of the results of washing is given in Fig. 5. Based on the results a general flowsheet adopted is given in Fig. 6.

The overall economics of beneficiation is given in Table 3.

1.3 Sintering of Iron Ore Fines from Bellary Hospet Area

About 16 tonnes of an iron ore sample of size 8 in. from Bellary-Hospet area was received from the Minerals & Metals Trading Corporation of India Ltd, to determine the percentage of additional −½ in. fines produced during crushing of −8+1/2 in. ore to −3 in. size and also to study the sintering characteristics of the fines so generated. As received, the sample consisted of lumps from 8 in. size down to fines. The original sample contained 5·1 per cent by weight of −1/2 in. fines which assayed only 40·2 per cent Fe, 14·6 per cent SiO_2 and 5·4 per cent Al_2O_3. Crushing of 8 in. ore to −3 in. size yielded an additional 4·1 per cent by weight of −1/2 in. fines assaying 67·2 per cent Fe, 1·23 per cent SiO_2, 2·0 per cent Al_2O_3 and 1·34 per cent CaO which was used for sintering tests.

Sintering characteristics of the −1/2 in. fines were studied for making unfluxed as well as fluxed sinters. In the case of unfluxed sinters it was found that 6 per cent water and 4 per cent coke was the optimum value for producing a good sinter. Fluxed sinters were made varying the basicity ratios from 1·0 to 2·2 by addition of limestone.

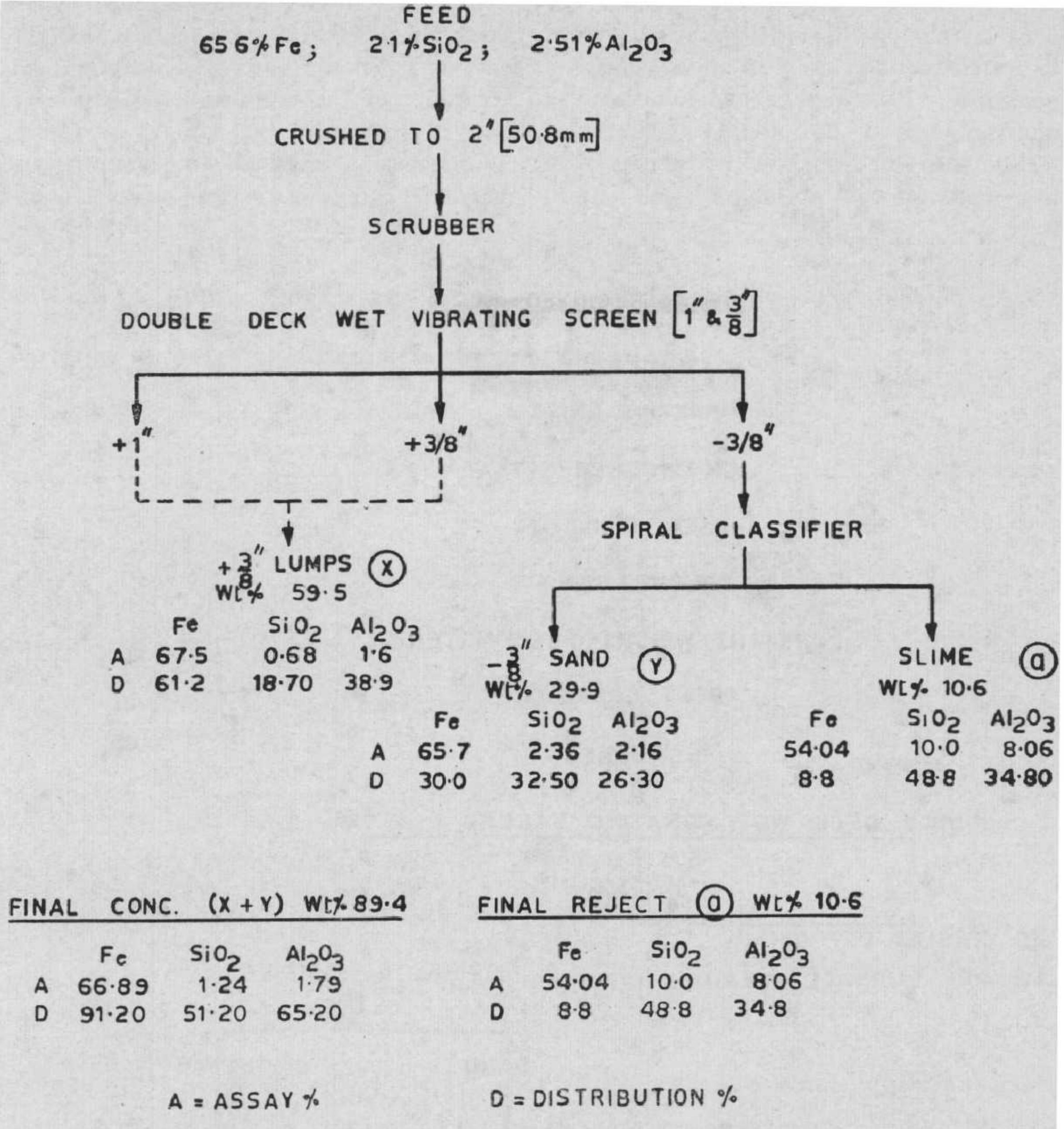


FIG. 5 — SUMMARY OF RESULTS OF WASHING OF THE MIXED SAMPLE OF MASSIVE AND LAMINATED IRON ORE FROM RAJHARA MINES

It was observed that strong sinters could be produced at lower basicities, but the strength gradually decreased with increase in basicity ratio. This might be due to the high grade ore fines employed for sintering. Fluxed sinters were highly oxidized as shown by their low FeO content, as compared to unfluxed sinters.

1.4 Beneficiation of a Low Grade Iron Ore from Goalpara, Assam

A sample of low grade iron ore from Goalpara district of Assam assaying 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, 0·073 per cent was received from the Director of

Mining & Geology, Shillong, Assam, for beneficiation studies. Microscopic studies indicated that the ore was banded hematite-quartzite. The chief gangue mineral was quartz followed by minor amounts of ferro-magnesium minerals. Hematite was free from quartz at about −48 mesh. Grindability tests on the sample showed that the ore was of a medium nature, i.e. between medium hard and medium soft according to the hardness classification of Denver Equipment Co., U.S.A.

Conventional methods like gravity, and magnetic and high tension separation and

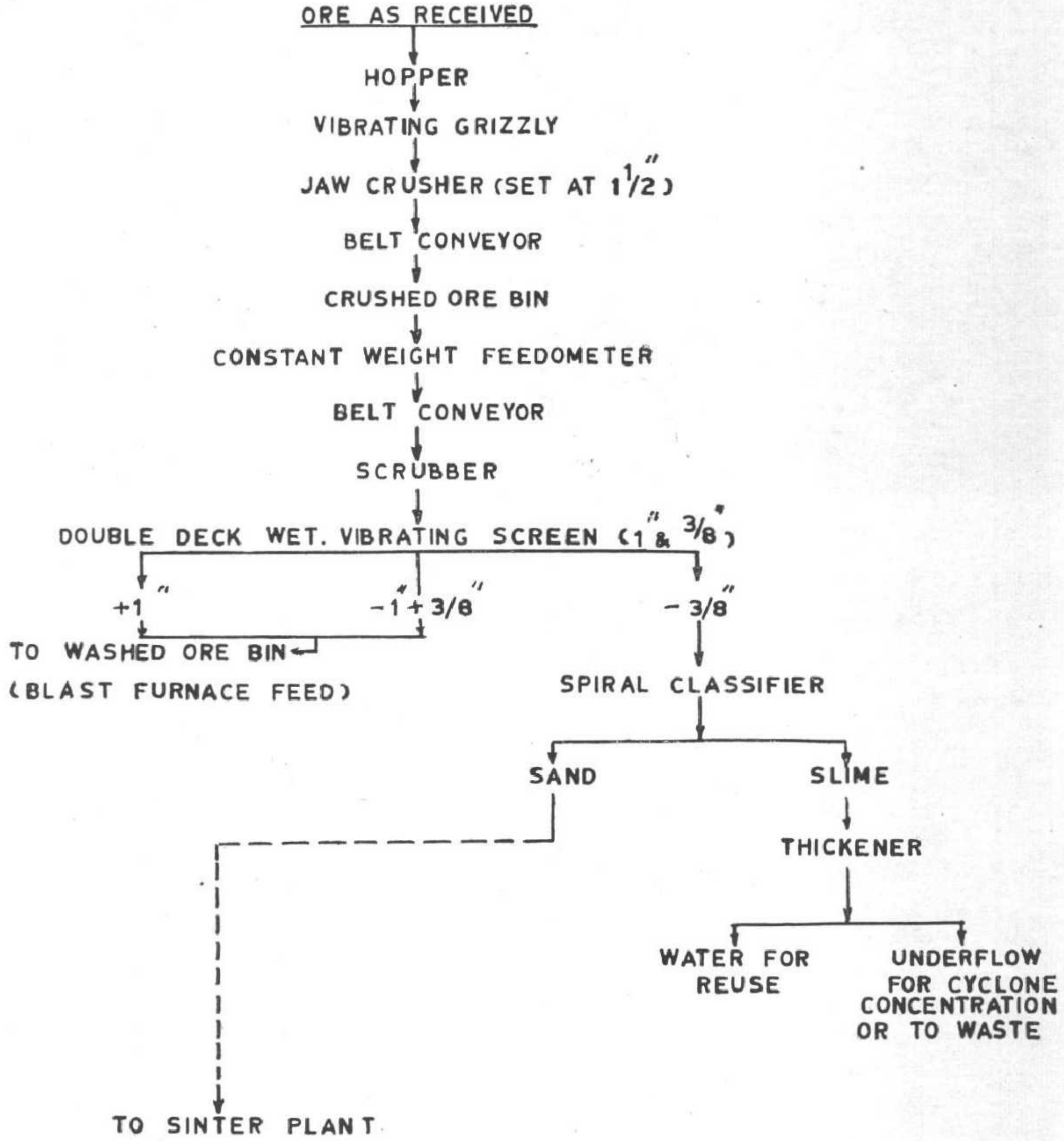


FIG. 6 — FLOWSHEET EMPLOYED IN NML FOR THE BENEFICIATION OF THE MIXED SAMPLE OF MASSIVE AND LAMINATED IRON ORE FROM RAJHARA MINES

Table 3 — Economic Evaluation of Beneficiation of Massive and Laminated Iron Ore from Rajhara Mines

	Crushing to 2″, dry screening and utilization of fines			Cushing to 2″, dry screening and rejection of $-\frac{3}{8}''$ fines			Crushing to 2″, washing and dewatering of $-\frac{3}{8}''$ fines		
	Fe	SiO_2	Al_2O_3	Fe	SiO_2	Al_2O_3	Fe	SiO_2	Al_2O_3
Analysis of final beneficiated ore, %	65·6	2·06	2·46	67·0	0·88	1·8	66·89	1·24	1·79
Yield, tonnes/day	14,000 (100%)			8,876 (63·4%)			12,516 (89·4%)		
Capital cost of ore treatment plant	Rs. 2·50 crores			Rs. 2·50 crores			Rs. 3·36 crores		
Cost of treatment per tonne of beneficiated ore									
(a) Capital cost	Rs. 0·894			Rs. 1·410			Rs. 1·344		
(b) Operating cost	Rs. 0·180			Rs. 0·284			Rs. 0·271		
(c) Cost of raw ore as mined	Rs. 5·000			Rs. 7·886			Rs. 5·593		
Total cost per tonne of prepared ore	Rs. 6·074			Rs. 9·580			Rs. 7·208		
Tonnes of ore required per tonne of pig iron	1·469			1·438			1·439		
Total cost of the ore required per tonne of pig iron	Rs. 8·925			Rs. 13·77			Rs. 10·37		
Difference in Alumina per cent	—			0·66			0·67		
Expected saving in production cost per tonne of pig iron in blast furnace smelting, due to decrease in alumina by beneficiation	—			3·30			3·35		

flotation techniques were employed for beneficiation. Straight tabling at −48 mesh yielded a concentrate assaying 66 per cent Fe with a recovery of 89·4 per cent Fe. Hydro-classification which gave a high grade coarse concentrate when followed by tabling of the medium and fine classified fractions yielded a combined concentrate assaying 66·89 per cent Fe with a recovery of 89·3 per cent Fe. Spiral followed by tabling of the spiral tailing yielded a combined concentrate assaying 64·4 per cent Fe with a recovery of 89·2 per cent Fe. Spiral and table treatment after hydro-classification yielded a combined concentrate assaying Fe, 66·33; FeO, 6·2; SiO_2, 4·28; Al_2O_3, 0·60; CaO, 0·08; MgO, 0·46 and S, 0·105 per cent with an iron recovery in the product of 91·0 per cent Fe.

The results obtained by magnetic separation of −48 mesh feed were not satisfactory. High tension separation could yield a concentrate assaying 67·71 per cent Fe but with a lower recovery of only 62·1 per cent Fe as separation in the −200 mesh fraction was not effective. Straight flotation after grinding the sample to 42·6 per cent −200 mesh and using sodium sulphonate in acid circuit at *p*H 4 yielded a concentrate assaying 66·21 per cent Fe with a recovery of 79·4 per cent Fe. Cationic flotation employing starch as depressant for iron bearing minerals and Aeromine 3037 as collector for siliceous gangue at a *p*H of 7·8 yielded a tailing (iron concentrate) assaying 67·4 per cent Fe with a recovery of 93·4 per cent Fe.

Spiral and table treatment of the ore after hydro-classification appears to be the best method of upgrading the sample as hydro-classification alone could produce a coarse classifier sand assaying between 66-67 per cent and constituting 33 per cent Fe by weight, thus necessitating only 61·0 per cent by weight of the sand for further treatment. The advantages of the process are (1) low capital and operational cost, and (2) small space requirement and possibility of fabricating spirals in India. Although cationic flotation yielded the best result by way of concentrate grade as well as recovery, the overall economics of the process will depend on the availability of the reagent Aeromine 3037 which is an imported product. The concentrate fines will require to be finally agglomerated before use in blast furnace and this aspect needs further study.

1.5 Beneficiation of an Iron Ore Sample from Kundremukh Deposits, Mysore

A sample of low grade iron ore collected from Kudremukh in Mysore was received for beneficiation studies. The sample assayed Fe, 42·17; FeO, 4·34; SiO_2, 36·3; Al_2O_3, 1·06; S, 0·074; P, 0·027 and L.O.I., 2·71 per cent. The sample was highly weathered as it has been collected from the surface. Petrological studies undertaken on the sample indicated that hematite, magnetite and goethite constituted the chief ore minerals. Quartz was the principal gangue and the iron minerals were liberated at about 100 mesh.

Straight tabling at 48 mesh size yielded a concentrate assaying Fe, 63·66; and SiO_2, 7·2 per cent with a recovery of 71·7 per cent Fe. Spiral treatment of a 48 mesh deslimed sample followed by tabling of spiral tailing yielded two iron ore concentrates which when mixed yielded a combined product assaying 60·63 per cent Fe with a total recovery of 73·2 per cent iron. Wet magnetic separation studies undertaken at various sizes indicated that good iron concentrates could be produced but the recoveries of Fe in the magnetic concentrates were low as magnetite was only one of the iron minerals in the ore. The concentrate obtained with a 65 mesh grind assayed Fe, 62·77 and SiO_2, 8·74 per cent with a recovery of 49·3 per cent Fe in it.

Flotation studies undertaken with the ore ground to 76·2 per cent —200 mesh indicated that sulphonate flotation at a *p*H of 3·5 could be successfully employed for producing good grade of iron concentrate with fairly high recoveries. The refloat concentrate produced under optimum conditions assayed Fe, 63·8 and SiO_2, 5·8 per cent with a recovery of 87·5 per cent Fe. Wet magnetic seperation followed by flotation of the iron minerals from the non-magnetic fraction using petroleum sulphonate as before, yielded two iron concentrates (viz., magnetic concentrate and refloat concentrate) which when mixed yielded a product assaying Fe, 62·9 and SiO_2, 6·2 per cent with a recovery of 90·1 per cent Fe in it.

1.6 Pilot Plant Studies on Beneficiation and Pelletization of Mixed Iron Ore Sample from Goa

Four different lumpy samples of iron ore from Sallitho, Surla, Pale and Velguem mines were received from M/s. Salgaocar, Goa, for beneficiation and pelletization studies. The sample received were of —6 in. lumps down to fines. The samples were mixed in the ratio of 46: 40: 26: 26, respectively, as desired by the firm. The mixed sample was crushed to —3/8 in. size by jaw crusher and cone crusher, and assayed Fe, 56·74; SiO_2, 2·54 and Al_2O_3, 8·1 per cent. The crushed —3/8 in. mixed ore was divided into two halves for beneficiation studies. The first part was treated by jigging and the other part by cycloning.

Jigging — About 3·5 tonnes of −3/8 in. mixed sample was taken and screened to three fractions, viz. −3/8+1/4 in., −1/4 in. +10 mesh and −10 mesh sizes. These screened fractions were jigged separately and the concentrate when mixed together assayed Fe, 60·5; SiO_2, 1·61 and Al_2O_3, 7·56 per cent with a recovery of 64·5 per cent Fe.

Cycloning — Cycloning tests were done after grinding the ore to −20 mesh and −35 mesh separately, but the results were not satisfactory. Another test with −65 mesh grind produced a concentrate assaying 62·5 per cent Fe with a recovery of 75 per cent Fe. Continuous cycloning with −65 mesh grind for obtaining a bulk quantity of concentrate for pelletization tests produced a concentrate assaying 61·7 per cent Fe with a recovery of 70·4 per cent Fe.

Pelletizing tests with the concentrate are in progress.

2.0 Pilot Plant Beneficiation Studies on Fluorspar Sample from Ambadongar, Gujarat State, Received from Gujarat Mineral Development Corporation

A 200 tonnes sample of fluorspar deposit in Ambadongar, Gujarat, was received from G.M.D.C. for beneficiation studies on a pilot plant scale and to recommend a flowsheet and detailed project report for setting up of a commercial plant to produce acid grade fluorspar. The specifications laid down for acid grade fluorspar is CaF_2, 97; $CaCO_3$, 1·25; SiO_2, 1·5 and S, 0·03 per cent. Six samples were received from Ambadongar area. Beneficiation studies were carried out after mixing the six different samples in such a way as to give the mill feed assaying nearly 30 per cent CaF_2.

Petrological and chemical analyses of the samples were carried out in details. The samples were then mixed in equal proportion and a composite sample was prepared for beneficiation studies. The mixed sample assayed CaF_2, 27·99; $CaCO_3$, 1·90; SiO_2, 49·12; Al_2O_3, 8·25; Fe_2O_3, 7·40; S, 0·16 per cent and minute traces of Pb. Petrological examination of the individual samples showed the presence of quartz, fluorite, feldspar, opaque minerals and small amounts of calcite. Hematite, magnetite, goethite, pyrite, chalcopyrite and galena mainly constituted the opaque minerals. It was also observed that most of the zones contain siderite while some zones contain traces of barite as well. Fluorite was present as fine veins as well as coarse crystalline vug deposition and the colour varied in shades of green, violet, yellow and amber. Complete liberation of fluorite was expected to be at about 100 mesh size. Since the samples contained clay, about 1 tonne of the mixed sample was crushed to about 2 in. size and subjected to a washing test (scrubbing and wet screening). This test indicated that the clay could be removed in the form of slime which assayed CaF_2, 10·94; $CaCO_3$, 1·97 and SiO_2, 46·48 per cent with a loss of only about 2 per cent CaF_2 in it. This washed ore was crushed to −10 mesh and batch flotation tests were carried out.

Grindability tests were carried out to determine the size of grinding equipment to be required in the mill. Batch flotation tests using feeds of different grinds indicated that the optimum grind for maximum recovery of fluorspar would be about 78 per cent −200 mesh. Sulphide flotations were carried out using different xanthates as collector. The use of activators like $CuSO_4$, Na_2S, etc., were studied. The effect of variation of *p*H were also investigated. The above sulphide flotation tests were carried out to reduce the S content of the ore as per specifications laid down for acid grade fluorspar. The results obtained so far could not produce a concentrate low in S. Further work in this direction is in progress.

Sulphide flotation followed by fatty acid flotation and cleaning of the rougher floats produced a fluorspar concentrate assaying CaF_2, 89·64; $CaCO_3$, 2·11 and S, 0·13 per cent with a recovery of 81·3 per cent CaF_2. Further batch flotation tests using (1) sodium silicate as a depressant both in the roughing and cleaning stages, (2) a combination of sodium silicate and *Katha* (tannin extract), (3) use of *Katha* only, (4) regrinding the rougher float, and (5) hot conditioning before cleaning were performed. The best result obtained was CaF_2, 95·7; $CaCO_3$, 0·82; SiO_2, 2·6 and S, 0·08 per cent with a recovery of 77·9 per cent CaF_2. Several other batch flotation tests were carried out using varying amounts of sodium carbonate as a depressant for quartz instead of sodium silicate and employing a finer feed for rougher flotation and a still finer grind for cleaning. The best grade in this series of tests was obtained by eight cleanings. The concentrate thus obtained assayed CaF_2, 97·14; $CaCO_3$, 0·46; SiO_2, 0·40 and S, 0·1 per cent with a recovery of 54·9 per cent CaF_2. Further batch flotation tests produced fluorspar concentrates assaying 97·5 to 98·6 per cent CaF_2 with less than 0·08 per cent S, but the recovery of CaF_2 was very low. Hence, further tests are being carried out to improve the recovery and lower the S content.

Pilot plant washing of 80 tonnes sample was completed. As requested by G.M.D.C. an interim flowsheet and a detailed list of crushing and grinding equipment with their capacities were prepared and sent to them.

2.1 Reduction of Sulphur from a Sample of Fluorspar from G.M.D.C.

The sample as received assayed 0·16 per cent S. Petrological examination studies indicated that part of sulphur was present as sulphides and the rest as barite. Xanthate flotation produced a sulphide float assaying 1·2 per cent S with a distribution of only 22 per cent S in it. Several flotation tests were performed for the reduction of S in the fluorspar float. Several reagents like dichromate, dextrine, $AlCl_3$, $FeCl_3$ and citric acid were tried for producing a fluorspar concentrate low in S. The best results from the tests performed so far were obtained when a combination of dichromate and dextrine were employed for depression of barite. The final fluorspar float assayed 0·05 S per cent and had a distribution of only 4 per cent S in it.

3.0 Pilot Plant Beneficiation Studies on a Low Grade Mixed 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. The samples, as received, from Itagarh, Garidih, Samaidih, contained 14·75, 22·45 13·65 and 25·87 per cent P_2O_5 respectively. A mixed sample was prepared as suggested by the Government of Bihar and was investigated to determine the possibility of beneficiating the mixed sample to a grade suitable for use as a natural fertilizer as well as for the production of superphosphate. The Government of Bihar also requested to recommend a suitable flowsheet which could produce a concentrate assaying not less than 30 per cent P_2O_5, with a maximum of 4 per cent total oxides ($FeO+Fe_2O_3+Al_2O_3$). The mixed sample assayed 17·34 P_2O_5 with 25 per cent total oxides.

Microscopic examination of the mixed sample indicated that apatite was the only phosphate mineral. It was associated with magnetite and other gangue minerals, such as quartz, feldspar, etc. Petrological examination of the —10 mesh sieve fractions also indicated that the sample was required to be ground finer than 65 mesh size for getting a clean apatite concentrate. Batch flotation tests with a feed ground to about 62·6 per cent —200 mesh using 0·69 kg./tonne of soap as collector for apatite, followed by one cleaning of the rougher concentrate produced an apatite concentrate assaying P_2O_5, 38·12 and Fe, 2·38 per cent with a recovery of 79·9 per cent P_2O_5.

Two cleanings of the above rougher concentrate produced a recleaner apatite concentrate assaying P_2O_5, 39·55 and Fe, 1·5 per cent with 71·3 per cent P_2O_5 recovery in it. Magnetic separation of the —10 mesh sample followed by flotation with one cleaning of the rougher concentrate yielded a refloat concentrate assaying P_2O_5, 38·20 and Fe, 1·83 per cent with a P_2O_5 recovery of 77·8 per cent. The magnetic fraction of the feed assayed Fe 48·69 per cent. The recovery of apatite by straight flotation could be improved from 79·9 to 85·4 per cent with the use of 0·5 kg./tonne of sodium silicate.

Based on the batch tests two series of tests with two lots of about 25 tonnes each of the sample were carried out on a pilot plant scale. In the first test, the classifier overflow was subjected to wet magnetic separation and the non-magnetic feed was subjected to flotation using sodium silicate as depressant for the gangue and sodium oleate as collector for apatite. In the second test, straight flotation of the classifier overflow was done followed by two cleanings. The results of tests 1 and 2 are presented in Tables 4 and 5, respectively. It was found that the low grade apatite sample of Singhbhum District, Bihar, can be upgraded to produce the desired apatite concentrate suitable for manufacture of superphosphate. A general flowsheet (Fig. 7) based on the first test has been recommended for the beneficiation of this sample.

4.0 Studies on Nundydroog Gold Ore with a View to Improve Gold Recovery

Extensive laboratory scale studies were undertaken on three samples, e.g. (1) crushed run-of-mine ore, (2) feed to the cyanidation plant, and (3) final cyanidation tailing, received from Nundydroog Mines, Kolar, with a view to minimize loss of gold in the final cyanidation residue. Petrological studies on the run-of-mine ore (Sample No. 1) indicated that gold was present in the native form associated with arsenopyrite and quartz. The run-of-mine ore assayed 5·6 dwt Au per ton.

Cyanidation studies undertaken on the feed to cyanidation plant sample (Sample No. 2) assaying 2·3 dwt Au per ton, indicated that it should be subjected to 27 hours agitation with sodium cyanide and lime to obtain a recovery of 87 per cent gold with respect to feed, equivalent to 94·6 per cent gold recovery with respect to run-of-mine ore. Removal of graphitic carbon, by flotation, prior to cyanidation did not improve the gold recovery. Wet magnetic separation followed by flotation of the cyanide residue yielded an arsenopyrite concentrate assaying 4·8 dwt Au per ton representing an additional gold recovery of 3·3 per cent with respect to run-of-mine sample. If wet magnetic separation is followed by tabling, a product assaying 5·2 dwt Au per ton could be recovered with a distribution of 3·3 per cent gold in it. The final rejects after tabling or flotation would assay 0·1 to 0·14 dwt gold per ton only, which would represent a loss of 1·6 to 2·2 per cent of the total gold or an overall gold recovery of nearly 98 per cent.

Cyanidation studies undertaken on the cyanide tailing (Sample No. 3) assaying

Table 4 — Day-to-day Data of the Pilot Plant Run with Apatite Sample: Test No. 1 — Magnetic Separation of the Classifier Overflow Followed by Flotation

Date Shift (7 hr duration)	Feed rate in kg./hr	Reagent addition in kg./ton				Assay per cent P_2O_5				Apatite conc.		Remarks
		Total quantity of silicate added to the conditioner	‡Soap (Oleic acid/NaOH emulsion)			Feed	Non-mag. feed	Final conc.	Final tailing	Wt %	Recovery of P_2O_5	
			Conditioner	Primary cells	Total quantity added							
27-6-66.G	1065	0·5	0·23	0·46	0·69	18·90	19·80	41·6†	6·05*	28·7	79·6	High tailing loss is due to the low iodine value of soap used (oleic acid).
30-6-66.A	940	0·5	0·23	0·69	0·92	18·01	19·14	37·8*	4·40*	40·4	84·8	Soap addition was increased by 25% to compensate the low iodine value of soap used.
30-6-66.B	1030	0·5	0·23	0·69	0·92	18·01	19·14	37·5*	5·76	38·5	80·2	
5-7-66.A	812	0·5	0·46	0·94	1·40	17·4	17·63	36·20*	0·71	46·8	97·3	Soap addition was doubled to compensate the low iodine value of soap, and feed rate decreased to 80% to reduce the tailing loss and to improve the recovery.
5-7-66.B	805	0·5	0·46	0·94	1·40	17·4	17·63	37·24	1·30	44·6	95·4	
6-7-66.A & B	780	0·5	0·46	0·94	1·40	16·0	18·31	35·72	1·96	39·6	88·5	
7-7-66.A	743	0·5	0·46	0·94	1·40	16·0	17·47	36·15*	1·45*	40·4	91·4	

*Autrometer analysis (X-ray).

†The conc. assayed 41·6% P_2O_5, 1·2% Fe and 0·52% Al_3O_2.

‡Iodine value of the oleic acid used for batch tests was 81 whereas the acid used for pilot plant tests had an iodine value of 73·3 which accounts for a higher oleic acid consumption for plant tests to get similar results.

Table 5 — Day-to-day Data of the Pilot Plaht Run with Apatite Sample: Test No. 2 — Straight Flotation

Date Shift (7 hr duration)	Feed rate in kg./hr	Reagent addition in kg./tonne				Assay % P_2O_5			Apatite conc.‡		Remarks
		Total quantity of silicate added to the conditioner	Soap (Oleic acid/NaOH emulsion)†			Feed	Final conc.	Final tailings	Wt %	Recovery of P_2O_5	
			Conditioner	Primary cells	Total quantity added						
18-7-66.B	677	0·5	0·46	0·94	1·4	17·22	33·27	2·38	48·0	92·8	Grade of concentrate is low due to inefficient cleaning
19-7-66.A	655	0·5	0·46	0·94	1·4	18·43	32·20*	1·58	55·0	96·2	
19-7-66.B	675	0·5	0·46	0·94	1·4	17·72	34·97	5·60	41·3	81·4	
20-7-66.A	506	0·5	0·46	0·59	1·05	16·48	37·80*	3·49*	37·9	86·9	Feed rate was reduced to 500 kg./hr. Soap addition was cut down in order to improve the grade of the concentrate. But, loss in tailings is high
20-7-66.B	532	0·5	0·46	0·59	1·05	16·37	37·81	4·64	35·4	81·7	
21-7-66.A	540	0·5	0·46	0·59	1·05	18·27	38·04	4·90*	40·5	84·0	
21-7-66.B	530	0·5	0·46	0·94	1·4	18·13	37·34	2·25	45·2	93·0	Soap addition was increased, and the rougher float has been cleaned twice in order to reduce the tailing loss and maintain the grade of the conc.
22-7-66.A	566	0·5	0·46	0·94	1·4	17·75	37·34	2·16	44·3	93·2	
22-7-66.B	528	0·5	0·46	0·94	1·4	18·65	37·70	1·84	46·9	94·8	
23-7-66.A	528	0·5	0·46	0·94	1·4	16·14	38·00*	2·32*	38·7	91·2	The discharge from the primary and secondary crushers was passed through guard magnet and the magnetic fraction amounting to 1·64% by weight and assaying 13·69% P_2O_5, 44·52%Fe and 14·4% FeO was rejected
23-7-66.B	530	0·5	0·46	0·94	1·4	16·71	38·95	2·86	38·4	89·5	

*Autrometer analysis (X-ray).

†Double the optimum quantity was added to compensate for the low iodine value of the oleic acid used in the pilot plant run.

‡The overall concentrate from Test II assayed 35·67% P_2O_5, 4·95% Fe, 1·3% FeO, 5·63% Fe_2O_3 and 0·98% A_2O_3.

0·54 dwt Au per ton indicated that an agitation time of 15 hours with NaCN and lime would recover 50 per cent gold in the feed equivalent to a recovery of 95·2 per cent Au with respect to run-of-mine ore. Straight tabling of the cyanide tailing yielded a heavy mineral concentrate assaying 3·6 dwt Au per ton and recovering 47·4 per cent gold in it and representing an additional gold recovery of 3·8 per cent with respect to run-of-mine ore. This product will have to be roasted before cyanidation for recovery of gold from it.

5.0 Beneficiation of Low Grade Graphite from Palamau, Bihar

A sample of low grade graphite from Palamau District of Bihar, was received from M/s. Singhania Commercial Company Ltd, Calcutta, for beneficiation studies. The sample assayed C, 15·5; ash, 76·2; S, 6·5; volatile matter 1·8 and Fe in ash 12·94 per cent. Petrological examination indicated that the graphite was of a soft and friable type, which was in intimate association with quartz, feldspar and pyrite. The liberation of graphite from gangue was incomplete even at —200 mesh.

Only the flotation process was investigated to upgrade the graphite sample. Preliminary flotation tests employing different grinds and using pine oil as frother-collector and sodium silicate as gangue depressant showed that a grinding having 79·4 per cent —200 mesh was optimum for flotation. Flotation tests using lime as depressant showed that 0·75 kg. of lime per ton of ore was optimum. The rougher float assayed 39·0 per cent ash with a distribution of 16·2 per cent ash in it. Flotation tests performed with other collectors showed that a combination of pine oil and light diesel oil could also be used for rougher flotation. The rougher float assayed 39·8 per cent and had a distribution of 15·7 per cent ash in it.

Reflotation of the rougher float thrice yielded a refloat concentrate assaying 14·34 per cent ash, with a distribution of only 3·1 per cent of the total ash in it. Regrinding of the rougher concentrate followed by reflotation of the ground product four times, yielded a final refloat concentrate assaying fixed C, 88; ash, 6·5; volatile matter, 4·4; moisture, 0·3; Cu trace and S, 0·8 per cent with distribution of C, 90·8 and ash, 1·4 per cent in it. This product should be suitable for use in battery manufacture, if necessary, after heating to expel the volatile matter.

5.1 Beneficiation of Graphite from M/s. National Carbon Co.

A sample of graphite assaying F.C., 77·85; ash, 17·60; V.M., 4·15; moisture, 0·40; S, 0·51 and Fe, 6·5 per cent in the ash, was received from M/s. National Carbon Co., Calcutta, for beneficiation studies. Petrological examination indicated that the graphite present was of crystalline flaky variety. The chief gangue was quartz which was accompanied by minor amounts of pyrite and hematite. Liberation of graphite from the gangue was incomplete even at —200 mesh size. Preliminary flotation tests employing different grinds using pine oil as frother-collector showed that grind having 60·5 per cent —200 mesh was optimum for rougher flotation. Flotation tests using sodium silicate as gangue depressant showed that 0·5 kg. per ton was optimum. The rougher float assayed 11·24 per cent ash with a distribution of 56·8 per cent ash in it. Flotation tests performed with other collectors showed that a combination of light diesel oil and pine oil could give satisfactory results. The rougher float assayed F.C., 85·33 and ash, 10·46 per cent, with a recovery of F.C., 99·4 per cent in it.

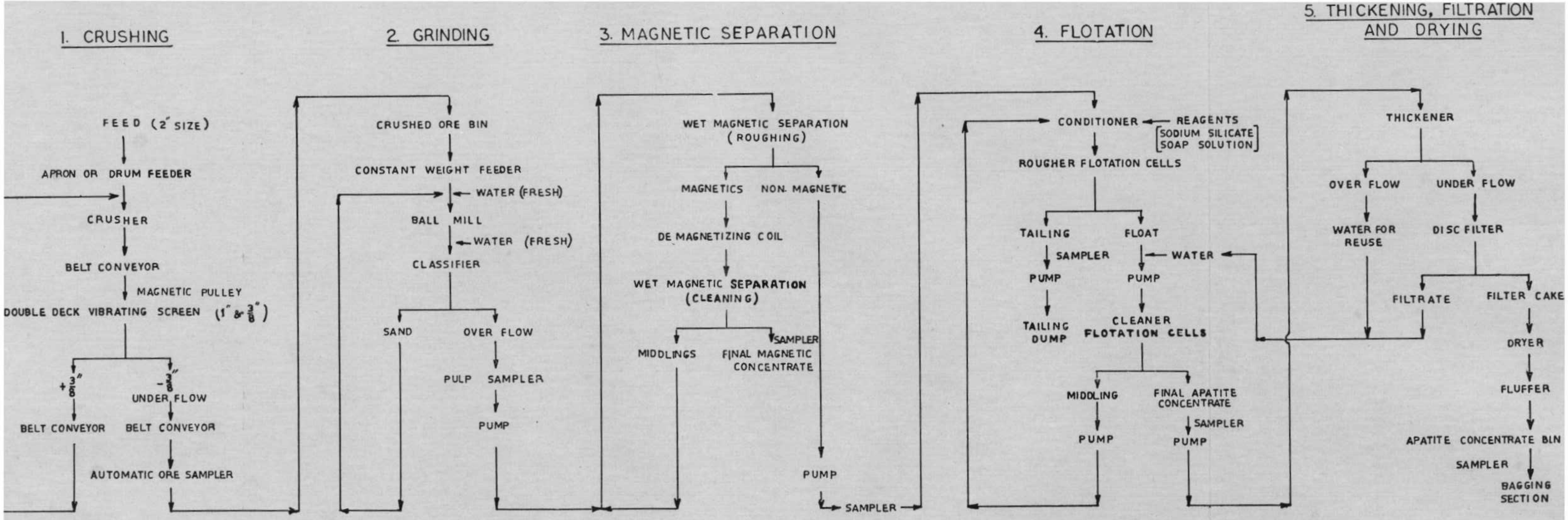


FIG. 7 — FLOWSHEET FOR THE BENEFICIATION OF LOW GRADE APATITE SAMPLE FROM SINGHBHUM

Reflotation of the rougher float thrice, yielded a refloat concentrate assaying F.C., 89·0; ash, 7·1; V.M. +Moisture, 3·9; S, 0·22; Cu trace and 7·89 per cent Fe in the ash with a recovery of 97·3 per cent F.C. in it. This product should be suitable for battery manufacture. Regrinding of the rougher float followed by reflotation thrice yielded a refloat concentrate assaying F.C., 90·0; Ash, 6·4; V.M. +Moisture, 3·6; and S, 0·21; Cu trace and 9·1 per cent Fe in the ash with a recovery of 99·0 per cent F.C. in it. This product also fulfills the grade requirements laid down for its use in battery manufacture. Heating the concentrate to expel the volatile matter can yield a final product of grade F.C., 93·6 and ash, 6·4 per cent.

6.0 Beneficiation of a Low Grade Limestone Sample from M/s. Rohtas Industries

A low grade limestone sample from M/s. Rohtas Industries Ltd, Dalmianagar, Bihar, assaying CaO, 38·68; CO_2, 30·48; MgO, 3·13; SiO_2, 17·92; Al_2O_3, 4·03; Fe_2O_3, 1·54; S, 0·09; P, 0·065 and total insolubles 21·24 per cent, was received for beneficiation tests. The object of the investigation was to produce a concentrate suitable for cement manufacture. Quartz was the principal gangue mineral in the sample which was not completely liberated even at −200 mesh. Fatty acid flotation employing different grinds indicated that a feed containing 94 per cent −200 mesh produced the best results.

An emulsion of oleic acid with sodium hydroxide was used as collector and pine oil as frother for limestone and sodium silicate as depressant for silicate minerals. The optimum amounts of reagents necessary for flotation of limestone were found to be 0·75 kg. per ton of sodium silicate, 1·0 kg. per ton of oleic acid emulsion and 0·017 kg. per ton of pine oil. Straight flotation of the ore after grinding to 94 per cent −200 mesh followed by one cleaning of the rougher float failed to yield a grade of concentrate acceptable for cement manufacture. However, two cleanings of the rougher float produced a cleaner concentrate assaying CaO, 47·43; SiO_2, 7·7 and insolubles, 9·5 per cent with a CaO recovery of 85·2 per cent. The concentrate should be acceptable to the cement industry. The calcite recovery is expected to further improve in actual plant practice when the cleaner tailings are recirculated.

The grade of the concentrate could be further improved after three cleanings to CaO, 49·29; MgO, 2·27; SiO_2, 6·31; Al_2O_3, 1·77; Fe_2O_3, 0·58; CO_2, 38·80; P, 0·04; S, 0·10 and total insolubles, 8·05 per cent with a recovery of 82·3 per cent CaO in the product. Settling tests with the concentrate as well as tailing, showed that these products could be made to settle fast by the use of flocculant. Grinding tests showed that the limestone sample could be classified as ' medium soft '.

7.0 Concentration of Cobalt from Cobalt Bearing Ferruginous Manganese Ore from Kalahandi, Orissa

A sample of cobalt bearing ferruginous manganese ore from Olatura, Kalahandi, Orissa, was received from the Geological Survey of India for production of cobalt concentrate. The sample as received assayed Co, 0·019; Mn, 15·0; Fe, 20·82; SiO_2, 26·43; Ni, 0·026; CaO, 0·63; BaO, 0·55 and L.O.I., 10·52 per cent. Wet magnetic separation of −10 mesh ore after a reducing roast to convert the iron mineral to magnetite yielded a cobalt concentrate (manganese fraction) assaying only 0·027 per cent Co. High intensity magnetic separation of the ore produced a concentrate assaying only 0·022 per cent Co. Cobalt was not present in the sample in the form of a distinct mineral, but was in colloidal combination with the manganese minerals. So the sample could not be beneficiated successfully for production of a concentrate rich in cobalt. Hydrometallurgical methods are suggested for recovery of cobalt and nickel present in the ore.

8.0 Reduction of Iron Content in a Manganese Ore from M/s. National Carbon Co.

Ten tonnes of a manganese ore sample assaying Mn, 50·6; MnO_2, 75·2; Fe, 5·32 and SiO_2, 4·72 per cent, was received from M/s. National Carbon Co. Ltd, Calcutta, for carrying out pilot plant tests to lower the iron content in order to make it suitable for manufacture of dry batteries. Pyrolusite and cryptomelane were the chief manganese minerals with some jacobsite and braunite. Magnetite, hematite and goethite were the iron-bearing minerals. Wet magnetic separation of the ore after grinding to —35 mesh produced a concentrate assaying Mn, 52·71 and Fe, 3·7 per cent with a recovery of 94·0 per cent Mn. High intensity dry magnetic separation of this concentrate could not be carried out successfully to reduce the iron content further. Wet magnetic separation of the ore after grinding to 200 mesh yielded a manganese concentrate assaying Mn, 53·5 and Fe, 3·89 per cent with a recovery of 90·8 per cent Mn.

Based on the results obtained, a flowsheet and a list of equipment have been drawn out for setting up of a commercial plant to produce 7500 tons of concentrate per year.

9.0 Beneficiation of Calcite from Sankaridrug, Mysore

A sample of low grade limestone assaying CaO, 43·05; CO_2, 35·9; MgO, 1·80; SiO_2, 15·40; Al_2O_3, 2·14; Fe_2O_3, 0·66; S, 0·09 P_2O_5, 0·19 per cent was received from the Government of Madras for beneficiation studies. Calcite was well liberated from the quartz even at 35 mesh. Soap flotation, using oleic acid emulsion as collector for calcite and sodium silicate as gangue depressant, yielded a rougher float assaying CaO, 52·90 and insolubles, 2·0 per cent with a recovery of 97·1 per cent CaO. Single reflotation of the rougher float improved the grade of the concentrate to CaO, 53·34; insolubles, 0·8 and P_2O_5, 0·26 per cent with a recovery of CaO, 94·7 per cent. Apatite flotation of the refloat conc, using *Katha* as a calcite depressant yielded a tailing (limestone concentrate) assaying CaO, 52·95; insolubles, 1·08 and P_2O_5, 0·10 per cent. This product fulfills the grade requirements laid down for its use in steel making.

Pelletization studies undertaken with the low-phosphorus limestone concentrate without further grinding indicated that good green pellets could be produced by using molasses, 4·0; and moisture, 8·1 per cent. Heat hardening at 110°C. yielded pellets having a compressive strength of 60·45 kg. per pellet. Calcination studies on the green pellets in a stationery gas fired kiln as well as in a rotary kiln at 1000-1050°C. showed that the calcined pellets retained their shape and strength well, though the original limestone lumps crumbled to 6·00 mm. and finer, even at 550-600°C.

10.0 Beneficiation of Low Grade Kyanite from Magarmohan Kyanite Mines, Pallahara, Orissa

A sample of low grade kyanite from Magarmohan Kyanite mines, Pallahara, Orissa, was received from M/s. S. Lal & Co. for beneficiation studies. 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+Na_2O, 1·6 and L.O.I., 1·7 per cent.

Quartz and mica were the chief gangue minerals. Kyanite was fairly well liberated from the gangue at about 48 mesh size. The ore after grinding to −48 and −65 mesh was subjected to hydro-classification followed by tabling of the different classified products. But this method did not yield satisfactory result. Separation of kyanite from micaceous gangue by tabling was found to be difficult due to the flaky nature of the latter. Flotation studies were next carried out with deslimed feed, at low *p*H, using lactic acid as depressant for mica and sodium sulphonate as collector for kyanite. Flotation at 4·9 *p*H, employing a deslimed feed containing 36 per cent by weight of −200 mesh and using lactic acid and sodium sulphonate for roughing followed by one cleaning yielded the best concentrate assaying Al_2O_3, 60·7 and Fe_2O_3, 1·07 per cent with a recovery of 67·4 per cent Al_2O_3. Use of hot pulp during conditioning did not improve the results. Flotation of mica using Aeromine 3037 and fuel oil followed by kyanite flotation using lactic acid and oleic acid emulsion yielded a kyanite concentrate assaying Al_2O_3, 58·8 per cent with a recovery of Al_2O_3, 70·7 per cent. In another test, use of sodium silicate, sodium hydroxide and oleic acid emulsion yielded a kyanite concentrate assaying Al_2O_3, 60·0 with a recovery of only 57·0 per cent Al_2O_3 in the concentrate.

11.0 Reduction of Silica Content of 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 (1) sample as received, (2) after crushing to about 1 in. (25·4 mm.) size, and (3) after crushing to about 1/2 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.

11.1 Reduction of Silica Content in a Magnesite Sample (No. 2) from Salem, Madras

Laboratory flotation studies were undertaken on a magnesite sample received from Dalmia Magnesite Corporation, Calcutta, assaying MgO, 45·86; CaO, 0·87; CO_2, 48·1; SiO_2, 3·0; Al_2O_3, 0·21; Fe_2O_3, 0·54; S, 0·24 and P, 0·028 per cent, to determine the optimum conditions for producing a magnesite concentrate low in silica. Quartz and serpentine were principal gangue in the sample. Magnesite was fairly liberated at about 100 mesh.

Rougher flotation employing nautral *p*H and a feed of 96 per cent −200 mesh size and using oleic acid emulsion (0·75 kg. per ton) as collector for magnesite and sodium silicate as depressant for siliceous gangue followed by cleaning of the rougher float produced magnesite concentrates assaying from 0·55 to 1·05 per cent SiO_2 with weight recoveries between 65 to 77·6 per cent depending upon the number of cleaning operations adopted. Recirculation of the cleaner tailings, as is done in plant practice, is expected to yield higher recoveries. Cationic flotation employing Armeen-12 in natural *p*H of 8 could be successfully employed for producing a magnesite concentrate assaying 0·72 per cent SiO_2 and constituting 81·2 per cent by weight. There was thus an extra recovery of 10·6 per cent by weight for the same grade of magnesite concentrate, when cationic flotation was employed. However, the economics as well as the indigenous availability of reagents will have to be considered before adopting cationic flotation.

12.0 Reduction of Insoluble Content of a Selenite Sample from Rajasthan

The sample as received assayed 1·06 per cent insolubles which was mainly due to the presence of clayey matter. Batch tests indicated that a two-stage washing would be necessary to yield the best results. The sample was first crushed to about 3/4 in. (18·9 mm.) size, soaked in water and then washed with water after scrubbing. The washed (deslimed) lumps were then ground to —20 mesh and washed again to give a clean finished sand assaying 0·41 per cent insolubles. In the pilot plant scale test, performed under same conditions, 45·3 per cent of the total insolubles were rejected in the primary slime weighing 6·5 per cent. The coarse washed sand assayed 0·59 per cent insolubles. In the second stage washing after grinding to —20 mesh, slime rejected weighed 20·3 per cent and carried 25·1 per cent of insolubles with it. The mixed washed sand weighing 73·2 per cent assayed 0·41 per cent insolubles (or equivalent to about 99·6 per cent selenite). This product has been sent to M/s. Bikaner Gypsum Ltd, for final processing and marketing trials. The results of pilot plant scale test are summarized in Fig. 8.

13.0 Removal of Mica from Jamuna River Sand

A sand sample from river Jamuna, assaying SiO_2, 84·0; Al_2O_3, 6·83; Fe_2O_3, 2·28; TiO_2, 0·39; CaO, 1·5; MgO, 1·18; alkali chlorides, 1·64 and L.O.I., 2·38 per cent was received from the National Building Organisation, Ministry of Works, Housing & Rehabilitation, New Delhi, for removal of mica so as to make it suitable for use in building industry. The sand as received consisted of quartz, feldspars, amphiboles, pyroxenes and flaky minerals such as muscovite, biotite, sericite and chlorite in order of abundance. Opaque minerals were present in traces. Flaky minerals, which constituted the bulk of the gangue, were present up to 5 per cent by weight of the sand.

Cationic flotation of the sand as received, employing 0·6 kg./t of Aeromine 3037 as collector and 2·0 kg./t of lead nitrate as activator for mica, at *p*H 3·0 yielded a sand tailing containing 1·3 per cent by weight of mica. Flotation employing washed sand under the same conditions yielded a tailing containing 0·84 per cent mica. Use of lesser quantity of lead nitrate viz., 1·0 kg./t, under the same conditions yielded a tailing containing 1·0 per cent mica by weight. Flotation of washed sand using 0·6 kg./t of Armac-CD as collector and 2·0 kg./t of lead nitrate, at the optimum *p*H of 3·0 produced the best result by yielding 76·4 per cent by weight of clean and containing only 0·2 per cent micaceous minerals thus rejecting almost all the mica in the float. Processes such as tabling, magnetic separation, or electrostatic separation did not yield satisfactory results.

Before flotation can be employed by the building industry for removal of the micaceous minerals from Jamuna River sand on an industrial scale, the economics of the process will have to be worked out on the basis of pilot plant test trials.

14.0 Beneficiation of Low Grade Pyrite-Pyrrhotite (Bore Hole No. 1) from Saladipura, Rajasthan

A pyrite-pyrrhotite sample assaying S, 40·8; Fe, 42·1; S as FeS, 1·9; S as FeS_2, 36·5; and SiO_2, 7·5 per cent with traces of Cu, Pb, Zn, Ni and As from Saladipura area of Rajasthan was received from the Geological Survey of India, Rajashthan circle, for beneficiation studies. Pyrite-pyrrhotite

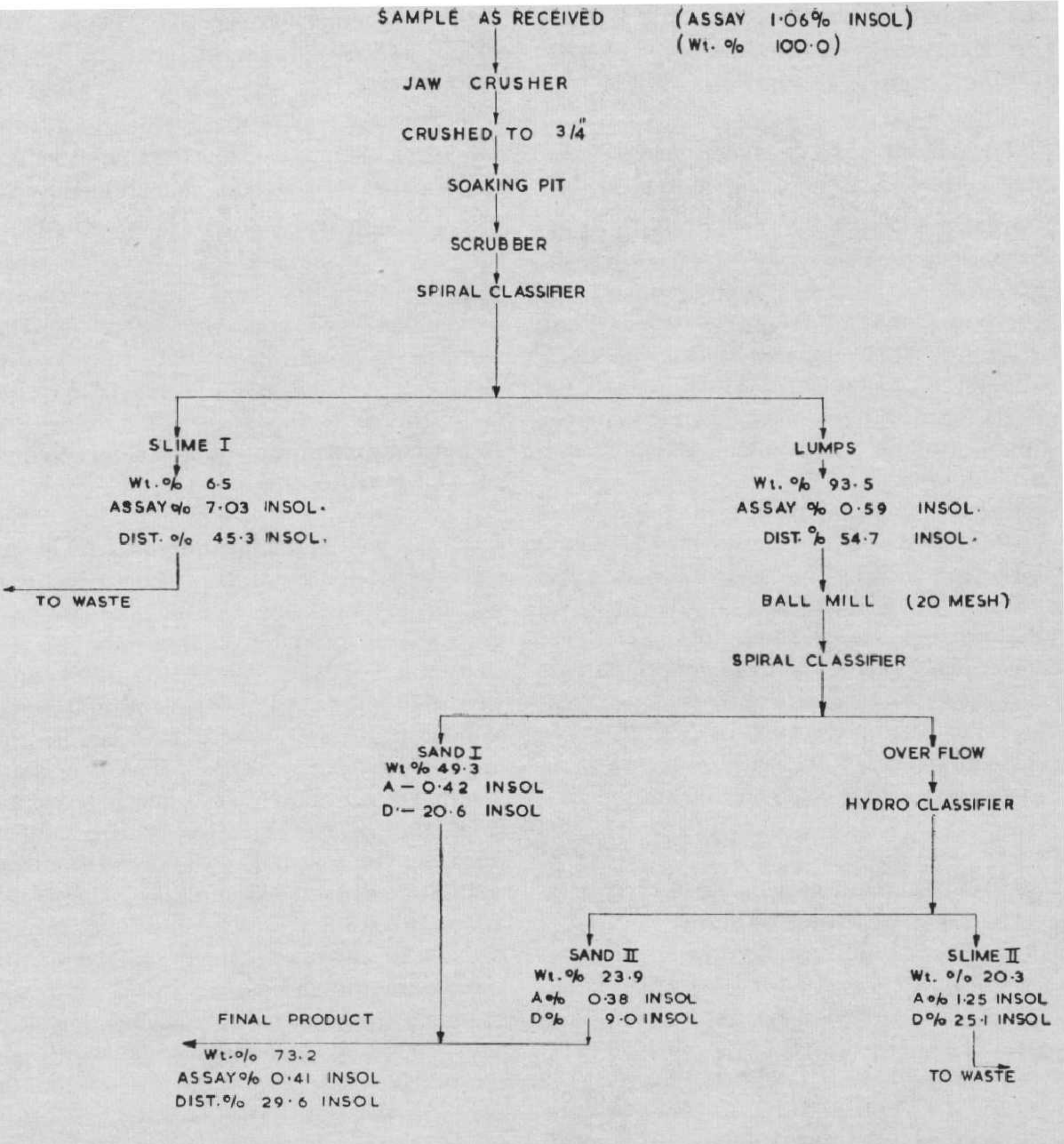


FIG. 8 — FLOWSHEET OF THE SUMMARY OF RESULTS OF BENEFICIATION OF SELENITE SAMPLE FROM RAJASTHAN

was the principal metallic mineral in the sample followed by minor amounts of magnetite and chalcopyrite. Quartz and ferro-magnesium minerals formed the major gangue. Liberation of pyrite and pyrrhotite from the gangue minerals occurred below 100 mesh. Straight flotation using potassium ethyl xanthate and pine oil yielded a rougher concentrate assaying S, 46·58 percent with a recovery of 95·2 per cent sulphur. Two cleanings of the rougher float improved the grade of the concentrate to total S, 48·43; S as FeS_2, 45·43; S as FeS, 3·0; Fe, 46·6; SiO_2, 0·94 and Cu, 0·083 per cent and traces of Pb, Zn and As with a sulphur recovery of 68·1 per cent. The concentrate was suitable for sulphuric acid manufacture.

14.1 Beneficiation of Low Grade Pyrite-Pyrrhotite (Bore Hole No. 2) from Saladipura, Rajasthan

The sample as received assayed Fe, 27·8; S, 20·8; S as FeS, 6·4; S as FeS_2, 14·4 and SiO_2, 29·0 per cent. Pyrite and pyrrhotite constituted most of the metallic contents of the ore. Minor amounts of sphalerite, magnetite, chalcopyrite and rutile formed the balance of the metallics. Non-metallic gangue consisted mostly of quartz and ferro-magnesium minerals. Chlorite and calcite in minor amounts were also observed.

Rougher flotation of a feed having 83·0 per cent —200 mesh employing 2 kg./ton of H_2SO_4, 0·12 kg./ton of potassium ethyl xanthate and 0·05 kg./ton of pine oil followed by three cleanings of the rougher concentrate yielded a cleaner concentrate assaying S, 45·1; S as FeS, 8·6; S as FeS_2, 36·5; Fe, 48·0; SiO_2, 0·9; Al_2O_3, 2·28 per cent and trace of As with a recovery of S, 61·1 per cent in the concentrate.

15.0 Beneficiation of a Sample of Quartz-Sillimanite Schist from Sonapahar, Assam

A sample of quartz-sillimanite schist assaying Al_2O_3, 21·8; SiO_2, 69·2; Fe_2O_3, 2·46; TiO_2, 0·80; CaO, 0·4; MgO, 0·14; K_2O+Na_2O, 0·70 and L.O.I., 4·6 per cent was received from the Director of Geology & Mining, Shillong, Assam, for beneficiation studies.

The sample consisted mainly of quartz, sillimanite, rutile, muscovite and sphene according to the order of abundance. According to the size analysis an extremely fine grind of the order of 8 to 25 microns will be necessary for a fair liberation of sillimanite. From the grindability test graph the sample could be designated as medium soft ore. Flotation tests using sodium silicate as gangue depressant and oleic acid emulsion as collector for sillimanite did not produce satisfactory results.

Flotation test employing a grind of 79·6 per cent —200 mesh and using lactic acid as a depressant for mica and sodium sulphonate as collector for sillimanite at *p*H 4 followed by two cleanings of the rougher float yielded a refloat concentrate assaying 40·11 per cent Al_2O_3 with a recovery of 47·8 per cent Al_2O_3 only. Further tests are in progress.

16.0 Beneficiation of Manganiferous Clay from Goa

A sample of manganiferous clay was received from Chowgule & Co., Goa, with a view to improving the Fe and Mn contents. The original sample assayed Fe, 33·2; Mn, 9·6; MnO_2, 10·4; Al_2O_3, 16·8; SiO_2, 13·3 and L.O.I., 14·0 per cent; and traces of FeO, CaO and MgO. The sample was predominantly fine in nature and contained about 58 per cent —200 mesh material. Magnetite was the chief iron-bearing mineral followed by hematite and subordinate amount of hydrated iron oxide. Manganese minerals were not present in a large enough size to be easily identified and were intimately associated with the clayey material.

Simple washing, hydro-classification, tabling, reduction roast followed by wet magnetic separation and some flotation tests were carried out. So far no encouraging results have been obtained.

17.0 Studies on Recovery of Apatite and Nickel from Copper Tailing from Ghatsila (Indian Copper Corporation Ltd)

A sample of copper tailing was received from the Indian Copper Corporation Ltd, Ghatsila, for the recovery of apatite and nickel. The sample as received assayed P_2O_5, 1·27; Ni, 0·073; Cu, 0·17; S, 1·45;

Fe, 9·33; Al_2O_3, 13·94 and SiO_2, 59·7 per cent. The sample was fine and contained 45 per cent −200 mesh material. Nickel has been found to occur as pentlandite and violerite associated with pyrrhotite. Magnetic separation at different intensities for the recovery of nickel is in progress. Flotation tests to recover apatite using oleic acid as collector are in progress.

18.0 Beneficiation of Ferruginous Bauxite from Amarkantak Area of Hindustan Aluminium Corporation

A ferruginous bauxite sample from Amarkantak of Hindusthan Aluminium Corporation Ltd was received for beneficiation studies. The sample as received assayed Al_2O_3, 39·00; SiO_2, 3·0; Fe_2O_3, 28·1; TiO_2, and L.O.I., 20·5 per cent. The sample consisted of bauxite, laterite, ferruginous clay, hematite, goethite, quartz and rutile. The aluminous minerals were free in the −100 +150 mesh size range while the hematites were free in the −150 +200 mesh size.

Tabling at −28 mesh after hydro-classification yielded a bauxite concentrate assaying Al_2O_3, 42·78 and Fe_2O_3 23·52 per cent with a recovery of 62·9 per cent Al_2O_3.

Reduction roast followed by magnetic separation at −100 mesh size yielded non-magnetic product (bauxite concentrate) assaying Al_2O_3, 68·14 and Fe, 9·25 per cent with a recovery of 55·3 per cent Al_2O_3. Preferential flotation of either bauxite or iron bearing minerals did not show any encouraging results. Further tests are in progress.

19.0 Production of Sized Sand for Glass Industry from a Sand Stone Sample from Gujarat Mineral Development Corporation, Gujarat

About 3 tonnes of a friable gritty white sand stone from Gujarat was tested for crushing and grinding characteristics and to produce sized sand for glass industry. The sample consisted mainly of quartz of about 5 mm. size to fines, with minor amounts of calcite and trace of sphene, tourmaline, rutile and black opaque spheroidal magnetic material. On crushing the sample by jaw crusher with 1 in. set 61·4 per cent by weight of the weathered sample was reduced to −20 mesh size. The jaw crushed product after passing through the roll crusher in two stages gave 28·6 per cent +28 mesh and 23·0 per cent −100 mesh material. Best results for sizing with yield was obtained by mechanical screening at 20 mesh followed by sizing of the −20 mesh material by hydrosizer.

20.0 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 output per day depending on the type of the ferro-alloy required. Raw materials are accepted on contract for researches on their amenability to electro-thermal reduction techniques. A full report giving recommendations on smelting practice, power consumption and size of installation required for a given output are made based on these smelting trials. During the period under review the pilot 500 kVA submerged-arc furnace was operated under Campaign No. 19 with the following two major objectives:

(i) In continuation of the earlier work done for ascertaining the operation characteristics using various reducing agents in the production of standard grades of high carbon ferro-chrome conforming to IS: 1170-1960, various sized fractions of Durgapur coke were employed in combination with chromite ore from M/s. Tullock & Co. taken as a standard ore and operational characteristics ascertained.

(ii) To ascertain the feasibility of using soft chromite ore from Sukinda area, Orissa, supplied by M/s. Tisco Ltd for production of various grades of high carbon ferro-chrome.

A total of 13 tons in the earlier stage and 10 tons in the later stage of high carbon ferro-chrome was produced. The following raw materials were used during the campaign period.

RAW MATERIALS

(i) *Chromite* — Chromite from M/s. Tullock & Co. taken as standard and that received from M/s. Tisco Ltd, were crushed and sieved to remove −1/8 in. fraction and the average sieve analysis and the chemical analysis of the ores are given below:

Sieve analysis of chromite ore from M/s. Tullock & Co.

Size fraction	Per cent
−1″ +3/4″	8·51
−3/4″+1/2″	16·00
−1/2″+1/4″	59·22
−1/4″+1/8″	9·22
−1/8″	7·05
	100·00

Chemical analysis of the ore

Cr_2O_3	51·45%
FeO	10·63%
Fe_2O_3	4·47%
SiO_2	6·50%
MgO	17·86%
CaO	0·20%
Al_2O_3	7·35%
P	0·01%

(ii) *Coke* — Durgapur coke used in the starting of the campaign analysed as below:

Sieve Analysis

−1″ +3/4″	50·55
−3/4″+1/2″	8·35
−1/2″+1/4″	12·50
−1/4″+1/8″	9·03
−1/8″	19·59

Analysis of Coke

Fixed Carbon	70·9%
Volatile matter	2·18%
Moisture	1·52%
Ash	25·4%

Ash Analysis

SiO_2	52·44%
Al_2O_3	22·40%
Fe_2O_3	11·48%
CaO	4·60%
MgO	3·60%
TiO_2	1·72%
S	1·20%
P	0·21%

(iii) *Fluxes* — Quartzite washed and sieved to −3/4 +1/8 in. with over 99 per cent SiO_2 was used throughout the campaign.

After the preliminary heating up of the furnace the charging was started and the furnace was tapped at intervals of 4 hours. The performance data relating to the two main phases of the campaign were found as under:

First Phase:

Size Variations in Reducing Agent

Total chromite charged	27,200 kg.
Total coke charged	8,515 ,,
Total quartzite charged	935 ,,
Total power consumed	54,620 kWh.
Metal tapped	13,205 kg.
Slag/metal ratio	0·80
Power consumption/tonne of ferro-chrome	4,140 kWh.

Analysis of the Alloy

Cr	59·05-65·4%
Si	1·10-5·20%
C	5·31-7·65%

Analysis of the Slag

SiO_2	37·70%
Al_2O_3	49·71%
MgO	8·24%
FeO	0·19%
Cr_2O_3	4·16%

Second Phase:

Studies with Soft Chromite from M/s. Tisco Ltd

Total chromite charged	18,000 kg.
Total coke charged	6,105 ,,
Total quartzite charged	653 ,,
Total power consumed	35,050 kWh.
Total metal tapped	9,253 kg.
Slag/metal ratio	0·78
Average power consumption/ tonne of ferro-chrome	3,800 kWh.

Analysis of the Alloy

Cr	57·06-65·46%
Si	2·54-5·37%
C	5·40-8·19%

Analysis of the Slag

SiO_2	32·30%
Cr_2O_3	27·68%
Al_2O_3	2·92%
MgO	35·81%

Further work establishing the optimum working characteristics for the production of standard grades of high carbon ferro-chrome using the soft chromite and the coke produced in Tisco coke ovens is being done and the raw materials preparation for the same is under way.

21.0 Electric Smelting of Titaniferous Magnetite Ore

The objective was to smelt the titaniferous magnetite ore to produce steel making grade or foundry grade pig iron and a by-product slag rich in titania.

A sample of titaniferous magnetite ore received from the Khursipar area, Bhandara district, Maharastra, containing FeO 12·86; Fe_3O_4, 63·10; SiO_2, 2·71; Al_2O_3, 3·54; TiO_2, 13·6; MgO, 1 and vanadium, 0·40 per cent and traces of CaO was used for the investigation. A sample of limestone containing CaO, 41·81; SiO_2, 6·44 and L.O.I. 36·29 per cent was used as a flux during smelting. Different reducing agents tried include charcoal containing volatiles, 32; moisture, 10·18; carbon, 48·12 and ash, 9·7 and a sample of Durgapur coke with moisture, 1·52; volatile matters, 2·18; carbon, 70·9 and ash, 25·4 per cent.

Smelting trials were carried out in a 50 kVA indirect-arc furnace with a magnesite lining. The ore and limestone were crushed to −3/4 in. size and coke to −1/2 +1/8 in. size. The initial experiments with Durgapur coke indicated that limestone can be completely eliminated from the charge mixture and the corrosive titania slags react badly with the magnesite lining. As a result titania content in the slag was very much decreased, the magnesia content was high and the lining life was very poor.

In order to obviate the above unfavourable conditions in the subsequent series of experiments, limestone was excluded from the charge. Also a magnesite shell coated on the working surface by 1 in. thickness with rutile was used. Two blank smelting experiments were conducted to season the lining. Coke was replaced by wood charcoal with low ash content to keep the titania content in the slag high. Table 6 shows the operational data obtained during the trials.

Slag volume was gradually decreased which showed a possible enrichment of

Table 6 — Operational Data During Smelting of Titaniferous Magnetite Ore

Experiment No.	8	9	10	11	12	15	16
Titaniferous magnetite ore (kg.)	7·00	7·00	7·00	7·00	7·00	7·00	7·00
Wood charcoal (kg.)	3·50	3·00	2·80	2·80	2·80	2·80	3·00
Duration of smelting (hr.)	3	3	2¾	2¾	2¾	2½	2¾
Power consumption (kWh)	70	70	69	67	70	70	65
Weight of met al obtained (kg.)	2·5	4·0	2·25	3·6	3·5	3·5	3·0
Slag characters	Over reduced viscous carbon	First fluid	Very fluid	Very fluid	Very fluid	Very fluid	Very fluid

titania in the slag. Over reduction by way of adding charcoal more than the optimum (which is 43 kg. per 100 kg. of ore) causes the slag to be very viscous and unworkable at the operating temperatures of the furnace which was 1500°C. Rutile magnesite lining was non-corrosive to the titania-rich slags and the lining life was very good. The entry of magnesia into the slag was prevented by rutile coating perhaps due to the formation of highly refractory titanium carbonitrides. The power consumption was very high due to considerably higher losses of heat by way of radiation from this furnace. One experiment conducted in a 50 kVA direct-arc furnace indicated that power consumption could be 4500 kWH per tonne of ore smelted.

Attempt is being made to enrich titania in the slag by using petroleum coke as reductant which contains very low ash. A complete material balance is being worked out on the basis of chemical analysis of the products obtained.

22.0 Electrolytic Recovery of Tin from Tinplate Scrap

Based on the investigation conducted earlier (reported in previous Annual Report) a preliminary Project Report was prepared on the basis of detinning 100 kg. of tin plate scrap in alkaline bath per day. Cost estimation of one tonne of recovered tin of 99·8 per cent purity was worked out which came to approximately Rs. 16,010/- per tonne.

23.0 Electrodeposition of High Purity Chromium from CrO_3 Acid and Fluoride Bath

The investigation was taken up with a view to electrodeposit pure metallic chromium from chromic acid and fluoride electrolytic bath.

During the period under review, studies were made to find out the suitable working range of the various variables like temperature of the bath, composition of the bath, anode and cathode materials with or without diaphragms, and the respective effect on the C.E. of Cr deposition. Tables 7 and 8 furnish the data under varying electrolyte composition, cathode current density and use of different anode mate-. rials.

24.0 Nitriding of Ferro-chrome

The investigation was taken up with a view to produce nitrogen bearing ferrochrome and to study the effect of absorption of nitrogen with respect to the particle size,

Table 7 — Effect of the Composition of the Electrolyte on C.E.

Temperature of the electrolyte ... 100°C.
C.D. at the cathode ... 1000 amp./sq. ft.
Electrodes — Copper cathode and 7% tin & lead alloy anode

Expt. No.	Composition CrO_3 g/L	NaF g/L	C.E.% (cathode)
1	250	5	18·0
2	300	5	21·8
3	350	5	26·3
4	400	5	26·0
5	350	6	26·0
6	350	6·5	26·2
7	350	7·0	26·3

temperature, time and the 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. Various factors studied include the effects of temperature, size of specimens and period of reaction. It was observed that the amount of nitrogen absorbed increased 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. Nitrogen absorption below 950°C. was quite insignificant.

Some samples cut to 1 × 1/2 × 1/2 in. were nitrided for 24 hours at 850°, 900°, 950°, 1000°, 1050° and 1100°C. The nitrided specimens were examined microscopically to study the type of nitride formed and the extent to which it is formed. Micro-hardness testing was also undertaken from the periphery to a centre of the cut specimens.

The micro-hardness values and the photomicrographs revealed that three types of nitrides were found. For a sample nitrided at 1100°C. for 48 hours, the nitride (Type A) formed very close to the surface was analogous to an eutectic with an average width of 1·5 mm., and a micro-hardness value of 1206. The second type of nitride (Type B) formed next to the first type towards the core was in the form of lamellar precipitates randomly oriented in the matrix with the zone width of 2·1 mm., and a micro-hardness value of 1132. The third type of nitride (Type C) found as small globular precipitates was uniformly distributed. The magnitude of the nitride precipitate decreased gradually towards the core and resulted in decreasing micro-hardness value. The average micro-hardness value

Table 8 — Effect of Cathode Current Density on C.E.

Temperature 100°C.

Electrode materials same as in Table 7

Expt. No.	Composition of electrolyte —350 g/L CrO_3 5 g/L NaF	
	C.D. in amp./sq. ft.	C.E.%
1	672	19
2	700	19
3	800	21·4
4	850	22·0
5	1000	26·3
6	1050	26·3

Effect of Different Anode Materials on C.E.

Temperature — as in Table 8
C.D. — 1000 amp./sq. ft.
Cathode — as in Tables 7 & 8
Electrolyte composition — as in Table 7 & 8

Expt. No.	Anode material	C.E.%
1	Pb anode	8
2	Ag+Pb alloy anode (7% Ag)	10
3	Sn+Pb ,, ,, (1% Su)	23·8
4	Su+Pb ,, ,, (7% Su) ·	26·3
5	Stainless Steel ,, ,, (rectangular)	40·0
6	Steel (cylindrical)	44·8

of the nitride rich zone was 72·5. The micro-hardness value of the unnitrided portion of the sample was 430.

In samples nitrided at 850° and 800°C. nitrides of types 'A' and 'B' could not be detected by metallographic means. The sample nitrided at 950°C. showed a very thin layer of type 'B' nitride but the type 'C' nitride was found to be predominant. The sample nitrided at 1000°C. contained all the three types of nitrides.

Nitrogenation of standard ferro-chrome produced by alumino-thermic process can best be carried out at a temperature of 1100°C. for 24 hours with the alloy crushed to 12 mm. size to get a product with 3·5 per cent nitrogen. Higher nitrogen contents up to 10 per cent can be obtained by nitriding 2 to 4 mm. size of the alloy. Fig. 9 shows the various types of nitride formation at different temperatures.

25.0 Reducibility of Lumpy Iron Ores of Dalli Pahar

Reducibility characteristics of four Dalli iron ore samples, viz. hydroxide, laminated, compact laminated and massive, were studied at the instance of M/s. Hindustan Steels Ltd. Reducibility tests were carried out on iron ore lumps of different sizes, e.g. $-2 + 1\frac{1}{2}$ in., $-1/2 + 1$ in. and $1 + \frac{1}{2}$ in. From each sample 500 grams was reduced with hydrogen gas at 800°C. with the gas flow rate of 3·5 litres per minute. The time for 90 per cent reduction for all the samples studied is recorded in Table 9. Period for 90 per cent reduction of the Bolani iron ore, which is presently used by Hindustan Steels Ltd for production of iron at Durgapur, is also included in Table 9 for comparison. It was observed that the time for 90 per cent reduction increased with increasing ore size. Massive variety predominated in the sample and the reduction rate of other varieties was likely to be faster than the massive type only. The reducibility of Dalli iron ores was found fully comparable to that of Bolani iron ore which is now used in the production of iron at Durgapur (Hindustan Steel Ltd).

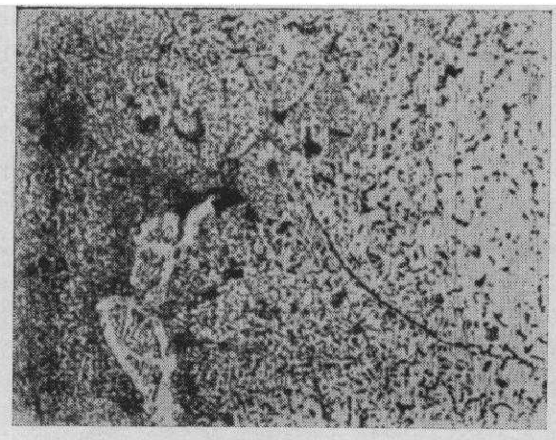
26.0 Beneficiation of Ilmenite by the Removal of Iron

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 problems. Laboratory scale investigations, have, therefore, been undertaken on the preferential removal of iron from ilmenite by roast reduction and subsequent aqueous

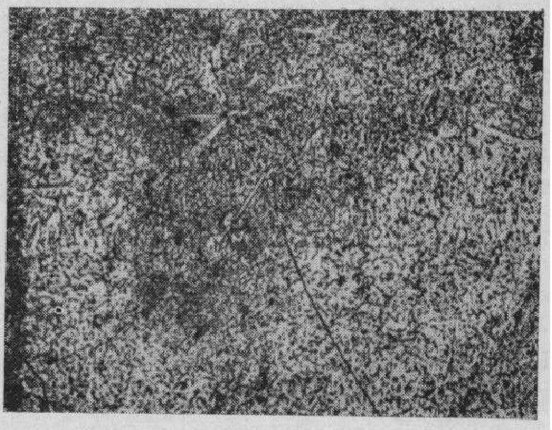
Table 9 — Effect of Size on Rate of Reduction of Lumpy Iron Ores

Weight of the sample ... 500 gm.
Temperature ... 800°C.
Hydrogen flow ... 3·5 litre/minute

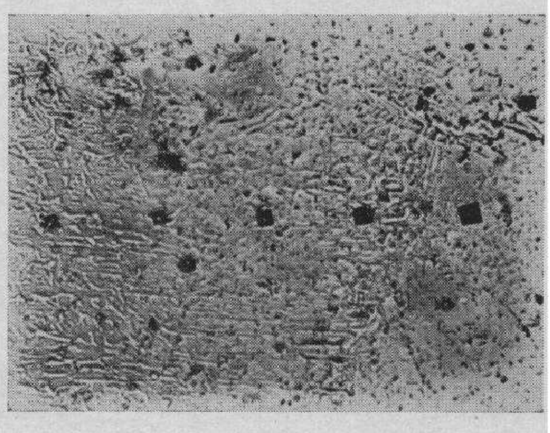
Sl No.	Ore size	Time for 90 per cent reduction (minutes)			
		Hydroxide ore	Laminated ore	Compact laminated ore	Massive ore Bolani
1	$-2'' + 1\frac{1}{2}''$	155	230	210	235
2	$-1\frac{1}{2}'' + 1''$	150	180	195	200
3	$-1'' + \frac{1}{2}''$	110	180	190	200



(a) Nitrided at 900°C. for 24 hr × 240. Etched in Aqua regia

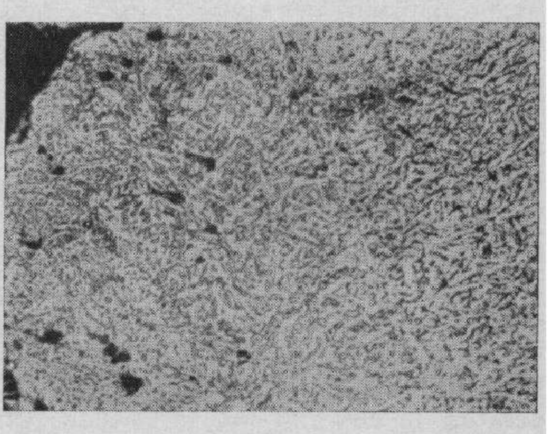


(b) Nitrided at 950°C. for 24 hr × 150. Etched in Aqua regia



(c) Nitrided at 950°C. for 24 hr × 450. Unetched

(d) Nitrided at 1000°C. for 24 hr × 150 Etched in Aqua regia



(e) Nitrided at 1100°C. for 48 hr × 450. Etched in Aqua regia

FIG. 9 — PHOTOMICROGRAPHS SHOWING THE VARIOUS TYPES OF NITRIDE FORMATION AT DIFFERENT TEMPERATURES

oxidation or by chlorination with hydrochloric acid gas.

Roast Reduction — Ilmenite samples from Travancore beach sands containing 55·44 per cent TiO_2 and total Fe 26·38 per cent was used during the semi pilot plant scale experiment. Based on earlier experiments reduction trials have been carried out with −100 mesh (BSS) size of ilmenite and −60 mesh (BSS) size of coke formed into briquettes and pellets. A rotary kiln has been used for reduction of ore. Oxidation studies are being carried out on 20-25 kg. lot of reduced ilmenite samples in a lead-lined M.S. tank having suitable arrangements for agitation and aeration.

At the initial stage reduction was carried out in refractory crucibles to find out the suitability of the different reducing agents. The temperature was maintained between 1150° and 1175°C., and reduction was carried out for 4 hours. Thirty per cent excess reducing agent over the stoichiometric amount were used for the tests. The extent of reduction of iron in ilmenite with charcoal, Durgapur coke and Giridih coke were found to be 74·4, 64·0 and 60 per cent, respectively. Higher carbon/ilmenite ratio gives better reduction and prevents the fusion of ilmenite but it increases carbide formation and affects subsequent oxidation of metallic iron. Attempt was made to bring down the reduction temperature by preoxidizing the sample. Pre-oxidation did not help as the FeO content of the sample was as low as 8 to 10 per cent.

Reduction trials were carried out in a small rotary kiln with hand made pellets. Bentonite, molasses or sulphite lye were used as binders for pelletization. The pellets disintegrated faster at higher temperatures causing increased losses of fines. Attempts to control the kiln temperatures between 1100° and 1150°C. was unsuccessful either by gas or oil-firing. The temperature in the kiln was either low, with incomplete reduction or high causing sintering of the charge. The maximum reduction obtained with the pellets was 7-10 per cent and the metallic iron content was low.

Loss of fines was considerably reduced by using briquettes made in a roller briquetting press with ilmenite to non-coking coal ratio at 1: 05, 1: 1, 1: 1·5 and 3 per cent molasses as binder. The extent of reduction was only between 15 and 20 per cent in all the tests having different proportions of the reductant due to oxidizing conditions in the furnace which could not be avoided.

Reduction tests in a pot type furnace with the briquettes or fines enclosed in a refractory curcible were encouraging and the extent of reduction in all the trials varied from 85 to 90 per cent. Owing to the slow rate of heat penetration to the centre of the charge, the time required to complete the reduction was long. Reduction of 15-20 kg. mix per batch is being carried out in a oil-fired furnace inside a refractory cruicible for carrying out the oxidation studies in a large leaching tank. Reduction studies with either a powder or briquetted charge would be carried out after a suitable kiln is made.

Chlorination of Ilmenite with HCl gas — The ilmenite concentrate employed in the investigation analysed TiO_2, 52·7; 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. Ilmenite was briquetted with 1 per cent dextrine as binder. Chlorination was carried out in a vertical silica reactor with HCl gas passing downwards from the top. The reaction products were continuously collected at the bottom. Systematic studies were carried out to find the effect of temperature, particle size and period of reaction, effect of pre-reduction and preoxidation and also the effects of hydrogen, chloride and oxygen in the chlorinating gas mixture on the removal of iron and other impurities.

Chlorination of ilmenite at 800°C. using hydrogen chloride alone has been observed to remove 92 per cent iron, and substantial quantities of chromium and vanadium yielding a product approaching rutile composition. The reaction product, however, contains a mixture of ferrous and ferric chloride and by the use of HCl-air mixture iron has been removed as $FeCl_3$ alone, thus considerably simplifying the entire operation. It was found possible to remove 80-90 per cent of the vanadium and 42-50 per cent of the chromium from the ilmenite with a HCl-air gas mixture containing 2-6 per cent air, and the residue analysing 95-97 per cent TiO_2 has been obtained with a rutile structure. It is proposed to carry out the chlorination of ilmenite with HCl in a fluidized bed reactor.

27.0 Extraction of Nickel from Nickeliferous Ores

Several low grade nickel ores of lateritic and serpentinous origin have been recently explored by the Geological Survey of India. In view of the strategic importance of nickel, the recovery of nickel from these low grade ores is of considerable importance and systematic investigations are in progress to study the extraction of nickel from the nickel-bearing ores from different regions.

(i) LATERITIC ORES FROM SUKINDA, ORISSA

Two samples of nickeliferous laterites were received from Sukinda area of Orissa. One of these consists of light brown light aggregates and the other dark brown dense aggregates.

(a) *The Light Aggregates* — The light grade nickeliferous laterites analysed NiO, 1·8; total Fe, 47·04; Cr_2O_3, 6·2; SiO_2, 6·8; MgO, 1·1; Al_2O_3, 12·36 and Co_2O_3, 2·9 per cent. The ore was pulverized and reduced with hydrogen. The reduced ore was leached with ammoniacal liquor in presence of gaseous oxygen. The various factors studied include the effect of temperature of reduction, particle size, rate of flow of hydrogen, kinetics of reduction, preconditioning and leaching, as well as the effect of different anions during the ammonical leaching. It was found that 82 per cent nickel extraction is possible by reducing −100 mesh ore at 750°C. for 60 minutes with a hydrogen flow-rate of 20 litres/hour, followed by one hour preconditioning prior to leaching with 7·5 per cent ammonium carbonate in 7·5 per cent ammonium hydroxide in presence of air at room temperature for a minimum period of 6 hours.

(b) *Dense Laterites* — The dense grade Sukinda nickeliferous laterite analysed, NiO, 0·70; total Fe, 39·5; Cr_2O_3, 2·9; SiO_2, 16·5; MgO, 1·12; Al_2O_3, 14·00; MnO, 1·85; and Cu, 0·2 per cent and traces of Co_2O_3. The ore was ground and reduced with hydrogen followed by ammonical leaching. The effects of variable factors indicated above was investigated and it was observed that 71 per cent Ni could be recovered by reduction with hydrogen at 600°C. flowing at 20 litres per hour. The reduced ore needed preconditioning for one hour and was leached for 6 hours with an ammonical liquor containing 7·5 per cent ammonium carbonate and 7·5 per cent (by wt) ammonium hydroxide.

(ii) NICKEL BEARING LATERITES FROM BADAM PAHAR, ORISSA

A sample of nickeliferous laterite containing NiO, 0·42; Fe_2O_3, 41·8; SiO_2, 15·5; Al_2O_3, 22·4; Cr_2O_3, 3·4 and V_2O_5, 0·27 per cent was received from the Badampahar area of Orissa. The sample was dark, brown, hard and lumpy. The ore was pulverized and reduced with hydrogen followed by ammonical leaching. The various factors studied include the temperature of reduction, particle size of the ore, flow rate of hydrogen; kinetics of reduction, preconditioning and leaching and the effect of nature of ammonium salts and their concentrations in the liquor on the nickel extraction.

It was observed that the ore could be best reduced at 650°C. with hydrogen for 60 minutes flowing at the rate of 10 litres per hour over a -170 mesh (BSS) size of the ore. The reduction needed an hour long preconditioning and leaching with 7·5 per cent of ammonium carbonate and 7·5 per cent ammonium hydroxide complete at room temperature, in presence of oxygen gas within 3 hours. Ammonium carbonate was found to be the best leachant as compared to sulphate, chloride, nitrate or hydroxide. The maximum nickel recovery obtained was restricted to 52 per cent by the method studied.

28.0 Extraction of Vanadium from Vanadium Bearing Titaniferous Magnetite

Vast reserves of vanadium bearing titaniferous magnetite occur in Orissa and Bihar. An ore received from southern border of Dalbhum in Bihar was taken up to study the extraction characteristics of vanadium from the ore by salt roasting.

An ore containing V, 1·05; total iron, 54·32; Al_2O_3, 3·50; SiO_2, 1·32; CaO, 1·39; MgO, 1·30; Cr_2O_3, 0·38 and S, 0·09 per cent was used for the investigation. The sample was subjected to an oxidizing roasting with NaCl and the product was water leached for the dissolution of vanadium. The factors studied include effects of temperature, particle size, soaking in NaCl solution, period of roasting varying amounts of NaCl and the effect of addition of a basic oxide during roasting. It was possible to get a vanadium recovery of 73·5 per cent by roasting the ore with 12 per cent NaCl at 820°C. for 4 hours and subsequent leaching of the roasted product in water. Further work for improving the recovery is in progress.

28.1 Removal of Iron from Vanadium Bearing Titaniferous Magnetite

Vast reserves of vanadium bearing titaniferous magnetite containing up to 2·5 per cent V_2O_5 occur in Orissa and ores cannot be upgraded by conventional ore-dressing methods as vanadium occurs in solid solution with iron and is an integral part of the magnetite grain. The investigation was taken up to beneficiate the vanadium bearing titaniferous magnetite ore through removal of iron by preferential roast reduction and aqueous oxidation in order to get a vanadium enriched product.

An ore containing V, 1·05; total iron, 54·32; Al_2O_3, 3·50; SiO_2, 1·32; CaO, 1·39; MgO, 1·30; Cr_2O_3, 0·38 and S, 0·096 per cent was used for beneficiation studies. The ore sample was reduced using coke as well as hydrogen gas. The factors studied include effect of temperature on iron reduction, effect of the amount of reductant, effect of particle size and kinetics of reduction. The reduced samples obtained after reducing coke and hydrogen gas were subjected to aqueous corrosive leaching to oxidize the reduced iron using NH_4Cl as an electrolyte. The liberated 'iron rust' can be separated by any known physical means. Factors studied during oxidation include effect of intermittent grinding, effect of different electrolytes, effect of various amounts of electrolytes and kinetics of oxidation.

It was possible to get an enriched residue containing V, 2·3 and total iron, 32·36 per cent by preferential reduction of iron, using coke as the reductant and subsequent oxidation of iron. When hydrogen gas was used for reduction an enriched product containing V, 2·78 and total iron, 28·56 per cent could be obtained.

29.0 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 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 conventional Bayer's process or any other suitable treatment consistent with the nature of the bauxite under investigation.

The bauxite sample contained Al_2O_3, 55·45; SiO_2, 22·4; Fe_2O_3, 5·3; TiO_2, 2·9 and L.O.I., 14·56 per cent. 4·65 per cent of its alumina content was present as gibbsite, 2·56 per cent as boehmite, 20·71 per cent as kaolinite and 72·08 per cent as diaspore.

Desilication of the bauxite sample was attempted by calcining at 850°C. followed by alkaline leaching with 15 per cent sodium hydroxide solution. Calcination of bauxite crushed to different sizes indicated that 89·7 per cent of silica could be removed using $-72 +100$ mesh size of the calcine. Silica removal improves only slightly with finer sizes but very fine grinding leads to very slow settling rate and affects the silica removal.

A bauxite sample crushed to -72 mesh (B.S.S.) size was calcined at 850°C. for various periods and was leached with 15 per cent NaOH for 1 hour at 95°C. It was found that 87·9 per cent of silica was rendered soluble by calcination at 850°C. for one hour and the silica removal thereafter gradually improved to 89·76 per cent with increasing period of calcination to 4 hours. The bauxite sample was calcined at 850°C. for 4 hours and was leached with 15 per cent NaOH solution for 1 hour at different temperature. It was found that minimum temperature of leaching for efficient silica removal should be 95°C. or boiling temperature of the solution. The kinetics of leaching was studied at 95°C. and the results are given in Table 10.

Table 10 — Kinetics of Leaching

Sl No.	Period of leaching (min.)	Desilication %	Percentage of alumina loss
1	30	80·17	8·53
2	60	89·76	9·36
3	90	89·84	13·02
4	120	90·03	15·10

It can be observed from the table that in one hour 89·76 per cent silica was removed with a minimum loss of alumina. With increasing period of leaching there was no appreciable change in the silica removal but corresponding alumina losses are high. Leaching with 15 per cent NaOH solution was carried out at 95°C. for 1 hour with varying quantities of NaOH. Desilication was found to be more effective with increasing Na_2O/calcine ratio. But the increased proportion of Na_2O leads to increased Na_2O/Al_2O_3 ratio and results in higher alumina loss during the desilication process. It was possible to obtain a desilicated residue with 4·4 per cent SiO_2 by calcining a -72 mesh (BSS) sieve bauxite at 850°C. for 4 hours and leaching the calcine with 15 per cent NaOH at 95°C. for one hour using 500 c.c. of leachant for 25 g. of the calcine.

30.0 Fluo-solid Roasting of Sulphide Ores

Fluo-solid roasting of sulphide ores is considered to be better than other processes as it helps in production of high SO_2 containing flue gases with a reasonably high capacity per unit chamber space, a good control over the roasting conditions and the production of roasted ore of constant composition. Fluo-solid roasting is taken up as a primary requisite to feed the leaching and electro-winning plants of copper and zinc for consistent operation. To enable a systematic study on roasting characteristics of non-ferrous sulphide ores, a six inch Dorr Oliver Reactor was installed.

A zinc concentrate was received from M/s. Hindustan Zinc Ltd, Udaipur, for roasting studies. The concentrate analysed Zn, 53·52; Pb, 2·34 and Fe, 5·51 per cent and 60 per cent of the particles were of −100 mesh (B.S.S.) size. To facilitate bench scale experiments on the roasting of sulphide ores, a six centimeter stainless steel reactor, 110 cm. long, with a suitable dust collector was fabricated to work up to a temperature of 1000°C. A suitable vertical furnace to house this unit with four burners working on coke oven gas was also made. Temperature of the furnace could be controlled within ±10°C. A six centimeter model glass unit similar to the reactor in all dimensions was made for space rate studies in order to ascertain the bed height, pressure drop across the bed and air velocity for various particle sizes of the ores. A continuous feeding device was incorporated to this model. Further work is in progress.

31.0 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 for pyrotechnique. The investigation was taken up at the instance of the defence establishment for improving the metallic content to over 99 per cent from oxidized powder containing high proportions of magnesium oxides.

From defence establishment 600 kg. of magnesium powder, grade V, was received for reconditioning. The metallic content of the oxidized powder varied from 77·40 to 91·31 per cent. The powders were treated and upgraded to over 98 per cent metallic magnesium.

32.0 Recovery of Germanium from Coal Ash

Germanium occurs as a minor constituent in coals and is discarded in the coal ashes. In view of its increasing importance in electronic industry, studies have been undertaken for extraction of germanium from coal ashes.

Representative samples of coals from various coal fields and fly ashes and flue dusts from various thermal power stations were obtained and analysed for their germanium contents by spectroscopy and X-ray fluorescence methods. Table 11 gives the results of analysis of eight samples which gave positive results out of fifteen samples analysed.

It can be observed from Table 11 that the fly ash from Jharia coals and flue dust from Singareni Collieries contain 56 and 58 ppm of Ge and extraction of germanium

Table 11 — Analysis of Fly Ashes and Flue Dusts for Germanium Extraction

Sl No.	Sources	Coal used	Ge (ppm)
Fly ashes			
1	Calcutta Electric Supply Corpn. Ltd, Calcutta	Lower Raniganj coal fields	17
2	Kothagudam Thermal Station, Andhra Pradesh	Singareni collieries	34
3	Tata Trombay Thermal Station, Bombay	Madhya Pradesh & Panel Valley fields	8
4	Sindri Thermal Station, Fertilizer Corporation of India, Sindri, Bihar	Jharia coal fields	56
5	Kalyan Power House, Thakurli, Maharashtra	Not known	36
6	Ramagudam Power Station, A.P.	Singareni collieries	17
Flue dusts			
7	Calcutta Electric Supply Corpn. Ltd, Calcutta	Raniganj coal fields	6
8	Kothagudam Thermal Power Station	Singareni collieries	58

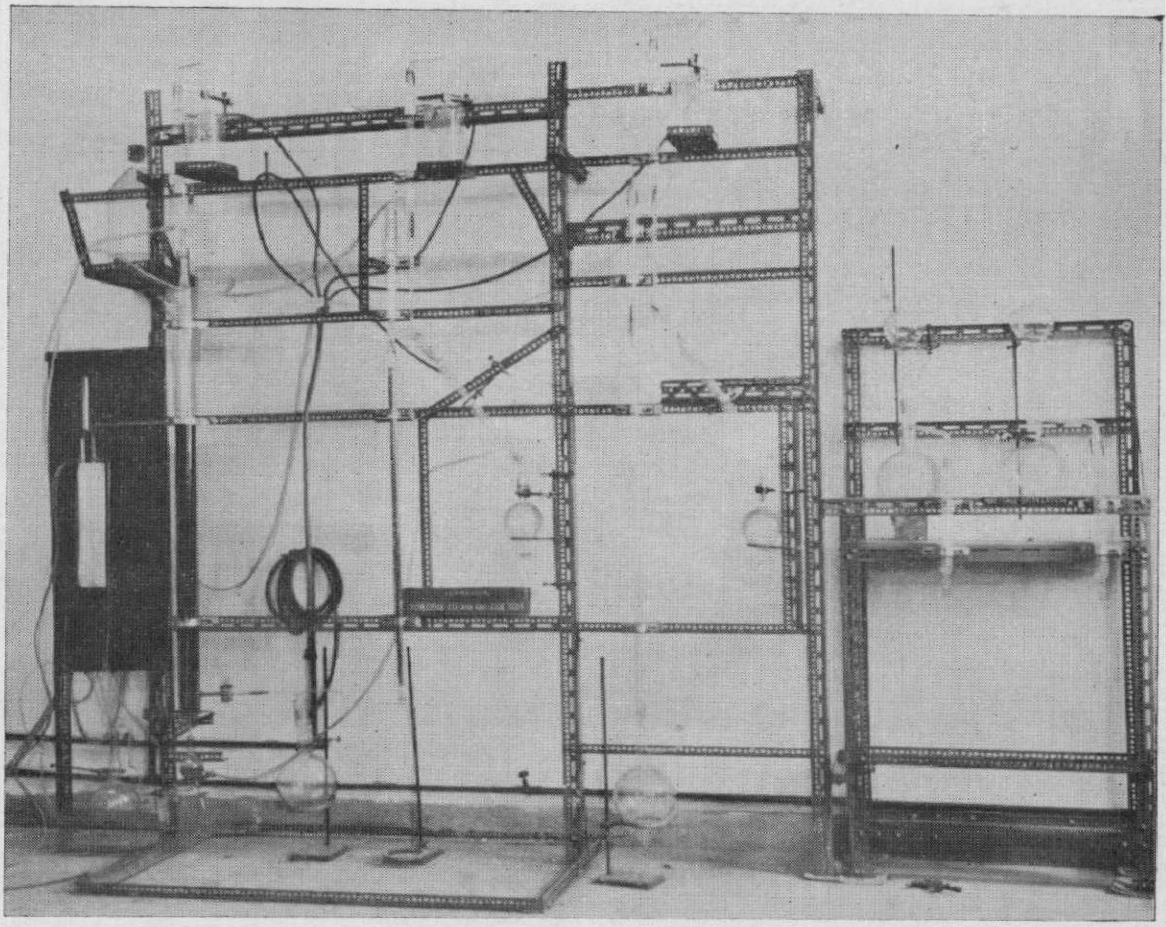


FIG. 10 — SET-UP FOR RECOVERY OF GERMANIUM FROM COAL ASH BY CHLORINATION

is being attempted, by smelting the fly ash and flue dust to obtain a germanium-rich regulus which can further be chlorinated with chlorine to extract germanium as germanium tetrachloride. An experimental set up for chlorination of the regulus with arrangements for fractionation and purification of germanium tetrachloride was designed and fabricated. The set up is shown in Fig. 10.

33.0 Recovery of Selenium and Tellurium from Copper Slimes

Copper slimes obtained as a by-product from electrolytic refining of blister copper at Indian Copper Corporation, Ghatshila, appear to be the main source of selenium and tellurium in India. In view of the wide applications of these rare metals specially in the manufacture of rectifiers and photocells, a systematic study on the recovery of selenium and tellurium from copper slimes was taken up.

A sample of anode slime obtained from Indian Copper Corporation, Ghatshila, analysed SiO_2, 6·5; Cu, 19·4; Ni, 31·5; Fe, 0·5; Se, 10·5; Te, 12·2; As, 5·44; S, 3·76; Ag, 0·772 and Au, 0·0928 per cent.

The slime was crushed and the crushed sample was roasted with sulphuric acid at different temperatures in the range of 350° to 575°C. in an all-glass apparatus. The roasting set up comprising the horizontal glass roaster along with furnace

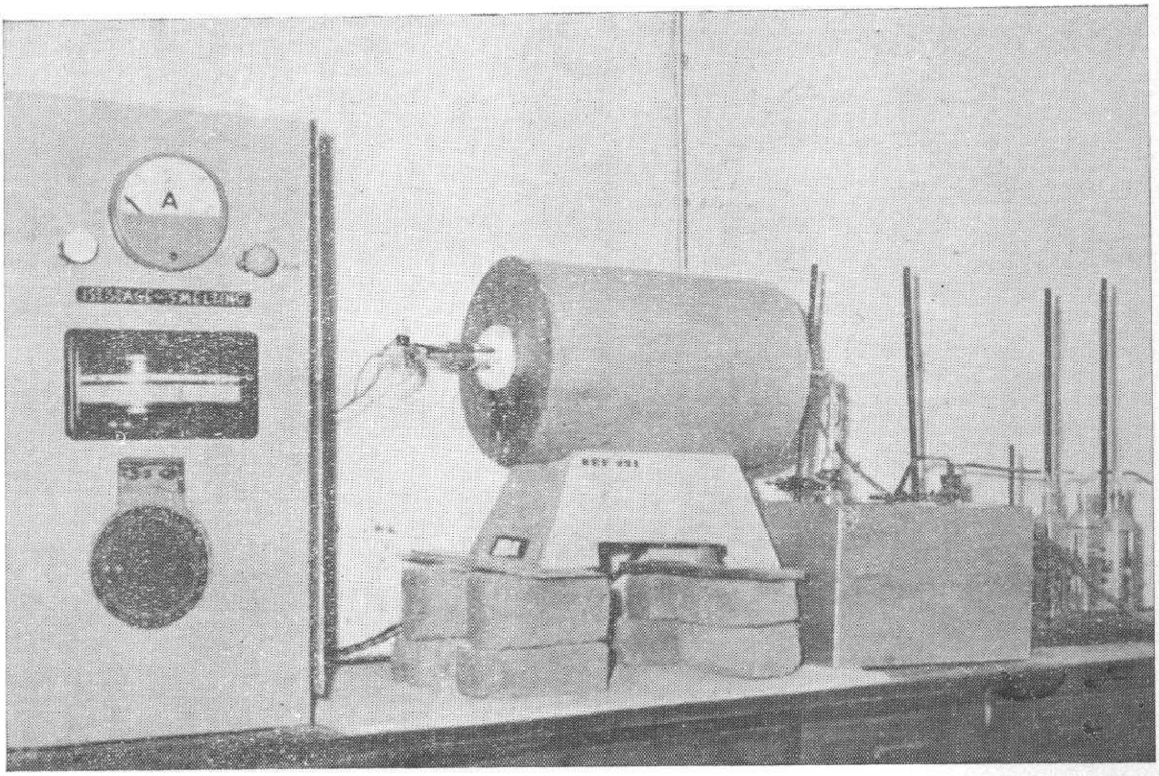


FIG. 11 — SET-UP FOR EXTRACTION OF SELENIUM FROM COPPER SLIMES BY SULPHURIC ACID ROASTING

control pannel, and the selenium collectors are shown in Fig. 11. It was found that during roasting, nickel and copper form water soluble sulphates and a substantial part of selenium was volatilized. The flue gas containing SeO_2 and entrained selenium was washed in water and dilute alkali to retain selenium, which could be separated later completely by passing sulphur dioxide to these acidified solutions. Over 90 per cent de-nickelization and 50 per cent de-copperization had so far been achieved by roasting the slime at 525°C. with double the quantity of sulphuric acid (by weight) for four hours. Further studies on the effect of varying percentage of acid in the charge and duration of roasting are under progress.

34.0 Preparation of Anhydrous Magnesium Chloride

At the instance of M/s. Hindustan Aeronautics Ltd work was taken up on the preparation of anhydrous magnesium chloride which can be used as fluxing material in melting, casting and refining of magnesium base alloys.

Due to difficulties experienced in preparing anhydrous salt by using various drying techniques, attempts were made to prepare it synthetically as per the following type of reactions:

$$MgO + C + Cl_2 \rightarrow MgCl_2 + CO$$
$$2MgO + C + Cl_2 \rightarrow 2MgCl_2 + CO$$

Theoretically both the reactions are exothermic and should enable the conversion without much practicable difficulty.

Different treatments were given to prepare the briquettes to achieve maximum possible porosity and crushing strength and its subsequent conversion to magnesium chloride. The average working temperature was around 920°±30°C., which favoured easy removal of magnesium chloride as liquid and readily drained. The adhering liquid

Table 12 — Results of Experiments on Preparation of Synthetic Cryolite

Sl No.	Fluospar added gm	H_2SO_4 added gm	Residual H_2SO_4 gm	$CaCO_3$ added gm	HBF_4 found gm	Hydrated alumina added gm	Na_2CO_3 added gm	Cryolite pptd gm	Remarks
1	200	243	26·0	26·0	85·0	72·0	76·0	135	—
2	200	243	31·3	32·0	102·4	82·5	96·0	163	—
3	200	243	28·6	29·7	95·5	77·0	85·0	148	—
4	200	243	31·8	32·0	94·2	76·0	83·0	146	—
5	200	243	35·1	36·0	93·3	76·0	83·0	147	—
6	200	243	33·6	34·0	90·6	74·0	81·0	142	—
7	200	243	33·6	34·0	100·2	81·0	89·0	163	—
8	200	243	33·0	33·0	100·8	82·0	82·0	157	—
9	200	243	30·4	31·0	82·8	68·0	74·0	128	—
10	200	243	26·4	27·0	106·2	86·0	88·0	162·5	—
11	200	243	35·0	36·0	92·1	75·0	79·0	146	—
12	200	243	32.7	33·0	96·7	78·0	86·0	150·5	—
13	200	243	31·2	32·0	94·1	76·5	84·0	148	—
14	200	243	33·3	34·0	95·7	78·0	83·0	147·5	—
15	200	243	33·3	34·0	95·7	78·0	83·0	148	—
16	200	243	34·9	35·0	99·8	81·0	120·0	144	Iron was removed from the liquor
17	200	243	45·7	47·0	92·0	75·0	75·0	135·5	—
18	200	243	43·6	44·0	92·8	75·0	75·0	137	—
19	200	243	41·0	42·0	97·5	79·0	80	148	—
20	200	243	41·9	42·0	89·4	75·0	83	131	—

layer should be thin to increase the gas diffusion for maximum conversion. The chlorination trials were conducted in a small 1 in. dia. vertical silica tube, internally heated unit. Average chlorine efficiency was only 60 per cent. The composition of the product averages between 97-98·4 per cent $MgCl_2$. Impurities from raw materials were found in the product as KCl, 0·75; NaCl, 0·8-1·2 and $CaCl_2$, 1-1·5 per cent; Fe, SiO_2 and Al not more than 0·067 per cent; and carbon, 0·3-0·8 per cent max. Further work is under progress to establish the conditions for successfully chlorinating the briquettes of magnesia-carbon mixture in a 6 in. dia. refractory tube, internally heated unit.

35.0 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 deposits of fluorspar in Rajasthan, Gujarat and Madhya Pradesh. Fluorspar is one of the chief raw materials used in the production of synthetic cryolite. The indigenous fluorspar is low-grade and required beneficiation in the first instance. Extensive work on laboratory and pilot plant scale on beneficiation of fluorspar was conducted.

Experiments for improvement of the process reported earlier were carried out to increase the number of cycles, so that the spent liquor can be circulated a number of times at reasonable leaching efficiency. By controlling certain variables it was possible to recycle the process liquor a number of times thus improving the process further. The results of the experiments are shown in Table 12.

Average cryolite analysed F, 54; Na, 23·9; Al, 17·5; SO_4, 3·5; Ca, 0·6; Fe, 0·05; SiO_2, 0·04 and moisture, 0·4 per cent. The salient feature of the experiment was that the precipitation of cryolite was carried out at a definite *p*H so that the precipitation was complete. The control of *p*H during the precipitation of cryolite was such that the residual aluminium was kept at the lowest level in the digest liquor. This control consequently allowed the cyclic experiment to proceed up to 20th cycle thus improving the overall utilization of boric acid. The loss of boric acid also dropped. Further experiments are being planned to increase the fluorine recovery and raise the purity of the cryolite product further.

36.0 Nickel-free Austenitic Stainless Steel

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

Comprehensive investigations were carried out on the determination of physical, mechanical, corrosion resistance 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 subzero tensile and impact properties, high temperature tensile and impact properties, high-temperature tensile strength, weldability, oxidation resistance and ageing characteristics of different compositions of the nickel-free austenitic stainless steels.

The complete data of the research and development work on substitute nickel-free stainless steels had been published in the *Journal of Iron & Steel Institute* (U.K.) March, 1967. This steel is being contemplated to be produced at the Alloy Steel Plant of Hindustan Steel, Durgapur.

36.1 Isothermal Transformations in 17 Per Cent Cr-Mn-N Stainless Steels

The object of this investigation is 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 is also under investigation.

In continuation of the work reported earlier, it was found that the addition of 10 per cent Mn to these steels at constant C+N level makes them entirely austenitic in structure. In the present investigation, main emphasis was laid on the behaviour of the diphase structure of delta+gamma when subjected to isothermal treatment at different temperatures. Two heats of the composition given in Table 13 were selected.

Table 13 — Composition of Steel

Heat	Cr,%	Mn,%	C,%	N,%
S_3	16·91	6·5	0·027	0·34
S_4	17·08	8·3	0·027	0·39

The steels were made in the high frequency induction furnace. Ingots were hot-forged and rolled to required sizes.

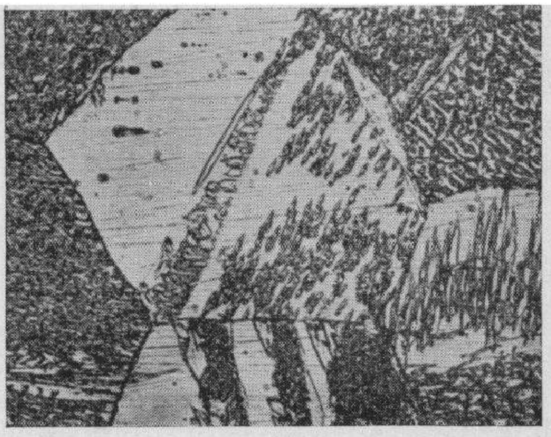


FIG. 12 — S_3-1200°C., 15 MIN. × 240. $\gamma+\delta$ WITH PRECIPITATION OF CARBIDES AT THE GRAIN BOUNDARY

FIG. 13 — S_3- 900°C., 2 HR × 240. THE DECOMPOSITION OF δ INTO AUSTENITE

The test pieces were solution treated at 1100°C. for 15 minutes. These were subjected to isothermal treatment at different holding temperatures in vacuum, and a record of their metallographic and X-ray data was obtained. Figs. 12 to 15 indicate the sequence of transformations as observed in these steels.

The mechanism of these transformations can be enumerated as follows for descending temperatures:

(a) Formation of austenite from delta ferrite.

(b) Precipitation at the grain-boundaries, delta-delta and delta-gamma, of a carbide phase $(Cr, Fe)_{23}C_6$ and Cr_2N.

(c) A partial transformation of delta-ferrite into a lamellar aggregate of carbides and an unstable austenite. This austenite is transformed to martensite during cooling.

(d) A regression of the austenite, becoming converted to alpha ferrite.

(e) Decomposition of the austenite, starting from the grain-boundaries delta-gamma, into an aggregate consisting of very fine carbides and alpha-ferrite.

(f) Partial transformation of the austenite into nitrogen pearlite (Cr_2N+, alpha).

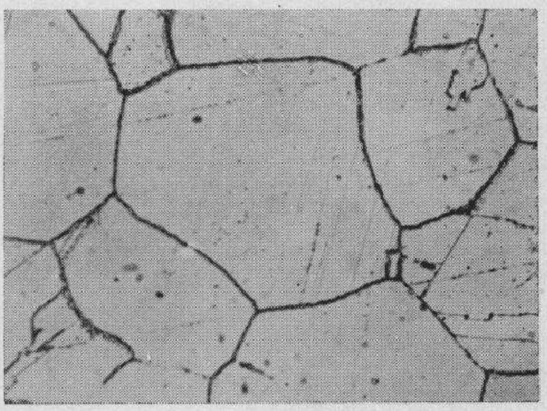


FIG. 14 — S_4-900°C., 2 HR × 240. COMPLETE TRANSFORMATION OF δ INTO AUSTENITE. GRAIN BOUNDARY PRECIPITATION OF THE CARBIDE PHASE

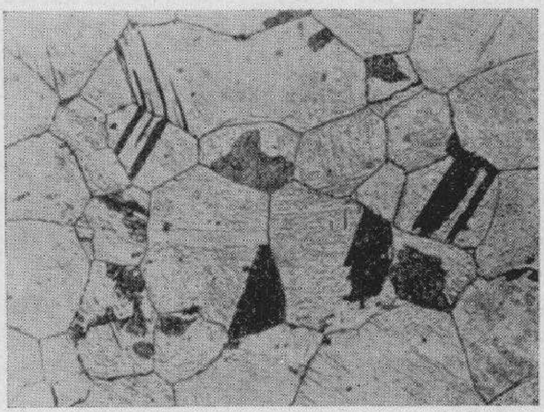


FIG. 15 — S_4-750°C. 100 HR × 240. PARTIAL TRANSFORMATION OF γ INTO A PEARLITE-LIKE PRECIPITATE OF Cr_2N

The validity of these postulations is being further examined with the help of the electron-microscopic study of these steels.

37.0 Studies on the Substitute High Strength Steels

Recently great demand of high strength steels (tensile levels above 100 tons/in.^{2}) has been felt in the aircraft and turbine industries. Attempts are being made at the National Metallurgical Laboratory to try some of the thermo-mechanical techniques such as 'Ausforming' and 'Marforming', recently developed in USA and other countries, for the purpose of developing high strength steels and also to study the underlying mechanism behind such type of strengthening. The following studies are being made presently:

(i) *Ausforming of Low Alloy Steels* — Ausforming process is the application of deformation to the metastable austenite at the bay of T-T-T curve (prior to transformation to bainite) followed by quenching to martensite. Studies were taken up on the effects of ausforming process together with composition and processing variables on the mechanical properties of substitute high strength steels of low alloy compositions. Low alloy steels of Ni, 1·5-3; Mn, 0-4 and Cr, 1-3 per cent with medium carbon content and with or without the additions of elements like Ti, Mo and V, etc., at the level of 0·2-1 per cent, were prepared for these studies. The deformation of metastable austenite, brought to the working temperature by quenching in a lead bath of desired temperature, is proposed to be effected by drawing or by forging. A suitable lead-bath furnace required for this purpose was specially designed and fabricated. Other preliminary preparations are in progress.

(ii) *Marstraining and Marforming* — Recent literature reports by a number of authors suggest that very high strength in steels can be produced by straining martensite to a limited extent which is termed 'marstraining' or 'marforming'. Studies were carried out on these steel samples of nominal compositions given below:

Steel No.	C,%	W,%	Mo,%
RX 1	0·95	2·45	0·10
RX 2	0·94	1·17	0·040
RX 3	0·88	2·73	0·046

For producing martensite a solution treatment at 1050°C. for one hour followed by quenching in iced brine was adopted. All the samples were given a systematic pretempering treatment from 250°-500°C. before straining (0-0·7%) and a retempering treatment at the same temperature range. The effect of straining caused increase in strength and a decrease in ductility. With the increase in retempering temperature (up to the pretempering temperature) these effects were enhanced until maxima was reached in the strength and a minima in the ductility (Figs. 16 & 17). Preliminary studies of the carbon replicas taken from the polished and etched samples indicated that the observed changes in mechanical behaviours of the steels are due to the fact that the carbides in the tempered martensite are dissolved and re-precipitated by the straining and retempering. Further studies on this aspect are being extended on the maraging type of steels. The effect of cold working on maraging type of steels known as 'marforming' has created a great deal of interest. Efforts are being made to develop some substitute maraging type steels and to study the marforming process on these steels and also on conventional type of maraging steels.

38.0 Development of Substitute Alloy Steels

Objectives of this project are based on the basic themes to develop indigenous

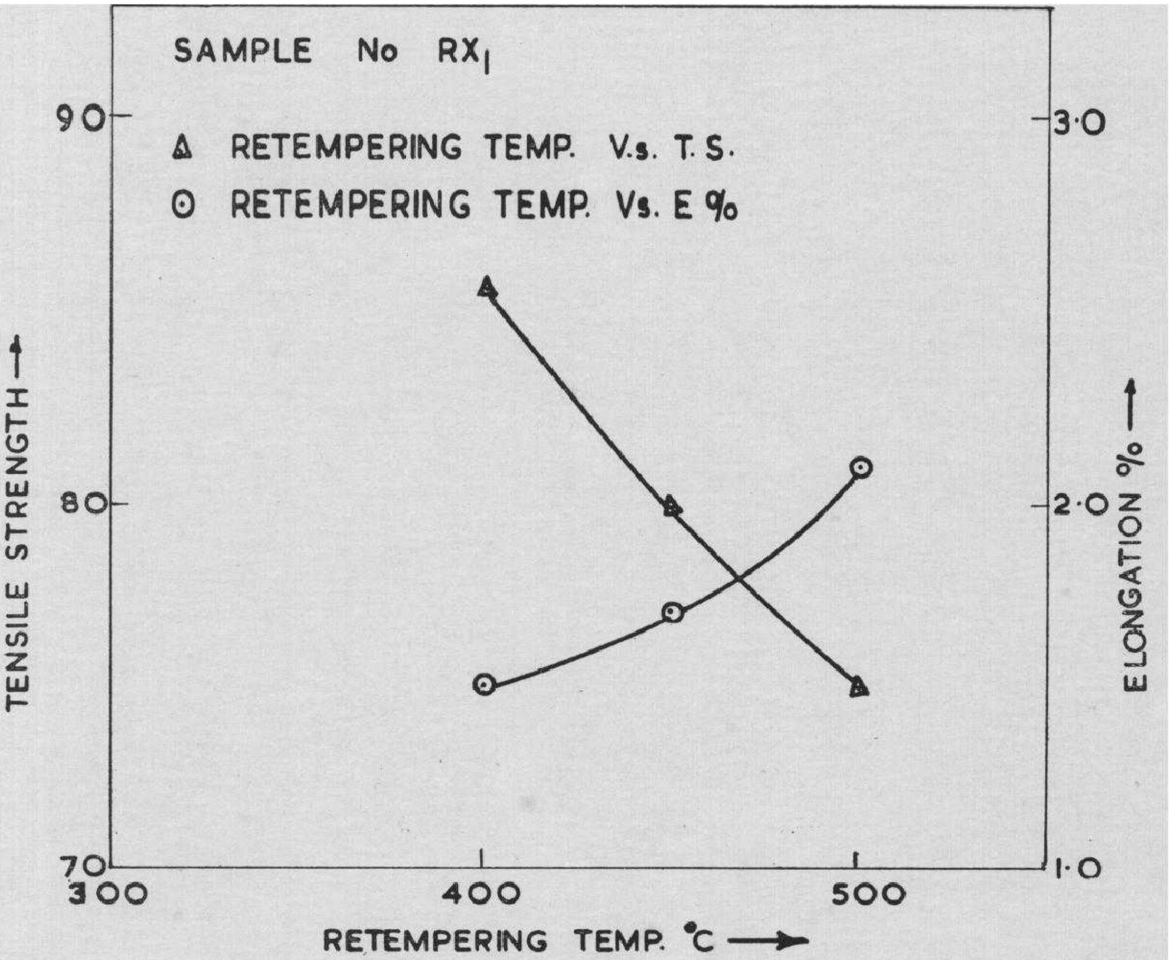


Fig. 16 — Effect of Retempering Temperature on Tensile Strength and Ductility Keeping the Degree of Prestrain 0·5%

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

(i) *Die Steels* — Tempering characteristics of steel DS_1 of composition given in Table 14 were investigated. Different tempering temperatures were selected to determine the correct tempering time and temperature of the steel investigated. The results obtained are shown in Table 15.

(ii) *Ausforming of Die Steel* — The effect of ausforming on high carbon high chromium cold work die steel was studied in relation to various percentages of deformation, tempering temperatures and sub-zero treatment. It was observed that by the application of ausforming on high carbon chromium die steel, a substantial increase in hardness was obtained.

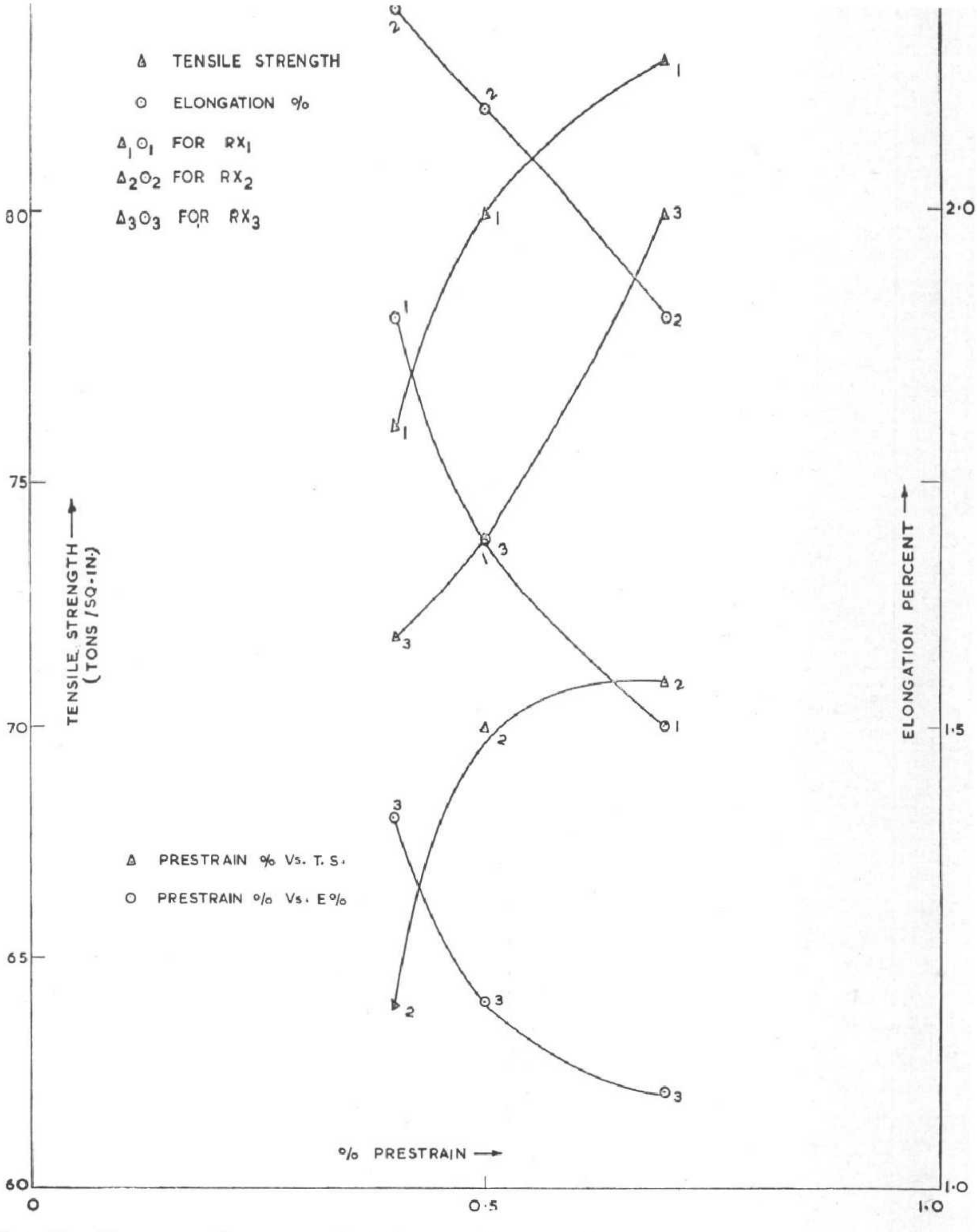


FIG. 17 — EFFECT OF DEGREE OF PRESTRAIN ON TENSILE STRENGTH AND DUCTILITY KEEPING THE RETEMPERING TEMPERATURE 450°C.

Table 14 — Composition of Steel DS_1 in Weight Per Cent

Steel	C, %	Mn, %	Si, %	Cr, %	V, %	Mo, %	W, %
DS_1	1·23	1·03	0·37	1·78	0·14	0·17	0·085

Table 15 — Results of Experiments done on Steel DS_1

Tempering temp. °C.	Tempering time				
	½ hr	1 hr	1½ hr	2 hr	2½ hr
150	836	830	830	815	796
200	823	810	802	790	784
300	700	685	675	666	660
350	650	648	639	631	629
400	615	610	602	595	580
450	548	550	552	548	547
500	—	490	—	480	—
550	—	383	—	374	—
600	—	362	—	358	353

Table 16 shows the hardness results obtained after various degree of deformation and subsequent tempering and sub-zero treatment.

(iii) *Bainitic Hardening of Tool Steel* — This project was undertaken with the view to heat treat high speed tool steels to a structure consisting wholly or partly of tempered bainite. It has been found that high speed steels consisting of almost entirely bainitic structures have satisfactory hardness for cutting tools.

The effect of holding period on the progress of bainite formation of 18/4/1 high speed steel at different transformation temperatures were studied. The hardness of the samples cooled in air and subsequently tempered for different periods and time are shown in Table 17.

(iv) *Isothermal Transformation Characteristics of Alloy Steels* — This project was taken up with a view to prepare an atlas of isothermal transformation diagram of Indian alloy steels, including substitute alloy steels under development at the National Metallurgical Laboratory.

Some of the isothermal transformation diagrams prepared from IS steels are shown in Figs. 18 to 20, and Table 18 gives the chemical composition of the steels.

(v) *Continuous-Cooling-Transformation of Alloy Steels* — The project was undertaken with a view to study the continuous-cooling-transformation characteristics of Indian alloy steels and to prepare an 'atlas' of the continuous-cooling-transformation diagrams for use in industry.

Table 16 — Hardness Values of Die Steel After Various Treatments

Percentage reduction	Hardness (VPN)									
	After Ausforming	After sub-zero treatment	Tempering temperature, °C.							
			350C°.		450°C.		550°C.		650°C.	
			Single tempering	Sbu-zero treatment	Single tempering	Sub-zero treatment	Single tempering	Sub-zero treatment	Single tempering	Sub-zero treatment
0	688	—	657	671	639	669	708	—	579	—
20	703	739	670	688	685	700	725	738	—	596
40	775	780	720	741	770	790	840	850	648	652
60	792	792	680	682	786	789	775	775	499	499

Table 17 — Hardness of Samples After Various Treatments at Different Transformation Temperatures and Holding Period

Hardness of specimens quenched in oil to room temperature from 1300°C.

850 VPN Tempered for 2 hr 835 VPN Tempered for (2+2) hr 815 VPN

Transformation temperature	Holding period	Approximate % of Bainite	Hardness VPN		Hardness VPN of samples after additional tempering at 565°C.			
			of samples cooled in air	of samples tempered at 565°C.	for 2 hours		for 2 hours	
					of samples cooled in air	of tempered specimens	of samples cooled in air	of tempered specimens
235°C.	10 min.	5	860	850	838	838	830	830
	30 min.	10	860	855	830	830	820	822
	1 hr	20	850	855	828	828	820	825
	4 hr	40	810	840	818	815	815	812
	8 hr	60	725	830	812	810	812	910
	16 hr	70	670	830	812	810	812	810
275°C.	10 min	5	865	860	830	830	815	818
	30 min	15	850	850	815	820	800	810
	1 hr	30	800	845	815	815	805	815
	4 hr	50	750	830	810	805	800	810
	8 hr	60	710	815	815	810	805	805
	16 hr	70	645	815	815	815	800	800
315°C.	10 min	10	860	865	850	855	830	835
	30 min	20	850	865	855	855	830	835
	1 hr	30	810	860	845	845	825	830
	4 hr	50	765	830	835	855	810	815
	8 hr	60	645	830	830	855	810	820
	16 hr	70	610	825	825	850	820	815

Experiments were conducted on steels of the compositions given in Table 19.

The diagrams of the continuous-cooling-transformation of the above steels are also furnished in Figs. 21 and 22.

(vi) *Development of Substitute Engineering Grade Alloy Steels* — In order to conserve some of the strategic alloying elements, like nickel and molybdenum used in the production of engineering steels for automotive and other industries, it was felt necessary to replace them by the indigenous and more economic materials. Work was, therefore, taken up to develop substitute steels of the Cr-Mn type which can be used in places of the conventional grades of steels.

Few preliminary heats of the hardenable and carburizing grades in the Cr-Mn-V and Cr-Mn series, respectively, were made in the 75 lb. capacity high frequency induction furnace. The analysis is given in Table 20. The forged bars from the hardenable grade of steel were solution treated and oil quenched from the austenitic range. The hardened specimens were tempered from 500-700°C. for different periods of time to study their tempering characteristics. The results are given in Fig. 23.

Metallographic and hardness data on some of the steels show a good agreement with the corresponding standard grades of steels. Figs. 24(a), (b), (c) and (d) show the progressive tempering behaviour of these steels. A systematic investigation of the tensile and impact properties of Mn steels is under progress. It is also proposed to study the mechanism of strengthening of quenched and tempered steels with the help of X-ray, electron diffraction and electron microscopy techniques.

TYPE OF STEEL							METHODS OF DETERMINATION	
IS 50 Cr I V 23							DILATOMETRIC & METALLOGRAPHIC	
ANALYSIS (WEIGHT %)							GRAIN SIZE	
C	Mn	Si	Ni	Cr	V	Mo	7	
0.51	0.72	0.27	0.15	0.95	0.21	0.04		
PREVIOUS TREATMENT							AUSTENITIZING CONDITION	
H. F. 70 LBS. INGOT 40" LBS. FORGING 0.5" SQ BAR ANNEALED.							TEMP.	TIME
							875 °C	30 MINS.

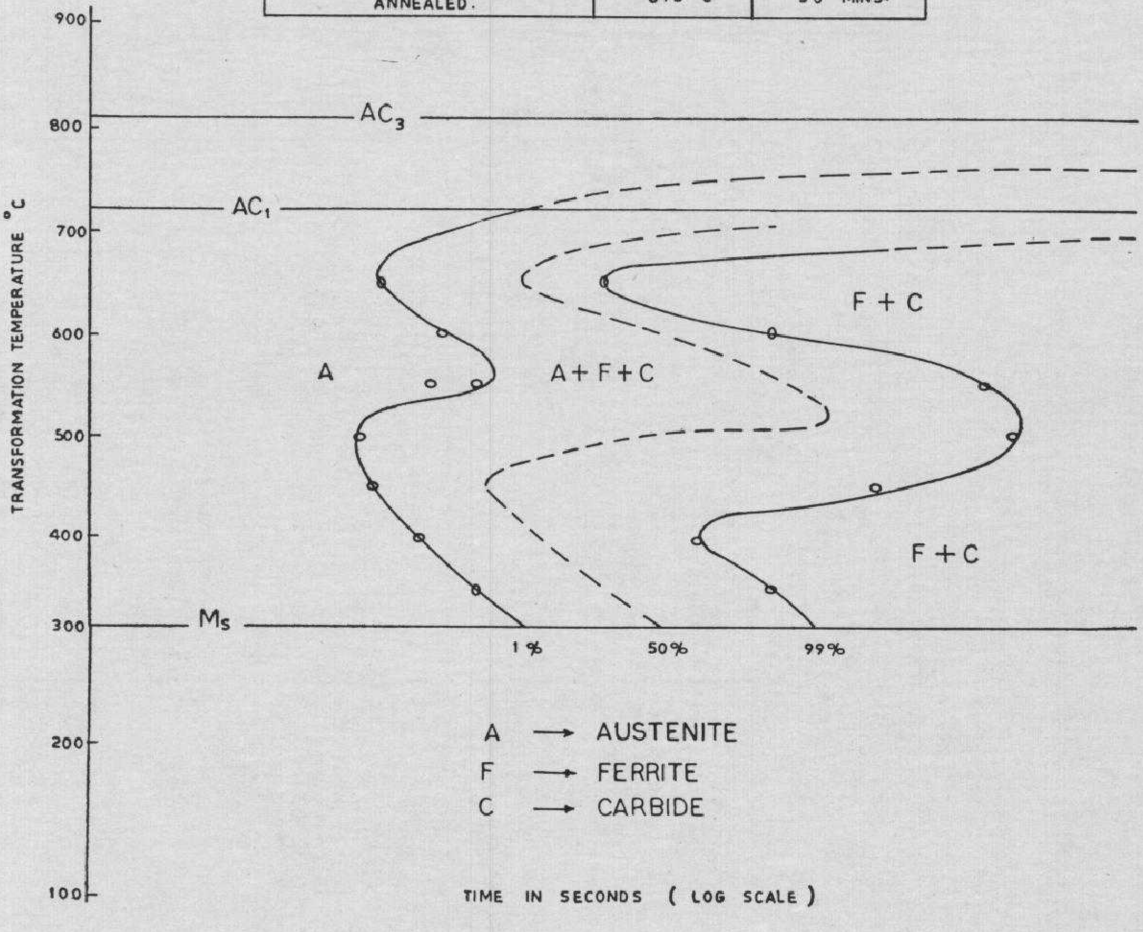


FIG. 18 — ISOTHERMAL TRANSFORMATION CURVE

39.0 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 through judicious modification in the percentages of alloying elements so that amount of peartite was reduced while

<table>
<tr><td colspan="7">TYPE OF STEEL</td><td colspan="2">METHODS OF DETERMINATION</td></tr>
<tr><td colspan="7">IS 40 Cr$_2$ AL 1 Mo 18</td><td colspan="2">DILATOMETRIC & METALLOGRAPHIC</td></tr>
<tr><td colspan="7">ANALYSIS (Wt %)</td><td colspan="2">GRAIN SIZE</td></tr>
<tr><td>C</td><td>Mn</td><td>Si</td><td>Cr</td><td>Mo</td><td>Al</td><td></td><td colspan="2" rowspan="2">7 – 8</td></tr>
<tr><td>41</td><td>57</td><td>–</td><td>1.57</td><td>.29</td><td>1.26</td><td></td></tr>
<tr><td colspan="7">PREVIOUS TREATMENT</td><td colspan="2">AUSTENITIZING CONDITION</td></tr>
<tr><td colspan="7" rowspan="2">H. F. 70 LBS
INGOT 40 LBS
FORGING 0.5" SQ. BAR
ANNEALED</td><td>TEMP.</td><td>TIME</td></tr>
<tr><td>930 °C</td><td>30 MINS</td></tr>
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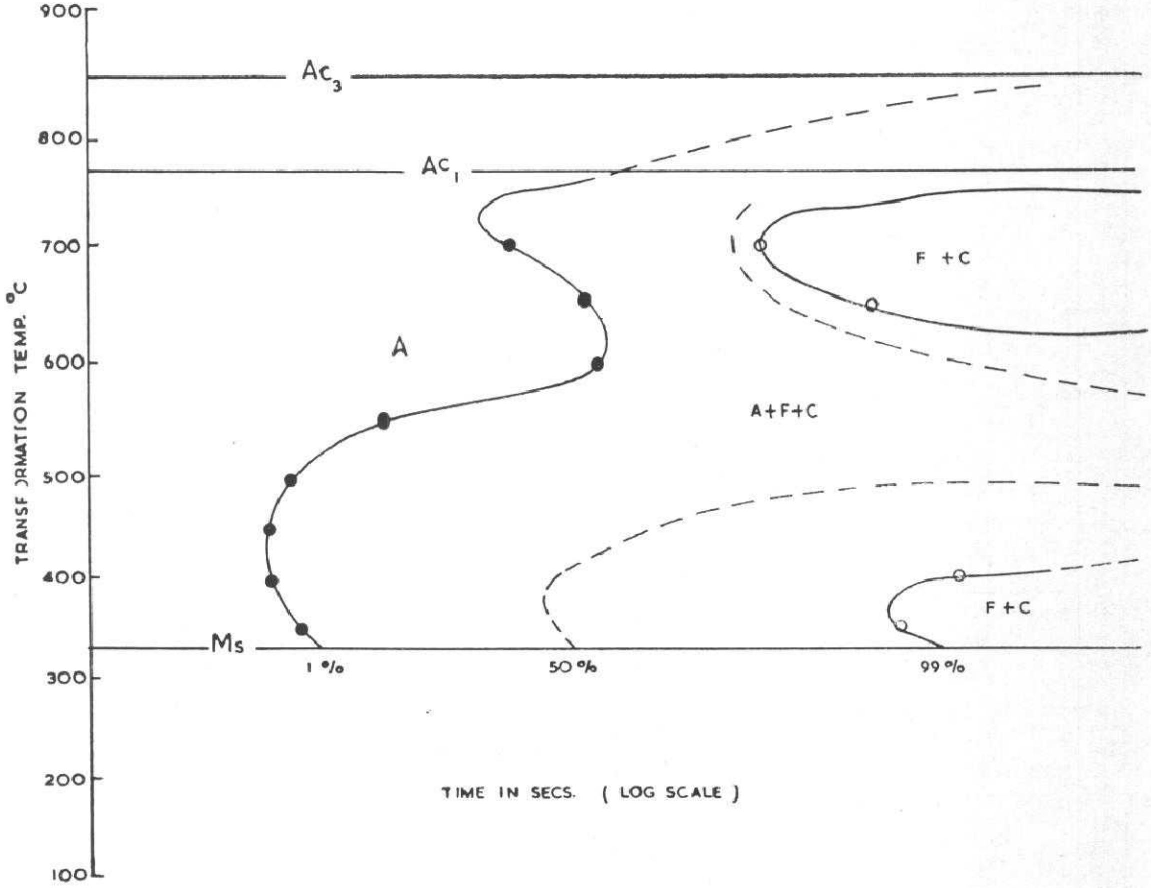
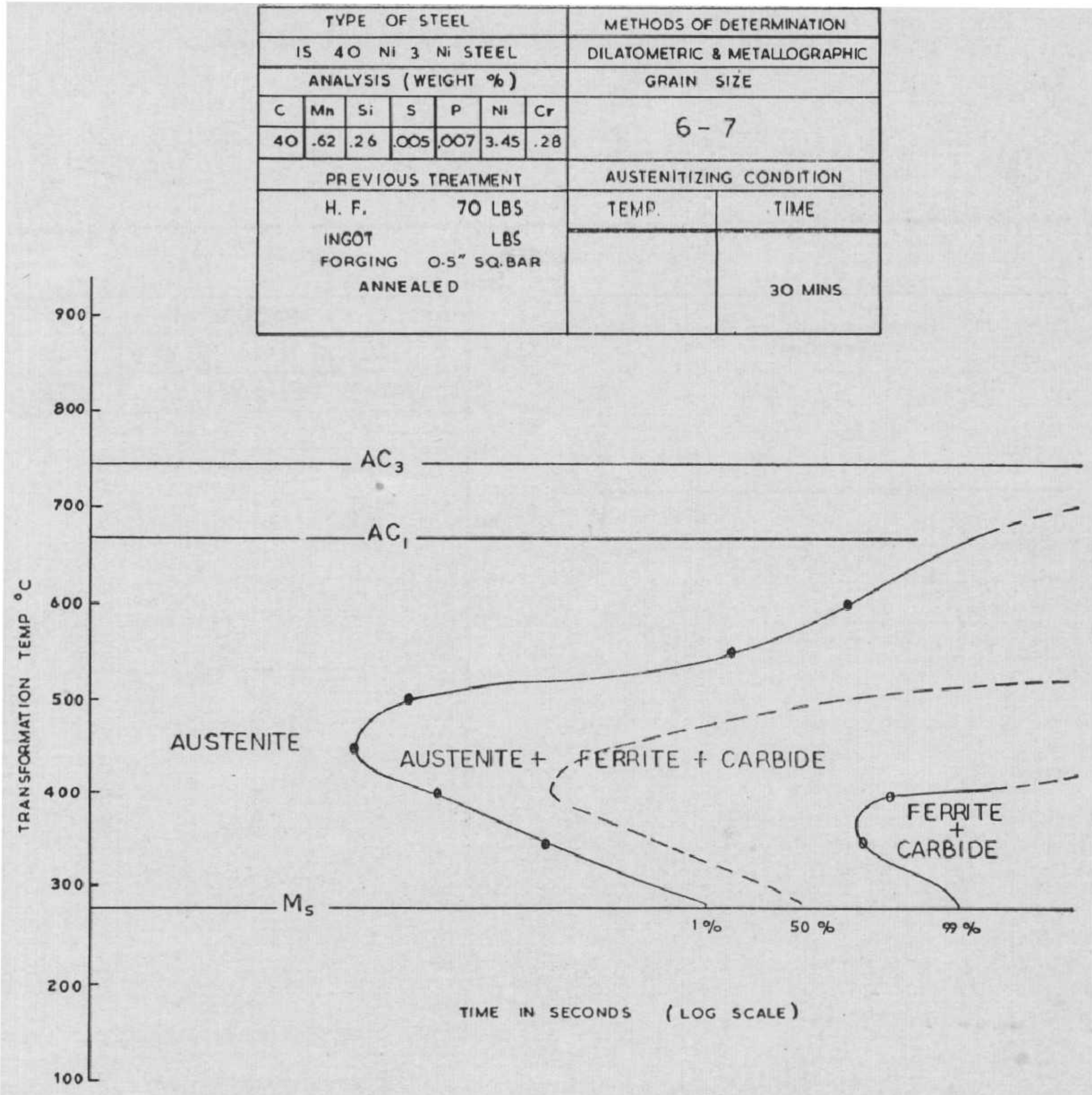


FIG. 19 — ISOTHERMAL TRANSFORMATION CURVE

strength was increased. Carbon, the principal factor in welding trouble, was maintained at a low level of about 0·15 per cent C. For improvement of the yield strength, elements such as aluminium, vanadium and titanium which act as grain refiners and in some cases give rise to precipitate dispersion hardening, were added to C-Mn steel. Copper of the order of 0·4 per cent was added to most of the steels to impart corrosion resistance. Various properties, such as tensile, impact resistance, weldability and corrosion resistance of most of the steels have been determined.

During the pericd under review, the grain refining effect of vanadium on C-Mn steel



TYPE OF STEEL	METHODS OF DETERMINATION
IS 40 Ni 3 Ni STEEL	DILATOMETRIC & METALLOGRAPHIC

ANALYSIS (WEIGHT %)

C	Mn	Si	S	P	Ni	Cr
40	.62	.26	.005	.007	3.45	.28

GRAIN SIZE
6 – 7

PREVIOUS TREATMENT	
H. F.	70 LBS
INGOT	LBS
FORGING	0.5" SQ. BAR
ANNEALED	

AUSTENITIZING CONDITION	
TEMP.	TIME
	30 MINS

FIG. 20 — ISOTHERMAL TRANSFORMATION CURVE

with 0·4 per cent copper was studied. It is known that the tensile strength and yield point of hot rolled C-Mn steels are markedly improved when alloyed with vanadium of the order of 0·1 per cent. Normalizing treatments again improve the impact strength and ductility without much changing the yield and tensile strength. It is also reported that vanadium carbide is an effective grain restrainer up to 1000°C. and this temperature is sufficient for all the vanadium carbide to go to the solution. Hence, the mechanical properties of the vanadium bearing steels referred to above were determined from the normalized bars.

Four different temperatures, e.g. 920°, 960°, 1000° and 1050°C. were selected for giving the normalizing treatments. In the present phase, work was carried out with the bars normalized from 920°, 1000° and

Table 18 — Composition of Steels in Weight Per Cent

C, %	Mn, %	Si, %	Ni, %	Cr, %	Mo, %	V, %	Al, %
0·51	0·72	0·27	0·15	0·95	0·04	0·12	—
0·41	0·57	—	—	1·57	0·29	—	1·26
0·40	0·62	0·26	3·45	0·28	—	—	—

Table 19 — Compositions of Steels in Weight Per Cent

C, %	Mn, %	Si, %	Ni, %	Cr, %	Mo, %
0·18	0·76	0·24	1·05	0·83	0·07
0·17	0·82	0·23	0·86	0·71	0·06

ANALYSIS (WEIGHT %)					
C	Mn	Si	Ni	Cr	Mo
0·18	0.76	0.24	1.05	0.83	0.07

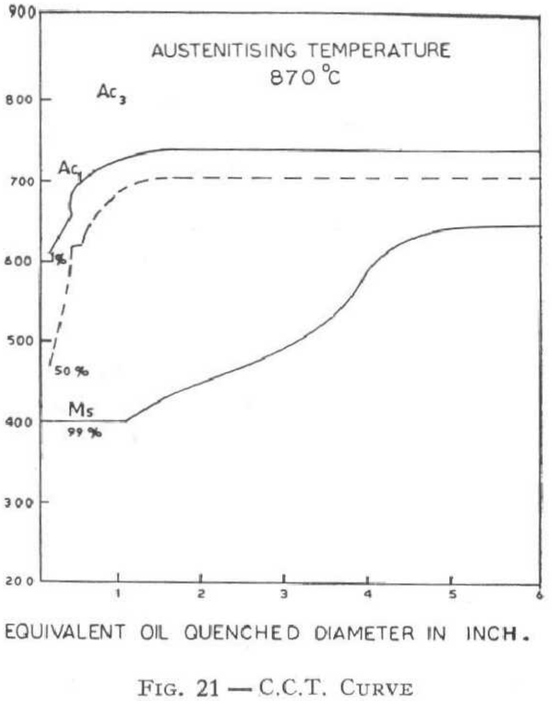


FIG. 21 — C.C.T. CURVE

1050°C. The grain size of the normalized pieces were determined by linear intercept method. From Table 21 it may be seen that the grain diameter increased with higher normalizing temperature and appreciable increase in size of the grains were observed from 1050°C.

From the hardness measurements of these normalized pieces, it may be noted that there is a trend of gradual increase in the hardness values with higher normalizing temperature up to 1050°C. This is probably due to solution hardening effect of vanadium. This has been verified by measuring hardness on ferrite grains. Yield and tensile strength were markedly improved when normalized from 1000°C. Elongation percentage, in other words, ductility remained

ANALYSIS (WEIGHT %)					
C	Mn	Si	Ni	Cr	Mo
0.17	0.82	0·23	0.86	0.71	0.06

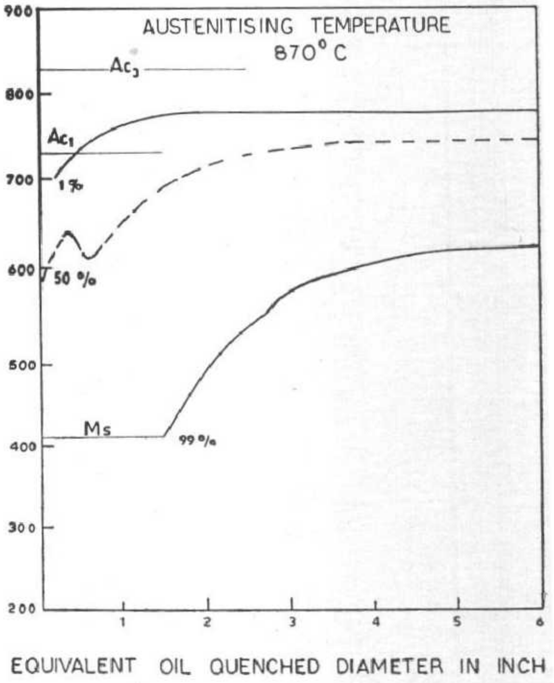


FIG. 22 — C.C.T. CURVE

Table 20 — Analyses of Heats

Sl No.	C, %	Si, %	Cr, %	Mo, %	Ni, %	Mn, %	N, %	V, %	S, %	P, %
H_1	0·24	0·31	3·20	0·52	0·40	0·65	—	—	0·050	0·048
H_2	0·20	0·34	1·50	0·50	—	2·00	0·05	0·15	0·037	0·032
H_3	0·25	0·15	1·51	0·41	—	1·87	0·02	0·15	0·010	0·028
C_1	0·19	0·27	—	0·23	1·70	0·56	—	—	0·042	0·039
C_3	0·25	0·19	—	0·30	—	1·50	—	—	0·026	0·031
C_3	0·15	0·12	—	0·34	—	1·27	—	—	0·012	0·029

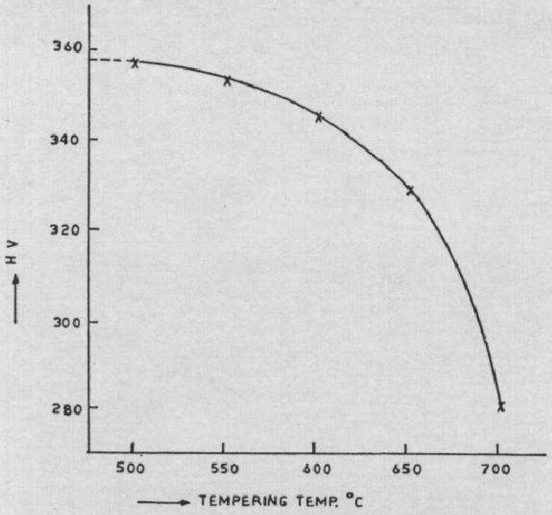


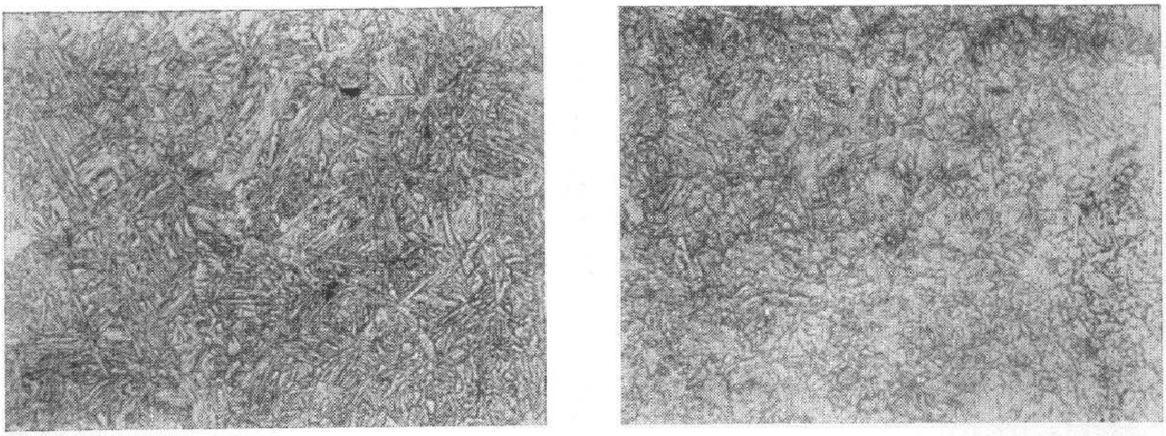
FIG. 23 — HARDNESS/TEMPERING TEMPERATURE CURVE FOR STEEL H_3

practically unchanged. Impact resistance of all the steels tested was found to be much lower both at room temperature and sub-zero temperature when normalized from 1000°C. than those normalized from 920°C. This may be due to grain coarsening effect of high temperature normalizing. However, to make a compromise between yield strength and impact value, the normalizing temperature has been kept between 920° and 1000°C. Further work in this line is in progress.

40.0 Improved Mild Steel for Structural Purpose

The object of the investigation is to improve the properties of mild steels by adding residual quantities of alloying elements which while contributing little to the increase in the cost of production would impart pronounced strengthening effect. With this purpose in view additions of niobium, vanadium, titanium and nitrogen were made in minute quantities to low carbon steel base. Manganese was added up to 1·5 per cent to some of the steels.

Work was carried out to establish the mechanism of strengthening of Nb-V-N steels which showed encouraging mechanical properties in a previous investigation. Work was extended to the evaluation of properties of Ti-Nb steels. In order to determine the structural constituents producing the strengthening effect in Nb-V-N steels, electron microscopic examination was carried out on a C, 0·1; Mn, 1·5; Nb, 0·06 and V, 0·02 per cent and 0·0085 N steel. Carbon extraction replicas were prepared and examined [Fig. 25(a) & (b)]. Both coarse and finely dispersed precipitates were observed. Selected area electron diffraction studies were carried out on the extraction replicas and the d-values of the strong lines were determined. The values are presented in Table 22 along with the lattice spacing data for NbC, VC and VN from A.S.T.M. index. As NbC, NbN, VC and VN are isomorphous with each other and have very close lattice parameter values, it was not possible to identify them individually by selected area electron diffraction technique. However, the electron diffraction values obtained indicate the precipitated phase to be VC.



(a) As quenched, 1050°C.-20 min.

(c) As quenched, tempered at 600°C.

(b) As quenched, tempered at 500°C.

(d) As quenched, tempered at 650°C.

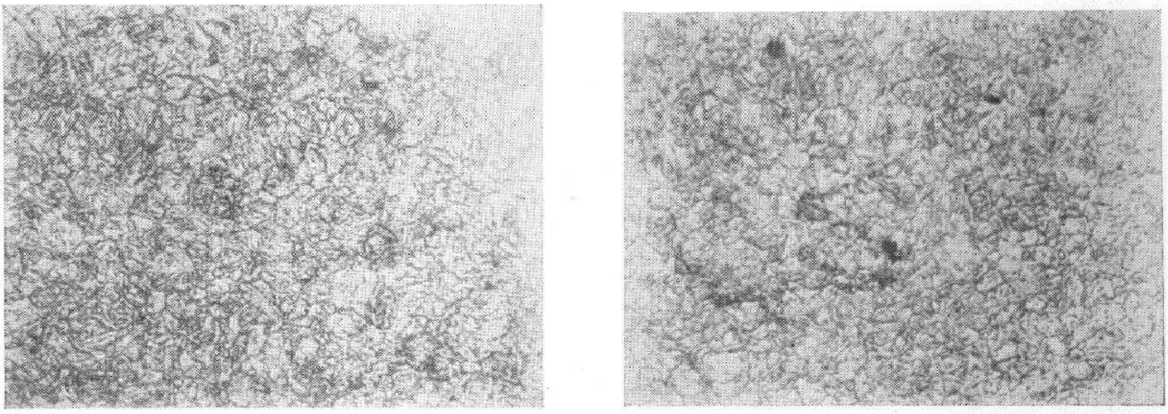


FIG. 24 — PHOTOMICROGRAPHS OF THE STEEL SPECIMEN SHOWING THE PROGRESSIVE TEMPERING BEHAVIOUR

Investigations on Ti-Mn, Ti-Nb and Ti-Nb-Mn steels were carried on heats of compositions shown in Table 23.

Tensile and impact properties of these steels were determined after normalizing at temperatures of 900°, 950°, 1000° and 1050°C. Results of tensile tests indicated a small increase in strength of the TiNb and TiMn steels and a pronounced strengthening of Ti-Nb-Mn steel with increasing normalizing temperature. Impact tests were carried out on V notch charpy specimens at room and sub-zero temperatures. The results which are plotted in Fig. 26 indicate in general an increase of tough-ductile transition temperature as the normalizing temperature increased. Of these three heats investigated the Nb-Ti-Mn steel showed the best combination of strength and impact toughness properties.

The future programme of work includes investigations on the effects of heat treatment variables and controlled rolling on the properties of low carbon steels containing residual quantities of alloying elements.

Table 21 — Properties of Heats After Normalizing Treatment at Different Temperatures

Heat No.	Treatment	Grain size ×10⁻⁴ cm.	Hardness V.P.N.	Micro-hardness on ferrite V.P.N.	Yield strength T.S.I.	U.T.S. T.S.I.	Percentage elongation	Charpy impact values in ft. lb.				
								R.T.	°C.	−20°C.	−40°C.	−60°C.
41/3R	Norm. 920°C.	—	197	—	33·6	36·8	28	42	28	24	35	—
	,, 1000°C.	12·75	214	148	35·6	46·08	28	30	22	20	4	—
	,, 1050°C.	17·70	226	177	—	—	—	—	—	—	—	—
41/4R	,, 920°C.	—	186	—	27·7	38·16	34·4	104	86	104	92	40
	,, 1000°C.	10·25	206	157	33·6	43·25	26·6	80	42	46	—	—
	,, 1050°C.	14·08	236	160	—	—	—	23	14	—	—	—
41/3B	,, 920°C.	—	178	—	26·4	37·88	34	170	110	186	46	6
	,, 1000°C.	12·4	194	137	33·6	44·68	33	44	22	—	10	8
	,, 1050°C.	15·1	206	144	—	—	—	—	—	—	—	—
41/3A	,, 920°C.	—	166	—	25·7	37·02	37·5	—	—	—	—	—
	,, 1000°C.	8·8	175	137	30·03	38·60	34·3	135	126	—	100	108
	,, 1050°C.	13·0	195	142	—	—	—	—	—	—	—	—

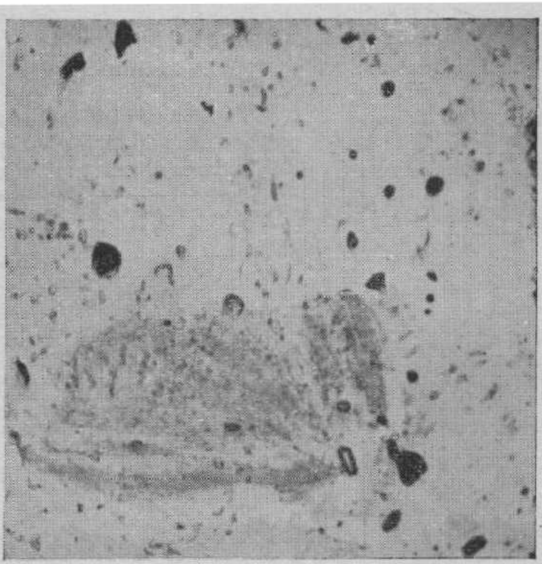


FIG. 25(a) — CARBON EXTRACTION REPLICA NORMALIZED AT 950°C. ×16,000

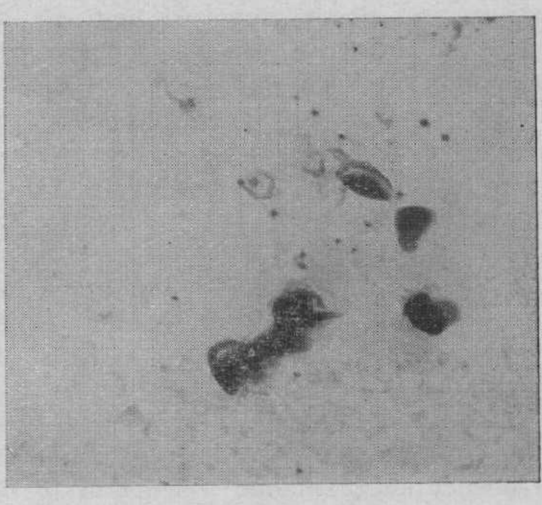


FIG. 25(b) — CARBON EXTRACTION REPLICA NORMALIZED AT 950°C. ×25,000

41.0 Development of Electrical Resistance Alloys for Heating Elements

The object of the project is to develop electrical resistance alloys with alloying elements available from indigenous resources, since the commonly used heating elements of the nichrome or kanthal

Table 22 — Results of Selected Area Electron Diffraction

Experimental d values of the strong lines A units		Corresponding d values of the different composition A unit		
Specimen 1	Specimen 2	VC	NbC/Nb_4C_3	VN
2·068	2·067	2·07	2·220	2·140
1·474	1·471	1·47	1·339	1·110
0·935	0·931	0·93	0·993	0·937
0·843	0·849	0·85	0·906	0·853

Table 23 — Chemical Composition

Heat No.	C, %	Mn, %	Si, %	Nb, %	Ti, %
6	0·14	0·39	0·063	0·020	0·039
7	0·10	1·41	0·061	0·034	0·052
8	0·10	1·53	0·068	Nil	0·047

group are mostly imported and contain large proportion of nickel and cobalt, the resources of which are lacking in India.

The major part of the development work on electrical resistance alloys starting from alloy composition to final production of wires was completed and the process was released for industrial production. During the period under review, the work was mainly conducted to study the welding characteristics of the alloy developed on resistance butt welds of wire samples. Welding of Fe-Cr-Al alloys offers great difficulty due to excessive grain coarsening tendency and oxidation which lead to the formation of weak, brittle zone resulting in failure of the joint during wire-drawing operation. For the purpose, a standard micro-resistance butt welding machine was employed. As reported in previous Annual Report, the welded zone of wire sample showed excessive grain boundary oxidation. To avoid this, welding was carried out in protected atmosphere. Several wires welded with tapered ends under optimum condition of current and pressure was found to be quite ductile and successfully drawn from 13 S.W.G. to 19 S.W.G.

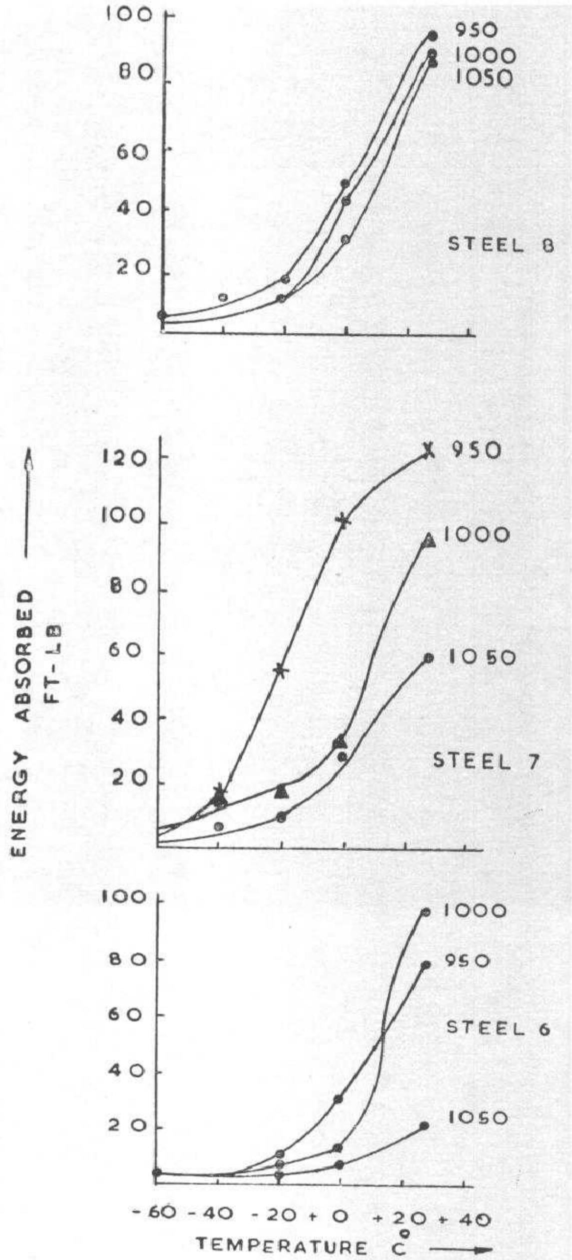


FIG. 26 — THE EFFECT OF NORMALIZING TEMPERATURE ON IMPACT TRANSITION CHARACTERISTICS OF STEELS 6, 7 AND 8. FIGURES INDICATE NORMALIZING TEMPERATURE IN °C.

From the micro-structures of the wire samples (Figs. 27a, 27b & 27c) welded under optimum condition with various tapered lengths it can be seen, the weld zone in case of $\frac{1}{16}$ in. tapered length was free from grain boundary voids and surrounded

FIG. 27(a) — BUTT WELDED WIRE SAMPLE HAVING $\frac{1}{16}''$ TAPERED LENGTH

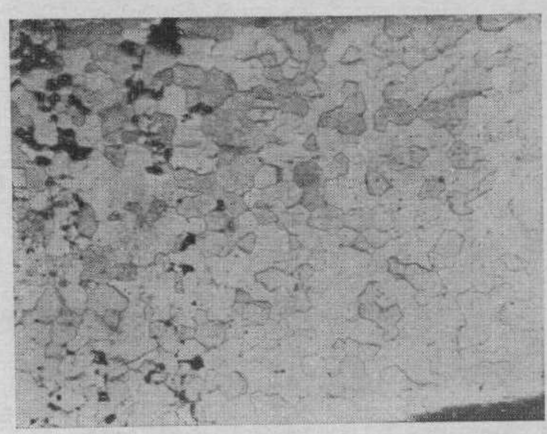


FIG. 27(b) — BUTT WELDED WIRE SAMPLE HAVING $\frac{1}{32}''$ TAPERED LENGTH

FIG. 27(c) — BUTT WELDED WIRE SAMPLE HAVING $\frac{1}{8}''$ TAPERED LENGTH

with more uniform grains which are desirable features for a good weld. The microstructures of the other two samples ($\frac{1}{32}$ and $\frac{1}{8}$ in. tapered lengths) revealed the welded zone, consists of non-uniform distribution of grains and some grain boundary defects. Further experiment in welding is being carried out with wires of large diameter.

With a view to study the effect of trace addition of misch metal with or without Zr on the working and physical properties of the alloys developed, some new heats were prepared in high frequency furnace. Assessment of various physical and mechanical properties of the heats is being made to evaluate the overall effect of controlled addition of one or more than one trace elements.

41.1 Development of Manganese Bearing Brasses

The use of brass utensils in Indian household has been a common practice for centuries. With the limited production of copper and the total dependence on foreign import for zinc, the problem was taken up to substitute the zinc in brass with the abundantly available manganese in India.

Corrosion resistance properties of binary and ternary Cu-Mn and Cu-Mn-Zn alloys were determined in organic acids, inorganic acids, alkalies and brine solutions of different concentrations in the range of 5-50 per cent by weight. The tests were carried out at 40°C. for 72 hours. The results indicated that Cu, 30 per cent Zn brass and alloys containing manganese have more or less similar corrosion rates in some media specially at higher concentrations but in others, e.g., alkali and sodium chloride, the corrosion rate of Cu, 30 per cent Zn is higher. Further work is in progress to determine the role of manganese in the alloy on the corrosion rate by selecting wider composition range of alloys.

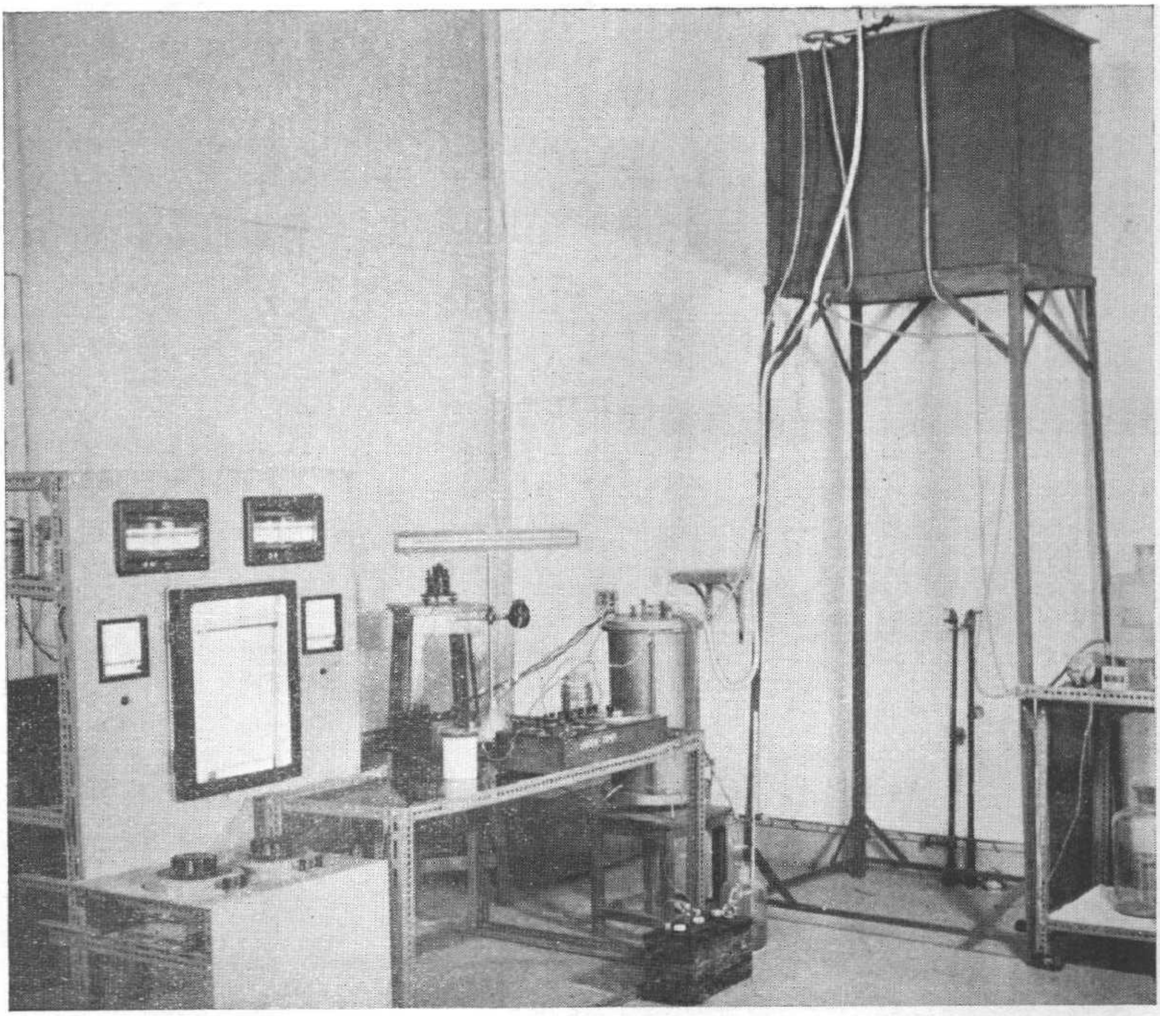


FIG. 28 — SETI-UP FOR THERMAL CONDUCTIVITY MEASUREMENT AT HIGH TEMPERATURE

The thermal and electrical conductivity of the manganese bearing brasses were studied up to a temperature of 100°C. In order to extend the region of studies to higher temperatures up to 600°C. an apparatus based on the steady-state heat flow method to measure the thermal conductivity was fabricated. The apparatus consists of a heater block made of copper to which is attached the test specimen of about 25 cm. in length. To the other end of the test specimen a cooling head also made of copper is fixed. Provision has been made in the cooling head to allow water to circulate completely round the specimen. The heat input at the hot end is thus taken away by the cooling block. After steady-state is achieved the temperature gradient along the test specimen is measured using a potentiometer. A guard-ring made of stainless steel has been provided to reduce the loss of heat due to radiation from the specimen. The insulated space between the specimen and the guard ring is flushed and filled with N_2 in order to reduce the oxidation of the test specimen at high temperatures. Fig. 28 shows the experimental set-up for the studies. Preliminary experiments for standardization are under progress.

42.0 Development of Magnetic Materials

(i) *Effect of Thermomagnetic Treatment on the Magnetic Properties of Some Alnico Type Magnetic Alloys* — Alnico alloys containing 23-24 per cent cobalt develop high magnetic strength on controlled cooling in magnetic field from solution temperature (1250/1300°C.) to 600°C. field. The recommended rate of cooling is approximately 0·5-5°C./sec. The important feature of this heat treatment is to cool the alloy quickly in the temperature range 1180-930°C. to suppress α-γ transformation. Since only the alpha phase is important for the development of magnetic strength the possibilities of adopting solution temperature in low temperature alpha range 900-930°C. were explored. Consequently thermomagnetic treatment was applied from 900°C., near the curie point when the alloy is in the α state and results compared with specimens solution treated at ∼ 1250°C.

The alloy studied contained Fe-Co, 23·2; Ni, 16·3; Cu, 3·1; Al, 8·6 per cent. The properties were determined by ballistic method and are given in Table 24 after various treatments in a magnetic field of 2500 oersteds and tempering for various durations at different temperatures. The results show that though the high temperature solution treatment gives better properties as compared to the low temperature solution treatment, later treatment gives high magnetic strength provided the alloy during casting cools very rapidly suppressing α-γ transformation.

Experiments were taken up to study the effect of trace additions of silicon, sulphur, zirconium, titanium, niobium, etc., on the critical cooling rate and magnetic properties of these alloys. It is envisaged to take up work on growing of crystals during casting with their easy direction of magnetization lying in the longitudinal direction of the mould.

(ii) *Effect of Low Melting Point Oxides on Magnetic Properties of Hard Ferrites* — During the period under review magnetic properties of strontium ferrite with and without addition of lead oxide were studied. A number of compositions were made in which lead oxide was added in increasing amounts and these were first calcined at high temperature and pulverized to fine powder by ball milling. The fine powder was compacted into tablets under a load

Table 24 — Magnetic Properties of Alnico Type Alloys

Heat treatment	Coercive force	Remanence	Energy product
	Hc oersted.	Br Gauss	BH max Mega-gauss oersted
Specimens originally cooled rapidly from 1250°C. to room temperature and then solution treated at 1250°C. and cooled to 600°C. in a magnetic field at 0·5°C./sec. and subsequently tempered at 650°C. for 6 hr, 560°C. for 10 hr	695	12900	5·7
After furnace cooling from 1250°C. to room temperature heated to 1250°C. and cooled to 600°C. at 0·5°C./sec. and tempering as above	590	11900	3·4
Specimens cooled from 1250°C. rapidly and then heated to 900°C. and cooled to 600°C. at 0·5°C./sec. and tempered as above	660	12500	4·9
Furnace cooled from 1250°C. heated to 900°C. and cooled to 600°C. in 0·5°C./sec. and tempered as above	540	11975	2·40

Table 25 — Magnetic Properties of Sintered Strontium Ferrite at Different Temperatures

Composition No.	1080°C. Hc Oersted	1080°C. Br Remanence	1180°C. Hc	1180°C. Br	1238°C. Hc	1238°C. Br	1280°C. Hc	1280°C. Br
SrO	1460	1820	1640	2250	1640	2280	1110	2060
Sr II	1180	1630	1200	1900	1380	2000	1040	2160
Sr III	1034	1720	890	1900	200	1920	—	—
Sr IV	1200	2040	920	1690	565	1230	—	—
Sr V	1150	2300	855	1740	420	1180	—	—

of 5 tons. The compacted tablets were then sintered at different temperatures varying from 1080° to 1280°C. and results are given in Table 25.

The shrinkage of the compacts after sintering are given in Table 26.

The magnetic properties and shrinkage results are being related with the structural changes which are under investigation by X-ray diffraction technique.

(iii) *Study of Electrical and Magnetic Properties of Some Low Manganese Low Aluminium Steels* — During the period under review two more heats containing Al, 3·8; Mn, 1·4; and Al, 5·7 and Mn, 1·5 per cent were made using high frequency induction furnace. The five pound ingots were hot forged and rolled at 1050°C. to 0·1 in. thick strips from which cylindrical rod specimens of 0·08 in dia. were made. These rods were annealed in vacuum for ninety hours at 1100-1200°C. and were then tested ballistically for their magnetization curve and hysteries loop. The maximum permeability of these rods were $\sim$ 11000 with coercive force about 0·5 and 0·36 oersted respectively. The electrical resistivity of these sheets was 60 and 64 micro ohm cm. respectively.

The steel containing 3·83 per cent Al was also cold rolled and its work hardening capacity studied. It was found that in the annealed condition the steel had hardness of about 140 VPN and the hardness increased to about 250 VPN when cold reduced to 90 per cent. Magnetic properties were also studied after cold rolling 0·08 in thick sheets to 60 per cent, and annealing to 900°C. for 30 sec., 1 minute, 5 minutes, 30 minutes and 1 hour. These annealed strips were then reduced to about 50 per cent and vacuum annealed at 1100°-1200°C. for 2-6 hours. It was found that after the first annealing treatment (900°C.) permeability 5000-7000 could be developed. On final annealing the permeability did not show any increase. The detailed study of properties after cold reduction and annealing are in progress.

Table 26 — Shrinkage Values at Various Temperatures

	1080°C.	1180°C.	1238°C.	1280°C.
SrO	9·54	21·025	25·25	27·34
Sr II	8·65 (1120°C.)	28·06	28·66	33·47
Sr III	9·92	23·21	29·9	29·73
Sr IV	25·61	27·92	26·35	29·74
Sr V	30·86	25·50	22·40	21·98

43.0 Light Metals and Their Alloys

(i) *Aluminium-Magnesium Alloys*—In continuation of the work done on aluminium-magnesium alloys in previous years, further experiments were carried out to cast ingots with a large section ($4 \times 1\frac{3}{4} \times 24$ in.) from aluminium 7-9 per cent Mg alloys. In earlier experiments, misch metal (containing 51 per cent Ce) was added to the melt for introducing cerium in the alloy and it was established that with the addition of misch metal, aluminium alloys containing higher percentages of magnesium could be made workable. In the present series of experiments a mixture of rare earth fluorides, obtained as a huge product during the processing of monazite sands by Indian Rare Earths Ltd was used to ascertain its suitability as a melting flux and for determination of cerium pick up. The cerium pick up was found to be encouraging and the mixture was quite suitable as a flux.

Attempts were made to cold reduce the slabs after initial hammer forging and dressing, in a four high rolling mill. After a few passes, the slabs did not take any further reduction. On the other hand superficial cracks appeared on the slab which were not deep seated in most of the cases. The slabs were found to have become work hardened and unsuitable for further cold reduction. Work is however, continued in this direction before arriving at a final conclusion.

Stress corrosion studies were carried out on Al, 8; Mg, 3 per cent misch metal alloy. Sheets, obtained by hot rolling down to a thickness of 0·125 in., were cold rolled to 0·050 in. thickness. These sheets were cut to the required size for making specimens for stress corrosion study. These specimens were mounted under stress on perspex stands; three specimens, which were given the same heat treatment, being fixed on each mount. The specimens were placed inside desiccators and subjected to a corrosive medium of synthetic sea water (NaCl, 26·5 g/l, KCl, 0·73 g/l, $MgCl_2$, 2·4 g/l, $CaCl_2$, 1·1 g/l, NaBr, 0·28 g/l, $MgSo_4$, 3·3 g/l, $NaHCO_3$, 0·20 g/l). Besides keeping water of the same composition in a bowl at the bottom of the desiccator, the specimens were moistened with it every day. Some specimens in the cold-rolled condition were only negligibly corroded even after two years.

Specimens were also subjected to similar corrosive attack (i) after solution treatment at 435°C., and (ii) after solution treatment followed by ageing at 150°C. for 50 hours, 100 hours, 150 hours and 1300 hours. Specimens aged for 150 hours and 300 hours showed foliation corrosion (Fig. 29a) all other specimens suffered only negligible corrosion even after two years (Fig. 29b).

Further stress corrosion studies of these alloys are being continued.

(ii) *Oxidation Studies on Aluminium and Aluminium Alloys by Electron Diffraction* — Investigations were undertaken mainly with a view to study the characteristics of the thin oxide layers formed on the surfaces of metallic aluminium and duralumin at elevated temperatures. The specimens were examined in a Finch type electron diffraction camera unit, by the reflection method.

Metallic aluminium having a purity of 99·9 per cent was used for preparation of samples. Suitable samples were prepared from remelted aluminium and machined in the form of small circular discs. These discs were polished mechanically and electrolytically etched in NaOH solution and electron diffraction patterns were taken. (Fig. 30). After taking the diffraction pattern the sample was placed in an oven maintained at a temperature of 200°C. It was noted that the initial pattern became gradually fainter and finally disappeared after about 286 hours of oxidation. Patterns of super purity aluminium were also taken using different etchants such as HF and NaOH (at room and elevated temperatures) to study whether the patterns obtained were characteristic of the etchant used or were truly representative of the surface under examination. It was observed that mild etching did not change the diffraction pattern.

Samples of duralumin were prepared from an extruded rod. The mechanically polished samples were vacuum sealed in

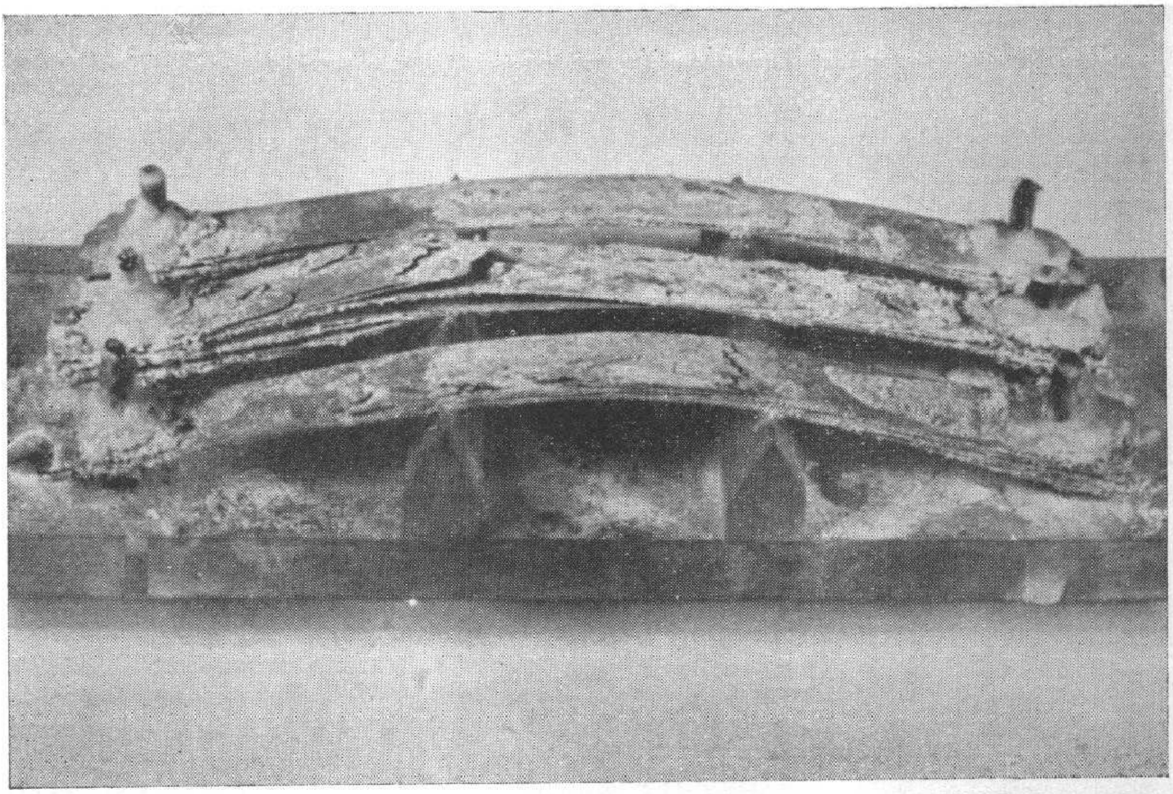


FIG. 29(a) — AL-Mg ALLOY AGED FOR 300 HR SHOWING EXFOLIATION CORROSION

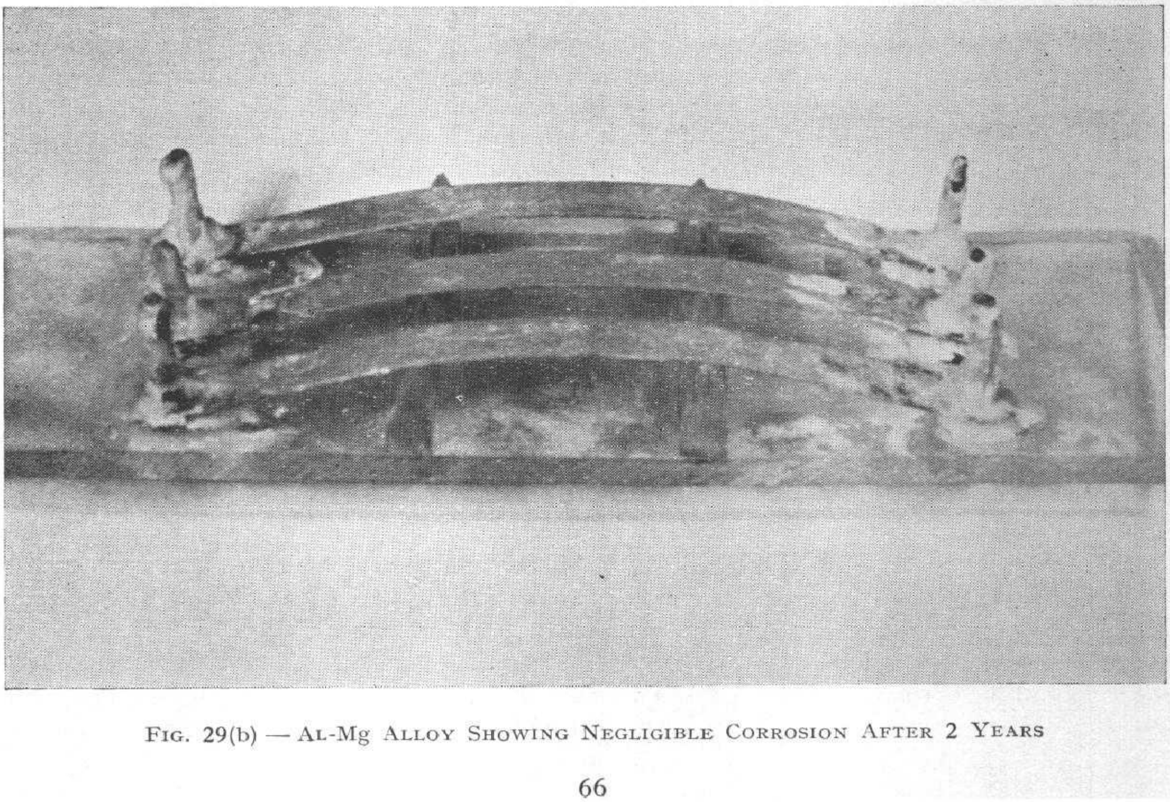


FIG. 29(b) — AL-Mg ALLOY SHOWING NEGLIGIBLE CORROSION AFTER 2 YEARS

FIG. 30 — ELECTRON DIFFRACTION PATTERN OF ELECTROPOLISHED POLYCRYSTALLINE, SUPERPURITY ALUMINIUM

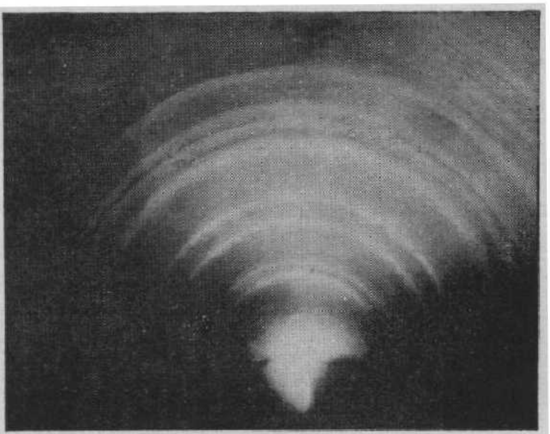


FIG. 31 — ELECTRON DIFFRACTION PATTERN FROM ELECTROPOLISHED EXTRUDED DURALUMIN

a pyrex glass tube, solution treated at 500°C. for one hour and then quenched in water at room temperature. These samples were then polished electrolytically and etched in 2 per cent NaOH solution and diffraction pattern was taken (Fig. 31), and caliberated by taking a mixed pattern with graphite. During oxidation at 200°C. the original pattern disappeared totally after 316 hours. Oxidation at 200°C., even for 1000 hours, did not produce any other pattern, indicating thereby that the amorphous oxide layer formed on the surface of the sample was non-porous, which did not permit the diffusion of aluminium and oxygen ions through it, thus preventing further oxidation.

(iii) *Preparation of Single Crystal of Aluminium* — Single crystals of aluminium were made by strain/anneal method. A $\frac{1}{4}$ in. thick slab of aluminium (purity 99·9 per cent) was cold rolled to about $\frac{1}{16}$ in. thick sheet. This sheet was made into pieces $2\frac{1}{4}$ in. by $\frac{1}{2}$ in., cleaned in aqua-regia, polished electrolytically and then vacuum annealed at about 505°C. for 15 hours. The annealed specimens were then strained by tension in a Amsler Tensile Testing machine. These lightly strained specimens were then given a second annealing treatment usually under the same conditions as the first one. The specimens were then electropolished and etched in HF and examined in an electron diffraction camera. The diffraction patterns obtained showed a spot pattern typical of a single crystal of aluminium along with some rings of polycrystalline grains also (Fig. 32). It appears that fairly large sized grains of single crystal aluminium could be obtained by this method. Further work on these

FIG. 32 — ELECTRON DIFFRACTION PATTERN FROM ALUMINIUM CRYSTAL PREPARED BY STRAIN/ANNEAL METHOD

lines will extend to the preparation of single crystals of aluminium-magnesium alloy intermetallic compounds.

44.0 Development of Iron-Aluminium Alloys

The project was taken up to develop iron-aluminium alloys comparable to stainless steel in heat and corrosion resistance.

In previous years, work was done on the binary alloys with minor alloying additions. During the period under review effect of larger amounts of alloying additions was taken up for study. Iron-aluminium alloys were made with addition of varying amounts of titanium with a view to improve their mechanical properties. Heats were made in a 6 kW high frequency furnace using magnesite crucible. Titanium additions were made to molten low carbon iron just before pouring. Ingots were buried in vermiculite for slow-cooling to prevent cracking. The mechanical working was carried out at 1000°C. by impact forging. The hardness of as cast and as forged alloys was taken and it was observed that the addition of titanium from 0·5 to 2·0 per cent to iron-aluminium alloys containing 8·0 to 11·0 per cent aluminium increases their hardness.

Table 27 gives the analysis of heats and their hardness in both as cast and as forged condition.

The as-cast and as-forged structure of Fe-Al-Ti alloys were studied after etching in ferric chloride solution. Their microstructure shows fewer oxide inclusions and finer grain structure as compared to binary alloys. Arrangements are being made for tensile and corrosion resistance tests. Similar studies with the addition of vanadium and zirconium to the iron-aluminium alloys are being made.

Table 27 — Composition and Hardness of Heats

No. of heats	Al, %	Ti, %	As cast hardness VPN	As forged hardness VPN
1	9·20	0·67	223	246
2	11·24	1·27	337	348
3	Results awaited		385	391
4	,,	,,	389	418

45.0 Development of Clad Bearing Metals

The investigation was taken up with a view to develop a suitable technique to bond dissimilar metals by rolling so as to obtain a composite product with properties which cannot be obtained by using single metal. The final objective was to utilize the technique to production of different engineering composites.

The detailed process developed for cladding aluminium on steel was reported earlier. Further work was carried out to utilize the same technique for cladding aluminium base bearing alloys on mild steel so as to obtain a bimetallic bearing which will give both strength as well as low frictional properties required for a good bearing.

Work was carried out in the first instance to develop a suitable aluminium base bearing alloy possessing good frictional properties. In the next phase the process for bonding of bearing alloy with mild steel by roll cladding was worked out. An analysis of behaviour of various alloying elements in aluminium in relation to their influence on bearing properties showed that elements such as tin, cadmium, lead, bismuth and indium provide low melting constituent and thereby improve antiscouring properties of aluminium. Whereas elements such as nickel, silicon, iron, manganese on alloying with aluminium produce new phase of greater hardness thereby improving wear and seizure resistance, copper, magnesium, zinc or silver may be used as alloying element with aluminium for strengthening the aluminium matrix where aluminium alloy bearing is to be used as solid bearing.

For developing soft bearing alloy for overlay application, series of heats were made of aluminium-lead-antimony group and mechanical and metallographic properties of the alloys were studied and reported. Further heats of the above mentioned two groups were made during the period and mechanical and metallographic studies were carried out. Experiments were also conducted on the development of solid bearing. Some of the heats of the solid bearing along with their mechanical properties as compared to the properties of standard copper base bushing aloys are shown in the Table 28. So far results obtained from various heats of different alloys indicate that there is possibility of achieving good bearing properties of aluminium base bearing alloys of the types under development which may replace high tin containing bearing alloys. Necessary arrangements for service trials of the bearings are also being made. Further work in this line is in progress.

46.0 Production of Phosphor-Bronze

At the instance of the Railway Design and Standards Organisation, investigation on production of phosphor-bronze spring was undertaken.

The work involved the development of a suitable melting and casting technique, determination of optimum hot and cold rolling schedule to produce strips and wires and the studies on the effect of cold working and subsequent thermal treatment on elastic properties so as to impart required spring temper to the phosphor bronze spring.

Virgin copper, tin and phosphor-copper (containing 13 per cent phosphorus) were used as the charge. Melting was carried out in the gas fired frit furnace under oxidizing atmosphere to avoid segregation. Effect of addition of oxidizing flux to the charge and optimum pouring temperature to get sound casting was determined. Hot workability of the alloy was found to be very poor compared to its cold working properties, though hot forging and rolling could be carried out to some extent under carefully controlled conditions at 500°C. The material responded well in cold working. At least 50 per cent cold reduction could be given before interstage annealing. Homogenizing treatment of 4 to 5 hours per 2 in. cross-section billets at 600°C. improved cold workability. It was observed that cold reduction of 60/70 per cent for flats and about 60 per cent for wires gave necessary spring temper to the product.

Table 28 — Properties of Solid Bearing Alloys

Heat No.	Nominal chemical composition	Mechanical properties			
		Ultimate tensile strength tons/sq. in.	Elongation %	Hardness BHN	Coefficient of friction under lubrication
AB 11	Al-base Major or Zn	19·5	1·56	136	0·016
AB 15	Small percentages of Pb-Mg-Cu-Sb	15·11	10	78	0·0247
AB 19	—	21·44	3·12	104	0·03
AB 3	—	25·7	1·56	136	0·04
Gun Metal	Cu-base	—	—	74	0·06
Phosphor Bronze	Cu-base	—	—	75	0·09

Stress relief annealing at 200°C. for different period varying according to cross-section was found essential for stabilizing the product. A report on 'Technology of Production of Phosphor Bronze Spring' was prepared and submitted to the Central Board of Railway Research. Service trial of the product in Railway signalling equipment is under progress.

46.1 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 over-load 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. Among the high expansion alloys various austenitic Fe-Ni-Cr-Mn alloys, brass, silicon bronze and Mn-Cu-Ni alloys were studied. Manufacturing technique for all ferrous thermo-bimetals were studied in detail and problems encountered in various stages of manufacturing were solved. The process developed has been released for commercial exploitation. Some small-scale supply of thermo-bimetals, particularly to public sector laboratories have been made.

The details for manufacturing of thermo-bimetals with copper base alloys such as brass, silicon, bronze, etc., as high expansion component was also worked out and small quantities of brass-invar bimetal were supplied to some industrial concern for use.

Stainless type bimetal with special property of corrosion and oxidation resistance was developed. Technique for manufacturing special type of extra high sensitive thermobimetal with Mn-Cu-Ni alloy as high expansion component is also being developed.

Progress was also made to develop improved technique for the production of composite strips and wire for use as substitute for springs and conductors in the electrical industry.

47.0 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. A summary of the work done during the period is given below.

(a) FOUNDRY SANDS

(i) *Sample No. M S-S-2* — This sample was collected from the south sand stone ridge at Phondaghat in Kankavali Mahal and received from Directorate of Geology & Mining, Government of Maharashtra. The sand grains were of sub-angular to sub-rounded shape and coarse to fine in size and had an A.F.S. grain fineness No. of 75·14 and a clay content of 6·9 per cent. The maximum distribution was spread over 30, 40, 50, 140 and 200 mesh sieves. Mineralogical composition of the sand consisted of mainly quartz while chemical analysis revealed it to contain SiO_2 94·9; Al_2O_3 2·78 and $FeO+Fe_2O_3$ 1·04 per cent. The sand did not sinter when put to test at 1350°C. Optimum moulding properties were found to be at 2·8 to 3·4 per cent moisture content of the sand mixture containing 10 per cent

Kutch bentonite. However, it was felt that a closer control over grinding, if exercised, would produce sand of better sieve distribution which could be more advantageous from the moulding point of view.

(ii) *Sample No. M-S-S-4* — This is a high silica sand having A.F.S.G.F. No. 26·7 and A.F.S. clay content 1·44 per cent was received from Directorate of Geology & Mining, Government of Maharashtra. The sand is coarse to fine grained and sub-angular to sub-rounded in shape. The sample mainly consisted of quartz. Other minerals like feldspar and muscovite were present in minor amounts. Pryoxene and zircon were present in traces only. Sintering range of the sample is above 1350°C. Study of its moulding characteistics were shown that the sample can be used in medium and heavy steel casting jobs.

(iii) *Sample No. M-S-S-5* — The sand received from D.G.M., Nagpur, Maharashtra, was reddish in colour and was a coarse to fine grained sand having an A.F.S. grain fineness No. 28 and A.F.S. clay content of 0·39 per cent. Grain shape was sub-angular to sub-rounded. It consisted mainly of quartz, assaying 97·94 per cent silica and 0·5 per cent alumina in the as received condition. The sintering range of the sand was above 1350°C. It showed optimum moulding properties with the addition of 10 per cent bentonite in the range of 2 to 3·8 per cent moisture content. For dry strength suitable percentage of dextrine was recommended. In view of its high permeability, high refractoriness, and high silica content, this sand can well be used in steel foundry practice and specially found suitable for core-making. Suitable mould washes will have to be applied to obtain good surface finish and to avoid metal penetration owing to coarse size of the grains.

(iv) *Silica Sand, Sample No.* 1 (*Kanhan river*) — The Kanhan river sand sample No. 1 received from the Directorate of Geology & Mining, Government of Maharashtra, was grey in colour and coarse to fine grained. The A.F.S. grain fineness number was 30 and had A.F.S. clay content of 15 per cent. It contained 78·93 and 80·69 silica in as received and washed conditions respectively. The mineral constitutents of the sand was predominantly quartz with small amounts of feldspar and other minerals. The sand contained black opaque grains in abundance. The sand grains were sub-angular to sub-rounded in shape. The sintering temperature was 1300°C. Satisfactory green properties were developed in the range of 1·4 to 1·9 per cent moisture when bounded with 5 per cent Kutch bentonite. In view of the medium refractoriness, five sieve distribution, which gives a close packing and good strength, it can be used in non-ferrous and medium cast iron castings.

(v) *Silica Sand, Sample No.* 2 (*Kanhan river*) —The sand was received from D.G.M., Government of Maharashtra. It was collected from the Kanhan river bed, east of the Kanhan railway bridge, at a distance of 12 miles from Nagpur, Maharashtra State. It was yellowish grey in colour, and coarse to fine grained having an A.F.S. grain fineness No. of 27·91 and A.F.S. clay content of 1·28 per cent. Grain shape was sub-angular to sub-rounded. The sample contained 83·37 silica. Petrological examination revealed that the sample consisted predominantly of quartz with minor amounts of feldspar; oxides of iron along with black opaque grains. Refractoriness of the sand was found to be moderate sintering temperature being 1300°C. In view of the medium refractoriness, four sieve distribution and sub-angular to sub-rounded shape which gives a close packing. This sand can be used for cast iron and non-ferrous casting jobs.

(vi) *Sherthalai Silica Sand* — The sand sample received from the Directorate of Geology & Mining, Kerala State, was dirty white in colour and medium to fine grained in size, having an A.F.S. grain fineness No. 58, and contained 0·26 per cent A.F.S. clay. It assayed 91·59 and 93·77 per cent of silica in the as received and washed conditions respectively. The sample mainly consisted of quartz with minor amounts of feldspar. Fine flakes of zircon, sillimanite and tourmaline were present. Traces of ferro-magnesium minerals and rutile were also present. Shape of the grains was sub-angular to sub-rounded. Sintering temperature was found to be above 1350°C. In view of the high refractoriness, good permeability and three sieve distribution, the sand was found to be suitable for core and mould making in medium and light steel foundries.

(b) BONDING CLAYS

(i) *Cutch Bentonite* — This bentonite sample was of brownish colour, dull waxy lustre and in the powder form. It was associated with moisture content of 11·00 per cent which is in agreement with the specification for western bentonites laid down by S.F.S.A. It contained SiO_2, 48·44 and Al_2O_3, 15·46 per cent. The ratio of sodium to calcium was 2·5. Its *p*H, gel and swelling index values were found to be 8·95, 36·5 and 12·5 respectively. The total base exchange capacity was 76 milliequivalent/100 gm. The sintering range of the sample was 1100°-1150°C. Petrological examination of the sample revealed that the predominant mineral constituent was montmorillonite. Study on the bonding characteristics of the sample with high silica Rajmahal sand showed that the sample developed satisfactory green and dry strengths. It was practically a sodium base bentonite and may be used as a binder in the foundries.

(ii) *Cutch Bentonite (Yellow)* — This bentonite sample was of yellow colour and in powder form. It was associated with moisture content of 13·11 per cent which is in agreement with the specifications for western bentonites laid down by S.F.S.A. Its *p*H, gel and swelling index values were found to be 9·2, 24·5 and 11·1 respectively. The total base exchange capacity was 72 mm. eq./100 gm. The sample sintered in the temperature range of 1050° to 1100°C. Petrological examination of the sample revealed that the predominant mineral constituent was montmorillonite. Study on the bonding characteristics of the sample with high silica Rajmahal sand showed that the sample developed satisfactory green and dry strengths. It was practically a sodium base bentonite and may be used as a binder in the foundries.

(c) SUITABILITY OF PORTLAND CEMENT AND SLAG CEMENT FOR CASTING PURPOSE

Cement is used as a binder in foundries where medium to heavy casting work is done. The main advantages which lead to its popularization in the foundries are viz., (i) Moulds are very hard, highly permeable, repairable and can be used for more than one castings. (ii) No drying ovens are required; instead air drying for 2-3 days is enough. (iii) Smooth casting finish is obtained. However, there are few disadvantages such as the high cast of the binder. Studies were conducted on the moulding properties of Portland and slag cement with a view to apprise the workability of both and to encourage the use of slag cement which is cheaper than portland cement without foregoing any of the advantages of the portland cement. In this present study slag cement was made from the slag obtained from low shaft furnace of National Metallurgical Laboratory. For portland cement ordinary masonry cement was used.

Various properties such as, moulding, setting and high temperature properties were studied. Moulding characteristics of both the cements were studied with varying amounts of moisture content and cement percentage. No notable difference in properties were noted between green properties of the two cements. Setting properties showed that portland cement set quickly and obtained more strength in less time whereas slag cement took more time to set; however, it attained the same strength as of portland cement. Setting time of 3 to 4 days was found to be enough for obtaining enough workable strength in the case of slag cement. Study of high temperature properties showed that slag cement resist high compression loads at elevated temperatures as compared to the portland cement, proving its safe workability for heavy castings. Studies are being conducted on the D.T.A. of the above cements. Casting characteristics also are under progress.

47.1 Shell Moulding

C.N.S.L. Resin Binder — The project was taken up with a view to study the suitability of indigenous raw materials for use in the shell moulding process under Indian conditions.

Work is being taken up on an indigenous resin called ' Polymerized C.N.S.L. ' resin received from Research Design and Standards Organisation, Ministry of Railways. This resin is made by heating phenol, a byproduct of cashewnut industry, with aldehydes to form higher polymer. Work is under progress to find out its physical properties so as to assess its workability in plant site. Studies are also being conducted in connection with mulling, moulding and core making problems. Work is also being carried out to evaluate its suitability with respect to increase in the dry strength and mould and core finish in the conventional foundry practice.

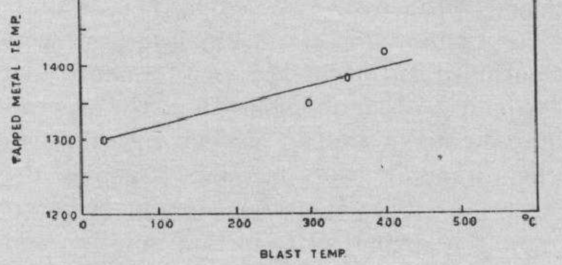


FIG. 33 — TEMPERATURE OF TAPPED METAL VS HOT BLAST TEMPERATURE

47.2 Hot Blast Cupola

Experiments were carried out, in the first instance, to assess the general characteristics of running of the hot blast cupola in comparison to the cold blast cupola, and hence, collect data for further work on it for different projects, such as, carbon pick-up, silicon pick-up, on injecting silica flour through the tuyers, saving of coke as well as its substitution with other fuels, evaluation of losses and pick up of elements, etc.

The usual charges consisted of 100 kg. of iron (basic and/or foundry pig) alone or with mild steel scrap. Each run consisted of about 10 charges. In each heat, bed coke of 100 kg. was employed while in the charge coke the quantity was either 10 or 16 kg. As flux 2·5 kg. lime stone per charge was used in the first few heats, while in the later heats this was increased to 3·5 kg. Fluorspar was added only in a few heats in the latter part of the heat. The following observations were made on the fourteen heats made so far.

Metal Temperature — It was observed that in cold blast practice the metal temperature at the sprout was about 1300°C. with 7: 1 iron/coke ratio, while on raising the blast temperature to 400°C. the metal was tapped at about 100°C. higher at the same iron/coke ratio. The rise in metal temperature with the increasing temperature of the blast is shown graphically in Fig. 33, which shows an average rise of 40°C. in the metal temperature for a rise of 50°C. of the blast temperature in the range of 300° to 400°C.

Metal Losses and Pick-up — In cold blast runs, the average silicon and manganese losses were about 20 and 15 per cent, respectively, while there was appreciable pick-up of both sulphur and phosphorus from the coke charge. These losses of silicon and manganese were found to be considerably reduced in the hot blast system due to increased affinity of the molten metal for these elements at higher temperature. Also, the amount of sulphur and phosphorus pick-up stood at a lower level in the cold blast system as in the hot blast system lower amount of coke could be used.

Incorporation of Steel Scrap in the Charge — Mild steel scrap was incorporated in the charge in the ratio of 50: 50 as mild steel to pig iron and was successfully melted.

Melting Rate — It was observed that the specific melting rate increased by about 40 per cent as a result of adopting hot blast in the cupola as compared to cold blast system.

47.3 Derivatograph

A derivatograph shown in Fig. 34 manufactured by Metrimpex (MOM) Budapest, Hungary; has been installed at NML. This is a complex thermo-analytical instrument designed to measure the temperature within the sample and determines the weight changes (TG), the rate of weight changes (DTG) and the enthalpy changes (DTA) of the sample simultaneously. The apparatus is fully automatic and the curves are registered photographically. A specimen copy of the drivatogram is shown in Fig. 34(a). The apparatus is versatile and can be used primarily in the qualitative detection of the component of samples such as mineral constituents of a rock and examination of clay minerals.

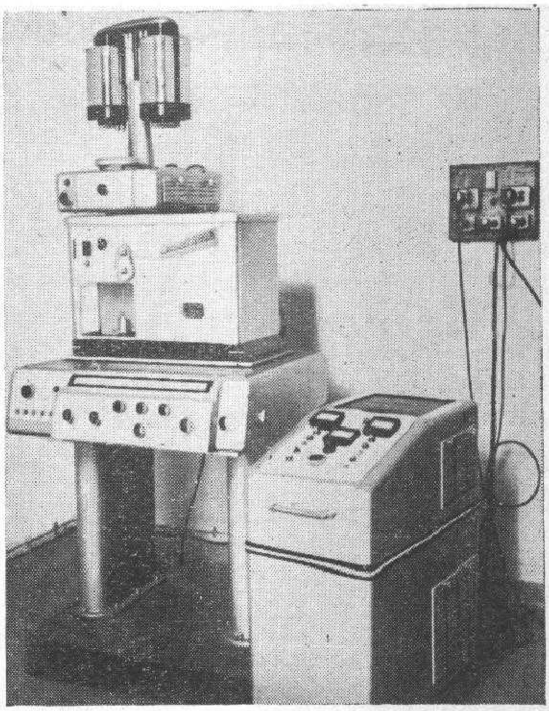


FIG. 34 — VIEW OF THE DERIVATOGRAPH INSTALLED AT THE NATIONAL METALLURGICAL LABORATORY

48.0 Powder Metallurgy — Production of Metallic Powders

Metal powders of aluminium, zinc, tin, copper, brass, lead, etc., are used in different grades for a variety of purposes including paint manufacture, chemical reduction and other powder metallurgy applications. Studies are 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 is also being considered.

(i) *Copper Powder* — Experiments were continued to study the exact quantity of soapnut powder to be used in the electrolyte to make copper powder according to the standard specification. During the experiments attempts were made to determine the amount of resin soap solution and the soapnut solution required for measured quantity of electrolyte. Table 29 gives

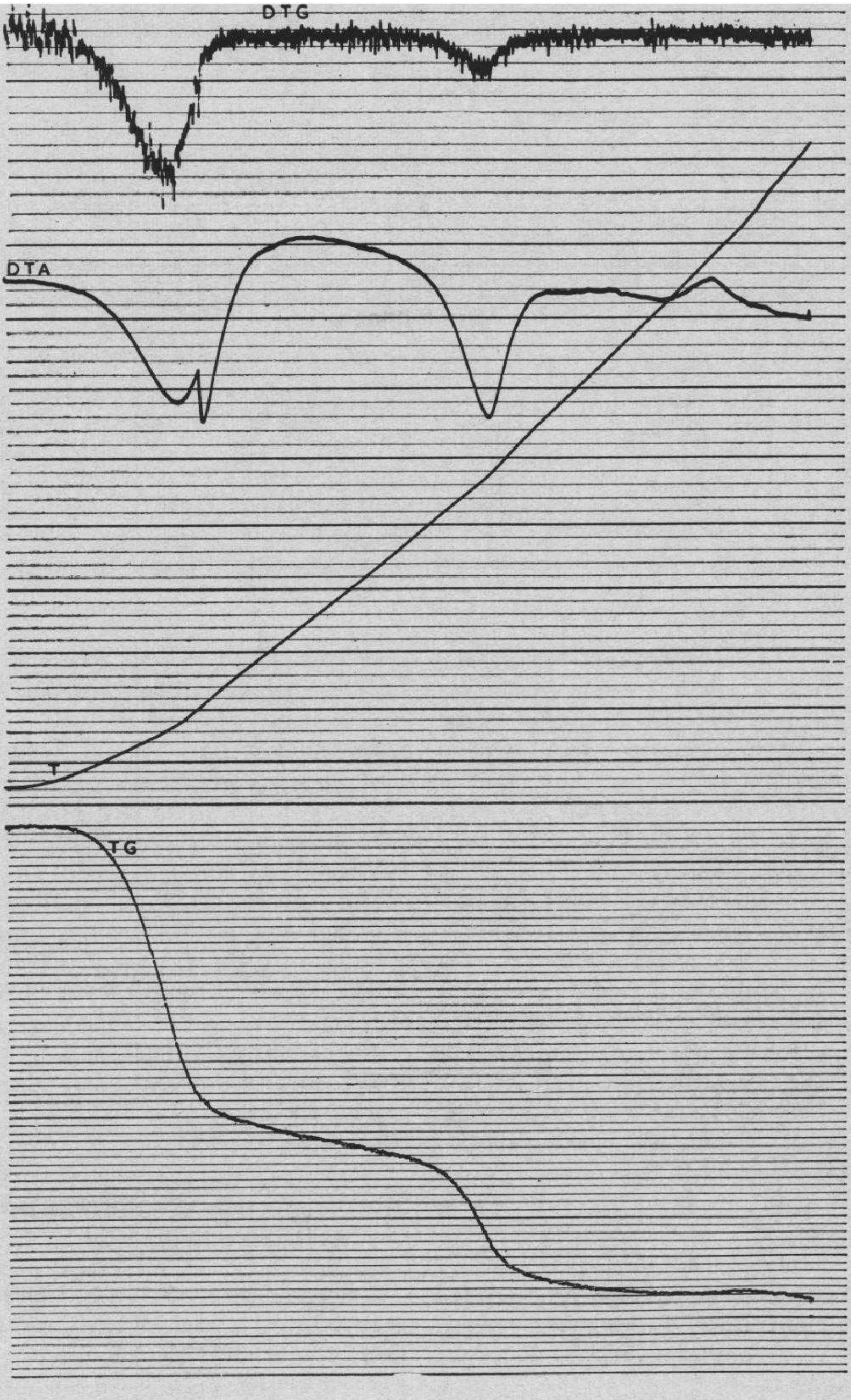


FIG. 34(a) — DERIVATOGRAPH OF A SAMPLE OF INDIAN BENTONITE SHOWING DIFFERENTIAL THERMOGRAVIMETRIC (DTG), DIFFERENTIAL THERMAL ANALYSIS (DTA), TEMPERATURE (T) & THERMOGRAVIMETRIC (TG) CURVES

Table 29 — Materials Used for Soapnut and Resin Soap Solution

	Wt of soapnut powder	Wt of the glycerine	Wt of the water	Wt of resin	Wt of borax
For soapnut powder solution	25 gm.	25 gm	250 cc. (approx.)	—	—
For resin soap solution	—	1·25 gm	50 cc. (approx.)	2·5 gm	2·5 gm

Table 30 — The Change in Apparent Density and Particle Size of Copper Powder Obtained After Milling

Sample No.	Apparent density before milling	Apparent density after milling	Particle size before milling				Particle size after milling				Time used for milling
			+140	+200	+270	−270	+140	+200	+270	−270	
1	2·15 gm/cc.	2·96 gm/cc.	18·4	12·3	19·8	49·5	7·7	7·7	12·0	72·6	10 min.
2	2·25 gm/cc.	3·36 gm/cc.	18·0	12·0	23·5	56·5	3·3	6·5	12·3	77·9	20 min.
3	2·25 gm/cc.	3·47 gm/cc.	8·1	13·1	23·9	54·9	2·7	5·7	11·2	80·4	30 min.
4	2·18 gm/cc.	3·56 gm/cc.	7·5	13·0	25·0	54·5	2·0	4·1	9·7	84·26	45 min.

the quantity of material used for making the extract of soapnut powder which was found suitable after experiments for depositing 1 kg. of the copper powder.

Experiments were conducted on milling the copper powder in a rod-mill to see its contamination with iron. Copper powder was pulverized in a rod mill running at 25 r.p.m.; of which inside lining together with rods were made of Mn-steel. The spectrographic analysis of the milled powder showed the presence of Fe in the sample only in traces (0·001-0·005 per cent). The change in the apparent density particle size of copper powder obtained of the milling is given in Table 30. Regarding the production of copper powder suggestions were made to Indian Copper Corporation, Ghatsila, to whom the process has been leased.

(ii) *Aluminium Lead and Tin Powder* — Experiments were undertaken to study the effect of temperature of the molten metal, pressure of atomization, nozzle diameter, height and temperature of the cooling medium and height of molten metal on the final particle size of the powder. Small-scale studies for making aluminium powder has been completed and a report is under preparation.

Preliminary tests for the production of lead and tin were also carried out. The temperature of the molten metal was kept at 350° and 450°C. for lead and tin respectively. It was observed that the atomizer used for the aluminium disintegration was not suitable for making tin or lead powder of very fine grade. A modified atomizer was designed for atomizing low melting metals and is under fabrication. A ladle, preheating furnace, and drying equipment, etc., were designed and fabricated for the production of 40 to 50 kg. of aluminium powder per batch.

48.1 Production of Iron Powder by Direct Reduction

Reducibility of iron ore samples received from M/s. V. M. Salgaocar E. IRMAO Ltd, Goa, was studied by the direct education method at temperatures of 900°, 1000° and 1100°C. with varying times as given in the Table 31.

Table 31 — Reducibility of Iron Ore Sample

Temperature °C.	Time hr.	Analysis of the reduced pellets				
		Fe, %	FeO, %	C, %	S, %	P, %
900	24	91·10	2·16	1·00	0·010	0·06
1000	16	90·13	3·36	0·04	0·030	0·08
1000	20	91·28	2·16	0·93	0·010	0·05
1100	6	91·84	0·72	1·15	0·025	0·06
1100	12	93·67	1·20	1·21	0·020	0·08
1100	16	93·93	1·10	1·06	0·030	0·08
1100	24	93·26	2·53	1·06	0·080	0·08

A number of batches of iron ore pellets were reduced at 1100°C., keeping the time of reduction constant at 8 hours. The reduced pellets were crushed for making iron powder. Pre-sintered pellets were used for direct reduction of the iron ore, so as to obtain high density iron powder.

49.0 Direct Reduction of Iron Ores by Naphtha

The investigation was taken up to study the direct reduction of iron ores by naphtha, a petroleum byproduct. Naphtha, being a mixture of paraffin hydrocarbon, consists of 10·15 per cent hydrogen and 80-85 per cent carbon, by weight, which acts as a reducing agent. High grade iron ore powders including blue dust were utilized for the study of direct reduction by naphtha and it was found that at a temperature of 850°-950°C. the reduction was complete in 60-90 minutes depending on the requisite amount of naphtha injected in the experimental tube within that time. The batch experiments of 100-200 gm. gave encouraging results and further study was completed by varying time and temperature.

This reduced powder was produced from iron ore of composition Fe_2O_3, 92·93; SiO_2, 1-1·7; Al_2O_3, 4·8 per cent and the reduced powder analysed as Fe, 88-90 and carbon 0·02-0·04 per cent. It was further purified to remove the gangue material and an iron powder of 92-93 per cent purity was obtained. The powder thus produced can find direct application in cutting of hard metallic ingots, scarfing, etc. Work is under way to prepare the powder on large scale batch experiments.

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

The measurement of electrical resistance of iron ore sample before, during and at the end of the reduction by hydrogen has proved to be a successful method, as reported earlier, for the study of the kinetics of hydrogen reduction. During period under review more work has progressed towards further standardization of the method and to reveal the reduction characteristics of completely unknown ores.

These experiments were performed by making compacts of pure iron oxide (99·89 per cent) by compacting at a pressure of 2·11 kg./mm.^{2} and subsequently heating at a temperature of 1100°C. These compacts have shown varying percentage of reduction behaviour of various ores along with pure iron oxide is shown in Fig. 35. Further work on the correlation of the electrical resistance and reducibility to porosity of ores, hydrogen flow, reaction temperature maintained during experiment, is under way.

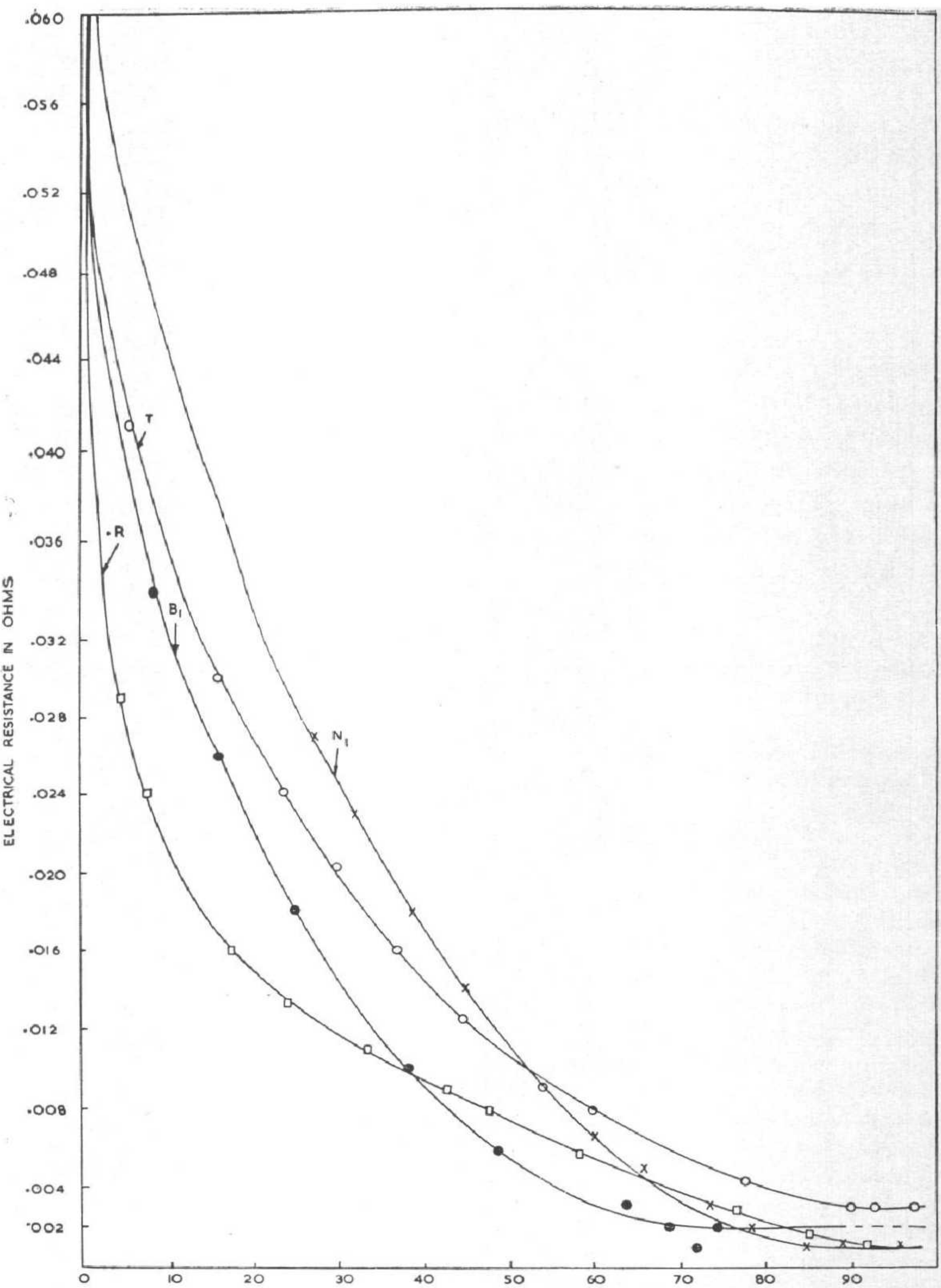


FIG. 35 — DEPENDENCE OF ELECTRICAL RESISTANCE OF IRON ORES ON THEIR PERCENTAGE REDUCTION

51.0 Determination of Porosity of Iron Ores and Sinters

Porosity of iron ores and sinters used in blast furnaces is one of the important factors in controlling the rate of the solid-gas reactions. This also determines the effective area of contact between the ores and the ascending reducing gases. An apparatus for the determination of micro and macro porosities of different iron ores and sinters was fabricated as shown in Fig. 36. The porosity determination of several indigenous iron ores collected from various sources is under progress. The ores are crushed to (—3 +4) mesh size (Tylor), in order to prepare a closely sized sample and are heated to 125°C. for 15 hours to remove moisture prior to porosity measurement. Kiriburu iron ore has shown macro and micro porosities 2·1 and 13·6 per cent while Bayaram showed 2·5 and 17·8 per cent respectively. More work is in progress.

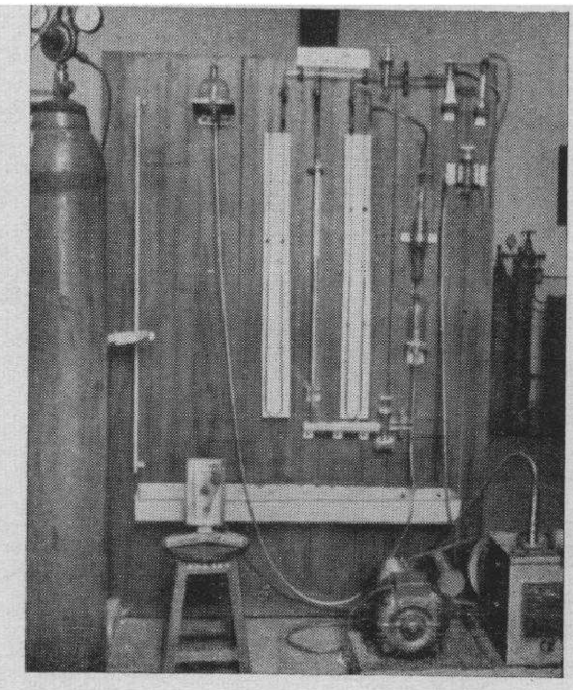


FIG. 36 — POROSITY DETERMINATION APPARATUS

52.0 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, Pb, Mg, Sn, Sb, retard the break-down of cementite. So study is 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 has been observed that C/Si ratio also plays an important part on mealleabilization. When it is 0·9-3·1 the malleabilization takes place and beyond this range no effect is observed.

Effects of copper addition and carbon/silicon ratio on white cast iron were reported in the previous Annual Report. Experiments were further conducted to study the effect of higher annealing temperatures on hardness. Fig. 37 indicates the plot of hardness for the samples mentioned in Table 32 against various temperatures of annealing for a period of 25 hours. It was observed that the hardness progressively decreased with higher annealing temperatures. The slope of the line indicates the rate of decrease in hardness with rise of temperature of annealing and is dependent on the composition of the samples.

Experiments were also carried out on white iron of the composition C, 2·29; Si, 1·10; Mn, 0·59; P, 0·18; S, 0·19; Cu, 1·26 per cent under nitrogen atmosphere for varying time periods. The results obtained were compared to those of packing method. The micro-photographs shown in Fig. 38 indicates that malleabilization effect was better pronounced under nitrogen atmosphere than when done under packing. These results were further confirmed by the hardness tests. Fig. 38(e) shows that for 5 hours treatment at 900°C. there was no appreciable difference in hardness in the

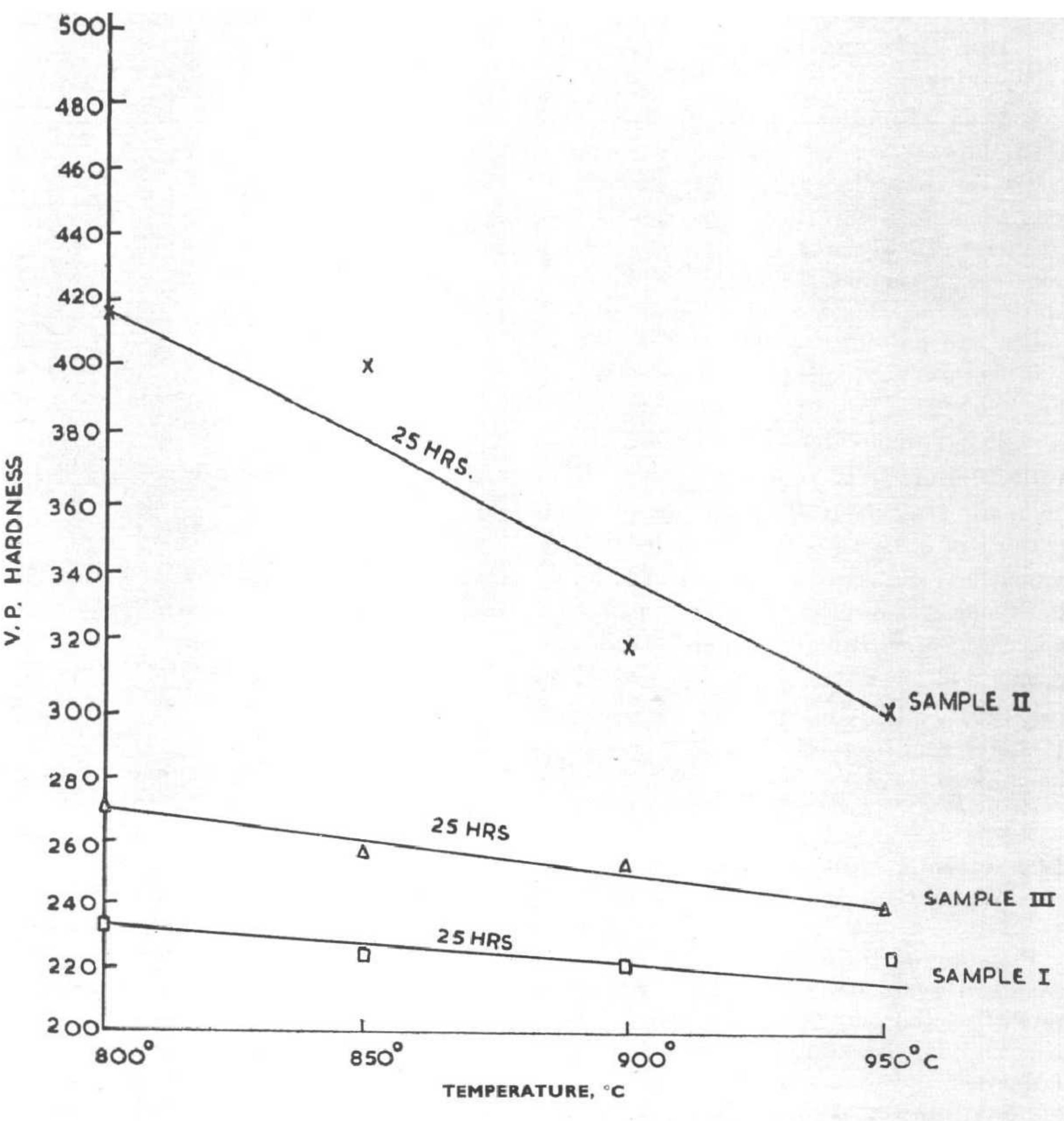
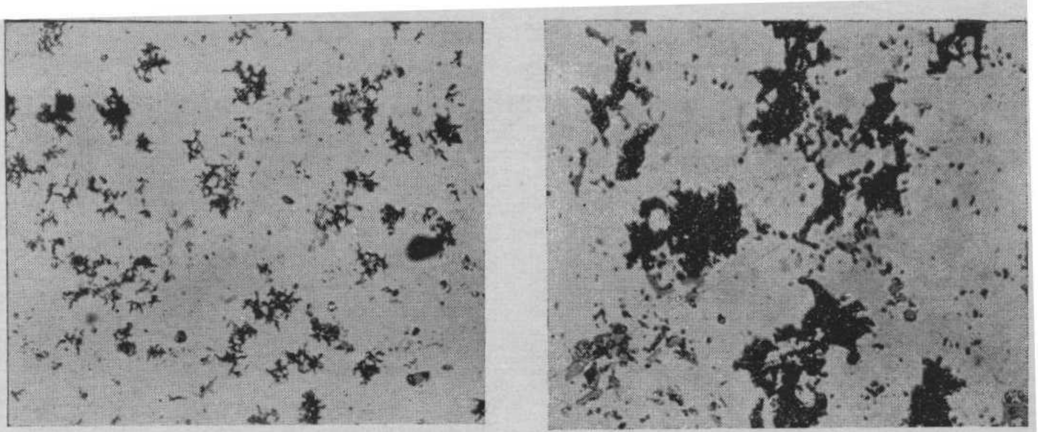


FIG. 37 — DECREASE IN HARDNESS ON ANNEALING AT DIFFERENT TEMPERATURES FOR 25 HOURS

Table 32 — Composition of Cast Irons

Sample No.	Compositions, %					
	C	Si	Mn	S	P	Cu
1	2·57	1·16	0·56	0·15	0·18	1·17
2	2·29	1·10	0·39	0·19	0·18	0·26
3	2·30	0·51	0·34	0·22	0·21	1·29

two cases, whereas for periods more than 5 hours, hardness of the samples treated under nitrogen atmosphere is much lower than the one obtained under the conditions of packing. The work on the effect of B, Al, Zn, etc., as inoculants in white cast iron, for its malleabilization characteristics, is under way.



(a) 10 hr. in nitrogen atmosphere at 900°C. ×150

(c) 15 hr. in nitrogen atmosphere at 900°C. ×150

(b) 10 hr. under pack at 900°C. ×150

(d) 15 hr. under pack at 900°C. ×150

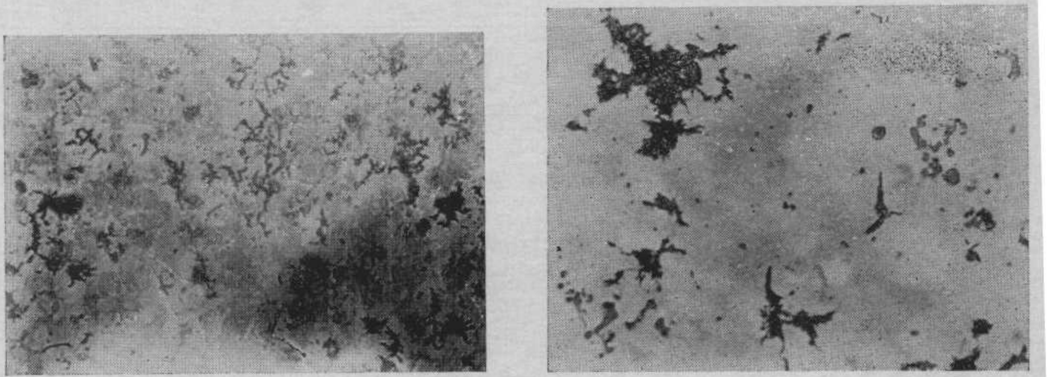


FIG. 38 — COMPARATIVE MICRO-STRUCTURES OF C.I. MALLEABILIZED UNDER NITROGEN ATMOSPHERE AND UNDER PACKING

53.0 Dephosphorization of Indian Cast Irons by Additions of Mixtures in the Ladle

Soda ash treatment is usually adopted for some degree of desulphurization of cupola metal. This, however, does not effect the removal of phosphorus because of acidic medium of the slags and hearths. However, the use of basic slags and the presence of basic constituents for the formation of stable phosphates in the oxidizing medium, it is probable that some dephosphorization may take place. It is with this idea that investigation was initiated to effect some degree of phosphorus removal using soda ash and oxidizing mixture like iron oxide or mill scale in a particular sequence in the ladle containing molten metal.

In a preheated basic ladle of 20 kg., batch experiments were performed by tapping the molten iron from a high frequency furnace at a temperature of 1400-1600°C. 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

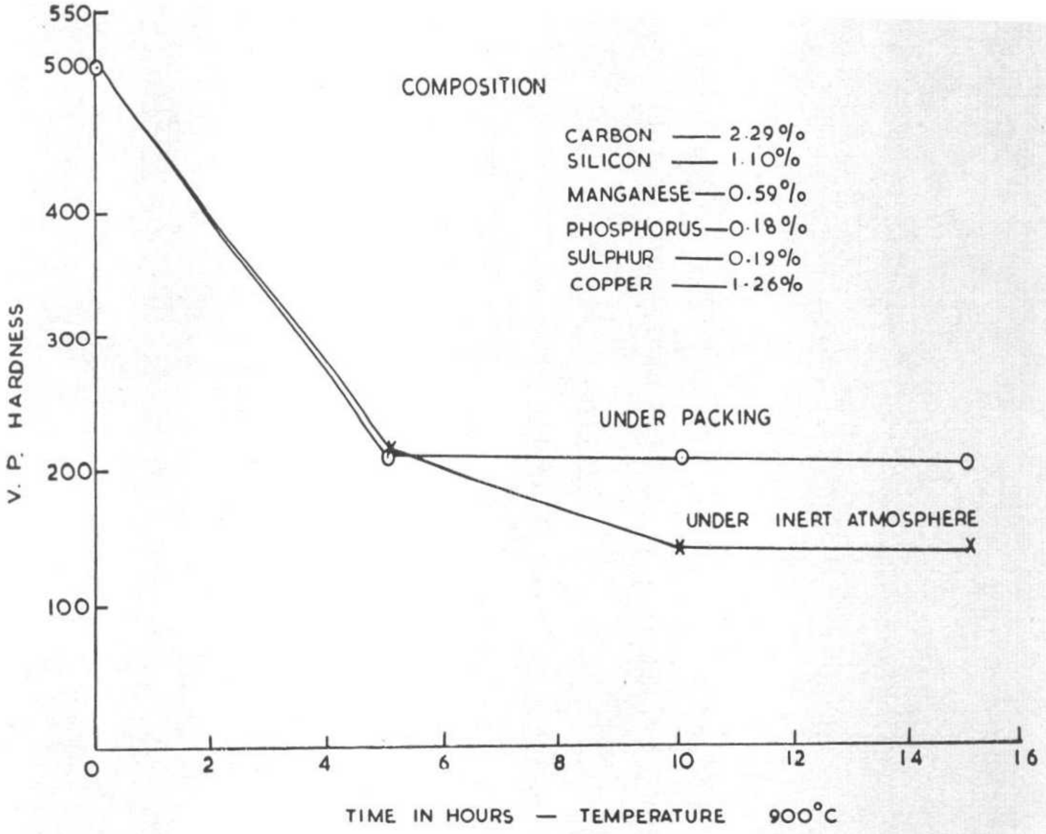


FIG. 38(e) — CHANGE IN HARDNESS WITH TIME AT 900°C.

after pouring the remaining metal. The reaction took place with flames on the surface. The metal was then poured in the mould, after removing the slag. These experiments were repeated by performing them in the furnace to see the effect at higher temperatures as in some cases the temperature has dropped down to 1250-1300°C. in the ladle. The results so far obtained shows phosphorus removal from 0·3 per cent in the original metal to 0·05-0·08 per cent in the treated metal. More experiments are in progress for working further details of the mixtures used and the temperatures employed for the efficient phosphorus removal of the cast iron. This has great industrial potentialities for applications where low phosphorus irons are employed in castings, etc.

54.0 Investigation into Steel Ingot Structures by Casting Stearine-Naphthalene Ingots

To study the solidification characteristics of big steel ingots, it is necessary to section the whole ingot, which becomes a difficult and costly affair. This is still more necessary, when changes are made in the ingot mould shape, sizes and type of ingots. The behaviour of mixture of stearine-naphthalene in fixed proportions on solidification shows a simulation in solidification to actual steel ingot. A study was undertaken to simulate and correlate the observations of the stearing-napthalene ingots to actual steel ingots. Plant data to this effect of actual steel ingots were collected from M/s. Tata Iron & Steel Company and Hindustan

Steel Limited, Rourkela, and it was found that percentage volume of pipe obtained was nearly the same when experimented with stearine-naphthalene mixture in small proportional ingots by applying proportional teeming conditions to the actual steel teeming. The ingots, mostly simulated killed steel ingots, showed central pipe with bridging in some cases. Thin skin indicating initial chill followed by dendritic and equiaxed crystals were obtained as solidified structure. The ingotism defect as obtained in actual steel ingots was also visible. Attempt was also made to make rimming ingots by the use of chemicals with molten stearine-naphthalene mixture. The rimming ingots showed blowholes at varying depths beneath the skin of the ingots and spongy top, indicative of a typical rimming steel ingot. Details of these ingots are given in Table 33 and are shown in Fig. 39. Though no dogmatic or empirical relationship could be obtained for correlation study, the attempt is still continued.

55.0 Study of the Reduction Mechanism of Iron Ores in Relation to the Gas Utilization Coefficient

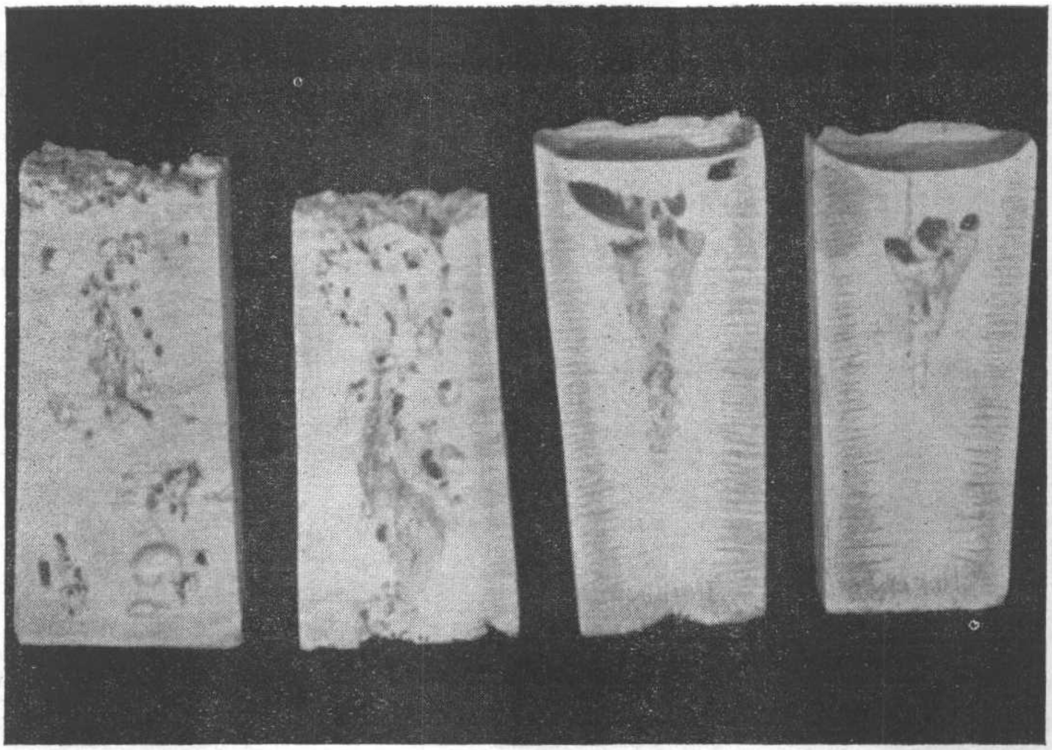
The reduction mechanism in an ore bed reduction by reducing gases like carbon monoxide and hydrogen is somewhat different from that of the reduction of ore lump. A point of fundamental interest is to achieve better understanding of the utilization of the reducing gas in reducing the ore present in excess in an ore bed. The purpose of the present work is to study the reduction mechanism in finely divided ore bed in relation to gas utilization coefficient [which is shown by $CO_2/(CO+CO_2)$ and $H_2O/(H_2+H_2O)$ for CO and H_2]. It is also hoped to study with typical Indian ores, the effects of various factors (like ore size, contact time, presence of other gases, etc.) on gas utilization coefficient.

Carbon monoxide was generated by passing carbon dioxide gas from the cylinder over a bed of graphite heated at 1050°C.

Table 33 — The Data of Different Types of Ingot Structures of Naphthalene and Stearine

Sl No.	Percentage mixture		Temperature °C.	Rate of pouring cc./sec.	Total volume of ingot cc.	Percentage volume of pipe %	Structure obtained
	Stearine %	Naphthalene %					
1	55	45	65	35 (T)	590	7·7	Central pipe with bridging formed. Dendritic crystal structure perpendicular to mould walls visible (BED)
2	55	45	65·5	9·2 (T)	582	6·5	do (BEU)
3	55	45	67	7·5 (T)	460	6·3	do (BED)
4	55	45	67	8·1 (B)	605	6·0	do (BEU)
5	55	45	67	8·1 (B)	610	5·8	do (BED)
6	55	45	69	8·6 (T)	468	5·3	do (BED)
7	58	42	67	8·0 (B)	605	7·7	do (BED)
8	55	45	69	5·9 (T)	581	—	Rimming ingot having deep seated blow holes
9	55	45	71	5·7 (B)	527	—	do

T — Top pouring.
B — Bottom pouring.
BEU— Big end up.
BED— Big end down.



(1) (2) (3) (4)

(1) & (2) — Rimming ingots; (3) & (4) — Killed ingots

FIG. 39 — STRUCTURE OF STEARINE-NAPHTHALENE INGOTS

Several samples of graphite were tested and it was found that with electrode graphite particles (—6·36 +3·12 mm. size), the conversion was very satisfactory. Unconverted carbon dioxide (minimum 10 per cent) in the gas mixture coming out of the conversion chamber was absorbed in a series of absorbents. After these absorptions, carbon monoxide was completely free from carbon dioxide and moisture. This pure gas was then passed to the reduction chamber through a flowmeter calibrated accurately for the carbon monoxide gas. Preliminary work for the reduction of the ores is in progress. A photograph of the complete set up of the apparatus is shown in Fig. 40.

56.0 Study of Reaction Between Solid and Liquid Metals

Many contradicting views have been furnished by different investigators on the subject due to the variations in materials and experimental techniques used. It was felt that a more systematic study of the factors controlling the kinetics of the reaction would be advantageous towards acquiring a better understanding of the whole process. Consequently in the present work the apparatus and experimental techniques were designed first to enable the reactions of iron and steel with molten zinc to be carried out under very closely controlled conditions.

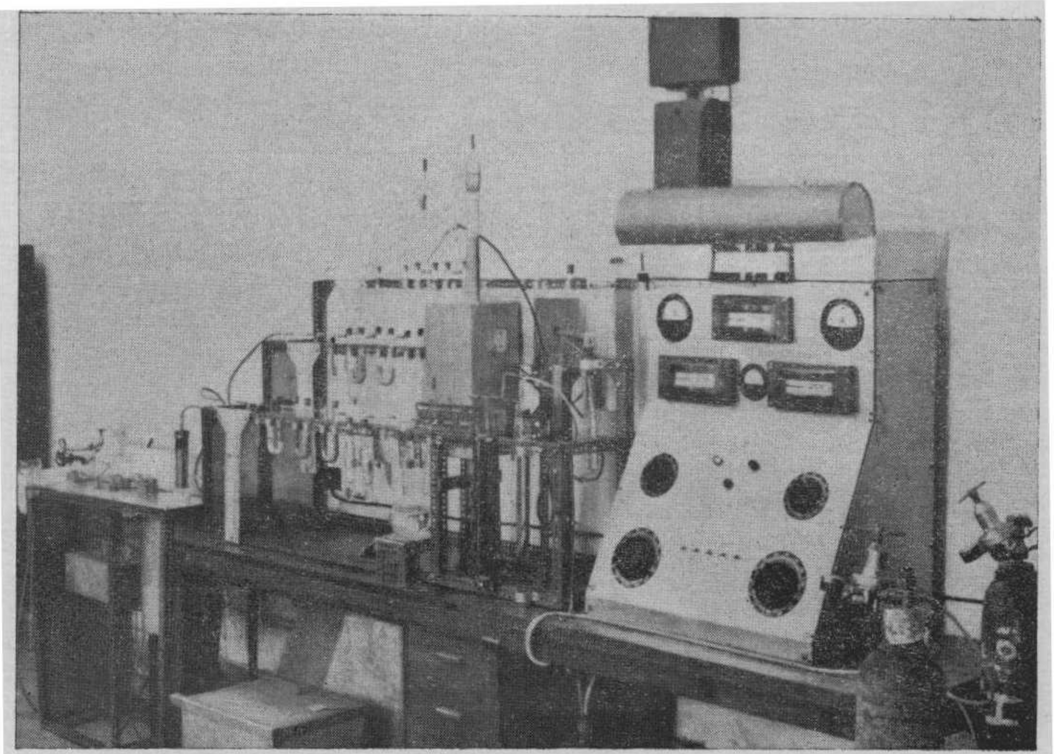


FIG. 40 — SET-UP FOR THE STUDY OF GAS UTILIZATION CO-EFFICIENT

The set-up consists of a sillimanite tube, some portion of which is kept under molten zinc-bath, into which purified argon gas was passed. When the desired temperature of the bath was attained, the specimen kept in tube was dipped into the molten bath for a specified time. The specimen was then taken out of the bath and quenched in water. The study was undertaken by using 0·1 per cent carbon steel and commercially pure zinc (99·3 per cent). Fig. 41 shows a photomicrograph of the zinc coated sample indicating three parts: outer layer has a similar composition as that of the bath, the middle alloy layer consisting of intermetallic phases (zeta, delta) and lastly a thin solid solution zone of iron and zinc (gamma). Further work is in progress.

57.0 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.

Arising from the work of curie temperature of iron alloys reported in the previous Annual Report, the work was extended in the following directions during the period under review:

(i) Measurement of curie temperature of cobalt alloys.

(ii) 'Mossbauer' effect studies of iron-aluminium and iron-silicon alloys.

(iii) Development of marageing type of steels based on iron-aluminium-silicon composition.

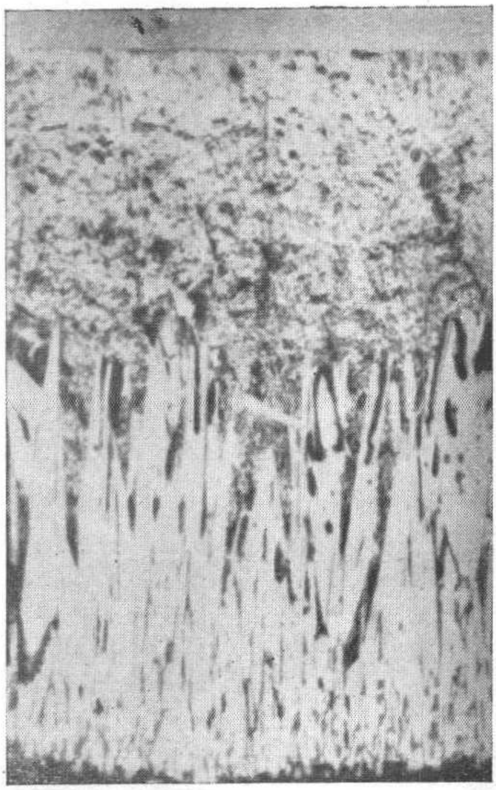


FIG. 41 — PHOTOMICROGRAPH OF THE ZINC COATED SAMPLE (450°C.)

(iv) Measurement of coefficient of thermal expansion of iron-aluminium, and iron-silicon alloys.

The progress made in the different directions are as follows:

(i) As mentioned in the previous report cobalt alloys presented difficulties in melting and after several trials with the melting in vacuum, two melts were made under vacuum. This cobalt was subsequently forged to be used as support rods for curie temperature measurements. Cobalt-aluminium alloys were made at the Atomic Fuels Division of the Bhaba Atomic Research Centre, Bombay; and eight compositions of cobalt-aluminium in the composition range 0·25 atom per cent aluminium were melted. Four of these melts on forging cracked into pieces and attempts are being made to remelt these compositions. The forged alloys are being welded to cobalt rods for curie temperature measurements.

(ii) The measurements of curie temperature of iron-aluminium and iron-silicon alloys suggested a difference in the nature of iron-aluminium and iron-silicon bonds in these alloys. To get a clearer picture of the nature of the bonds, 'Mossbauer' effect studies were contemplated and the Tata Institute of Fundamental Research, Bombay, has kindly extended this facility. Samples of alloys were prepared and sent to Tata Institute of Fundamental Research for 'Mossbauer' effect measurement.

(iii) The nature of iron-aluminium, iron-silicon bonds in Fe_3Al and Fe_3Si suggested the possibility that marageing type of alloys based on controlled super lattice formation could be developed. From the knowledge gathered about the nature of these bonds from studies conducted, a few compositions were made and heat treatment was carried out on the alloys for different periods of time and temperatures. After initial exploratory experiments four new compositions were melted on which work is now in progress.

(iv) As data available in literature on co-efficient of thermal expansion of iron-aluminium and iron-silicon alloys are not only meagre but also conflicting, it was considered worthwhile to measure the coefficient thermal expansion of iron-aluminium and iron-silicon alloys, and work is now in progress on these lines.

It may also be mentioned that there appears to be a relationship between magnetism and crystal structure. From such a correlation an empirical rule for the occurence of sigma phases in binary alloy systems of transition metals was formulated.

58.0 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 present 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 progress in commercial aluminium.

In continuation of the work on the improvement of electrical conductivity of commercial aluminium by (1) addition of alloying elements, and (2) thermal treatment, a series of aluminium alloys, designated PM4 to PM8, were prepared by melting the constituent metals with suitable fluxes and degassers in an oil-fired furnace and were cast into ingots (10 sq cm. × 60 cm) in metal moulds. The ingots were rolled and drawn to 0·110 in. diameter wires at M/s. Indian Cable Company Ltd, Jamshedpur, and subsequently tested by them for mechanical (wrapping and breaking load) and electrical tests specified in Indian Standards. It was then decided to extend the scope of the work and another series of alloys, PM 6 to PM 16, were made and drawn into wires. Whilst no difficulty was experienced in rolling and drawing without any alteration in the rolling and drawing sequences, PM 16 could not be rolled probably due to inhomogeneous distribution of alloying elements.

Preliminary tests on the corrosion of the wires have shown that their corrosion resistance is superior to that of the wires now available in the market. Although a synthesis of good conductivity, high strength and high corrosion resistance was obtained, attempts are being made to bring about further improvements in the conductivity.

59.0 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, Pb-Cd and Pb-Sb alloys in the liquid state.
(b) Study of liquid metals and solidification.
(c) Enthalpy of liquid alloys.

STUDY OF THE INTERACTION BETWEEN ALUMINIUM-COPPER ALLOYS

The investigation on the structure of liquid aluminium-copper alloys was completed during the year. Aluminium-copper alloys containing 4, 14, 33 and 40 per cent copper were centrifuged at 700° and 800°C. under centrifugal forces of 40 g and 141 g, where g is a gravitational force, for periods up to 7-9 hours. The aluminium-33 per cent copper alloy was additionally centrifuged at 600°C. Chemical and metallographic examinations showed that a concentration gradient was set up in all alloys as a result of centrifuging. The cast grain sizes also showed the variation across the longitutional section of the specimens. The concentration gradient was analysed to yield cluster size. It was found that the cluster size (1) decreased with increase of temperature, (2) is a function of composition and varies between 30-84Å with a minimum at the eutectic composition, and (3) decreases withi ncrease of centrifugal forces. It was shown that there is usually a distribution in cluster size. The cluster size could be correlated with the viscosity of the alloys. It was also shown that the volume fraction of the clusters in liquid aluminium-copper alloys is between 3-7 per cent in the temperature ranges of metallurgical interests.

A significant feature of the investigation is that a synthesis of viscosity, cluster size and volume fraction of clusters enables the calculation of heat of formation of the cluster, which was shown to vary between 5·5 to 8 Kcal/gm. atom. A comparison of heat of formation with the published thermodynamic values suggests that the composition of the cluster is $CuAl_n$ ($2 > n > 0{\cdot}5$).

STUDY OF THE INTERACTION IN THE LEAD-CADMIUM ALLOYS

Three lead-cadmium alloys containing, respectively, 10, 17·5 and 25 per cent cadmium were studied between 300-600°C. under centrifugal force 40 g and 141 g by the experimental technique previously developed. It was found that a concentration gradient was set up in the liquid state. An analysis of the concentration gradient showed that the size of the lead and cadmium clusters was almost unchanged with temperature, but an initial decrease was observed. The size of the clusters varied with composition with the minimum at the eutectic composition. The existence of lead-rich and cadmium-rich clusters could be justified on the basis of published thermodynamic data of the system.

INTERACTION IN LEAD-ANTIMONY SYSTEM

A number of lead-antimony alloys containing 6, 11, 2, 30 and 65 per cent antimony were examined in the liquid state according to the technique developed. The results are at present being analysed but are of very complex nature. It was observed that whilst a gradual concentration gradient was set up in case of 6 and 11·2 per cent antimony alloys at lower speeds of centrifugation, the 30 per cent antimony alloy behaved in a peculiar manner. It showed separation into the three distinct regions which could be observed on macro-examination of the sectioned ingot. The 65 per cent antimony alloy showed the usual horizontal macro-demarcation when temperature was slightly above the liquidus at all speeds of centrifuging; when centrifuged at 700°C. only, a gradual change in concentration occurred. The complexity of the system is further borne out when the published values of the volume change on mixing are examined. Work has not yet advanced to the stage where any explanation could be proposed.

LIQUID METALS AND SOLIDIFICATION

(i) *Spat Cooling* — Since liquid state of metals is the first step in almost all metallurgical processes and because the structure of solidified ingot or casting is important from the point of view of mechanical properties and performance of metals, it was decided to study the effect of liquid structure on solidification. It was proposed to study solidification under (1) conditions of spat cooling, and (2) conventional cooling. Solidification characteristics of pure aluminium and antimony were first investigated on rapid cooling from the liquid state (900°C.). A simple but effective experimental set up was designed to ensure the rapid solidification. The apparatus (Fig. 42) essentially involves centrifuging the molten metal contained in crucible and ejecting the liquid metal in a thin stream under centrifugal force on to an annular copper drum. The thickness of the film thus solidified was between —0·15-0·2 mm. Thin films were metallographically examined. Figure 43 shows the metallographic structure for aluminium film and shows that plastic deformation occurred immediately after solidification and may be caused by the associated volume change. In the case of antimony the structure varied between typically dentritic to almost structureless phases. The word 'structureless' has been used for want of a suitable word but does not imply an amorphous structure. The crystallinity of thin film was examined with the help of X-ray diffraction. The observations suggest that rapid solidification does not lead to the formation of perfect crystalline lattice and that there is a general lack of crystallinity.

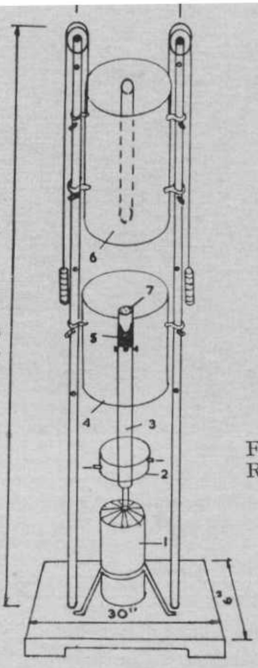


FIG. 42 — SET-UP FOR RAPID SOLIDIFICATION OF METALS

It is now proposed to extend this work to aluminium-copper alloys and to study any possible effect of liquid structure on mechanical properties of the films.

The effect of liquid structure of solidification by conventional methods is being studied on the aluminium-titanium system with a view to study the mechanism on grain refinement of aluminium and its alloys. Alloys containing 0·05, 0·1 and 0·5 per cent titanium were prepared and initially centrifuged at 700°, 750° and 800°C. in a stainless steel crucible and later in a refractory crucible to avoid any contamination. As the results obtained so far are preliminary, no generalized conclusions are given.

(ii) *Effect of Holding Time* — The effect of holding time in the liquid state on

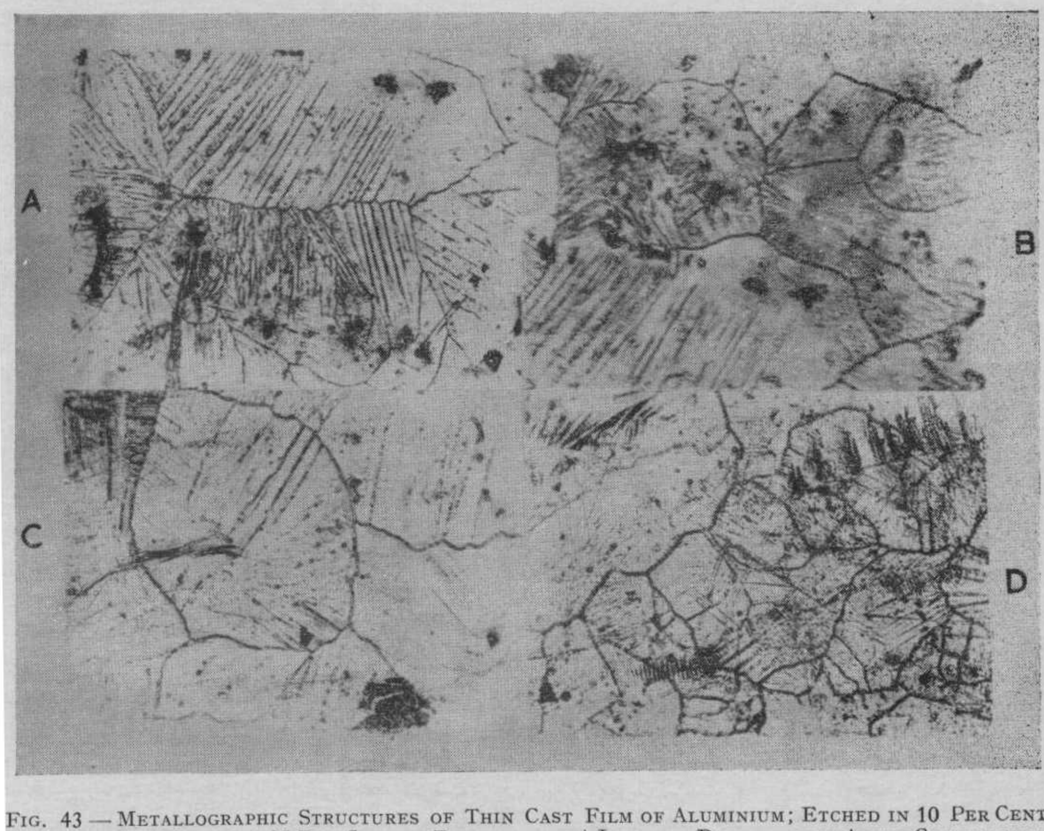


FIG. 43 — METALLOGRAPHIC STRUCTURES OF THIN CAST FILM OF ALUMINIUM; ETCHED IN 10 PER CENT NaOH. × 270: (REDUCED 35 PER CENT IN REPRODUCTION) INDICATE DEFORMATION AFTER CRYSTALLIZATION. A & C ALSO SUGGEST GRAIN BOUNDARY MOVEMENT

solidification was studied for the aluminium-copper eutectic alloys at temperatures of 600°, 650°, 700°, 800° and 900°C. The effect of holding time up to 200 hours was studied. The samples were quenched from the liquid state and were metallographically examined. It was found that a copper rich phase which appeared in the micro-structures of specimens, held for shorter holding times in liquid state, progressively disappeared as the holding time was increased as shown in Figs. 44 and 45. This could be due to structural changes in the liquid state. A relationship between time and temperature was established for the disappearance of the phase from the micro-structure.

ENTHALPY OF LIQUID ALLOYS

During the year, the heat contents and heat capacities of three binary lead-antimony alloys and a ternary lead-antimony-tin alloy were determined as a function of temperature utilizing the experimental technique earlier reported. The results obtained are shown in Figs. 46 and 47 respectively. The figures indicate that the relationship between heat capacity and temperature is not continuous but exhibits one or more irregularities. There is general agreement that discontinuties in the variation of heat capacity with temperature are indicative of internal rearrangement of atoms, but their precise nature in these alloys cannot be predicted at this stage. Although no unequivocal evidence has so far been presented for the atomic configurational rearrangement in the liquid state, results reported by other workers on the volume change in lead-antimony alloys suggest that the interaction is complex. Attempts are now being made to correlate the heat capacity data with the cluster size.

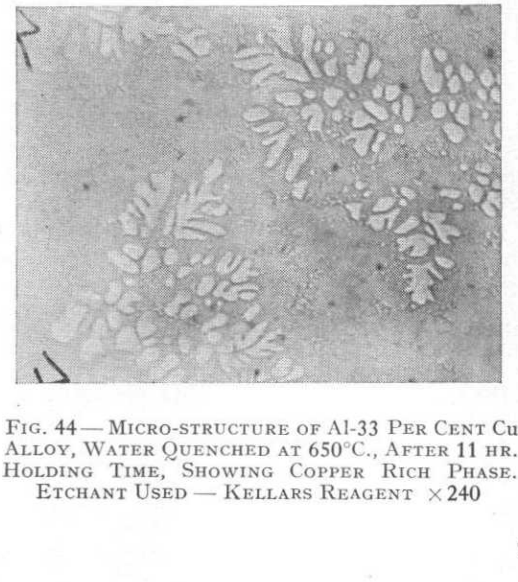


FIG. 44 — MICRO-STRUCTURE OF Al-33 PER CENT Cu ALLOY, WATER QUENCHED AT 650°C., AFTER 11 HR. HOLDING TIME, SHOWING COPPER RICH PHASE. ETCHANT USED — KELLARS REAGENT ×240

FIG. 45 — MICRO-STRUCTURE OF Al-33 PER CENT Cu ALLOY, WATER QUENCHED AT 650°C., AFTER 30 HR. HOLDING TIME, SHOWING DISAPPEARANCE OF THE COPPER RICH PHASE. ETCHANT USED — KELLARS REAGENT ×240

60.0 Effect of Titanium on Iron and Steel

(a) *Titanium in Steel* — The project was undertaken to study the transformation and precipitation hardening characteristics of titanium 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

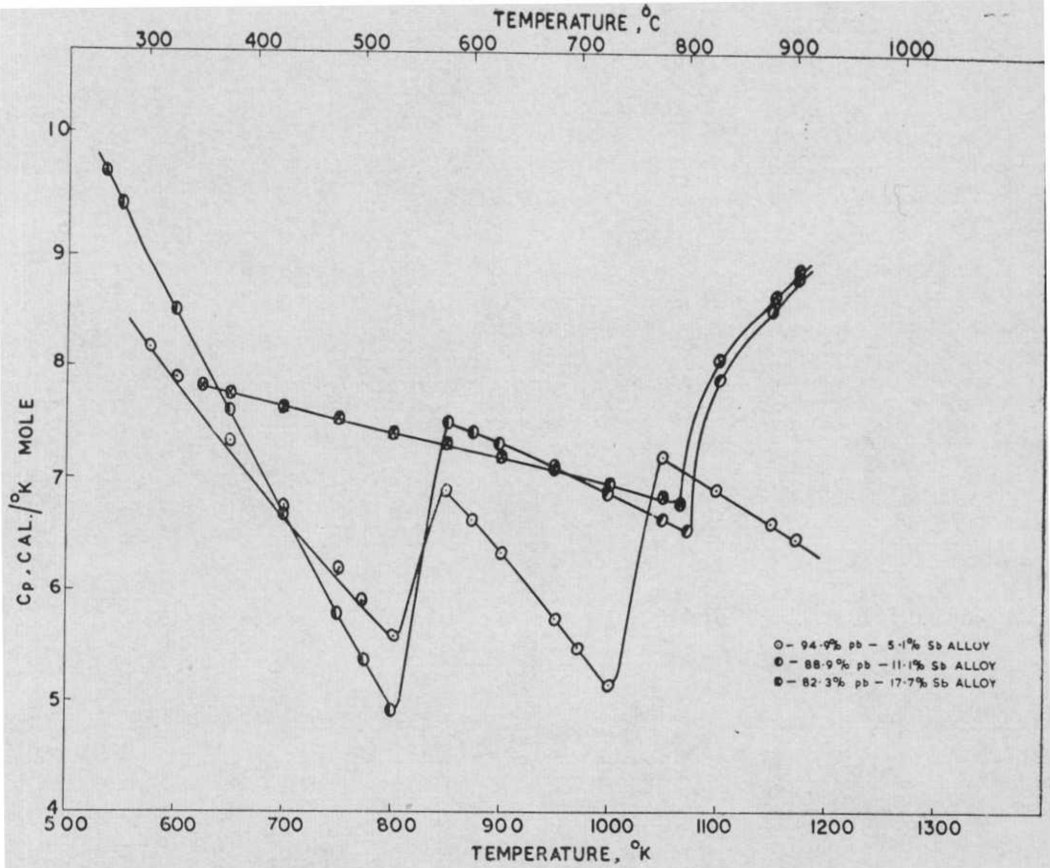


FIG. 46 — HEAT CAPACITIES OF THREE LEAD ANTIMONY ALLOYS IN LIQUID STATE

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. Studies on the strengthening of iron by titanium were continued from the previous year. The age hardening characteristics of solution treated specimens of iron, 2·20; Ti and iron, 3·8 per cent Ti steels were studied at 500° and 600°C. for different periods of ageing between 2-26 hours. Tensile tests on specimens with optimum ageing were also carried out. In order to reinforce the precipitation strengthening due to Fe_2Ti, a number of iron-titanium alloys were made with additions of manganese and silicon, etc., work on which is in progress.

(b) *Titanium in Cast Iron* — The results of the investigation carried out to study the effect of titanium addition up to 2·6 per cent to white cast iron can be summarized as follows:

(i) Titanium first exercises graphitizing effect and the originally white fracture of the titanium-free cast iron

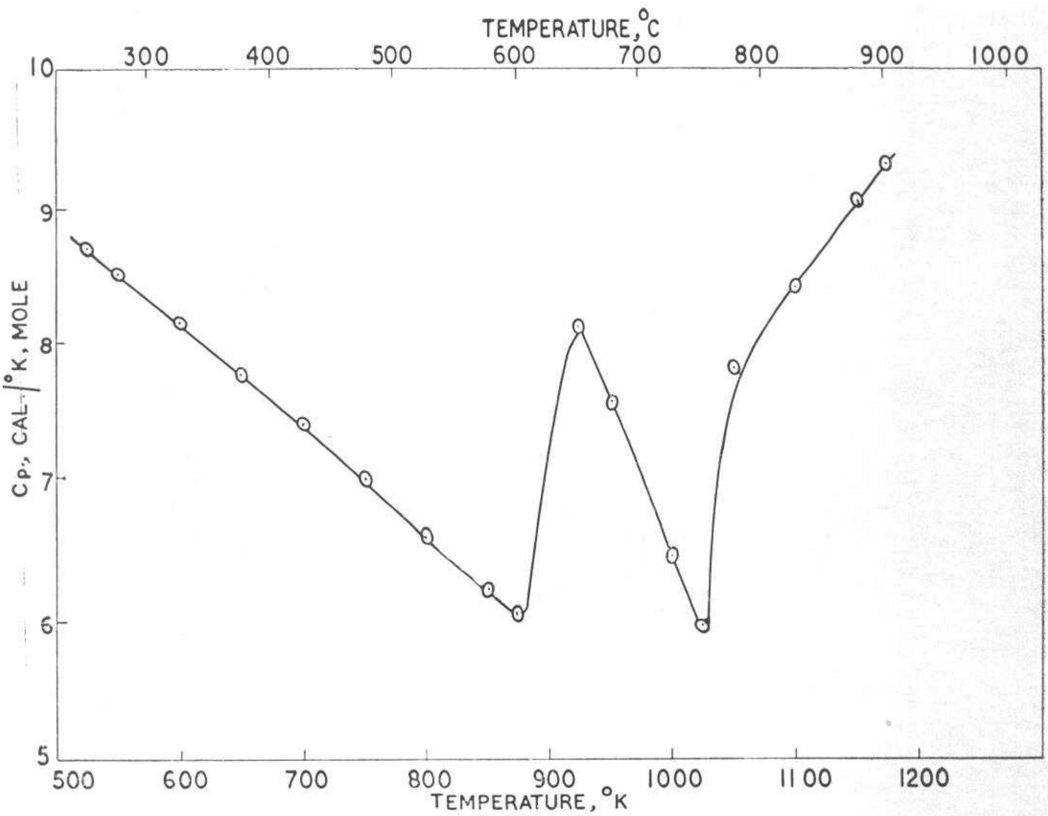


FIG. 47 — HEAT CAPACITY OF Pb, 83·76 PER CENT-Sb, 13·49 PER CENT-Sn, 2·84 PER CENT ALLOY IN LIQUID STATE

became fully grey before again becoming white as the titanium content was increased.

- (ii) The graphitizing tendency of titanium synchronized with a coarser distribution of graphite flakes and a marked tendency towards the formation of rudimentary spherulitic graphite.
- (iii) Titanium carbide could be identified as a separate phase in specimens containing more than 1·2 per cent titanium.
- (iv) Cementite and graphite were usually simultaneously present in all the specimens, though to varying extent.
- (v) A new morphological form of graphite was observed in the thermally cycled titanium bearing grey cast iron samples.
- (vi) Titanium appears to decrease the c/a ratio of graphite.
- (vii) Titanium promotes stacking faults in cast iron.

It is now proposed to study the effect of titanium addition on grey cast iron.

61.0 Marageing 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 marageing, 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.

Work was continued on the Fe-2ONi-3 per cent Al base alloys containing various amounts of manganese. It was observed that the addition of manganese exercises a beneficial effect on marageing. The tensile strength and other mechanical properties were also examined. The effect of replacing nickel by larger amounts of manganese was also studied. It was observed that larger amounts of manganese in general are not conductive of the development of marageing. Work is in progress on more complex steels.

62.0 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 treatment is not completely understood. In particular, the following points remain obscure:

(a) Nature of the nucleii formed in the two processes 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 year under review, the grain sizes of dilute binary magnesium alloys with manganese, nickel, chromium, iron and vanadium were measured in specimens which were cast after preheating to 900°C. (casting temperature 850°C.) and 700°C. in preheated graphite moulds 550°C. Work was then extended to study the grain refinement in magnesium-1 per cent aluminium alloys by the addition of hexachloroethane. Hexachloroethane up to 0·3 per cent was added in different specimens which were cast from a temperature of 780°C. into preheated graphite moulds at 300°C. It was observed that after initial grain refinement, the effect was reversed as the amount of hexachloroethane was increased beyond 0·5 per cent. The effect of holding time in liquid state on cast grain size was also investigated. Grain refinement of lay graphite of magnesium-aluminium alloys from casting temperature of 750° and 780°C. was also investigated. No such reversal of the grain refining tendency was observed but maximum grain refinement was achieved at 0·02 per cent graphite and thereafter there was no change in grain refinement as the amount of graphite was increased.

Work is in progress to study the mechanism of grain refinement in magnesium alloys utilizing the recently developed technique of centrifuging in liquid state. Subsequently it is proposed to study the mechanical properties of some magnesium alloys.

63.0 Transformations in Iron-Manganese System

Work was taken up with a view to study the phase transformation in iron-manganese system. It was reported earlier that in iron-15 per cent manganese alloys, austenite transforms to either alpha or epsilon martensites. If stacking faults are introduced in austenitic range through plastic deformation, gamma to epsilon transformation is promoted. In order to study the possibility of supressing the gamma to epsilon transformation by alloy additions which are known to increase the stacking fault energy, heats were made of iron-15 per cent manganese-1 per cent Al, iron-15 per cent manganese-3 per cent Ni, and iron-15 per cent manganese-1 per cent Ti alloys to study the effect of the additions of aluminium, nickel and titanium on the transformation. Work is now in progress on (1) study of the dilation and ageing characteristics, and (2) the effect of plastic deformation at room and elevated temperatures on transformations.

64.0 Studies on the Phase Equilibrium Diagram of the Binary Mn-Ni, Mn-Zn and Mn-Sn Systems

Due to shortage of nickel in the country efforts are being made to substitute nickel by manganese in most of the alloys where nickel is an essential part. It is, therefore, quite natural and essential that the phase relationship of manganese with other element or elements are to be understood in a more detailed way from the practical point of utilizing these alloys. The literature on the phase relationship of Mn with Ni, Zn and Sn are not very exhaustive and quite a good amount of work is still left. Work was, therefore, taken up on the thermal alloys Mn-Ni, Mn-Zn and Mn-Sn systems. So far four Mn-Ni alloys were prepared under argon atmosphere. The weighed elements were sealed in silica tubes under argon and were melted in a high frequency furnace by placing the sealed tubes inside a graphite block. These alloys were then homogenized. X-ray diffraction photographs were taken of homogenized and other heat treated samples. Heat treatments were given to these alloys to ensure the formation of different phases as per published informations and/or other additional ones.

Due to wide difference of melting points between manganese and tin and manganese and zinc it was difficult to prepare these alloys in conventional ways of melting and casting. Attempts are, therefore, being made to prepare these alloys by sintering these powder compacts at suitable higher temperatures under some protective atmosphere. The chemical composition of the manganese-nickel alloys are given in Table 34. X-ray diffraction analyses of the Mn-Ni alloys showed the presence of some unknown phase/phases apart from the conventional phases reported by the earlier workers shown in Table 35 along with their heat treatments. Accurate lattice parameter measurements from powder diffraction pattern of the alloys, using Nelson and Rilay's methods are being done, both for identification and structural investigation purposes. Photographs were taken in FeK$_\alpha$ radiation and in an 11·46 cm. dia. Philips powder camera using straumanis mounting.

Alloys of Mn-Zn and Mn-Sn are being investigated.

X-ray Diffractometric Work — The following main studies were carried out.

(i) X-ray diffraction studies on Fe-Mn-Al alloys (matrix and the precipitated phases).

Table 34 — Chemical Composition of the Mn-Ni Alloys

Alloy No.	Mn %	Ni %
N 1	47·88	52·12
N 2	53·00	47·00
N 3	32·00	68·00
N 4	61·60	38·40

Table 35 — X-ray Diffraction Phase Analysis Results of the Manganese-Nickel Alloys

Alloy No.	Heat treatments given	Phase identified (X-ray diffraction analyses results)
N 1	As cast	Predominant phase is δ' (f.c.t.) with a=3·732 Å C=3·537 Å and an unknown phase, the structure of which is being investigated
	Annealed at 950°C. for 100 hr furnace cooled	
	Annealed at 950°C. for 50 hr and water quenched	
N 2	As cast	An unknown phase (x+y)
N 2	Annealed at 950°C. for 50 hr and furnace cooled	Predominant phase is δ' with a=3·732 Å, c=3·537 Å and the unknown phase (X)
	Annealed at 950°C. for 50 hr and water quenched	
N 3	As cast	do
N 3	Annealed at 950°C. for 100 hr and furnace cooled	Not yet studied
	Annealed at 950°C. for 50 hr and water quenched	do
N 4	As cast	Predominant phase is δ' (f.c.t.) with a = 3·732 Å, c = 3·537 Å. Other phases are γ (f.c.c.) with a=3·695 Å and δ (b.c.c.) with a=2·974.
N 4	Annealed at 950°C. for 100 hr furnace cooled	Not yet studied
N 4	Annealed at 950°C. for 50 hr and water quenched	do

(ii) X-ray diffraction studies on the precipitation characteristics in Al and Ti treated steels in connection with the problem on grain size control in steel.

(iii) X-ray diffraction studies on kyanites, blast furnace and open hearth slags from different steel projects, carbon and hydrogen reduced illmenite samples, phase relationship studies on the oxide systems of zirconium-calcium, aluminium-niobium.

(iv) X-ray diffraction studies of the phases present in high strength steels developed.

65.0 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 (1) temperature of extrusion, (2) speed of extrusion, and (3) 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.

The conventional X-ray diffraction technique was used in which the axis of the extruded rod was kept horizontal and perpendicular to the X-radiation which was incidentally just on one edge of the specimen. The measurement of the azimuthal positions of the intensity maxima on the semi-debye-scherrer rings obtained in the diffraction photographs, enables to deduce the preferred orientations developed in the extruded rods.

During the period under review, the variation of the texture from the surface to the centre of the extruded rods of duralumin was studied. Duralumin ingots were chill cast and machined to 3 in. dia. by 10 in. long extrusion billets and extruded at 400°C. with exit speed of 5·3 ft/min. to a rod of 0·785 in. dia. The X-ray diffraction photographs taken for 20 positions of the duralumin extruded rod at a pitch of 0·0392 in. (5 per cent of the original diameter of the extruded rod) indicate three zones: (a) surface, (b) intermediate, and (c) central zones.

(a) *Surface Zone* (*Thickness* 0·157 *in.*) — The surface zone has three different textures: [111], [100] and [112]. From the intensity of spots on semi-debye rings it is estimated that the prominent [111] texture is approximately 80 per cent, the [100] texture is approximately 20 per cent and the [112] texture is present only in traces.

(b) *Intermediate Zone* (*Thickness* 0·196 *in.*) — The result of analysis of this zone shows that the [111] texture decreases from about 80 to 50 per cent, the [100] texture increases from about 20 to 50 per cent in the radial direction towards the centre of the extruded rod and [112] texture present, remains in traces only without any appreciable change.

(c) *Central Zone* (*Thickness* 0·392 *in.*) — The central zone consists of 50 per cent of the [111] texture; 50 per cent of the [100] texture and traces of the [112] texture. No change in any of the textures present is noticed with the position in the radial direction towards centre. The textures of three specimens of the above duralumin extruded rods, (1) front, (2) middle and rear portion, have been studied. No appreciable change in textures has been found in the above three specimens.

66.0 Carburizing Characteristics of Grain-refined Steels

The object of the investigation is to study the carburizing characteristics of grain-refined steels. A good carburizing steel should produce on carburizing a hard wear-resisting case and tough core with minimum number of post-carburizing heat treatment cycles. A fine-grained steel, which is capable of maintaining the fine-grain size during carburizing, has the advantages of maintaining a tough core and does not require any post-carburizing heat-treatment for core-refining. The reduction in the number of heat-treatments reduces the cost of the process and risk of distortion.

Compositions of the heats studied are given in Table 36. The heats were forged at 1150°C. into $\frac{3}{4}$ in. square bar. The specimen blanks were normalized at 950°C. and cylinders of $\frac{1}{2}$ in. diameter and 3 in. long were made for carburizing. Pack-carburizing was carried out in a mixture of charcoal and barium carbonate (charcoal, 90; barium carbonate, 10 per cent) at 920°C. for 8 hours. The specimens after carburizing were cooled in the box. Each carburized specimen was cut into three pieces (each 1 in long). Two pieces of each were reheated to 810°C. in argon atmosphere and one piece of each was quenched in oil and other pieces were furnace cooled. Experimental results are given in Table 37.

Table 36 — Composition of Heats Studied

Heat No.	C %	Mn %	Nb %	Ti %	P %	S %
1	0·15	1·00	—	—	0·02	0·03
2	0·10	0·75	0·026	0·040	0·02	0·03
3	0·14	0·30	0·019	0·039	0·02	0·03

67.0 Activity of Carbon in Iron-Chromium 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.

Measurement of activity of carbon in iron-chromium-carbon alloys containing 4·5 per cent chromium was completed at 1000°C. Experiments were carried out with an iron-chromium alloy containing 6·5 per cent chromium at 1000°C. and the typical results obtained are recorded in Table 38.

68.0 Photoelastic Stress Analysis

Photoelastic evaluation of stress concentration factor due to multiple notches was undertaken to obtain qualitative measure of stress relieving effect of neighbouring notches. The theoretical determination of stress concentration factors due to multiple

Table 37 — Results of Experiments

Heat No.	Specimen No.	Hardness (V.P.N.)		Apparent case depth
		Core	Case	
1	1A	232	330	1·2 mm
	1B	143	214	1·2 mm
2	2A	187	432	1·0 mm
	2B	94	190	1·0 mm
3	3A	205	614	1·0 mm
	3B	122	236	1·0 mm

Note: (1) 1B, 2B, 3B are furnace cooling.
(2) 1A, 5A, 6A are quenched in oil.

Table 38 — Results of Equilibration at 1000°C. with Fe-Cr 65 Per Cent Alloy

Expt No.	Wt % C	p^2CO/pCO_2 in atmospheres	Activity of carbon aCr C
1	0·18	3·094	0·022
2	0·22	3·06	0·0223
3	0·23	3·558	0·026
4	0·265	4·834	0·035
5	0·453	7·108	0·051
6	0·23	4·55	0·033
7	0·248	6·097	0·044
8	0·478	12·02	0·087
9	0·816	20·68	0·1508
10	1·271	31·90	0·232
11	1·485	46·40	0·338
12	1·322	46·58	0·3394
13	0·953	35·46	0·251
14	1·193	35·72	0·2602
15	1·466	29·19	0·2127
16	3·309	66·43	0·4842
17	3·433	69·92	0·5095
18	2·903	74·25	0·5412

notches with various spacing and relative depth of the notches is a difficult problem and experimental data is too scanty. Photoelastic evaluation of stress concentration factor due to multiple notches with different spacing and relative depth was undertaken to provide further data to assist the designers in utilizing the stress relieving effect of notches.

Stress concentration factors due to three notches in a beam under pure bending have been 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. The work was completed.

69.0 Production of Basic Refractories from Indigenous Resources

(i) *Development of Refractories from Almorah Magnesite* — Magnesite, a principal raw material for the basic refractories, occurs in the Almorah region of Uttar Pradesh and its suitability for the production of magnesite refractories was investigated earlier in this Laboratory. The present work was taken up to assess the suitability of Almorah magnesite for the production of chrome-magnesite and magnesite-chrome refractories. The magnesite from two sectors of Almorah region (1) Agargirichchina and (2) Dewaldhar was taken up for the study. Magnesite-chrome and chrome-magnesite compositions were prepared using Tullock's chrome ore. In the first phase of the project Agargirichchina magnesite was taken up. The physical properties have been reported earlier. In the second stage Dewaldhar magnesite was taken up.

Dewaldhar magnesite as compared to Agargirichchina contains less impurities. The chemical analysis is given in Table 39. Two chrome-magnesite compositions (70: 30 and 60: 40) and one magnesite-chrome composition (85: 15) were prepared. The test specimens were then fired at 1400°, 1500°

Table 39 — Chemical Analysis of Magnesite Fired at 1650°C.

	Per Cent
MgO	90·20
CaO	1·91
Al_2O_3	0·36
SiO_2	2·46
Fe_2O_3	5·28
Moisture	0·06
L.O.I.	0·34

and 1600°C. and their physical properties were studied (Table 40). In the first phase of the work it was observed that the texture of the fired specimens was not good. In order to improve the texture some selected compositions were made using −10, −18 and −36 mesh B.S.S. chrome ore and fine magnesite. The samples made of these compositions were then fired at different temperatures and their physical properties are being studied.

(ii) *Studies on Sintering and Stabilization of Dolomite* — Dolomite from Nattam area was studied for assessing its suitability as a fettling material for tar-bonded bricks and for making completely stabilized bricks.

The dolomite was found to be coarsely crystalline with siliceous impurities. By fine grinding and addition of mill scale, magnesite-dolomite clinker suitable for use in fettling in the open hearth furnace as well as tar-bonded dolomite for L-D furnace lining can be produced by firing the mix at a temperature above 1600°C. Bulk density, porosity, hydration resistance and other refractory properties of this fired brick are comparable to other basic refractory products. By suitable addition of dunite to this dolomite and firing, completely stabilized dolomite refractory can also be produced from this raw material.

70.0 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.

(i) *Development of Dense Carbon Aggregate from Imported Anthracite Coke* — During the period under review work was done on formulating compositions of raw mixes based on Gauhati coke on bench scale which consisted of compounding mixes, their briquetting, baking and physical

Table 40 — Physical Properties of Chromite and Magnesite Compositions Fired at Different Temperatures

Composition	Sp. No.	Specific gravity		Bulk density			App. porosity %			Linear change after firing (%)			R.U.L.
Temp. of firing, °C.		1500	1600	1400	1500	1600	1400	1500	1600	1400	1500	1600	1600
70% chromite and 30% magnesite	M_{30}	4·006	4·030	3·221	3·283	3·280	19·91	18·86	19·53	0·074 contr.	0·187 exp.	0·44 contr.	ta 1700°C. te 1820°C.
60% chromite and 30% magnesie	M_{10}	3·951	3·999	3·166	3·204	3·228	19·59	19·27	18·56	0·4 contr.	0·68 contr.	0·5 contr.	ta 1630°C. te 1800°C.
85% magnesite and 15% chromite	M_{85}	3·681	3·683	2·882	2·950	2·934	22·01	20·40	19·35	0·62 contr.	1·43 contr.	1·71 contr.	ta 1650°C. te 1860°C.

testing. For this purpose, three binders, a road tar from M/s. Shalimar Tar Products Ltd, a pitch from Bhilai Steel Plant and bitumen supplied by M/s. India Carbon were utilized as binders. Based on the knowledge previously obtained, the petroleum coke samples were crushed and graded and a number of compositions were formulated, the range for which are given in Table 41.

Table 41 — Powder, Binder Percentages, Mixing and Fabricating Details of Various Composition

Grain size of petroleum coke	22 mesh BSS to −72 BSS
Binder percentages	20 to 30% on the coke
Mixing temperature	90 to 150°C.
Mixing time	10 to 20 min.
Forming pressure	12000 to 1600 lb./sq. in.
Calcination temperature	1300°C.

Physical properties of these compacts were studied and based on these figures, the optimum grain size of petroleum coke, the best binder (both from the point of view of availability and economics), optimum binder percentage and mixing temperatures and the optimum forming pressure and heat treatment schedule were all determined. Physical properties of dense aggregates made from Gauhati coke are given in Table 42. Fig 48 shows a photograph of dense aggregates.

(ii) *Characteristics of Certain Indian Petroleum Cokes* — During the period under review work was conducted to examine the effect of variation in calcination temperature on the physical properties as well as the cokes' suceptibility to oxidation. The materials were crushed in jaw and roll crusher and graded to obtain −10, 18 mesh B.S.S. powders. These were then calcined in a tube furnace in nitrogen atmosphere at temperatures in the range of 400° to 1100°C. at intervals of 100°C. Calcination at 1300° and 1600°C. were also done by keeping the material packed in graphite crucibles in a down draft kiln held at the desired temperatures. The following properties of the calcines were determined.

(a) Proximate analyses
(b) True density
(c) Electrical resistivity
(d) D.T.A. in oxygen atmosphere

(a) Proximate analyses: Volatile matter determination was done in a thermal balance at 925°C. with N_2 atmosphere. For ash content, oxidizing atmosphere was maintained at 925°C. Table 43 shows the proximate analyses.

(b) Specific gravity (true density) was determined according to the ASTM standard procedure for refractory materials using kerosene. Table 44 gives the specific

Table 42 — Physical Properties of Dense Aggregate from Gauhati Coke

Aggregate	Binder	Porosity %	Bulk density gm./cc.	App. sp. gr.	Crushing strength lb./sq. in.	Elect. resistivity ×10³ ohm/cm.³
No. 1	A	19·8	1·61	2·02	8,500	
	B	19·0	1·63	2·02	10,200	
	C	19·6	1·63	2·02	13,000	In the order of 11 to 14
No. II	A	23·3	1·55	2·02	5,000	
	B	23·7	1·56	2·05	8,000	
	C	25·6	1·52	2·05	7,400	
No. III	—	31·1	1·41	2·05	5,000	

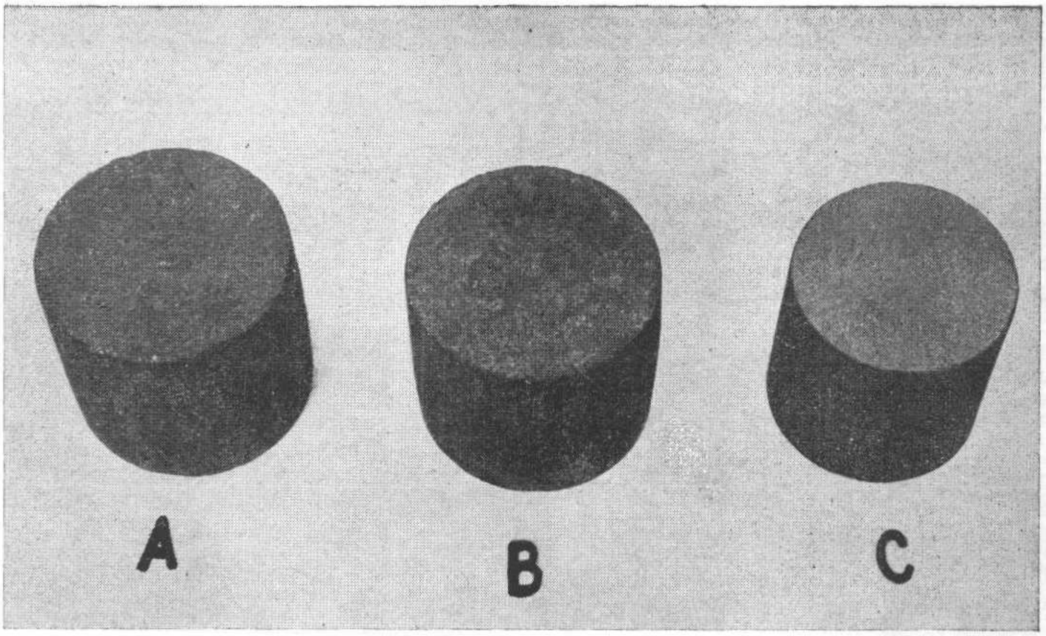


FIG. 48—(A) DENSE AGGREGATE OF DIGBOI PETROLEUM COKE; (B) DENSE AGGREGATE OF LOW ASH ANTHRACITE; AND (C) DENSE AGGREGATE OF GAUHATI PETROLEUM COKE

gravities of different samples calcined at different temperatures.

(c) Electrical resistivities were determined with the apparatus built in the Laboratory and are given in Table 45. Fig. 49 shows the plot of specific gravity against the logaritham of resistivity.

(d) Differential thermal analysis was used as a qualitative method for assessing the comparative resistances of the raw cokes and their calcines to oxidation. Fig. 50 shows a graph, the specific gravity being plotted against the maximum temperature of oxidation resistance. It shows that the maximum temperature of oxidation increases with the increasing specific gravity within a certain range. Table 46 furnishes the data from differential thermal analysis.

Table 43 — Proximate Analysis of Petroleum Coke

Calcination temperature °C.	Petroleum coke (Digboi)				Petroleum coke (Gauhati)			
	Moisture %	V.M. %	Ash %	Fixed carbon %	Moisture %	V.M. %	Ash %	Fixed carbon %
Uncalcined	0·300	8·800	0·199	90·701	0·410	7·400	0·204	91·986
400	0·730	8·300	0·219	90·751	0·507	6·780	0·216	92·497
600	2·100	5·400	0·228	92·272	1·530	5·020	0·230	93·220
800	0·380	1·280	0·240	98·100	0·572	0·842	0·265	98·321
1000	0·190	0·200	0·255	99·355	0·240	0·500	0·282	98·979
1300	0·140	Nil	0·268	99·592	0·168	Nil	0·296	99·536
1600	0·090	Nil	0·276	99·634	0·124	Nil	0·380	99·496

Table 44 — Specific Gravities of Calcined Samples of Petroleum Coke

Calcination temperature °C.	Petroleum coke (Digboi)	Petroleum coke (Gauhati)
Uncalcined	1·335	1·327
400	1·364	1·342
500	1·391	1·363
600	1·512	1·494
700	1·692	1·669
800	1·797	1·765
900	1·885	1·885
1000	1·975	1·939
1100	2·015	1·964
1300	2·030	1·992
1600	2·110	2·084

In connection with this investigation the following apparatus were fabricated.

1. Calcination furnace used for calcining the coke samples up to a temperature of 1100°C. (Fig. 51).

2. D.T.A. sample holder (Fig. 52) for conducting the thermal analyses of carbonaceous samples in N_2 atmospheres as well as to measure the suceptibility towards oxidation resistance in oxidizing atmosphere.

Table 45 — Electrical Resistivities of Samples of Petroleum Coke

Calcination temperature °C.	Petroleum coke (Digboi)		Petroleum coke (Gauhati)	
	Resistivity ohm/in.^{3}	Resistivity ohm/cm.^{3}	Resistivity ohm/in.^{3}	Resistivity ohm/cm.^{3}
Uncalcined	—	—	—	—
400	—	—	—	—
500	$0{\cdot}34\times10^{-6}$	$0{\cdot}879\times10^{-6}$	$0{\cdot}71\times10^{-6}$	$1{\cdot}8\times10^{-6}$
600	40840×10^{-3}	10365×10^{-3}	201500×10^{-3}	511800×10^{-3}
700	$127{\cdot}6\times10^{-3}$	$324{\cdot}6\times10^{-3}$	$241{\cdot}1\times10^{-3}$	$628{\cdot}6\times10^{-3}$
800	$30{\cdot}66\times10^{-3}$	$77{\cdot}64\times10^{-3}$	$35{\cdot}95\times10^{-3}$	$90{\cdot}20\times10^{-3}$
900	$16{\cdot}39\times10^{-2}$	$41{\cdot}63\times10^{-3}$	$14{\cdot}53\times10^{-3}$	$35{\cdot}62\times10^{-3}$
1000	$11{\cdot}76\times10^{-3}$	$29{\cdot}84\times10^{-3}$	$13{\cdot}50\times10^{-3}$	$34{\cdot}27\times10^{-3}$
1100	$10{\cdot}04\times10^{-3}$	$25{\cdot}49\times10^{-3}$	$11{\cdot}37\times10^{-3}$	$28{\cdot}88\times10^{-3}$
1300	$7{\cdot}34\times10^{-3}$	$18{\cdot}65\times10^{-3}$	$7{\cdot}91\times10^{-3}$	$20{\cdot}1\times10^{-3}$
1600	$5{\cdot}43\times10^{-3}$	$13{\cdot}8\times10^{-3}$	$6{\cdot}89\times10^{-3}$	$17{\cdot}5\times10^{-3}$

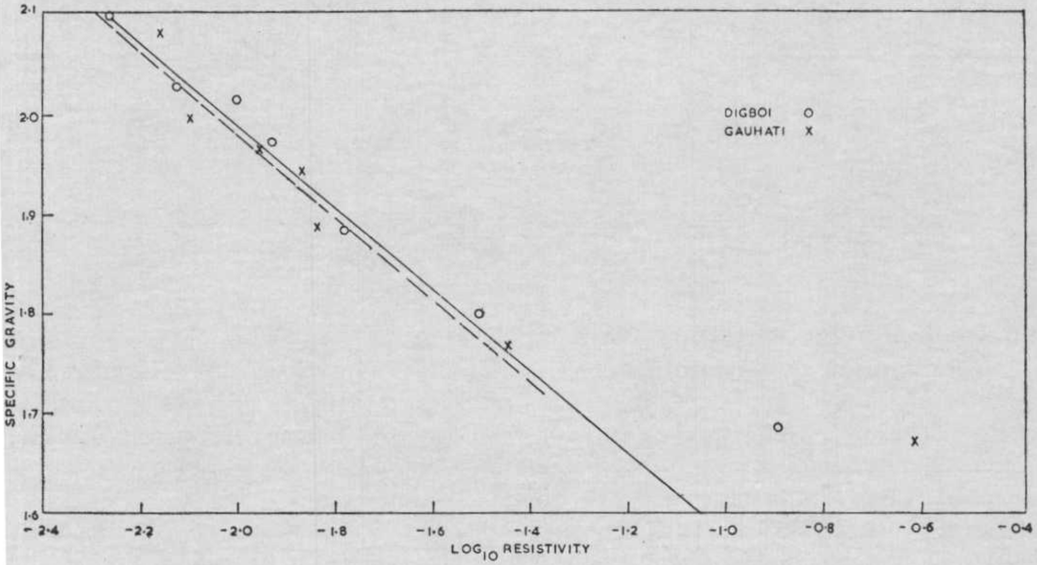


FIG. 49 — PLOT OF SPECIFIC GRAVITY VS LOG_{10} RESISTIVITY

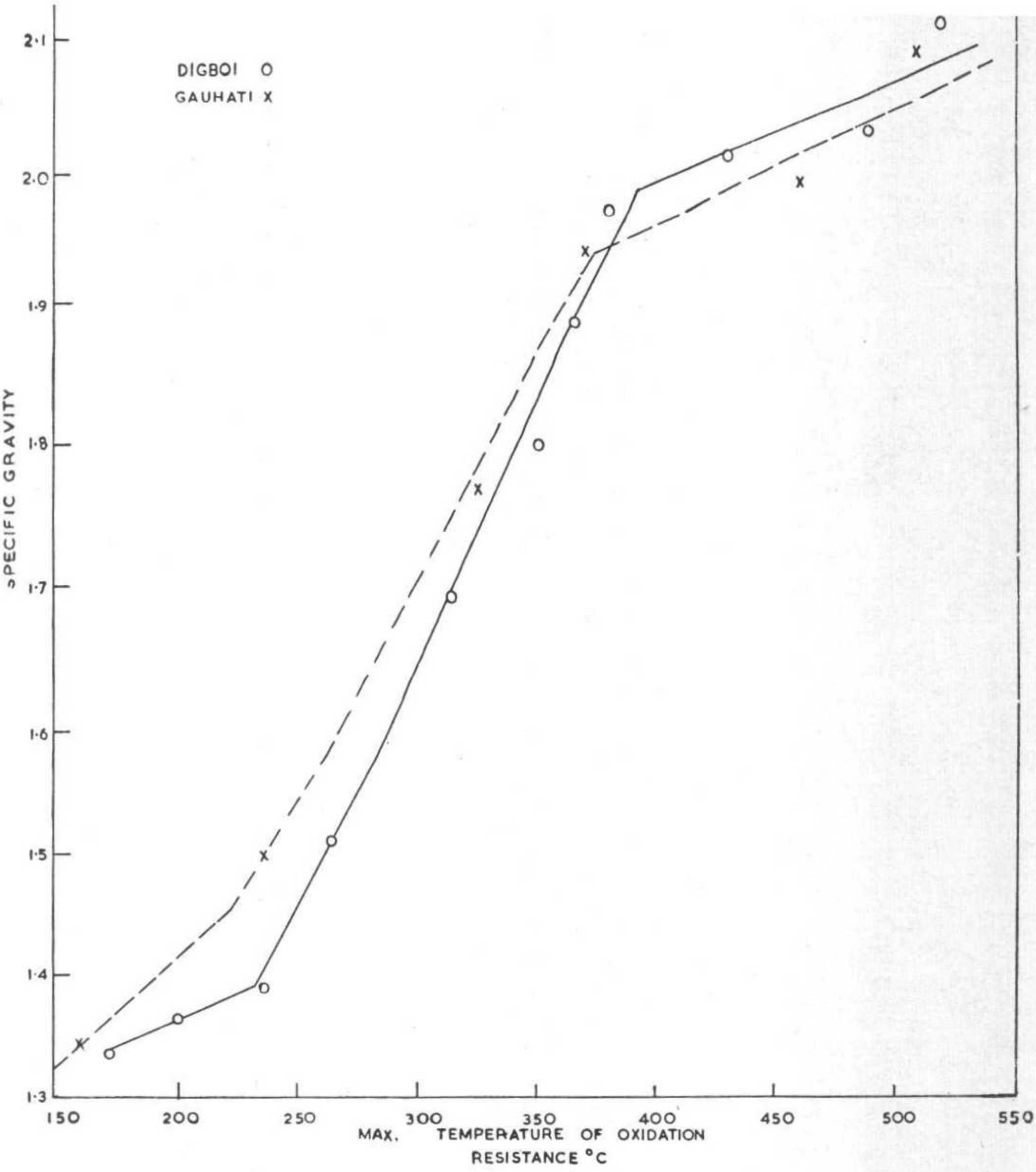


FIG. 50 — PLOT OF SPECIFIC GRAVITY VS MAXIMUM TEMPERATURE OF OXIDATION RESISTANCE °C.

71.0 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 Riasi Tehsil in the Udhampur District of Jammu Province and the resources are considered to be large. Samples of clays collected from different pits in this area were received from Director of Geology & Mining, Jammu & Kashmir Government, Jammu; for investigation for their suitability in the manufacture of fireclay refractories.

Studies were made on the physico-chemical properties of high Al_2O_3 type of clays associated with bauxitic deposits of

Table 46 — Data from Differential Thermal Analysis of Samples of Petroleum Coke

Calcination temperature °C.	Petroleum coke (Digboi)		Petroleum coke (Gauhati)	
	Oxidation starts at temp. °C.	Oxidation finishes at temp. °C.	Oxidation starts at temp. °C.	Oxidation finishes at temp. °C.
Uncalcined	170	620	150	660
400	200	650	160	670
500	235	660	—	—
600	265	670	235	710
700	315	750	—	—
800	350	815	325	800
900	365	840	—	—
1000	380	870	370	850
1100	430	880	—	—
1300	490	890	460	930
1600	520	945	510	940

Jammu. The results indicated that these clays could be used for the manufacture of super-duty fireclay refractories. On the basis of their mineralogical constitution, these clays were classified into two categories, (1) kaolinitic clays, and (2) clays with kaolinite and diaspore intimately mixed in varying proportions.

Out of these, four kaolinitic type clays were investigated in the present study to find out their suitability for the manufacture of super-duty fire-clay refractories of high grog type. The chemical analyses of these clays are given in Table 47. The effects of grog firing temperature, amount of bonding clay employed and final brick firing temperature on the physical properties of the high grog compositions made from these clays were studied. The grog making temperatures examined were 1300° and 1500°C. The bond clay content was varied between 5 and 20 per cent and the brick firing temperatures considered were 1350° and 1500°C. Four mixes from each clay were made with the ratio of grog and clays as shown in Table 48.

From the trend of results of the various physical properties of the above compositions it was found that these clays could be utilized for the manufacture of super-duty fireclay bricks by (1) firing the grog at 1500°C., (2) using bond clay between 10 and 25 per cent, and (3) firing the bricks at 1500°C. The results on physical properties of some selected compositions are given in Table 49.

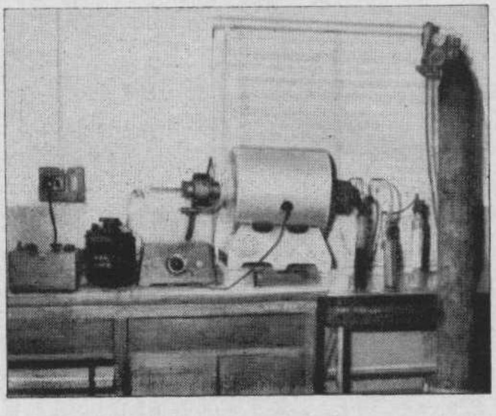


FIG. 51 — APPARATUS FOR CALCINING COKE SAMPLE

72.0 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 which do not have binding properties in the raw state without the addition of binders which alter chemical composition and more or less adversely affect their high-temperature properties. Pure oxides like alumina, magnesia and thoria are examples of such refractories. 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.

As a result of extensive bench trials, details for a process for forming stable and

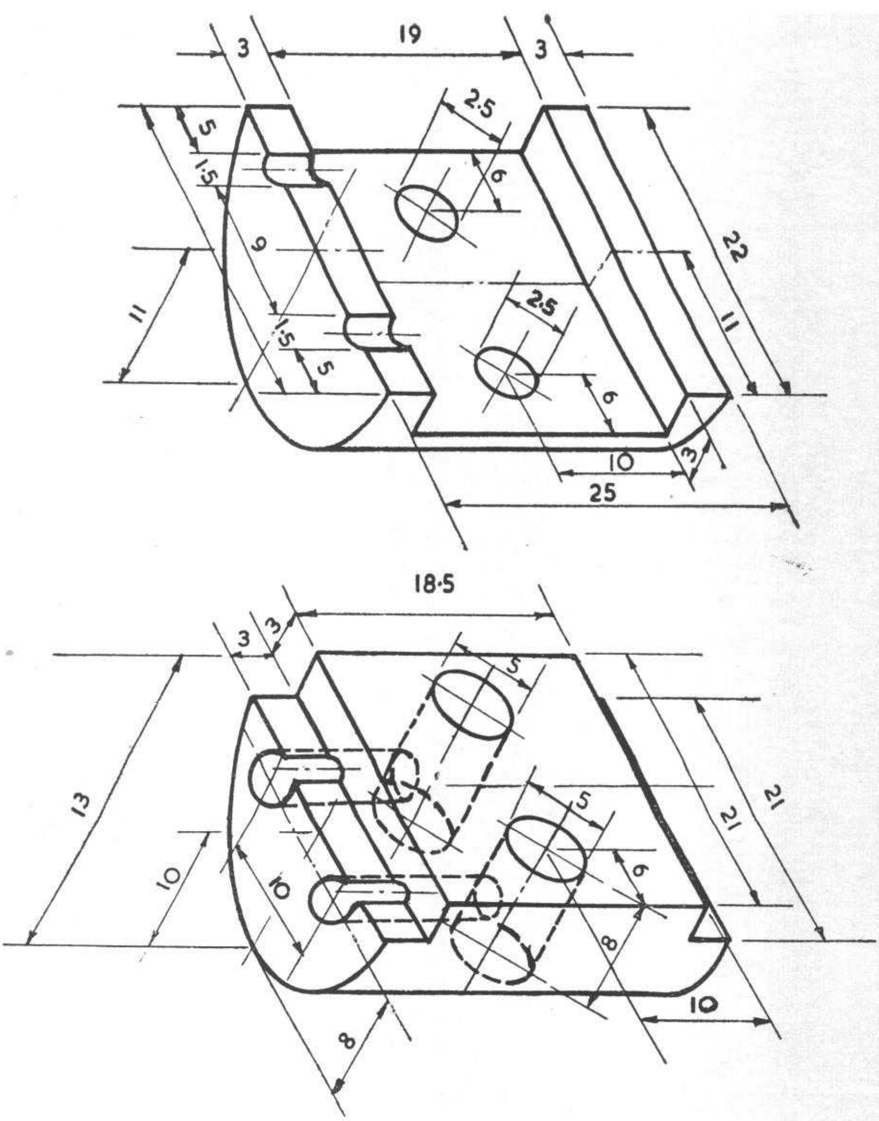


FIG. 52 — D.T.A. SAMPLE HOLDER

castable suspensions from non-plastic refractory materials like sillimanite, kyanite, etc., were worked out and a patent application has been filed. During this period, Orissa kyanite lumps with 54 per cent Al_2O_3 were treated. The work consisted of forming stable and castable suspensions of kyanite, casting test pieces and finding out their fired properties. After preliminary crushing, the raw material was ball-milled for 80 hours and the particle size distribution was determined. Stable slips from this material were formed and their fluidities, casting rates and stabilities determined. Some of the typical values obtained are as follows:

Water content in slip — 30%

Fluidity by mariotte tube. Flow time in seconds from 30 cc. mark to 60 cc. mark — 28·0

Sp. gravity of the slip — 2·00 gm./cc.

Casting rate — 0·30-0·4 cm./min.

Stability expressed as change in solids content at the top of a stoppered glass

Table 47 — Chemical Analysis of the Clay from Jammu

Clay No.	Moisture %	L.O.I. %	SiO_2 %	Al_2O_3 %	Fe_2O_3 %	TiO_2 %	CaO %	MgO %	Na_2O %	K_2O %	Total fluxes	Ratio SiO_2/Al_2O_3	P.C.E
C_3	0·32	12·88	43·84	38·31	1·44	1·05	Tr	0·180	0·80	0·69	4·16	0·145	34
C_5	0·32	12·30	43·48	38·84	0·76	1·20	Tr	0·109	0·80	0·01	4·88	1·119	34
C_6	0·20	13·60	42·40	39·98	1·92	1·70	Tr	0·140	0·45	Tr	4·21	1·060	34
C_9	0·16	14·88	44·80	36·80	1·52	1·10	Tr	0·540	0·32	0·40	3·88	1·214	34

Table 48 — Mixes Made with Different Proportion of Grog and Clay

	C_3-5 to C_9-5	C_3-10 to C_9-10	C_3-15 to C_9-15	C_3-20 to C_9-20
Grog	95	90	85	80
Bond Clay (C_5)	5	10	15	20

tube after 24 hours — Original 70%, after 24 hours 60%

Average particle size — 7·0

Some casts were fired at cone 18, i.e. 1500°C. and cone 30 (about 1640°C.) The fired properties are given in Table 50.

Some more material was treated in ball mill to obtain finer powders. The casting properties of the fired test pieces are being studied.

73.0 Studies on Development of High-Alumina Cements

The study on high alumina cements carried out during the year, was mostly confined to examining the effect of the following factors on the properties of cement compositions prepared by sintering process:

(i) Methods of batch preparation

(ii) Temperature of sintering

(iii) Soaking time

With regard to batch preparation, the effect of both wet and dry grinding of the batch on the sintering behaviour of the cements were studied. The results showed that wet grinding produced a homogeneous mix with uniform sintering of the briquettes. Accordingly, further studies were carried out on wet ground batch materials. The effect of temperature of sintering on the bulk density and on the free-lime test of sintered cements made from one of the compositions is given in Table 51. The bulk density of the compacts was taken as a measure of sintering. The completion of reactions between lime and bauxite was

Table 49 — Physical Properties of Some Selected Compositions of Clay

Clay used	C_3	C_5	C_6	C_6	C_9
Grog firing temperature, °C.	1500	1500	1300	1500	1300
Brick firing temperature, °C.	1500	1500	1500	1500	1500
Grog, %	85	85	80	95	80
Bond clay, %	15	15	20	5	20
Linear shrinkage, %	1·373	1·473	0·39	1·92	0·67
Apparent porosity, %	17·83	18·27	15·60	20·10	14·51
Bulk density	2·22	2·20	2·18	2·11	2·296
Cold compression strength lb./sq. in.^{2}	3232·0	5060·0	14300·0	4410·0	9425·0
MOR lb./sq. in.^{2}	1398·0	1850·0	1886·0	792·0	2361·0
Reheat shrinkage volume	3·29	2·81	2·38	2·98	1·77
Refractoriness under load	ta-1560 te-1700	ta-1580 te-1740	ta-1530 te-1680	ta-1580 te-1700	ta-1520 te-1700
Spalling	30	30	30	30	30

assessed by White's free-lime test. The results show that the optimum temperature for sintering of the cement compositions is between 1300° and 1400°C.

After determining the optimum temperature of sintering for the various compositions, the effect of soaking time on the properties of cements was studied. Table 52 gives the bulk densities of sinters prepared with different periods of soaking at 1350°C. and the cold compression strength of final cements determined according to B.S. specification. The strength values showed an increase with increase in soaking period from two to twelve hours. For the sake of comparison, the cold compression strength of cement prepared from the same composition by the melting method is also given in the same table. It is seen that the strength of the sintered cement prepared with twelve hours soaking has almost approached the strength of the cement prepared by melting method.

Table 50 — Properties of Some Fired Specimen

Firing temp. °C.	Apparent porosity %	Bulk density g./c.	Linear shrinkage %
1500	33	1·9	4·0
1640	0·2	2·7	8·0

Tabel 51 — Effect of Temperature of Sintering on Bulk Density and Free-lime Test

Sample No.	Temperature of firing °C.	Bulk density	White's free lime test
SM_{6-6}	1200	n.d.	positive
,,	1250	n.d.	do
,,	1300	n.d.	slightly positive
,,	1350	1·310	negative
,,	1400	1·788	do

A pilot scale continuous furnace for melting high alumina cement designed and fabricated in this laboratory is shown in Fig. 53. Some preliminary experiments on this furnace gave a continuous production of cement at the rate of about 20 kg./hr when coke oven gas and preheated air are used.

Table 52 — Bulk Density of Sinter at Various Soaking Time and Cold Compression Strength of Cement

Sl No.	Soaking time	Bulk density	Cold compression strength
1	2	1·460	2529
2	4	1·478	2930
3	6	1·788	3150
4	8	1·794	5096
5	10	1·790	5077
6	12	1·798	6177
7	(melted cement)	—	7300

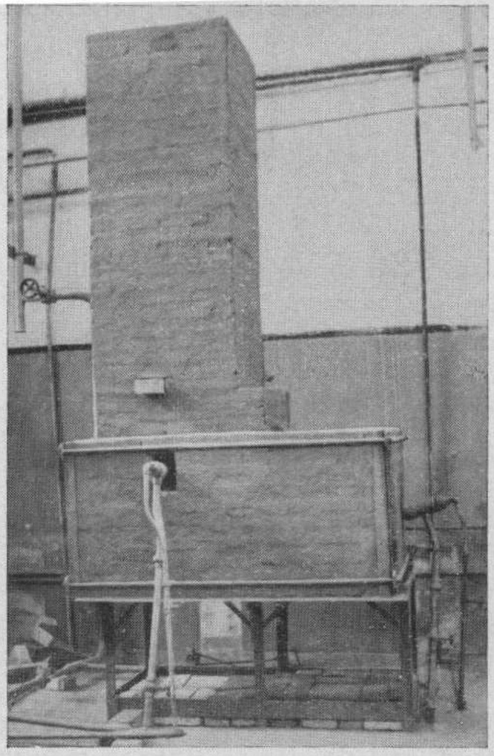


FIG. 53 — PILOT FURNACE FOR MELTING HIGH ALMUINA CEMENT, DESIGNED AND FABRICATED AT THE NATIONAL METALLURGICAL LABORATORY

74.0 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 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 twofold, 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.

Starting with Rajhara clay and grog of the same clay of various grain sizes, the mixes given in Table 53 were prepared and their packing characteristics and extrudabilities were studied.

Table 53 — Packing Density and Extrudability of Different Mixes

Mix No.	Grog %	Grain size (mesh) BSS	Packing density gm./cc.	Extrud-ability
1	50	−5 mesh	1·648	Extrudable
2	60	,,	1·663	,,
3	70	,,	1·650	,,
4	80	,,	1·690	Difficult
5	50	−10 mesh	1·640	Extrudable
6	60	,,	1·649	,,
7	60	,,	1·670	,,
8	80	,,	1·720	Difficult
9	50	18 mesh	1·640	Extrudable
10	60	,,	1·659	,,
11	70	,,	1·710	,,
12	80	,,	1·730	Difficult
13	50	−36 mesh	1·631	Extrudable
14	60	,,	1·636	,,
15	70	,,	1·659	Extrudable
16	80	,,	1·800	Dfficult
17	50	*−72 mesh	1·580	Extrudable
18	60	,,	1·583	,,
19	70	,,	1·630	,,
20	80	,,	1·640	Difficult

*This fraction was obtained by pot milling −36 mesh BSS size grog. Entirely different grain size distribution was obtained. The trend in packing density with increasing grog percentage was, however, comparable.

It was observed that addition of water increased the packing density almost in the same order as dry values. Clay-grog samples are being prepared for extrusion. In connection with the standardization runs of erosion test apparatus, it was found that with a gas fired furnace, the melting period for steel before the test would be about four hours. A temperature of about 1600°C. could be reached in the furnace fitted with one tangential burner. Size of the support for crucible that contains molten steel was also standardized. Some nozzle samples were tested in this way.

75.0 Determination of the Properties of Indigenous Nozzles and Stoppers

Refractories are heterogeneous materials and devising standards for these on the basis of a few physical tests is quite difficult. This is particularly so in the case of nozzles and stopper heads used in the teeming of molten steel. A number of variables influence the performance of these refractories and their textural as well as other properties together form only a few of these variables. It is, however, necessary that some workable specification is to be drawn up so that it could serve at least as guide line for manufacturers and consumers.

With the above end in view the Indian Standards Institution have requested the National Metallurgical Laboratory to test samples of nozzles and stoppers produced in this country. A few imported varieties have also been included as these are at present being used by some of the steel plants.

(i) *Nozzles* — Initially it had been agreed upon that the properties to be determined would be (a) chemical analysis, (b) refractoriness, (c) refractoriness under load, (d) apparent porosity, bulk density and apparent specific gravity, (e) permanent linear change, (f) spalling resistance, (g) cold crushing strength, and (h) permeability. Standard methods of testing was used to conduct the above tests. The following observations were made:

(a) Chemical analyses: The variation in alumina content of the nozzles was from 51 to 31·92 per cent. Compared to foreign samples, the Al_2O_3 content is consistently high. Variation in flux content of 5 to 11 per cent is comparable to that of the foreign samples. High alumina content indicated incorporation of bauxite or other high alumina raw materials.

(b) The P.C.E. was in the range of orton cone 29-30 which when compared to the foreign samples is on the higher side. Ta values were also on the higher side whereas lower values are preferred in foreign countries.

(c) Permanent linear change on reheating varied markedly from foreign samples as they showed shrinkage or negligible expansion. Judged this way, the Indian nozzles did not appear to have good pyroplastic properties.

(d) Apparent porosity, bulk density and permeability: In general the apparent porosity was higher than 24 per cent.

(e) Cold crushing strength compared favourably with that of foreign samples.

(f) In case of spalling resistance, no inference could be drawn as the test procedure was inadequate and needed modification. The failure by spalling is shown in Fig. 54.

(ii) *Stoppers* — This part deals with only fireclay and high alumina type stoppers as graphite based stoppers are presently not being produced in any sizeable quantity in the country. In the case of stoppers considerable importance is given to good spalling resistance. Other important properties are high refractoriness, good refractoriness under load and good abrasion resistance at high temperature. The experimental procedure adopted was the same as in the case of nozzles and the following results were obtained.

(a) Chemical analysis: The alumina content was 31-35 per cent and should be considered quite satisfactory. The flux contents varied from 5 to 12·7 per cent. The P.C.E. values were in the range of orton cone 29-30 viz. the same as the nozzles. This is not a very desirable feature as the stopper head should be more refractory than the nozzles at the steel pouring temperatures. By bringing down the flux content, it may be possible to improve refractoriness. Refractoriness under load was not determined as test samples could not be drilled out of the stoppers.

(b) Permanent linear changes were remarkable for the expansion, the maximum recorded being 8·3 to 9·5 per cent. The

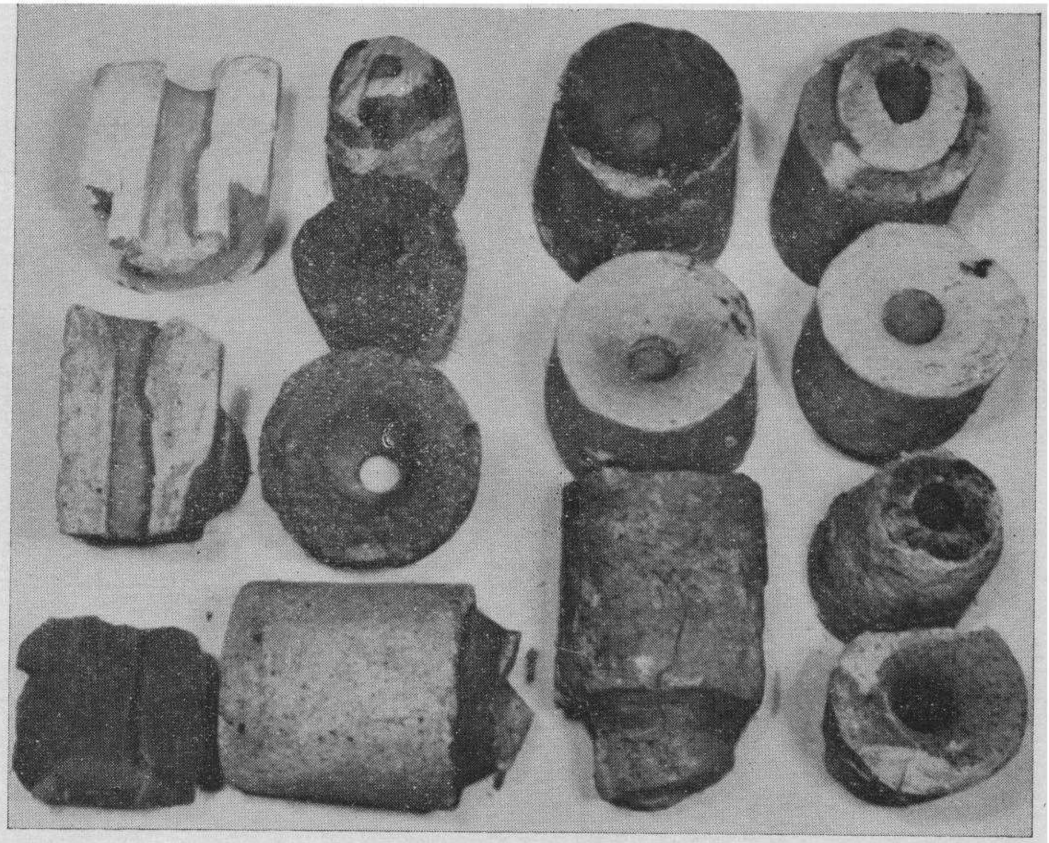


FIG. 54 — PHOTOGRAPH SHOWING THE APPEARANCE OF DIFFERENT NOZZLES AFTER THE SPALLING TEST ACCORDING TO B.R.R.A. METHOD

foreign stopper tested in the series showed an expansion of the order to 4 to 5 per cent.

(c) Apparent porosity varied over wide range, viz. from 13 to 35 per cent. A very low porosity is to be avoided from the point of view of spalling resistance as according to Louise Halm a porosity lower than 20-23 per cent may be hazardous.

(d) Bulk densities appeared to be high enough while the permeabilities were quite low. Both of these are comparable to those of foreign samples.

(e) Cold crushing strengths were quite adequate. Spalling resistance of almost all the stoppers fell within the range of 1-3 cycles. Fig. 55 indicates the manner of failure in general. A better spalling resistance is essential for the satisfactory performance of stoppers.

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

The A.S.T.M. apparatus based on the standard test procedures C201-47, C202-47 and C182-47 is widely used for determining thermal conductivity of refractory materials. A less used apparatus is that originally designed by Blakeley and Cobb —

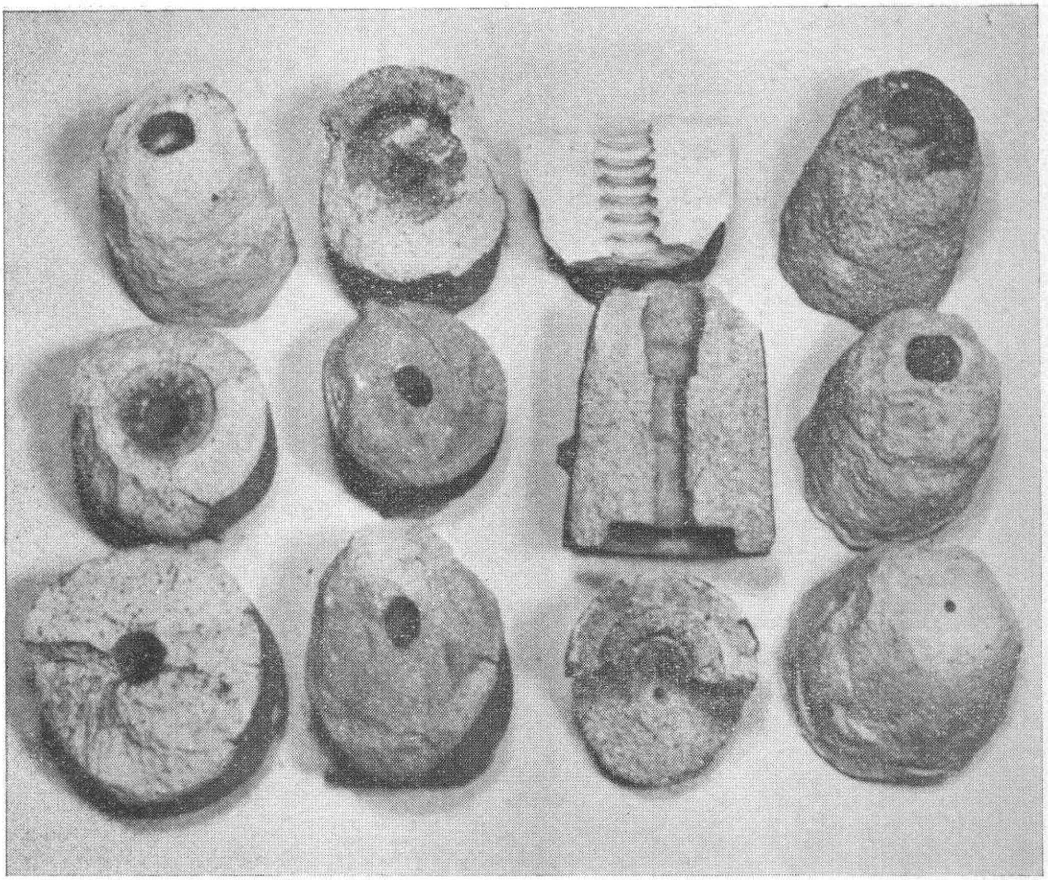


FIG. 55 — PHOTOGRAPH SHOWING THE EXTENT OF SPALLING OF THE DIFFERENT STOPPERS AFTER THE SPALLING TEST ACCORDING TO B.R.R.A. METHOD

both depend on the same basic principles but the A.S.T.M. apparatus is more elaborate and can be used up to hot face temperatures of the order of 1300°-1350°C. With the Blakeley & Cobb apparatus, as in use at present, hot face temperatures are limited by the resistance-elements employed and are never usually employed beyond 1000°C. In the A.S.T.M. apparatus, a water calorimeter is employed to measure the heat transmission. Under favourable conditions and availability of spares and good maintenance, the A.S.T.M. apparatus is a very reliable instrument and can be expected to yield repeatable results. In India where most of the spare parts are to be imported and maintenance is difficult, keeping such an apparatus in a good operational condition is an arduous job. Its initial cost also is considerably high. The Blakely-Cobb apparatus is much simpler and cheaper and can be built from components which are easily available in India, making its maintenance very easy. Moreover, the former needs a highly skilled person to conduct the determinations as control has to be exercised over a number of variables whereas the latter can be handled by less skilled personnel.

A study was made on both types of the apparatus to determine how far the results obtained in each apparatus are comparable, so that recommendations could be made to Indian Standards Institution for the more suitable apparatus to be employed for routine thermal conductivity determinations at the manufacturer's works as well as at the consumer's end, under Indian conditions.

The results indicated that in the range of temperatures tested, the thermal conductivity values obtained with Blakeley-Cobb apparatus were mostly 0·82 to 0·98 (the average being 0·89) of the values obtained with the ASTM apparatus. Only two values deviated appreciably, one being 0·68 for dense brick and another 1·22 for a siliceous insulation brick both at a mean temperature of 100°C. If a mean temperature in the range of 200°-300°C. is chosen, then agreement between the two sets of values was considerably enhanced, the average Blakeley-Cobb apparatus is appreciably similar to that of the A.S.T.M. apparatus. It may, therefore, be advantageous to employ the former apparatus as a routine check apparatus using the A.S.T.M. apparatus for occasional control purposes.

A point which needs attention is the spread of values obtained with both the instruments. At lower hot face temperatures, i.e. up to 200°C., this spread was appreciable, while above 300°C., the minimum and maximum values obtained were not far apart. This may indicate that in framing standards for thermal conductivities, the acceptable value must fall within a range, rather than at one point, and temperatures at which conductivities are determined must be carefully stipulated.

In conclusion it may be stated that it is possible to obtain repeatable values for thermal conductivity within a certain range using the Blakeley-Cobb apparatus. This range is quite narrow and is of the order of hot face temperatures of 400-500°C. The spread of values obtained for the same specimen is more or less the same as in the case of the A.S.T.M. apparatus. The conductivity values given by Blakeley-Cobb are consistently lower than those obtained with A.S.T.M. apparatus — the trend with rise in mean temperature, however, being more or less the same. A point which may be of interest is the spread in values obtained on the same specimen under identical experimental conditions. This indicates that in framing standard specifications, an acceptability range will have to be specified for each conductivity value and the range will have to be determined carefully. It may also be necessary to stipulate a range of hot-face temperatures depending on the nature of the apparatus employed.

77.0 Determination of Properties of Some Indian Fire Bricks

Both, moderate-heat duty (IS:6) and high heat duty bricks sent by seven manufacturers were tested for their variance in physical properties, viz. apparent porosity, bulk density, apparent specific gravity, cold crushing strength and modulus of rupture. Wherever full bricks were available, the modulus of rupture was done on the full brick also. All these tests were done as per I.S. specifications.

Apparent porosities of IS:6 bricks were mostly in the range of 25 per cent, though one showed 31 per cent and another 19 per cent. The IS:8 bricks had porosities in the range of 26 per cent. The bulk density of IS:6 was in the range of 2 gm/cc. and that of the IS:8 is in the range of 1·8 to 1·85 gm./cc. The thermal expansion in the case of IS:6 bricks was linear with temperature up to 900°C. beyond which temperature the rate fell down and at or near 1000°C. shrinkage started. The property could not be followed beyond 1000°C. IS:8 bricks also showed the same trend.

78.0 Development of Ramming Mixes for Low Frequency Induction Furnaces

The objective of this investigation is to develop suitable indigenous substitutes for the imported mixes which are currently being used.

Table 54 — Chemical Analysis

SiO_2 %	Al_2O_3 %	Fe_2O_3 %	TiO_2 %	CaO %	MgO %	NaO_2 & K_2O %	L.O.I. %
7·96	83·02	2·40	2·78	1·26	1·00	Not determined	0·90

Table 55 — Sieve Analysis and Green and Dry Properties

Sieve No.	+5	−5+10	−10+25	−25+60	−60+85	−85+100	−100
BSS	0·80	24·02	27·02	8·45	1·31	1·13	38·08

Green and Dry Properties

Packing density, gm/cc.—2·822
Green compression strength, lb./sq. in.—32
Green shear strength, lb./sq. in.—5·3
Dry modulus of rupture, lb/sq. in.—72·0
Dry compression strength, lb./sq. in.—416·0

The imported mixes are used as linings for melting the following metals:

Mix No.	Metals or alloys melted
Mix No. 1	Steel
Mix No. 2	Brass & aluminium
Mix No. 3	Cast iron
Mix No. 4	Copper

Work was conducted during the period under review towards the development of a mix equivalent to the imported mix No. 3. The physical and chemical properties of the imported mix No. 3 were examined and the results are given in Tables 54, 55 and 56.

A number of trial mixes were compounded from high alumina base raw materials and plastic refractory clays, available in the country. The test specimens prepared from these mixes were fired to 1000, 1200 and 1400°C. and their physical properties were studied. Propertes of a trial mix finally chosen are given in Table 57. Its green and dry properties are given below.

Green and Dry Properties

Packing density gm/cc.— 2·742
Green compression strength lb./sq. in. — 41·3
Green shear strength lb./sq. in. — 8·1
Dry modulus of rupture lb./sq. in. — 121·0
Dry compression strength lb./sq. in. — 417·0

Table 56 — Properties When Fired to Various Temperatures

Firing temp. °C.	Apparent porosity %	Bulk density gm/cc.	Linear change %	Modulus of rupture lb./sq. in.
200	Disintegrated in water	—	—	63·4
400	28·96	2·776	—	102·3
600	23·95	2·750	—	171·8
800	23·95	2·918	—	164·6
1000	23·90	2·890	*0·31	701·0
1200	25·09	2·803	*0·71	921·0
1400	27·30	2·728	*0·92	1022·0

*Indicates expansion.

Table 57 — Properties When Fired to Various Temperatures

Firing temp. °C.	Apparent porosity %	Bulk density gm./cc.	Linear change %	Modulus of rupture lb./sq. in.
1000	24·1	2·714	0·63	264
1200	20·7	2·756	0·43	872
1400	21·0	2·743	0·56	1272

Preliminary trials on the performance of this mix on an induction furnace in the laboratory are in progress.

79.0 Study of Pyrophyllite for Use as Refractory Material

Two varieties of pyrophyllite — white and pink, from U.P., were studied. Physical properties, e.g. bulk density, porosity, crushing strength, as well as fired properties of the rock sample, were determined. Some pyrophyllite-clay bodies were also prepared and refractory properties were studied.

The P.C.E. of pyrophyllite was between orton cones 7-28 (1630-1650°C.). The bulk density of the pyrophyllite bodies varied between 1·80-1·95 gm/cc. and porosity between 2·24 per cent. From the preliminary studies it was found that since the P.C.E. of pyrophyllite was low, the pyrophyllite could not be used for general refractory purpose. The advantages of pyrophyllite compositions are due to its permanent linear expansion and bloating tendency. The chemical analysis, bulk density and porosity were comparable to other alumino-silicate refractories, so special articles in casting pit refractories as ladle brick, nozzles, stoppers and sleeves can be made with this pyrophyllite.

80.0 Mineralogy of Indian Metallurgical Slags

This project was taken up with a view to make a detailed study of the mineralogy of slags from Indian iron and steel plants. Mineralogical study is of importance in slag chemistry for its utilization. In the earlier report, studies on the mineralogy of blast furnace and open hearth slags from TISCO, IISCO and Durgapur Steel Plant were reported. During the period under review different slags from Bhilai, Bhadravati and Durgapur Steel Plants were studied. The blast furnace slags from Bhilai Steel Plant were predominantly composed of well-developed melilite crystals, and smaller amount of diopside (pyroxene). In some of the samples glass was also present in considerable amounts. Open hearth slags from Bhilai Steel Plant were moderate to well crystallized with aggregates of black opaque phase enclosed or surrounded by silicate phases. Beta-dicalcium silicate, oxide phase (wustite type) and monticellite were the predominant phases, negelschmidtite was present in small amount.

The blast furnace slags from Bhadravati also showed melilite as major phase. Melilite occurred as small laths or dendrites interlocked with other crystalline phases or glass. Spinel was present in considerable amount. It occurred as well crystallized octahedra often in aggregate forms (Fig. 56). Diopside was well crystallized as rosettes; some oldhamite was also noticed in powder mounts. X-ray analysis of this slag indicated the presence of beta-dicalciumsilicate but its presence could not be detected under microscope, possibly due to fine crystal size. The open hearth slag from Bhadravati appeared to be well crystallized having predominantly beta-dicalcium silicate. Other minor phase present was isotropic oxide phase having very high refractive index. Nagelschmidtite was also present. The L.D. slags from

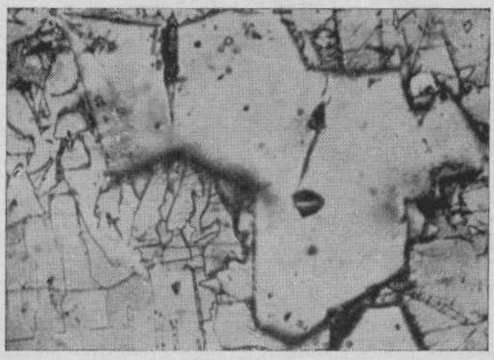


FIG. 56 — WELL CRYSTALLIZED OCTAHEDRA CRYSTAL OF SPINEL IN BHADRAVATI BLAST FURNACE SLAG

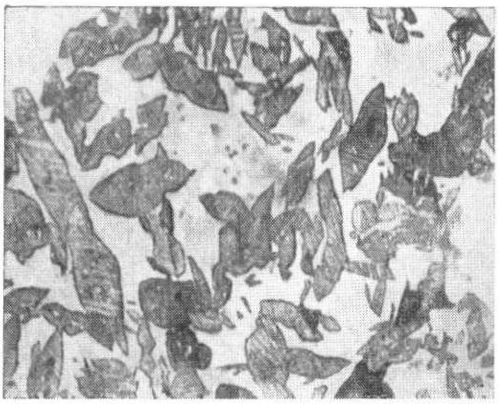


FIG. 57 — TWINNED CRYSTALS OF β-DICALCIUM SILICATE IN BHADRAVATI L.D. SLAG

Bhadravati also consisted of predominantly beta-dicalcium silicate showing beautiful lameller twinning (Fig. 57). Oxide phases were also present. The presence of tricalcium silicate and some aluminate phase was suspected. The E.P.I.F. (electric pig iron furnace) slag from Bhadravati was well crystallized. Well developed crystals of melilite occurred as laths and tablets. Interstitial matrix occuring between melilite crystals was dark brown ferriferous diopside. Spinel was also present. Disiliconizing slag from Durgapur was also studied. It was mainly composed of alpha cristobalite and some iron manganese oxide solid solution. Metallic iron were also present in small amount.

81.0 Study on Reflectivity and Micro-hardness of Ore Minerals

About hundred minerals were collected from different sources. In addition, manganese and iron ore samples from about 10 different localities were collected for comparative studies of reflectivity and micro-hardness values of various minerals present in the ores. About 3-5 polished section were prepared from each area. In all about 100 polished sections were prepared for the above purpose. The samples were mounted with cold setting plastic so as to avoid the effects of thermoplastic mounting on the physical properties of minerals. The effect of chemical and mechanical polishing to remove the fine scratches was studied for all the minerals.

Polished sections were examined with the help of ore microscope to identify the constituent minerals emplying conventional ore microscopic methods including microchemical test and structure etching. X-ray diffraction and X-ray powder photographs techniques were employed for nearly 30 minerals for confirming the ore microscope results.

Reflectivity Measurements — The following standards to be used in reflectivity measurements at different ranges were prepared as suggested by the International Committee on Ore Microscopy:

Type of the standard	Reflectivity range
1. Black glass of known R.I.	0-15
2. Synthetic carborandum	15-25
3. Silicon	25-45
4. Pyrite	45-65
5. Silver-backed glass	65-100

Reflectivity measurements are being carried out by using the above standards.

Micro-hardness Tests — Micro-hardness measurements for Cu, Pb, Zn, Ag and Ni were completed. Micro-hardness measurements for iron and manganese are being carried out. Arrangements are being made to give metallic coating to the surface of the polished sections so as to carry out analysis for the trace elements present in the minerals which affect reflectivity and micro-hardness of ore minerals.

82.0 Studies on Corrosion of Metals and Alloys

(i) *Atmospheric Corrosion* — Long term corrosion tests are being continued with different metals such as mild steel, aluminium, aluminium alloys, copper, brass, zinc, monel and the progress of corrosion attack on these metals is being noted both at Jamshedpur (industrial atmosphere) and Digha (marine atmosphere). The corrosion rate of mild steel at Digha was noted to be about 10 times of the corrosion rate of zinc and amongst various other non-ferrous metals and alloys, the corrosion rates for aluminium and aluminium alloys were found to be minimum.

Short term field tests on yearly and monthly corrosion rates of samples exposed in different months were also in progress for determining the correlation between the corrosion rates and the environmental factors accompanied with the seasonal changes, such as, atmospheric pollution and variations in humidities, temperatures, rainfall, dew fall, etc. In addition to the above, studies on daily corrosion and day and night time corrosion are also in progress at Marine Corrosion Research Station, Digha. From the results obtained so far it was clearly evident that the salinity of the atmosphere has greater influence on the corrosion rate. From the variation of (monthwise) yearly corrosion rate the maximum corrosive period was noted between October and February at Digha and between June to October at Jamshedpur. The corrosion rate of mild steel and the amount of salinity of the atmosphere at Digha during different months showed straight line relationship as shown in Fig. 58. No such relationship of corrosion rate vs. SO_2 pollution at Jamshedpur was noted; the main controlling factor there being the amount of rainfall and to some extent relative humidity of the atmosphere.

(ii) *Sea Water Corrosion* — Systematic studies on corrosion of various ferrous and non-ferrous metals in sea water under nature and laboratory test conditions were taken up to determine the various factors which influence the corrosion rate.

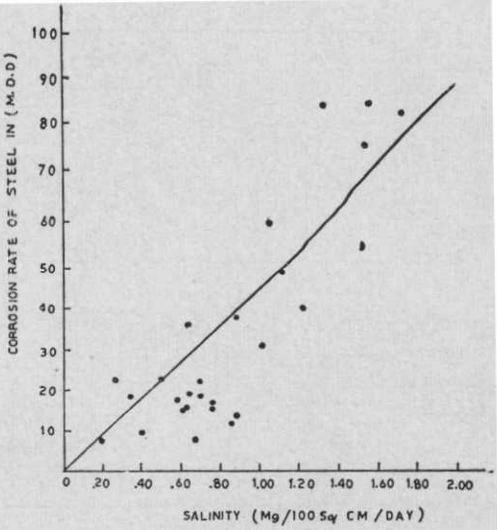


FIG. 58 — CORROSION RATE OF MILD STEEL AND AMOUNT OF SALINITY

Samples of different metals, such as, mild steel, galvanized steel, copper, brass, nickel, monel, zinc, Al-2S, Al-3S, Al-57S, etc., were exposed in the sea under alternate immersion conditions at half tide level and also in the sea-bed at a depth of about two feet. Hydrology of sea at Digha is being recorded and Fig. 59 shows the changes in chlorinity, salinity, temperature, specific gravity and conductivity of sea water for the period from December 1965 to February 1966. In spite of the differences in the chemical and biological characteristics and also in the degree of pollution the corrosion rate for different metals in sea water at Digha was similar to the results in other parts of the world. Of all the metals tested copper and brass showed minimum corrosion at half tide level and nickel and monel showed minimum corrosion in the sea-bed. The corrosion rate for certain metals like nickel, monel, zinc, aluminium and aluminium alloys and galvanized steel was more in the case of exposure at half tide level than in the sea-bed. However, for copper and brass, the corrosion rate was almost same under both the conditions. There are various reasons for such differences in the corrosion rate

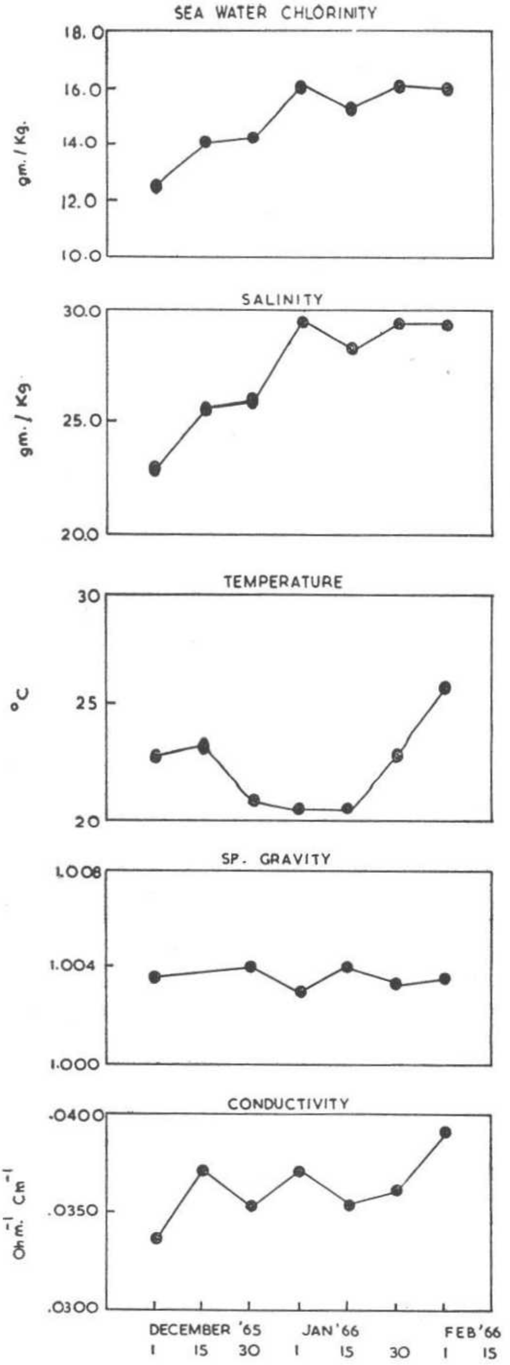


FIG. 59 — HYDROLOGY OF SEA WATER AT DIGHA

in the two types of exposure, one of them being the effect of fouling due to some oyster type of bacteria which lodged on the surface of nickel, monel, zinc, aluminium, aluminium alloys and galvanized steel exposed at half tide level only.

Data obtained in laboratory test showed that amongst various factors velocity, temperature and pH of the sea water have considerable influence on the corrosion rate.

(iii) *Fuel Ash Corrosion and Its Prevention* — With progressive increase in operating temperatures and pressure of boilers and increase in the use of gas turbine for power generation, the use of oils and low grade coals has increased manifold, simultaneously increasing the importance of corrosion by fuel ashes. For avoiding breakdown in power production because of failure of super heater tubes and turbine blades, the importance of minimizing corrosion due to fuel ash has increased considerably for which a detailed study has been undertaken at National Metallurgical Laboratory. The programme of studies includes the investigation on the mechanism of fuel ash corrosion and reduction of this type of attack by other possible means, mainly development of new alloy compositions which are more resistant than the presently used ones.

The first phase of the studies has been completed using sulphated vanadium slags containing V_2O_5 and Na_2SO_4 in different proportions. The performance of the nickel-free stainless steel developed by the National Metallurgical Laboratory was compared to the conventional 18/8 Cr/Ni stainless steel and inconel at different temperatures ranging from 700 to 950°C. The threshold temperature at which the rate of attack increases rapidly was found to lie between 700° and 800°C. depending on the characteristics of the individual alloys and on the composition of the slag used.

The time relationships of the reactions involved in the corrosion process were also determined. In the general equation for the rate processes $y = kt^n$, the value of 'n' can be used to describe the relationship of corrosion with time. The function was found in the present case in general to follow more than one relationship depending on temperature, slag composition and type of materials. In general the initial corrosion rate was high and followed a straight line relationship (n = 1·3 to 0·95). With larger periods the rate became parabolic but fell off rapidly in some cases where the value of ' n ' was found to be as low as 0·1.

Metallographic studies of the oxidized alloys had shown that while 18/8 stainless steel had a massive frontal attack, the attack on nickel-free steel and inconel was more or less uniform and the metal/slag interfaces were smooth and sharp. The first phase of the studies has indicated that compared to 18/8 Cr/Ni stainless steel, nickel-free stainless steel had much superior resistance to sulphated vanadium slag. The corrosion resistance of this alloy at high temperatures in the slag mixtures was found to be equal or superior to inconel.

(iv) *Oxidation of Copper and Copper Alloys* — The investigation was undertaken to study the role of various metallurgical factors, such as, alloy composition, heat-treatment, cold working and environmental conditions, i.e., temperature, partial pressure, gas composition, etc., on the oxidation kinetics of copper and its alloys (Cu-Zn, Cu-Al, Cu-Mn, etc.). The main emphasis of the work is to throw more light on the mechanism of oxidation.

To study the mechanism of oxidation of copper and its alloys by absorption method, an experimental set-up, based on the principle of measuring the oxygen consumed during the oxidation process, was designed and fabricated. Oxidation rates of copper and Cu-Al (0·45, 1·56 per cent Al by weight) and Cu-Mn (2·03, 5·86, 13·91 per cent Mn by weight) alloys have been determined at various temperatures (400°, 500°, 550°, 600° and 750°C.) using wire and sheet specimens. The data indicated that the degree of oxidation is controlled by more than one law and the nature of corrosion rate vs. time curve needs to be studied in detail for which a more sensitive apparatus is being designed.

To analyse the amount and constituents of films formed on copper and its alloys in different environmental conditions, an apparatus based on coulometric reduction technique was designed and set-up (Fig. 60). The apparatus is based on the principle of measuring the amount of current consumed in reducing the film formed during oxidation, by subjecting the measured surface area of the specimen to cathodic reduction in 0·1M NH_4Cl solution at a constant current. The apparatus has been standardized by preliminary tests using electrolytically pure copper sheet specimens, when presence of both Cu_2O and CuO were detected. The films were obtained by heating the cleaned specimens in an electric furnace in atmospheric air or oxygen at different temperatures. Potential-time curves have been obtained by means of a continuous electronic strip-chart. Fig. 61 gives a typical curve for copper oxidized at 250°C. for 2 hours. From the potential of inflection points the nature of oxide was determined. The time required for the respective cathodic-reduction to be completed gave the amount of oxide.

(v) *Role of Inhibitors in the Dissolution of Iron and Its Alloys* — The investigation was undertaken with a view to study the kinetics of metal dissolution in the presence and absence of polar organic inhibitors of thiourea and mercaptan groups. The role of various factors, e.g. adsorption of inhibitor, anodic and cathodic polarization, etc., in controlling the metal dissolution is being studied. Effect of inhibitor on adsorption and diffusion of hydrogen in the metal are also being investigated.

To study the diffusion of hydrogen through steel, an apparatus was designed and fabricated (Fig. 62). Circular specimen (6·0 in. dia.) of desired thickness was tightly fixed between two hemispherical cups having ground flange. Acid was added to the upper half of the cup, connected to a burette containing mercury, where metal dissolution takes place. The lower half of the cup is connected to a micro-burette

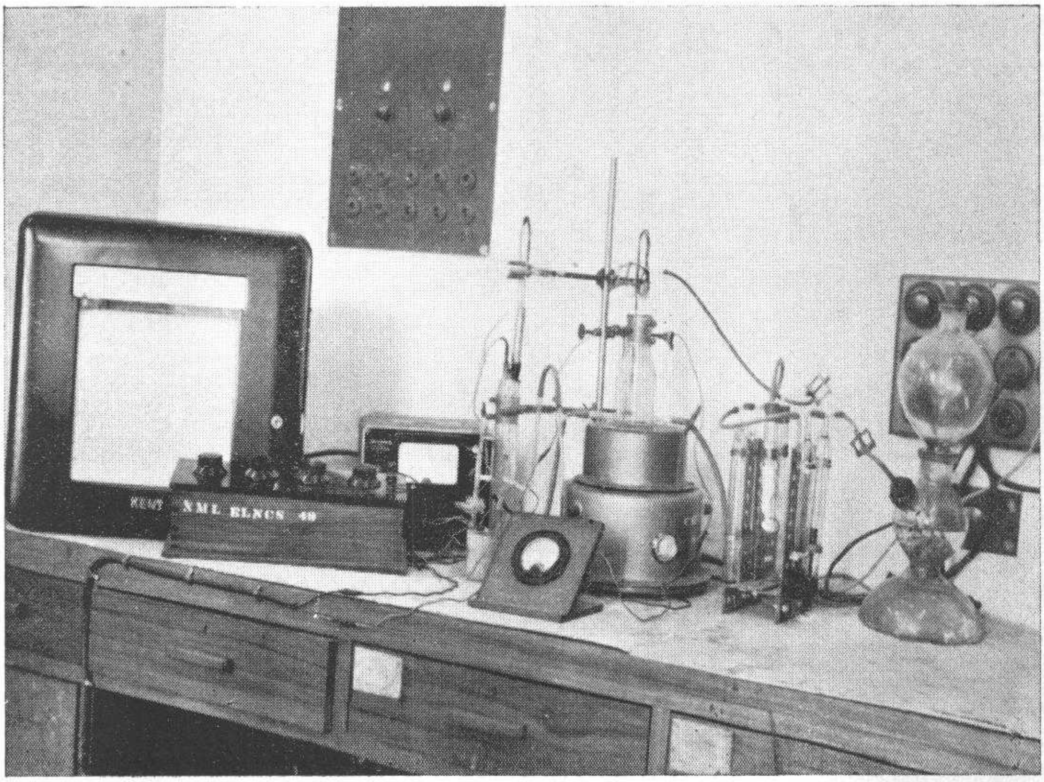


FIG. 60 — SET-UP TO STUDY THE OXIDE FILM FORMATION ON COPPER AND ITS ALLOYS

containing saturated sodium sulphate solution where the hydrogen diffusing through the steel plate is collected and measured. It is possible with this apparatus to measure simultaneously the rate of hydrogen evolution, diffusion and the potential change during the acid attack in the presence and absence of inhibitors. The apparatus has been standardized on the basis of series of studies using mild steel plates of different thickness, such as 0·05, 0·04, 0·03 and 0·02 in. The specimens used were in two conditions, i.e. cold rolled and cold rolled followed by annealing at 720°C. The results indicated that compared to cold rolled steel the rate of diffusion of hydrogen was more in case of annealed sample in spite of the fact that the extent of hydrogen evolution was less. With addition of inhibitors appreciable decrease in the rate of diffusion and evolution of hydrogen was obtained.

During dissolution of steel in acid a part of hydrogen formed at cathode diffuses inside the steel which has a significant influence on the properties of steel. To measure the total amount present in the sample, pickled samples were heated over mercury for 6 hours at 200°C. The hydrogen evolved from the sample was collected and analysed using Ambler's apparatus. Treatment at 200°C. was sufficient to remove bulk of hydrogen from the metal and as such high temperature extraction was not carried out. Moreover the H_2 extracted below 200°C. is more important from the point of view of hydrogen embrittlement.

To carry out cathodic and anodic polarization, a polarization cell (Fig. 63) was so designed that tests can be carried out at different temperatures. The deaeration of the solution can also be done in the apparatus itself. Polarization studies in the presence of various amount of inhibitors of thiourea and mercaptan groups in 1N sulphuric acid at 40°C. were made and compared with that of non-inhibited acid.

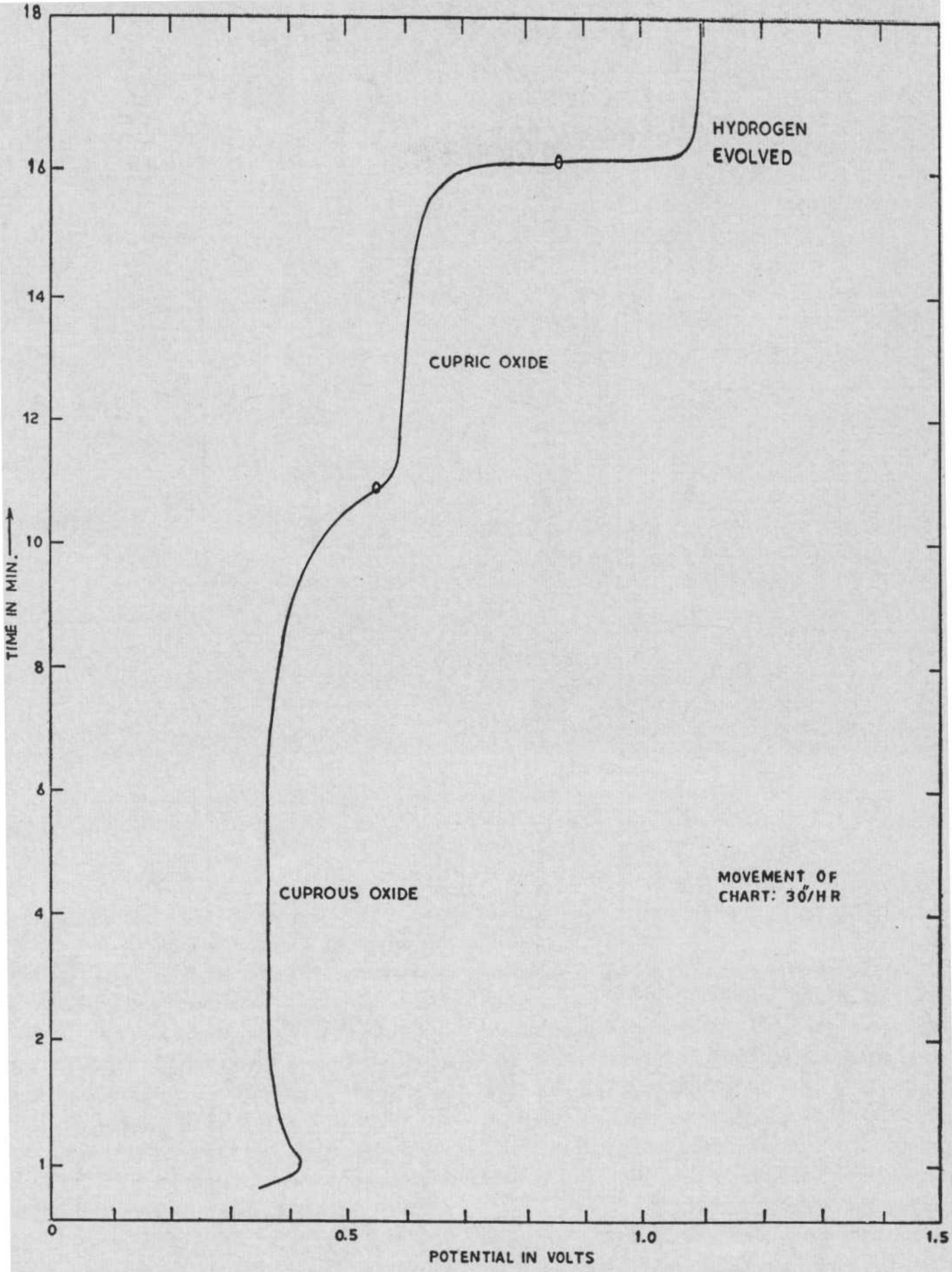


FIG. 61 — POTENTIAL-TIME CURVE OF COPPER OXIDIZED AT 250°C. FOR 2 HOURS

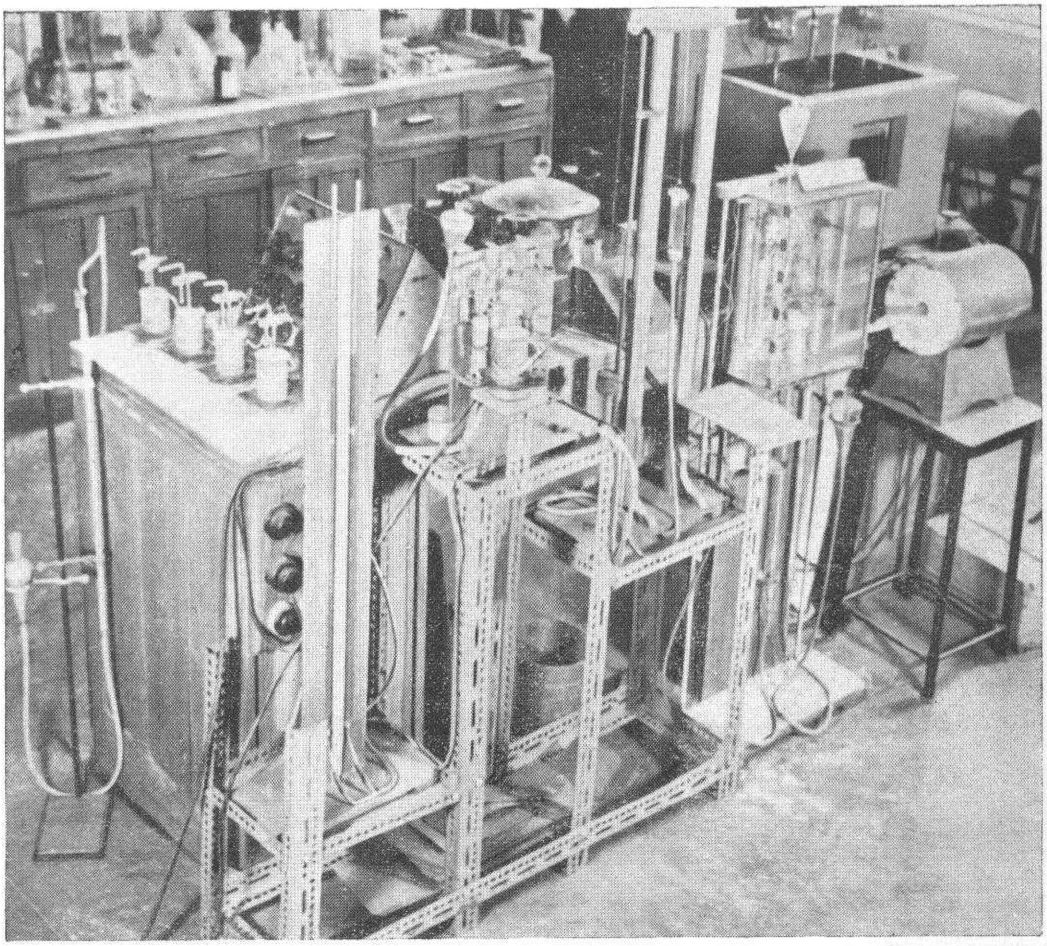


FIG. 62 — SET-UP TO DETERMINE DIFFUSION CO-EFFICIENT OF HYDROGEN THROUGH STEEL

Cold rolled samples were used for all experiments.

(vi) *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 various existing theories. A number of copper alloys, viz. alpha-brass, Cu-Mn, Cu-Sn and Cu-Al, are under investigation.

Earlier, work was carried out to study the effect of various physical and metallurgical factors, viz. applied stress, cold work, prior strain, solute atom concentration, etc., on the stress corrosion behaviour of these alloys. The testing solution constituted $CuSO_4$, $5H_2O$ and $(NH_4)_2SO_4$; the effect of solution *p*H had been studied elaborately. Effect of grain-size on the stress corrosion behaviour of alpha-brass and Cu-23 per cent Mn alloy was studied. Cracking time was found to decrease with increase in grain-size. When cracking time was plotted against $1/\sqrt{d}$, where d is the grain diameter in mm., a straight line relationship was obtained.

Effect of temperature on cracking time was studied with alpha-brass and Cu-Mn alloys of 7 and 23 per cent Mn. Brass has shown to crack rapidly with an increase in testing temperature. Alloy containing 7 per cent Mn showed a minimum in the cracking time vs. temperature curve at

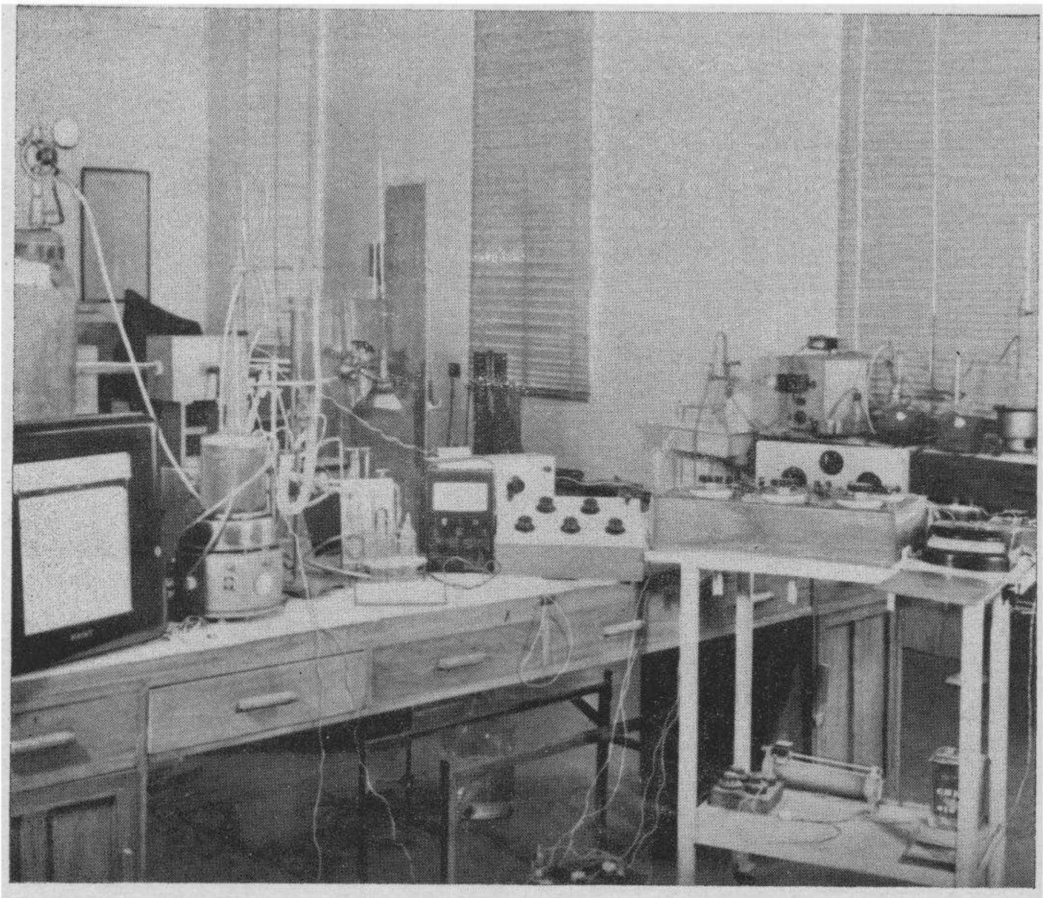


FIG. 63 — SET-UP FOR ANODIC AND CATHODIC POLARIZATION STUDIES IN PRESENCE OF INHIBITOR

about 40°C. when tested in a solution of $pH = 6{\cdot}8$ (at room temperature). The higher manganese alloy behaved like α-brass in a solution of $pH = 6{\cdot}8$ (at room temperature). The same alloy, however, showed a cracking time variation with temperature in the same way as the other Cu-Mn alloy, but with a wider range, when tested in a solution of $pH = 6{\cdot}1$ (at room temperature). In all these cases reciprocal of absolute temperature shows straight line relationship with the logarithm of reciprocal of cracking time expressed in minutes, though the slope of the curve varies with temperature.

Experiments were carried out with interrupted loading to study its effect on the initiation of cracks. A systematic variation in cracking time with respect to the time needed in constant loading was observed in brass and Cu, 23 per cent Mn alloy when the load was above the yield stress values. Loads below the yield stress did not show any such effect.

(vii) *Industrial Corrosion Problems* — During the period under review enquiries relating to corrosion problems were received from various industrial and other organizations and remedial measures were suggested to minimize the corrosion attack. During the year the following two important tests were carried out in this connection.

(a) For electrification of railway traction, the Indian Railways desire to replace the galvanized structures by aluminized ones which will be exposed to corrosive atmosphere in addition to high temperature steam as both electrical and steam locomotives ply on the same tracks. The comparative performance of the two types of coattings was determined by exposing the samples daily to high temperature steam (2 hours), salt spray (6 hours) and high humidity (16 hours). Tests were continued for a period of 30-40 days. Number of hardware items, angles, etc., were tested by visually noting the changes in the surface condition of the specimen and appearance of rust. The tests indicated that as far as resistance to corrosion is concerned aluminized articles can replace the galvanized ones.

(b) Corrosion of lacquer coated high silicon steel sheets developed unusual corrosion during transport and storage. The nature of failure was investigated and found to be due to filiform type of attack. The failure developed due to some defects introduced during manufacturing process combined with ambient humidity conditions during transport and storage.

83.0 Gases in Metals

Various samples of metals and alloys were received from different divisions of NML and from other research laboratories for the determination of O_2, N_2 and H_2 in them. These gases were estimated by vacuum fusion method. Solid soluble nitrogen gas was also estimated in some creep resistant alloys, by electrochemical method developed in the laboratory.

83.1 The Form of Hydrogen in Steel and Alloys

V. I. Lakomskii made the first attempt to show experimentaly that hydrogen is present in iron and steel in the form of positively and negatively charged ions. He charged an iron wire of $\frac{1}{8}$ in. dia. with hydrogen and direct current was passed for few hours. He found in case of mild steel wire a higher concentration of hydrogen at the cathode then at the anode. It happened, according to him, due to the existence of both positively and negatively charged hydrogen ions. They migrated under the influence of direct current. The experimental data shown by Lakomskii was not conclusive.

He could not show in all the experiments a similar enrichment of hydrogen in both the anode and cathode. But in most cases he got enrichment of hydrogen in cathode and not in anode. He explained that migration of proton was more rapid than negatively charged hydrogen ion due to small diameter of proton. Secondly, according to him, the element with completed electronic level as in Fe-Si alloy and Fi-Al alloy, created the condition when dissolved in iron, for negative polarization of the hydrogen in the alloy. Systematic study was taken up at National Metallurgical Laboratory to establish that some of the hydrogen in steel remain in ionized form.

M.S. wire was charged cathodically in a sulphuric acid-bath (10 per cent) at a current density of 0·1 amp. cm.^{2} for $1\frac{1}{2}$ hours. Two gm of sodium sulphide was 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 so that some of the surface hydrogen may diffuse out of the sample. Two pieces from both the ends were cut to find out of the initial hydrogen at two ends. A direct current of 15 amperes was passed through this wire for 2, 3 and 4 hours. Test pieces from anode and cathode area of the wire were cut and analysed for hydrogen in the vacuum heating hydrogen determination apparatus. Keeping the wire under the influence of

Table 58 — Migration of Hydrogen Ion Under Electric Field

(H_2 in cc. per 100 gm)

Mode of charging	Time of passing D.C.	End 1 (+) Before passing D.C.	End 1 (+) After passing D.C.	End 1 (+) Change	End 2 (−) Before passing D.C.	End 2 (−) After passing D.C.	End 2 (−) Change
Cathodically	4 hr	11·47	13·44	+1·97	10·78	12·47	+1·69
,,	,,	5·70	4·49	−1·21	5·28	5·56	+0·28
,,	,,	4·63	5·11	+0·48	6·98	6·78	−0·20
,,	,,	7·67	8·02	+0·35	8·07	9·73	+1·66
,,	3 hr	7·44	6·04	−1·40	9·39	9·85	+0·46
,,	,,	4·71	4·62	−0·09	3·35	4·22	+0·87
,,	,,	3·99	4·92	+0·93	4·45	3·94	−0·51
,,	,,	14·18	13·98	−0·20	12·47	16·60	+4·13
,,	,,	13·24	12·15	−1·09	9·49	13·42	+3·93
Thermally	2 hr	3·21	3·32	+0·11	3·15	3·29	+0·14
,,	,,	2·80	2·73	−0·07	2·92	2·80	−0·12
,,	,,	3·15	3·28	+0·13	3·23	3·14	−0·09
,,	,,	3·62	3·61	−0·01	3·54	3·62	+0·08

electric field for more than 2 hours further change in the concentration was observed. In most cases an increase in the concentration of hydrogen took place at the cathode. In few cases the increase in connection of hydrogen was observed in the anode. Table 58 shows migration of hydrogen ion under electric field.

Few samples, charged thermally with hydrogen do not show any change of concentration either in the anode or in cathode when similarly D.C. was passed for 2 hours. When the sample was charged electrolytically there was a chance that some protons enter into the iron matrix which are migrating towards the cathode during the passage of the D.C.

83.2 Pickling of Ferrous Materials in Hydrochloric Acid-Bath

Pickling is done generally with sulphuric acid. The chief raw material for the manufacture of sulphuric acid is sulphur, which has to be imported, where as hydrochloric acid preparation does not involve any foreign exchange. To study the suitability of hydrochloric acid for pickling purpose under Indian conditions a few preliminary experiments carried out to determine the comparative rate of dissolution of the basic metal by both the acids, without any inhibitor. The results are given in Table 59.

From the experimental data it was found that for the same percentage of acid, HCl attack is less vigorous than H_2SO_4. Hydrogen pick up is also definitely less than that in H_2SO_4. It was also observed that the attack was uniform and slow in HCl and future study will be to compare the rate of pickling in presence of different inhibitors and the corresponding hydrogen pick up. Pickling of rusted material will also be studied.

Table 59 — Dissolution of Metal in HCl and H_2SO_4 Pickling Bath

Time of dissolution hr	Percentage of acid %	Dissolution in gm/m.^{2} HCl	Dissolution in gm/m.^{2} H_2SO_4	H_2 pick up in cc./m.^{2} HCl	H_2 pick up in cc./m.^{2} H_2SO_4
2	5	0·86	1·28	2·13	7·52
2	10	1·27	2·32	2·56	9·15
2	15	1·45	2·42	3·62	9·83
2	20	1·51	3·23	4·01	11·21
2	25	1·26	3·77	4·17	12·10
2	30	1·27	4·12	4·25	12·82

84.0 Spectrographic, Polarographic and Other Analytical Work

(a) *Spectrographic Analysis of Copper-Beryllium Alloy* — This work was taken up to develop a suitable spectrographic method of analysis of beryllium cobalt and nickel in copper-beryllium alloy.

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 alloying elements like beryllium, cobalt and nickel in this alloy is important. Chemical methods 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, Co and Ni. Samples also were taken into solution. Few drops of the solution were dried on spectrographic pure graphite rod 10 mm. dia., which served as lower electrode. The upper electrode was a pointed carbon rod. A series of experiments were conducted to standardize the conditions of exposure and excitation to obtain smooth working curves. The reproducibility and accuracy of the method is also found out. The work was completed with satisfactory results.

(b) *Spectrochemical Determination of Acid Soluble and Acid Insoluble Aluminium in Steel* — This work was taken up to develop a suitable method to determine spectrographically the small amount of both soluble and insoluble aluminium in steel.

For acid insoluble aluminium, the sample was dissolved in HCl with a few drops of HNO_3. When solution was complete, it was filtered on paper pulp and washed throughly with hot HCl. The filtrate was reserved for determination of acid soluble aluminium. The residue was dried and ignited in a muffle furnace. After cooling this was treated with 2 drops of H_2SO_4 and 2 to 3 ml. of HF, evaporated to dryness and then fused with spec. pure sodium carbonate in a platinum crucible. This was subsequently taken into solution with HCl. After mixing it with proper amount of pure iron solution, this was used for determination of acid insouble aluminium.

Synthetic standards were prepared (for both the determination) by dissolving separately spectroscopically pure materials in extra pure appropriate acid and these are mixed in such a proportion that aluminium content of the resulting solution was 0·001 per cent in the lower limit and 0·2 per cent in the upper limit. Few experiments were carried out in Hilger large quartz spectrograph to establish the best working conditions in order to get smooth working curves for both the cases and to get reproducible results. It was found that rotating electrode with excitation technique gave quite satisfactory results. Further work is being carried out to determine the precision and reproducibility of the method.

(c) *Petrological Analysis of Low Grade Ores* — Detailed petrological study of various low grade ores received for beneficiation tests was performed. In addition to the study of the representative samples, constant support was provided to the ore-dressing investigators by examination of the products of beneficiation tests. Detailed petrological studies of the following were conducted.

In case of iron ores, manganese ores, pyrite-pyrrhotite samples, graphite, apatite bearing samples, gold ores, fluorspar, sandstone, bauxite, sillimanite, kyanite, magnesite, selenite and limestone, the physical character, mineralogical composition, nature and degree of interlocking between the desirable and undesirable constituents, ore texture, etc., were indicated. In the year under review nearly 40 samples were subjected to detailed petrological examination.

The number of test products examined runs into hundreds.

(d) *Analysis of Ores, Minerals, Metals, Alloys and Other Metallurgical and Mineral Processing Products Employing X-ray Fluorescence* — The non-destructive, highly selective, sensitive and very quick method of X-ray fluorescence analysis has been employed to expedite analysis of routine test and processing products of various divisions of the National Metallurgical Laboratory as well as for specialized analytical problems.

The results of analysis obtained 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 or better compared with analysis using conventional time-consuming analytical techniques. The testing process involves comparing the sample of unknown analysis with an analysed sample having similar element environment as the unknown. For greater accuracy calibration curves are prepared using a few carefully analysed samples. When analysed samples of similar nature as the unknown are not available synthetic standards are prepared for comparison purposes. The range of concentration of the elements of interest varied from trace amount (P.P.M.) to 100 per cent. Following is the list of products and the elements or radicals for which the fluorescence analyses were performed:

(i) Flotation products of low grade apatite sample from Bihar for P_2O_5.
(ii) Flotation products of graphite for S and Fe.
(iii) Flotation products of fluorspar from Ambadonagar, Gujarat, for Ca and S.
(iv) Flotation products of copper ore from Khetri (Rajasthan) for Cu.
(v) Flotation product of limestone from Tisco for CaO.
(vi) Beneficiation products of bauxite from Hindusthan Aluminium Corporation for Fe/Fe_2O_3.
(vii) Coal ash, fly ash and flue dust samples for Ge.

During the period under review, nearly 625 elements or radical analyses were performed on about 550 samples.

(e) *Chemical Analysis*—During the period under review nearly 400 samples were qualitatively and quantitatively analysed for different radicals.

85.0 Preparation of Plating Salts

The electroplating industry in India currently employs imported plating salts, polishing compositions and plating equipments. 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, cyanide cadmium plating salt, etc., was taken up, based essentially on the use of raw materials and chemicals made in India. Addition of special ingredient, beneficial in increasing conductivity, good throwing power, brightness and other related properties for good plating was also investigated from indigenous sources.

The common plating salts mostly used by light engineering industries for decorative and protective finishes 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 electro-chemistry studies and the characteristics of the plating electrolytes derived from the salt mixture such as conductivity, cathodic and anodic polarization, 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 and wear 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 was tested and compared in continuous production plating practice. All these 'NML-formulated' salts have been satisfactory in performance, as reported by consumers in light engineering industry.

(i) *Replacement of $NiCl_2$ by NaCl in Nickel Plating Bath* — In the dull nickel plating solution the possibility of replacing one of the ingredient, viz. NiCl by NaCl was investigated keeping the other constituents unaltered. Behaviour of cast and depolarized nickel anodes was studied in the same bath containing NaCl. The performance of cast nickel anode (Ni, 98; Fe, 0·75; Cu, 0·24; Sn, 0·10; C, 0·82 and Mn, 0·10 per cent) was found better than the depolarized nickel anode (Ni, 98·9; Fe, 0·53; Cu, 0·17; Mn, 0·01; C, 0·09 and O_2, 0·118) per cent in NaCl containing nickel plating electrolyte. Thus the formulation of dull nickel plating salt with NaCl replacing NiCl will entail less capital cost towards plating and nickel anodes. It is, therefore, advocated for commercial plating practice.

(ii) *Development of Bright Nickel Plating Salt* — The process and the technique for bright nickel plating which requires no buffing after plating have been established with organic reagents containing a suitable brightener, viz. naphthalene sulphonic acid, a leveller like 'coumarin', and an antistress reagent 'saccharine 'together with a sodium salt of weak organic acid in the usual dull nickel plating bath. The reagents were added in minute traces in right proportion in nickel plating solution. Bright, minor finish coating of nickel under the rigid control of *p*H, and current density and temperature was obtained on properly polished and degreased cathode surface. Such bright nickel plating solution as developed was put under trial on continuous plating practice in commercial plant to assess its suitability in production plating. The results as reported are quite satisfactory and the process has been offered for commercial exploitation.

A systematic study has been made of the effects of certain aromatic sulphonic acid derivatives, coumarin and the related compounds added to a Watts nickel bath. Benzene derivatives contribute sulphur but no carbon to the deposits. Deposit obtained from baths containing napthalene derivatives usually contain sulphur but no carbon. Mechanical and physical properties of electrodeposited nickel are also altered by these addition agents incorporated in the plating solution. An explanation to mechanism of bright nickel plating has been tried to establish from the above experimental findings.

(iii) *Behaviour of Nickel Anodes in Nickel Plating Electrolyte* — The physical and electrochemical characteristics of the different types of nickel anodes, viz. cast, depolarized, etc., having the compositions given in Table 60 were studied in nickel plating solution. Their performance in the plating electrolyte have been assessed, viz. anodic current efficiency and anodic polarizaton, are represented graphically in Figs. 64 and 65.

The controlled amount of impurities, viz. C, S, Fe, Mn, Cu, P, Si and O_2 present in the nickel anodes has been found to affect the anodic efficiency, its electrochemical reactivity and its performance in plating solution. Oxygen and sulphur have important role in the depolarization of nickel and increase the reactivity of nickel and inhibit passivity.

(iv) *Crack-free Bright Chromium-plating from S.R.H.S. Bath* — A process for crack-free bright chromium plating from self regulating high speed bath containing $SrSO_4$ and K_2SiF_6 was established. The

Table 60 — Composition of Different Types of Nickel Anodes

Percentage by wt	Electrolytic nickel	Cast nickel	Dipolarized nickel	High carbon cont. nickel	Cast rolled nickel	Remarks
Ni, %	99·85	98·86	98·9	99·47	99·57	Ni by difference
Co, %	0·05	0·63	0·68	Not found	Not found	
C, %	0·04	0·13	0·03	0·29	0·17	
Fe, %	0·02	0·11	0·18	0·09	0·08	
Si, %	0·007	0·009	0·005	0·085	0·126	
Cu, %	0·01	0·08	0·08	0·02	0·03	
Sn, %	Not detected	Not detected	Not detected	Trace	Trace	
S, %	0·003	0·006	0·006	0·027	0·006	
Mn, %	Trace	0·001	0·001	0·001	0·047	
P, %	0·002	0·001	Trace	Trace	Trace	
O_2, %	0·023	0·170	0·120	0·018	0·038	

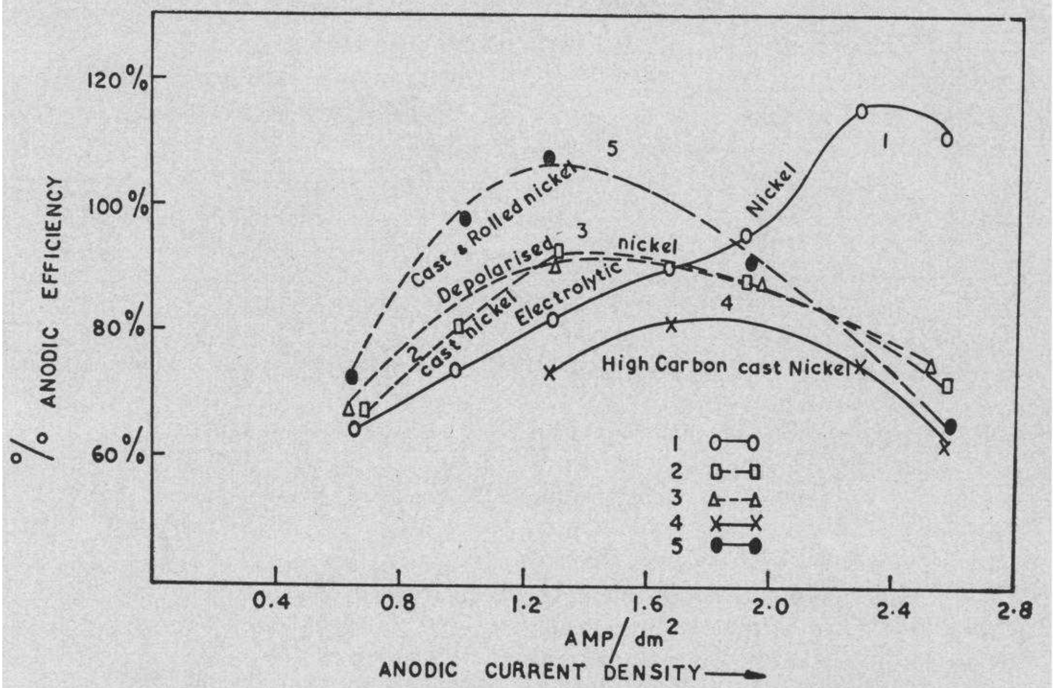


FIG. 64 — ANODIC EFFICIENCY OF DIFFERENT NICKEL ANODES

ordinary conventional chromic acid-sulphuric acid bath for chromium plating does not throw well and has a low current efficiency and develop crack in chromium deposits after a certain thickness of plating. Self-regulating high-speed chromium plating bath as developed contains, besides chromium acid, $SrSO_4$ and K_2SiF_6 in suitable proportion.

A bright, crack-free chromium plate with a thickness of at least 0·00005 in. from a bath of chromic acid and a mixture of

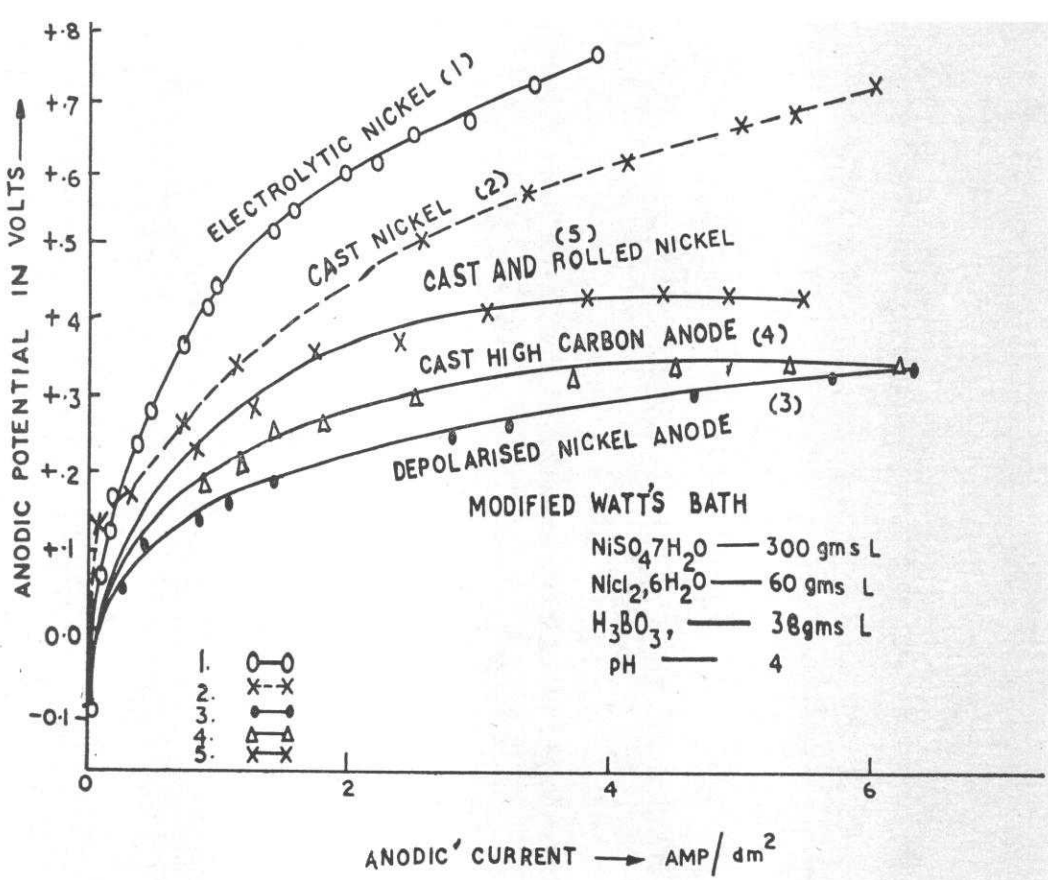


Fig. 65 — Anodic Polarization of Different Nickel Anodes

sulphate and fluorosilicate catalysts, as $SrSO_4$ and K_2SiF_6 with the bath containing 1·2 gm/l to 6·3 gm/l total catalyst ions has been found suitable. The ratio of the weight of CrO_3 (180 gm/l-540 gm/l) to catalyst ions found suitable in the range of 80: 1 to 150: 1, sulphate ion content 20-85 per cent of total catalyst concentration. Operation of the bath was carried out for a temperature range of 120-140°F.

A certain portion of chromic acid in trivalent state by reduction with tartaric acid is to be maintained in the S.R.H.S. bath to get the bright crack-free chromium plate. This is an alternative method that has been found suitable compared to the method mentioned earlier.

86.0 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* — Detailed studies were carried out to recover zinc from zinc dross by vacuum distillation in a mild steel pot of 300 kg. capacity. The 24 in. dia. mild steel retort which was fabricated earlier was slightly modified for adequate temperature measurements. Distillation tests were carried out with different amounts of charge at an average furnace temperature of 750°C., but as the rate of distillation at 750°C. was not very encouraging, some experiments were carried out at an average furnace temperature of 800°C. with different amounts of charge and varying periods of distillation. The results are shown in Table 61.

Table 61 — Distillation of Zinc at 800°C.

Expt. No.	Wt of charge kg.	Period of distillation hr	Wt of condensate kg.	Rate of distillation kg./hr	Zinc recovery %
22	300	17	122	7·18	—
21	300	21	182	8·7	—
30	200	21	165	7·86	85·94
20	200	23	172	7·48	89·6
18	150	13·5	114	8·45	79·2
19	150	19	130	6·84	90·2
32	100	8·5	45	5·29	—
34	100	13	78	6·0	81·5
31	100	13·5	71	5·26	—

The rates of distillation at 800°C. were faster than that at 750°C. for a particular charge weight. The rate of distillation was, however, found to be slowed down when the charge weight was decreased from 200 to 100 kg. The rate of distillation is governed principally by the thermal conductivity and diffusivity.

The present unit fabricated out of mild steel has already withstood 550 hours of batch-wise distillation operating in the temperature range of 750-800°C. and about 3·5 tons of pure zinc has so far been collected. It is proposed to carry out a few studies with premelted charge for uniform heat distribution and heat propagation through the charge.

(ii) *Recovery of Zinc from Zinc Dross received from Post & Telegraph Department, Government of India* — Zinc dross in the form of slabs were received from Post & Telegraph Department, Government of India, for recovery of zinc. The sample analysed Fe, 3·01; Pb, 1·53 and Al, 0·26 per cent. Distillation studies were carried out in an 8 in. dia. mild steel pot with 6 kg. of zinc dross per batch for different periods at varying distillation temperatures. It was observed that a recovery of 98-99 per cent zinc could be obtained in the temperature range between 550-650°C. The distillation rate is faster at higher distillation temperatures. The sample was found to be free from flux-contamination and the operation was smooth and the order of vacuum was better as compared to the various other samples of dross so far tested.

The condensate obtained at lower temperature of distillation are found to be purer than the condensates at higher distillation temperatures. The impurities in the condensate varied between 0·003 to 0·046 per cent Fe and 0·39 to 0·56 per cent Pb depending on the temperature and period of distillation.

87.0 Utilization of Emery Flour

This investigation was taken up to examine the suitability of emery flour obtained as a waste product in the manufacture of synthetic emery from bauxite for industrial usage. The results of investigation showed the possibility of utilizing this material (1) for making sharpening sticks and blocks for polishing razor blades, etc., (2) for manufacture of non-slip tiles, and (3) as a feed back to the cupola in the manufacture of synthetic emery from bauxite.

87.1 Recovery of Metallic Values from Red Mud

With the expansion of the aluminium industry in India, the quantity of red mud discarded by the aluminium industry has considerably increased thus creating problems of its disposal. Indian red mud contains 15 to 20 per cent Al_2O_3, 20 to 30 per cent Fe_2O_3, 25 to 30 per cent TiO_2 and 4 to 6 per cent alkali. Investigations have been taken up to recover Al_2O_3, TiO_2 and Fe from the red mud.

Initial attempts were made to recover alumina by roasting a sample of red mud with Na_2CO_3, lime and coke at 1100-1150°C. and leaching the roasted product with caustic soda solution. Sixty per cent of Al_2O_3 could be removed by roasting red mud with 17 per cent Na_2SO_3, 3·75 per cent $CaCO_3$ and 8·5 per cent coke followed by leaching of the calcine with 10 per cent NaOH. Further systematic studies are under progress.

87.2 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.

Liming of agricultural soils by basic slag for different crops, such as wheat, groundnut, maize and gram, has already given encouraging results. In order to evaluate the economics of the use of basic slag as liming material, the main factor may be the cost of crushing the slag and as such it is essential to determine the optimum grain size of the basic slag which is required for the agricultural sector for most efficient liming purposes. In view of the above, experiments are under way to find out the best possible way to crush the slag at minimum cost.

Rourkela open hearth slag which has already given good results in the first field trials has been crushed to different mesh sizes and 100 kg. of each size has been sent to the Agricultural Research Institute, Kanke, Ranchi, for application to the soils in different plots under idential conditions to determine the optimum size of the slag required to be crushed for liming purposes.

87.3 Utilization of Blast Furnace Slag as Rail-road Ballast

This investigation is also a part of a 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. The comparative study indicates that blast furnace slag can replace granite, limestone, quartz, etc., which are at present 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. Indian Railways are interested to initiate trials with the representative samples of Tisco blast furnace slag for arranging field trials in the rail-track near Tatanagar.

PILOT PLANTS

88.0 Low Shaft Furnace Project

The pioneering and challenging investigations on the direct injection of liquid naphtha into the hearth of an iron smelting furnace with simultaneous enrichment of the blast with oxygen were successfully conducted.

As desired by the Government of Maharashtra, investigations were undertaken with the titania-free and high titania iron ores, lime stone and low temperature carbonized coke made from wholly non-coking Wardha Valley coals from iron smelting in the Low Shaft Furnace Pilot Plant. Considerable headway has been made in multitude of industrially oriented projects on iron and steel production technology.

Programme of Research and Development Work

Phase I

(i) Iron ores from Orissa and Bihar with non-coking coals from Raniganj and Dishergarh coalfields either in bedded charge or by briquetting iron ore, limestone and non-coking coal.

(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 ferro-manganese with or without oxygen injection in the low-shaft furnace pilot plant.

Extensive investigations under Phase I(i) to (vi) and Phase II have already been completed and several investigation project reports have been issued.

The investigations on iron smelting undertaken during the period under review can be broadly classified into the following campaigns.

Thirty-fourth Campaign

Smelting trials were conducted with iron ore fines (Orissa Minerals), low temperature carbonized coke (C.F.R.I.) and blended fluxes with the object of direct injection of liquid naphtha into the hearth of an iron smelting furnace with simultaneous enrichment of the blast with oxygen and also to study the effect of the blast additives on the smelting characteristics.

Thirty-fifth Campaign

Smelting trials were conducted with iron ore from Deulgaon and limestone from Rajur of Maharashtra state employing low temperature carbonized coke made from Wardha Valley non-coking coals at the Central Fuel Research Institute, Jealgora, and also with low temperature carbonized coke made at C.F.R.I. with non-coking coals of Raniganj.

Thirty-sixth Campaign

Investigation No. I

Smelting trials were conducted with Deulgaon iron ore and Rajur limestone of Maharashtra employing nut coke as fuel and reductant.

Investigation No. II

Smelting trials were conducted with blended iron ore, comprising titania-free Deulgaon iron ore with high titania Khursipar iron ore with Rajur limestone of Maharashtra employing nut coke as fuel and reductant.

Investigation No. III

Smelting trials were conducted with blended iron burden comprising titania-free Deulgaon iron ore and high titania Khursipar iron ore and Rajur limestone of Maharashtra employing low temperature carbonized coke (C.F.R.I.).

Thirty-seventh Campaign

Smelting trials were conducted with Deulgaon iron ore and Rajur limestone of Maharashtra employing pearl coke as the fuel and reductant.

DETAILS OF FURNACE CAMPAIGNS

Thirty-fourth Campaign

The preliminary trials with naphtha injection and simultaneous enrichment of the blast with oxygen have been reported earlier. In this trial the relative merits of either fuel oil or naphtha injection with simultaneous enrichment of blast with 1, 2 and 3 per cent oxygen, respectively, were investigated.

The burden consisted of iron ore fines (Orissa Minerals), low temperature carbonized coke made from non-coking coals (C.F.R.I.) and blended fluxes. The chemical and physical characteristics of raw materials employed are given in Tables 62 and 63. The furnace was blown in under the following operational conditions:

Tuyere diameter, mm.	75
Hot blast temperature, °C.	575
Hot blast volume Nm/hr	2400-2750
Hot blast pressure mm. W.G.	1800-1820
Top gas temperatures, °C.	325-360

Table 62 — Chemical Analysis of Raw Materials

1. Chemical analysis of iron ore (Orissa):

Fe %	SiO_2 %	Al_2O_3 %	S %	P %
59·42 to 64·50	3·20 to 6·34	4·10 to 5·20	0·01 to 0·29	0·02

2. Chemical Analysis of Fluxes:

	CaO %	SiO_2 %	Al_2O_3 %	MgO %
(i) Limestone (Madras)	54·31	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

The basically calculated degree of the slag CaO/SiO_2 was maintained between 1·2 to 1·3 and the alterations in the operational variables were confined with in very narrow limits. The smelting operation in each individual stage was continued for a reasonably long period for attaining equilibrium smelting conditions for ensuring correct data, which were compared to the base period without any injection. The operational results with fuel oil injection and different degrees of oxygen enrichment are summarized in Table 64.

After the assessment of the smelting performance with fuel oil, the technological aspects and industrial implications of direct injection of liquid naptha was extensively studied and the results are summarized in Table 65.

Both fuel oil and naphtha injection with simultaneous enrichment of the blast with oxygen which was primarily necessitated by low blast temperature, significantly reduced the fuel rate and increased the production.

The injection rate of naphtha was lower than that of fuel oil and, therefore, the oxygen enrichment of the blast in relation to the injection rate was higher.

Under smelting conditions, the replacement ratio with the naphtha was better than that of fuel oil. The replacement ratios were little higher than those obtained with fuel oil injection in the industrial scale. At the present prices of fuel and naphtha, the utilization of naphtha is economically more attractive. Apart from the test results, the investigation has

Table 63 — Screen Analyses of the Raw Materials Employed

Raw materials	−50·8 +25·4 mm.	−25·2 +12·7 mm.	−12·7 +6·35 mm.	−6·36 +3·17 mm.	−3·17 mm.
Low temperature carbonized coke (C.F.R.I.)	59·50	37·50	Nil	Nil	3·00
Iron ore (Orissa)	Nil	2·00	49·85	42·25	5·90
Limestone (Madras)	93·00	4·75	0·60	0·50	0·5
Dolomite (Assam)	15·95	79·80	4·00	Nil	0·25

indicated the possibility of injection of highly inflammable petroleum product directly into the hearth of an iron smelting furnace and no operational dangers were faced. It was found that naphtha can be successfully injected for iron-smelting provided adequate safety precautions are adopted.

Table 64 — Chemical Analysis of Pig Iron, Slag and Gas

Data on	Base period	1st stage	2nd stage	3rd stage
Quantity of fuel oil/tonne of pig iron, kg.	Nil	31·0	30·7	30·6
Average oxygen enrichment, %	Nil	1	2	3
Average composition of pig iron, %				
C	2·5	2·8	2·6	2·70
Si	3·5	4·12	3·30	3·20
S	0·07	0·07	0·08	0·09
Mn	0·18	0·04	0·14	0·12
Average composition of slag, %				
CaO	34·3	31·60	32·27	34·70
SiO_2	35·6	38·48	35·08	37·1
Al_2O_3	22·8	22·50	20·40	21·20
MgO	3·8	4·30	4·01	4·82
FeO	1·2	1·60	1·80	1·40
Average composition of gas by by volume, %				
CO	26·0	26·1	26·9	27·2
CO_2	4·3	4·5	4·8	5·3
CH_4	3·6	3·10	3·0	3·0
H_2	0·6	0·57	Nil	Nil
CO/CO_2 Ratio	6·0	5·8	5·5	5·1

Table 65 — Operational Results with Naphtha Injection and Oxygen Enrichment of the Blast

Data on	Base period	1st stage	2nd stage	3rd stage
Quantity of naphtha/tonne of pig iron, kg.	Nil	18·4	17·8	17·8
Average O_2 enrichment, %	Nil	0·74	1·4	2·6
Average Metal Analysis:				
C, %	2·5	2·7	2·51	2·6
Si, %	3·5	3·6	3·4	3·5
S, %	0·07	0·07	0·064	0·05
Average Slag Analysis:				
CaO, %	34·8	33·9	36·2	36·5
SiO_2, %	35·6	35·7	35·8	35·6
FeO, %	1·2	1·3	1·2	1·0
Average Top Gas Analysis:				
CO, %	26·0	26·0	25·8	26·6
CO_2, %	4·3	4·5	4·67	5·3
CH_4, %	3·6	3·1	3·2	3·5
CO/CO_2 Ratio	6·0	5·8	5·5	5·0

Thirty-fifth Campaign

It has been mentioned in the previous Annual Report that for ascertaining the possibilities of establishment of a regional iron works based on the exploitation of the raw-materials occuring in the State, the Government of Maharashtra had sent iron ore, limestone and low temperature carbonized coke for conducting extensive smelting trials. The chemical and the particle size classification of raw-materials are recorded in Tables 66 to 70.

The smelting of Deulgaon iron ore with low temperature carbonized coke made from Wardha Valley coals at the Regional Research Laboratory, Hyderabad, has already been reported. In this campaign, investigations were conducted with iron ore from Deulgaon, low temperature carbonized coke made at the C.F.R.I. from Majri, Ghughus, Ballarpur, Chanda, and Rayatwari collieries of the Wardha Valley coal fields.

Before undertaking the smelting trials, the reducibility of Deulgaon and titaniferrous Khursipar iron ores were compared with Orissa Mineral iron ore, when it was found that the reducibility of Maharashtra ores were inferior to the Orissa Mineral iron ore. After the completion of investigations with low-temperature carbonized coke made at R.R.L., the smelting characteristics of Deulgaon iron ore and Rajur limestone were known and consequently the trial was initiated with Deulgaon iron ore and Rajur limestone employing nut coke as fuel and reductant. After reasonable duration of trial, nut coke was progressively replaced by low

Table 66 — Proximate Analyses of the Fuels

Fuels	Fixed carbon %	V.M. %	M %	Ash %	S %
Nut coke (Lodna)	68·42	4·45	1·68	25·45	0·61
Pearl coke (Tisco)	71·35	4·35	1·10	23·20	0·35
*Low temperature carbonized coke (C.F.R.I.)	61·50	4·10	8·50	25·90	0·23
*Low temperature carbonized coke (R.R.L.)	54·42	13·09	0·75	31.74	0·36

*Made from the Wardha Valley non-coking coals.

Table 67 — Ash Analyses of Fuels

Fuels	CaO %	SiO_2 %	MgO %	Al_2O_3 %	Fe_2O_3 %	P %	S %
Nut coke (Lodna)	2·20	56·90	Trace	25·80	14·80	0·40	—
Pearl coke (Tisco)	4·85	55·28	2·5	25·10	11·20	0·40	—
Low tmperature carbonized coke (C.F.R.I.)	5·68	53·80	1·88	28·00	6·60	N.D.	—
Low temperature carbonized coke (R.R.L.)	9·47	50·10	Trace	31·02	8·78	N.D.	—

Table 68 — Analyses of the Iron Ores

Origin	Fe %	SiO_2 %	Al_2O_3 %	MgO %	S %	P %	TiO_2 %
Deulgaon iron ore	64·40	3·72	2·00	Trace	0·089	Trace	0·10
Khursipar iron ore	54·44	1·04	5·12	Trace	0·096	0·027	14·50

Table 69 — Analyses of the Fluxes

Origin	CaO %	SiO_2 %	MgO %	Al_2O_3 %	S %
Limestone-Rajpur (Maharashtra)	48·03	5·56	2·70	1·6	Trace
Dolomite — Assam	31·30	0·40	20·70	0·60	Trace

temperature carbonized coke made at C.F.R.I. from the Wardha Valley coals with proper adjustment of operational conditions and basicity degree of slag, so that minimum fluctuation took place. The operational conditions of smelting with soft coke are given in Table 71.

The addition of the flux was calculated to result in a lime basicity degree CaO/SiO_2 in

Table 70 — Screen Analyses of the Raw Materials Employed in Different Investigations

Raw material	−50 mm. %	−50 +25 mm. %	−25 +12 mm. %	−12 +6 mm. %	−6 +3 mm. %	−3 mm. %
Nut coke (Lodna)	10·0	45·3	31·0	6·0	2·5	5·20
Pearl coke (Tisco)	—	—	41·5	42·1	8·2	8·20
L.T.C. (C.F.R.I.)	10·30	48·2	37·7	2·7	0·5	0·60
L.T.C. (R.R.L.)	10·37	48·30	31·37	4·25	0·50	4·70
Deulgaon iron ore	Nil	19·25	60·00	14·50	3·95	2·50
Khursipar iron ore	Nil	19·50	57·50	15·25	4·00	3·75
Limestone (Rajur)	Nil	7·50	74·87	13·00	2·25	2·37
Dolomite (Assam)	Nil	9·50	87·30	3·20	Nil	Nil

Table 71 — Operational Conditions of Smelting with Soft Coke (C.F.R.I.)

Operational variables	Data
Tuyere diameter	75 mm.
Av. blast volume Nm^3/hr	2800
Av. blast pressure mm.WG	1950
Hot blast temperature, °C.	550
Top gas temperature, °C.	460

Table 72 — Analysis of Pig Iron, Slag and Top Gas

Average analysis of pig iron

C %	Si %	S %	Mn %	P %
2·70	3·50	0·08	0·17	0·28

Average analysis of slag

CaO %	SiO_2 %	FeO %	Al_2O_3 %	MgO %	S %
34·1	35·5	1·8	26·2	7·4	0·70

Average top gas analysis

CO %	CO %	CH_4 %	N_2 %
24·5	3·7	4·2	Balance

the range of 1·2-1·3. The chemical analysis of pig iron, slag and top gas are recorded in Table 72.

No significant difference in smelting behaviour between the two varieties of low temperature carbonized cokes made by different process was seen. The inferior strength of the soft coke seriously reflected on the regularity of the descent of the burden and generated the exceedingly high amount of flue dust. It was considered that the poor physical strength of the soft coke and excessive degradation will not enable its employment under industrial conditions.

Thirty-sixth Campaign

Investigation No. I

It was observed during the two previous campaigns conducted either with L.T.C. coke made at R.R.L. or at the C.F.R.I. from the wholly non-coking coals of Wardha Valley that iron smelting in low shaft furnace pilot plant although possible were never satisfactory due to the severe degradation of the soft coke particles during the descent of the burden in the furnace shaft. In an industrial furnace of higher shaft weight and different blowing conditions, the suitability of such soft coke for iron smelting is extremely doubtful if not entirely impracticable. It was, therefore, considered that smelting trials with nut coke and pearl coke, which are available as metallurgical wastes in an integrated iron and steel plant, will be of interest to the Government of Maharashtra for setting up of a regional iron works with the utilization of surplus nut and pearl coke from Bhilai Steel Plant. In this investigation, therefore, the smelting trial was conducted with Deulgaon iron ore, Rajur limestone and nut coke. The furnace was operated under the following conditions:

Tuyere diameter, mm.	75
Average hot blast volume, Nm^3/hr	2350
Average hot blast pressure, mm.WG	2000
Average hot blast temp., °C.	500
Average top gas temp., °C.	475

In burdening the furnace, adequate amount of limestone was added to result in lime basicity CaO/SiO_2 of 1·2 and dolomite was added to yield 6-8 per cent MgO in the slag. The data obtained under the steady state of smelting is recorded in Table 73. The results were entirely satisfactory.

Investigation No. II

It has been mentioned earlier that the Government of Maharashtra had sent two varieties of iron ores, namely Deulgaon iron ore free from TiO_2 and high titania iron ore (14·50 per cent TiO_2) from Khursipar. It was initially intended that the high titania ore should be smelted exclusively for making high titania slag, for the possible recovery

Table 73 — Analyses of Pig Iron, Slag and Top Gas Employing Deulgaon Ore and Nut Coke

Average analysis of pig iron

C %	Si %	S %	Mn %	P %
2·6	3·5	0·06	0·18	0·37

Average analysis of slag

CaO %	SiO_2 %	FeO %	Al_2O_3 %	MgO %	S %
36·5	30·3	0·9	18·7	6·7	0·65

Average top gas analysis

CO %	CO_2 %	CH_4 %	N_2 %
25·2	4·2	3·6	Balance

of TiO_2 from it for utilization in a chemical industry as in paint manufacture. But in view of the impracticability of the smelting operation in coke blast furnace with high TiO_2 in the burden, it was mutually agreed to find out the maximum utilization of high titania ore by blending it with TiO_2 Deulgaon iron ore. The smelting trials with high titania iron ore can, therefore, be divided in two categories, namely with the utilization of nut coke as fuel or with the utilization of low temperature carbonized coke made at C.F.R.I. from Wardha Valley non-coking coals. After attaining a steady state of smelting with Deulgaon iron ore, Rajur limestone and nut coke, the Deulgaon iron ore was progressively replaced with Khursipar ore to find out the optimum amount of it in the ore blend. It was assumed that the replacement of Deulgaon iron ore with Khursipar iron ore will not significantly alter the fuel rate and, therefore, the flux rate. A burden calculation showed that replacement of 25 per cent Deulgaon iron ore with Khursipar will yield a slag containing 5 per cent TiO_2 theoretically. The furnace was blown under the conditions given in Table 74.

Twenty-five per cent of the Deulgaon iron ore was replaced with 25 per cent titaniferous ore and it was observed that it did not contribute specifically to any irregularities in the smelting operation. The analysis of pig iron, slag and top gas with 25 per cent titaniferrous ore in the ore blend are given Table 75.

In the next stage ore burden constituted with 50 per cent high titania ore and 50 per cent Deulgaon ore which theoretically would result in a slag containing 10 per cent TiO_2. With the increase of Khursipar iron ore in the ore blend, the slag became more viscous and it was considered that further increase of Khursipar iron ore in the blend will not be practicable. The chemical analysis of pig iron and slag with 50 per cent Khursipar ore in the ore burden is given in Table 76.

It was observed that the pig iron contained 3·24 per cent C which was above

Table 74 — Operational Conditions with 25 Per Cent High Titania Iron Ore in the Ore Burden

Operational variables	Data
Tuyere diameter	75 mm.
Average blast volume, Nm^3/hr	2300
Average blast pressure, mm.WG	2200
Hot blast temperature, °C.	500
Top gas temperature, °C.	500

Table 75 — Analyses of Pig Iron, Slag and Top Gas with 25 Per Cent Titaniferous Ore

Average analysis of pig iron

C %	Si %	S %	Mn %	P %	Ti %
3·0	3·8	0·01	0·7	0·27	0·21

Average analysis of slag

CaO %	SiO_2 %	FeO %	Al_2O_3 %	Mg %	TiO_2 %	S %
30·20	35·80	1·50	20·50	6·8	4·2	0·74

Average top gas analysis

CO %	CO_2 %	CH_4 %	N_2 %
25·4	4·21	3·6	Balance

Table 76 — Chemical Analysis of Pig Iron and Slag with 50 Per Cent High Titania Iron Ore in Ore Burden

Material	C %	Si %	S %	P %	Mn %	Ti %
Pig iron (1)	3·24	3·74	0·006	0·303	0·210	0·54
Pig iron (2)	3·24	3·64	0·024	0·238	0·114	0·42

	CaO %	SiO_2 %	FeO %	MgO %	Al_2O_3 %	TiO_2 %	S %
Slag	31·0	34·45	1·5	6·5	19·8	5·2	0·65

	CO %	CO_2 %	CH_4 %	N_2 %
Top gas	25·6	4·1	3·8	Balance

the normal average obtained during the low shaft furnace iron smelting and it was considered as due to the presence of titanium in pig iron. It was also noticed that partition of sulphur between the metal and the slag had also improved presumably due to the presence of titanium in the metal.

Investigation No. III

In this part of the investigation the low temperature carbonized coke made at the Central Fuel Research Institute from the wholly non-coking Wardha Valley coals constituted the fuel burden while the Khursipar ore mixed with Deulgaon iron ore yield, 25 and 50 per cent, in the blend. The furnace was operated under almost similar operational conditions and the smelting behaviour was somewhat similar with the exception that the coke rates with 25 and 50 per cent of iron ore in the blend were higher and irregularities in burden descent were observed due to the poor physical strength of the fuel. The analysis of pig iron, slag and top gas with 25 and 50 per cent high TiO_2 ore in blend are given in Tables 77 and 78.

Despite the addition of 25 and 50 per cent Khursipar ore in the blend, the high fuel rate necessitated the addition of large amount of limestone as flux with the result that TiO_2 in the slag was low.

Table 77 — Analysis of Pig Iron, Slag and Top Gas with 25 Per Cent Titaniferous Ore and Low Temperature Carbonized Coke (C.F.R.I.)

Average analysis of pig iron

C %	Si %	S %	Mn %	P %	Ti %
3·15	3·5	0·03	0·17	0·26	0·24

Average analysis of slag

CaO %	SiO_2 %	FeO %	Al_2O_3 %	MgO %	TiO_2 %	S %
33·3	31·5	1·2	22·2	7·0	3·8	0·7

Average top gas analysis

CO %	CO_2 %	CH_4 %	N_2 %
24·8	3·8	6·7	Balance

Table 78 — Analysis of Pig Iron, Slag and Top Gas with 50 Per Cent Titaniferous Ore and Low Temperature Carbonized Coke (C.F.R.I.)

Average analysis of pig iron

C %	Si %	S %	Mn %	P %	Ti %
2·23	2·66	0·006	0·14	0·43	0·45

Average analysis of slag

CaO %	SiO_2 %	FeO %	Al_2O_3 %	MgO %	TiO_2 %	S %
33·64	32·28	1·5	21·00	5·95	4·4	0·59

Average top gas analysis

CO %	CO_2 %	CH_4 %	N_2 %
26·00	4·0	3·9	Balance

Thirty-seventh Campaign

In this campaign the smelting trials were undertaken with Deulgaon iron ore, Rajur limestone of Maharashtra, dolomite employing pearl coke as the fuel and reductant. In view of the low cost of pearl coke in comparison with nut coke, it was considered that even a partial utilization of pearl coke in the fuel burden will assist considerably in improving the economics of iron production. However, in this trial pearl coke (40 per cent between 25 per cent +12 mm. and 40 per cent −12+6 mm.) was employed to the extent of 90 per cent while 10 per cent L.T.C. coke was added to assure permeability to the burden. The lime basicity of slag was kept at 1·0-1·2 and adequate dolomite was added to result 8-10 per cent MgO in the slag. The operational conditions were almost identical as have been mentioned earlier. The analysis of pig iron, slag and top gas in the steady state of operation are mentioned in Table 79.

In this campaign the top gas temperature was lower than that of nut coke, which was due to the lower particle size of the fuel. Higher blast pressure was not required for the desired blast volume and, therefore, the employment of pearl coke did not affect permeability of the burden. It was felt that industrial operation with blended fuel burden was technically and economically feasible.

Table 79 — Analyses of Pig Iron, Slag and Top Gas Employing Deulgaon Ore and Pearl Coke

Average analysis of pig iron

C %	Si %	S %	Mn %	P %
2·8	3·2	0·07	0·20	0·35

Average analysis of slag

CaO %	SiO_2 %	FeO %	Al_2O_3 %	MgO %	S %
38·3	35·3	0·6	20·7	4·3	0·6

Average top gas analysis

CO %	CO_2 %	CH_4 %	N_2 %
25·6	4·5	3·5	Balance

88.1 Investigation of Briquetting

Briquetting of Iron Ore Fines — In continuation of the investigations reported in the last Annual Report, further experiments were conducted for studying the bonding due to presence of coking coal from salt ore from Poniati collieries. The addition of coking coal was increased from 5, 10, 15 and 20 per cent with the particle size of −0·2 mm. The mix of iron ore fines and coal was preheated to temperature of 350-500°C. and fed to the roller briquetting press.

The determination of physical properties by shatter test and crushing strength showed that the room temperature crushing strength was poor due to the low preheating temperature and low pressure of the available roller briquetting press. The chemical analysis of the iron ore and the proximate analyses of the coals are given in Table 80.

It was considered that hot-briquetting would not be possible with the existing facilities.

88.2 Experimental Baby Blast Furnace for Studying the Effect of Nature of Metallic Burden and Injection of Auxiliary Fuels on the Smelting Efficiency

The objective of smelting in the baby blast furnace is to evaluate the effect of burden preparation, oxygen and fuel injection on the fuel rate and productivity.

Smelting trials were conducted exclusively with oxygen at room temperature. The

Table 80 — Analyses of Iron Ore and Coking Coals

I. Analyses of iron ore:

	64·14	3·28	4·57	0·01	0·02

II. Proximate analysis of coals (air dried basis):

	Moisture %	Ash %	V.M. %	Fixed carbon (by diff.) %	B.S.S. Coking Index
(i) Saltore	2·50	12·30	36·40	48·80	15
(ii) Poniati coal	2·37	14·63	32·38	50·62	14

attainment of temperature in the smelting zone was very high and affected the refractory lining. Further trials with prepared burdens are under way.

Table 81 — Change in Slag Composition with Blowing Time

	10 min.	13 min.	16 min.	20 min.
CaO, %	18·5	24·50	23·10	22·50
SiO_2, %	22·4	21·60	22·50	21·20
FeO, %	3·0	2·60	4·25	2·48
Fe_2O_3, %	24·10	19·40	18·40	19·85
MgO, %	28·20	24·80	24·60	27·20
P_2O_5, %	0·84	1·6	2·30	1·80
Al_2O_3, %	3·20	4·8	4·10	4·40

89.0 Malleabilization Characteristics of Low Shaft Furnace Pig Iron

In order to ascertain the malleabilization characteristics by dilatation technique of low phosphorus pig iron made with low-phosphorous non-coking coals in low-shaft furnace pilot plant, a pot furnace with salt-bath and a suitable dilatometer were fabricated. After the equipment offers consistent results, it will be used to study the effect of addition of boron and aluminium on the malleabilization cycle with a view to reduce it.

90.0 Pneumatic Method of Steel Making in Basic-lined Side-blown Converter

The processing of hot metal containing 0·9-1·4 per cent Si and 0·3-0·4 per cent P to low carbon steel in basic-lined side-blown converter was successfully developed and a patent on ' A Process for the Conversion of Molten Iron of Various Compositions to Different Grades of Steel by Employing Air for Oxidizing Reactions ' has been filed. The process has gained considerable industrial importance.

In order to study the factors affecting the removal of phosphorus, samples of slag were taken at different blowing periods. The analytical results of a typical blow are given in Table 81.

The results showed that dephosphorization commenced almost from the beginning of the blow. After attaining a maximum dephosphorization, the elimination of phosphorous decreased presumably due to its lower content in the molten metal and the higher temperature of the bath. Based on the kinetics of the removal of carbon and phosphorus with the adjustment of the operational conditions, attempts to preferentially oxidize phosphorus without significant elimination of carbon were not entirely successful, and further trials are under way. Arrangements for lime injection are being made, which is expected to accelerate phosphorus removal. Modification in the tuyeres are being done to improve the heat balance stirring of the metal and absorption of larger amount of scrap.

91.0 Investigations on the Cupola Melting Practice — Gas Analyses with Substitute Fuels

Extensive iron melting trials were conducted in an 1 tonne/hr experimental cupola to assess the possibility of substitution of foundry coke with large varieties of solid fuels. While several non-metallurgical fuels like low temperature carbonized cokes made from certain non-coking coals were satisfactory, the other varieties were unsuitable. It was, therefore, considered that the analyses of the combustion gases collected from the different stack heights of the cupola would indicate its thermal state. Arrangements for probes were provided in the cupola shaft for drawing gas samples and determination of temperature at these heights. The gas analyses were compared with that of hard coke. It was observed through a plot of compositional change of the gas with the stack height presented almost similar contour with the various alternative solid fuels, significant difference in the gas analyses at identical heights were observed. An attempt was made to judge the suitability of the fuel from the stack gas analyses, thermal efficiency and combustion ratio. The effect of the oxygen enrichment of the blast on the cupola performance employing either hard coke or various alternative fuels were thoroughly investigated.

92.0 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 investigations on the industrially oriented scrap-oxygen-steel making were undertaken. Although the oxidation of the undesirable elements occurs as in basic oxygen process, the employment of solid scrap at the atmospheric temperature in the vessel from the very beginning of the blow constitutes the major break through. The process will not necessitate installation of costly electric steel making furnace and dependence on cheap source of power. In continuation of the study of the various variables affecting the process, the effect of addition of progressively increasing amounts of steel scrap to pig iron scrap was investigated and it was noticed that 25 per cent of the former could be absorbed.

Effect of different additions of Fe-Mn on desulphurization was evaluated by its additions after the melting of the scrap in which the basicity degree of the slag and addition of solid fuel were held constant. The results are given in Table 82.

Table 82 — Ferro-Manganese Addition and Desulphurization

Fe-Mn addition %	'S' in scrap kg.	'S' in blown metal kg.	'S' removed %
Nil	0·08	0·079	1·25
1	0·093	0·090	3
2	0·08	0·076	5
3	0·08	0·069	13·7
4	0·08	0·048	40
5	0·08	0·040	50

It was found that higher amount of manganese in molten metal favoured desulphurization.

The effect of basicity (CaO/SiO_2) degree of the slag on dephosphorization was thoroughly examined by its variation from 1·4 to 2·5 at a constant fuel rate. The phosphorus contents in the blown metal decreased with the increase in the basicity degree of the slag. Results are given in Table 83.

Table 83 — Dependence of Phosphorus Removal on Basicity Degree of Slag

Basicity degree of slag CaO/SiO_2	'P' in scrap %	'P' in blown metal %
1·4	0·39	0·073
1·5	0·45	0·061
1·6	0·40	0·050
2·2	0·46	0·025
2·5	0·47	0·019

It is considered that the process has ample industrial potential for adaptation in steel foundries.

93.0 Reactivity of Solid Fuels Employed for Iron Smelting in Low Shaft Furnace

Reactivity of the fuel greatly influences the gaseous reduction in the stack of the blast furnace and thereby reflects on the fuel consumed. It is of greater significance in a low shaft furnace as the limited stack height requires fuel of optimum reactivity.

The general principle underlying the various empirical methods for determination of the reactivity of a solid fuel depends on oxidation with carbon dioxide, steam or air. The conditions of test are as follows:

(i) Size of coke — Through 14 & 22 mesh B.S.S.
(ii) I.D. of the transparent silica reaction tube — 2·0 cm.
(iii) Length of the portion of the reaction tube occupied by coke sample having thermocouple in position — 9·0 cm.
(iv) Temperature of coke — 950°C.
(v) Flow rate of CO_2 — 5 ml./min.
(vi) Volume of CO_2 used for each determination — 50 ml.

The temperature of coke is first raised to 950°C. within one hour in a current of pure, dry nitrogen and maintained at this temperature for another hour. Pure CO_2 stored in the gas holder of 200 ml. capacity is passed through the coke bed by displacement with conc. magnesium chloride solution.

The first 150 ml. of CO_2 is utilized to flush the system. The gases resulting from the passage of the remaining 50 ml. of CO_2 are collected in a nitrometer over caustic soda solution to absorb the unconverted CO_2. The volume of gases collected mainly consisting of CO_2 is directly taken as an index of reactivity of the coke towards CO_2.

Experiments were carried out with the following coke samples:

(i) Pearl coke (Bhilai).
(ii) Lodhna coke.
(iii) L.T.C. (Ballarpur) (C.F.R.I.).
(iv) C.F.R.I., C.T.C. (Raniganj coals).
(v) L.T.C. (Mazri), (C.F.R.I.).
(vi) L.T.C. (Wardha Valley coals) (R.R.L.).
(vii) L.T.C. (Ghughus) (C.F.R.I.).

The reactivity values together with the proximate and ash analyses of these cokes are given in Table 84. Attempts are made to correlate the reactivity values with the fuel rate in low-shaft furnace.

94.0 Production of Pig Iron from Run-of-mine Iron Ore, Lime Stone Fines and Coke or Coal Breeze, in a Rotary Furnace

Extensive investigations were undertaken to obtain molten pig iron from iron ore fines, limestone dust and small particle of solid fuels such as coke breeze or low temperature carbonized coke which are unsuitable to produce hot metal by any of the conventional processes.

Several varieties of iron ore fines were smelted with coke breeze, while two different varieties of limestone were used. Investigations were then conducted with a suitable ore employing L.T.C. breeze and also non-coking coal breeze as fuel. The chemical analyses of raw-materials and screen analysis are given in Tables 85 to 90.

Table 84 — The Reactivity of Various Indian Cokes

Coke	Reactivity av. of 3	Proximate analysis					Analysis of ash				
		Moisture %	Ash %	V.M. %	F.C. %	CaO %	SiO_2 %	MgO %	Al_2O_3 %	Fe_2O_3 %	P %
Pearl Coke (Bhilai)	51·90	0·8	21·7	1·4	76·1	3·2	53·94	4·9	29·2	—	0·3
Lodhna Coke	63·70	1·7	25·5	4·5	68·4	2·2	56·9	Tr.	25·8	14·8	0·4
Ballarpur L.T.C.	71·70	2·5	34·7	8·2	54·6	6·1	54·6	0·8	28·96	6·5	0·6
C.F.R.I. L.T.C·	85·10	8·5	25·9	4·1	61·5	5·68	83·8	1·88	28·0	4·6	0·08
Mazri L.T.C.	88·96	4·1	40·2	3·7	52·0	1·2	60·8	0·6	35·9	0·9	0·1
Maharashtra Kolshit	92·10	0·75	31·7	13·1	54·4	9·5	50·1	Tr.	31·0	8·8	0·1
Ghughus L.T.C.	98·70	2·1	48·1	6·3	43·5	2·6	54·2	0·6	33·2	8·8	0·2

Table 85 — Analyses of Raw Materials

	H_2O %	Ash %	V.M. %	F.C. %	S %
Coke breeze (T)	2·50	26·0	1·50	70·10	0·50
Non-coking coal(s)	7·0	17·4	26·1	49·50	—
L.T.C. (K)	2·4	19·3	9·2	69·1	—

After obtaining a metal pool in the revolving furnace, the precalculated charge consisting of iron ore dust, limestone fines and the coke or coal breeze was charged intermittently into the furnace. The CO liberated inside the furnace was burnt by oxygen through consumable lance, which mainly provided the thermal requirements.

Regardless of the solid fuel employed, the pig iron analysed C, 3·5 and silicon, 0·3; phosphorus, 0·3-0·05 and sulphur, 0·1 per cent. The average lime/silica ratio in slag was 1:1 and the FeO contents between 2·5 to 4·5 per cent. The high sulphur was due to low basicity degree of the slag.

The results of investigations are very promising and the process is suitable for industrial adaptation.

95.0 Apparaisal of Raw Material for Iron Making

(i) *Iron Ore: Physical & Chemical Characteristics* — The determination of the reducibility and the decrepitation characteristics of iron ore and dissociation behaviour of limestone are of great importance for iron smelting, apart from the other equally important properties, viz. physical strength, porosity, etc.

Results of physical tests, porosity and decrepitation characteristics of Khursipar ore of Maharashtra and some iron ores from Madhya Pradesh are reported in Table 91. The decrepitation temperature varied from 360 to 460°C. The shatter strength varied

Table 86 — Analysis of the Ashes of Fuels

	SiO_2 %	CaO %	Al_2O_3 %	MgO %	Fe %	P %
Coke breeze (T)	50·90	3·10	25·50	1·80	4·8	0·27
Non-coking coals (S)	65·6	2·3	22·80	1·4	6·3	0·058
L.T.C. (K)	66·3	0·7	23·3	2·2	4·2	0·028

Table 87 — Analysis of Limestone (B)

CaO %	SiO_2 %	Al_2O_3 %	MgO %
44·88	6·96	1·60	3·27

Table 88 — Analysis of Iron Ore (O.M.)

Fe %	SiO_2 %	Al_2O_3 %	CaO %	MgO %
64·14	3·28	4·57	Trace	Trace

from 88·8 to 98·7 per cent on +12 mm. fraction whereas abrasion strength varied from 65·1 to 90 per cent on +60 mm. fraction. Result of the reducibility test with H_2 gas as reductant of Orissa Minerals, Khursipar, Deulgaon, B. Patnaik, Gua, Meghatuburu, Bolani and Taldih iron ores are recorded in Table 92. The particle size was maintained at −6·4+3·2 mm. In view of the lowest fuel rate obtained with Orissa Mineral iron ore, it was taken as standard ore for comparing the reducibility of other iron ores. In comparison, all the ores had inferior reducibility as shown in Fig. 66. The decrepitation test simulate the effect of time, temperature and furnace atmosphere on iron bearing materials during their descent in furnace stack and may probably indicate the breakage due to these factors. Results are given in Table 91.

(ii) *Limestone*: *Dissociation Characteristics of Limestone* —During the descent of the burden in the stack of the smelting furnace the limestone in the burden, due to endothermal dissociation, consumes large amount of heat. The dissociation reaction of limestone should, however, be completed before it descends to the bosh area of the furnace or before reaching the high temperature zone, to minimize the solution loss reaction as far as practicable.

The dissociation characteristics were studied by heating the sample at predetermined rate up to 950°C., which to a certain extent simulated the progressive increase in temperature during the descent in the furnace stack. The results are given in Fig. 67. The dissociation commenced between 730° and 800°C., and was completed in the range of 900-950°C. On attaining 950°C. isothermal dissociation rate was determined. The results are given in Table 93.

96.0 Study on Sulphide Capacities of Slag

The experimental set up for studying partition of sulphur between carbon saturated iron and slags of different basicity

Table 89 — Screen Analysis of the Raw Materials

Name of 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 below
Iron ore (OM)	—	—	2·30	19·54	26·36	51·80
Limestone (B)	—	2·79	59·81	29·38	4·84	3·18

Table 90 — Particle Size of Fuels Employed for Smelting

Name of the fuel	+7 mesh sieve	−7 mesh sieve +16 mesh sieve	−16 mesh sieve +30 mesh sieve	−30 mesh sieve +60 mesh sieve	−60 mesh sieve +100 mesh sieve	−100 mesh sieve & below
Coke breeze (T)	8·60	21·66	30·14	15·27	10·67	13·66
Non-coking coals (S)	20·50	18·43	16·03	10·37	14·07	20·60
L.T.C. Breeze (K)	24·30	17·69	20·39	19·70	12·54	5·40

Table 91 — Physical Properties of Iron Ore

Iron ore	State	Shatter strength on 12·0 mm. size		Abrasion strength on 6·0 mm. size			Macro-structure examination	Decrepitation temp. °C.	Screen analysis after decrepitation test			Porosity %
		−76 +50 mm. %	−50 +25 mm. %	−76 +50 mm. %	−50 +25 mm. %	−25 +12 mm. %			−12·7 +6·4 mm. %	−6·4 +3·2 mm. %	−3·2 mm. %	
Khursipar	Maharashtra	—	98·7	—	90·0	92·5	Massive & hard	400-440	98·6	1·0	0·4	2·86
Goethete variety	Madhya Pradesh	83·4	79·9	38·0	38·8	52·5	do	400-412	97·7	1·8	0·5	10·04
Friable variety	do	93·8	94·7	80·8	84·1	82·8	Mostly laminated	375-406	98·0	0·75	1·25	21·94
Compact variety	do	94·5	93·0	61·5	61·5	74·5	Porous	400-460	99·6	0·3	0·1	23·12
Compact laterite variety	do	91·0	88·8	—	—	—	do	360-385	98·0	1·5	0·5	29·7

Table 92 — Reducibility Test Results

Iron ore	State	Size −6·4 +3·2 mm. at 900°C. 800 cc./mm.		Size −12·7 +6·4 mm. at 900°C. 800 cc./mm.	
		Reduction	Time min.	Reduction	Time min.
Orissa minerals	Orissa	92·1	32	91·7	32
Kursipar	Maharashtra	75·4	65	66·7	65
Deulgaon	do	78·0	50	55·0	60
B. Patnaik	Orissa	89·1	90	85·1	90
Gua	Bihar	78·5	70	70·1	85
Meghatuburu	Orissa	85·9	90	64·6	75
Bolani	do	88·9	72	81·7	80
Taldih	do	84·2	95	79·6	90

Table 93 — Calcination Characteristics of Limestone

Limestone	Initial temperature of calcination °C.	Total CO_2 evolved in litres	Time min.	Size of limestone mm.
Madras	785	20·7	140	−190 +12·7
Andhra Pradesh	745	6·5	100	do
Nepal	790	17·97	150	do
Rajur (Maharashtra)	820	15·5	100	do
Dabok (Rajasthan)	845	14·85	180	do
Bisra	780	22·6	190	do
K.K.T.	800	16·8	190	do
Ch. & Dutta	730	24·6	210	do

Table 94 — Analyses of Synthetically Made Irons

Sample No.	C %	Si %	Mn %	S %
1	2·93	1·79	0·80	0·13
2	2·84	1·79	0·74	0·21
3	2·66	0·79	0·80	0·08
4	2·79	1·85	0·79	0·05

degrees under various oxygen potentials was described in the previous Annual Report. For studying the transfer of sulphur for the metal to slag phase, synthetic iron mainly differing in sulphur contents

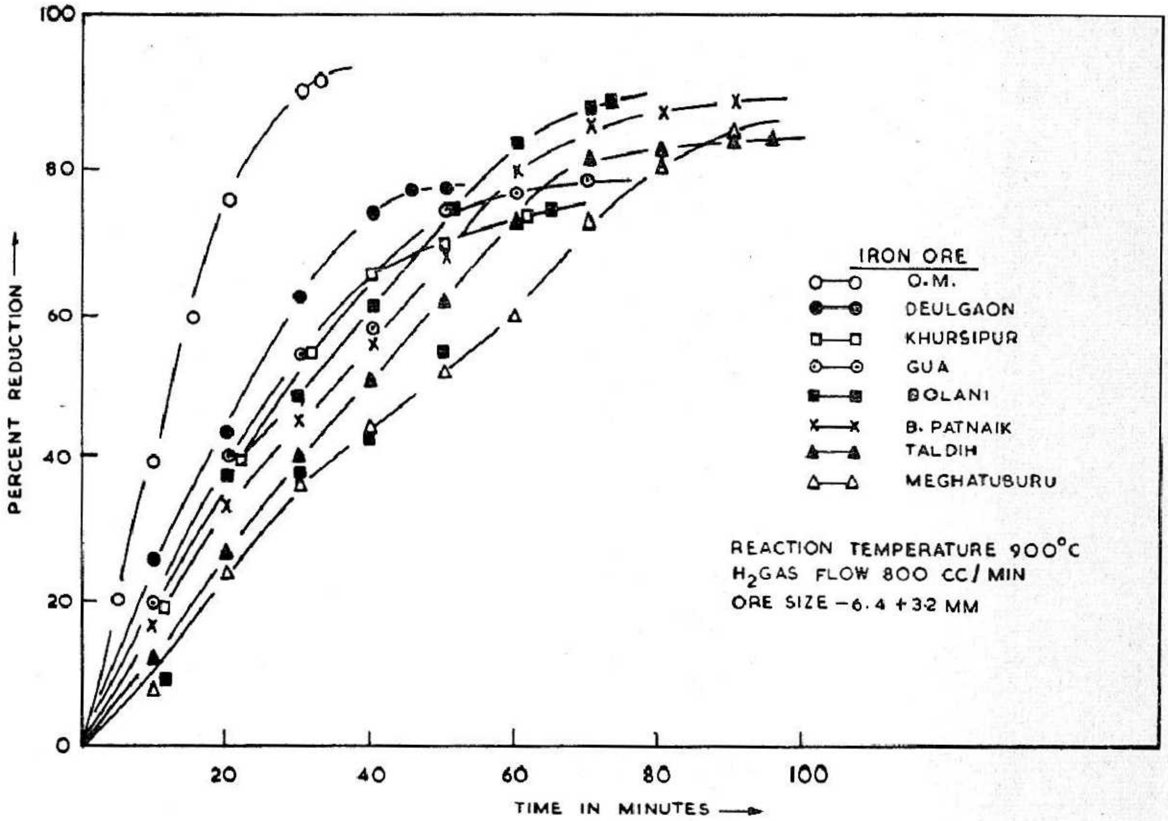


FIG. 66 — COMPARISON OF THE REDUCIBILITY OF DIFFERENT IRON ORE SAMPLES

are made, compositions of which are given in Table 94.

Apart from these samples, pig iron made in the low shaft furnace pilot plant analysing C, 3·64; Si, 1·77; Mn, 0·97 and S, 0·34 per cent was employed. Due to the limitation of the temperature attained in the platinum —10 per cent rhodium furnace, the study of transfer of sulphur to a large variety of slags of different compositions was not practicable and restricted to slags mentioned in Table 95, for initial studies.

Table 95 — Composition of Slags Employed

Sample No.	CaO %	SiO_2 %	Al_2O_3 %	MgO %
1	48·0	52·0	—	—
2	46	40	13	—
3	41	45	—	14
4	36	38	—	26

The melt was kept at 1450-1480°C. surrounded by a gas mixture of CO, CO_2 and N_2 flowing at the rate of 120, 60 and 20 cc./min., respectively, for measuring the rate of transfer of sulphur from the metal to slag phase. Further work is under way.

97.0 Electrolytic Manganese Metal Pilot Plant

During the year under review, two types of ores having the following compositions were investigated.

	I	II
Mn, %	37	52·7
Fe, %	17·6	3·8
SiO_2, %	4·28	4·6
MnO_2, %	52·6	77·1

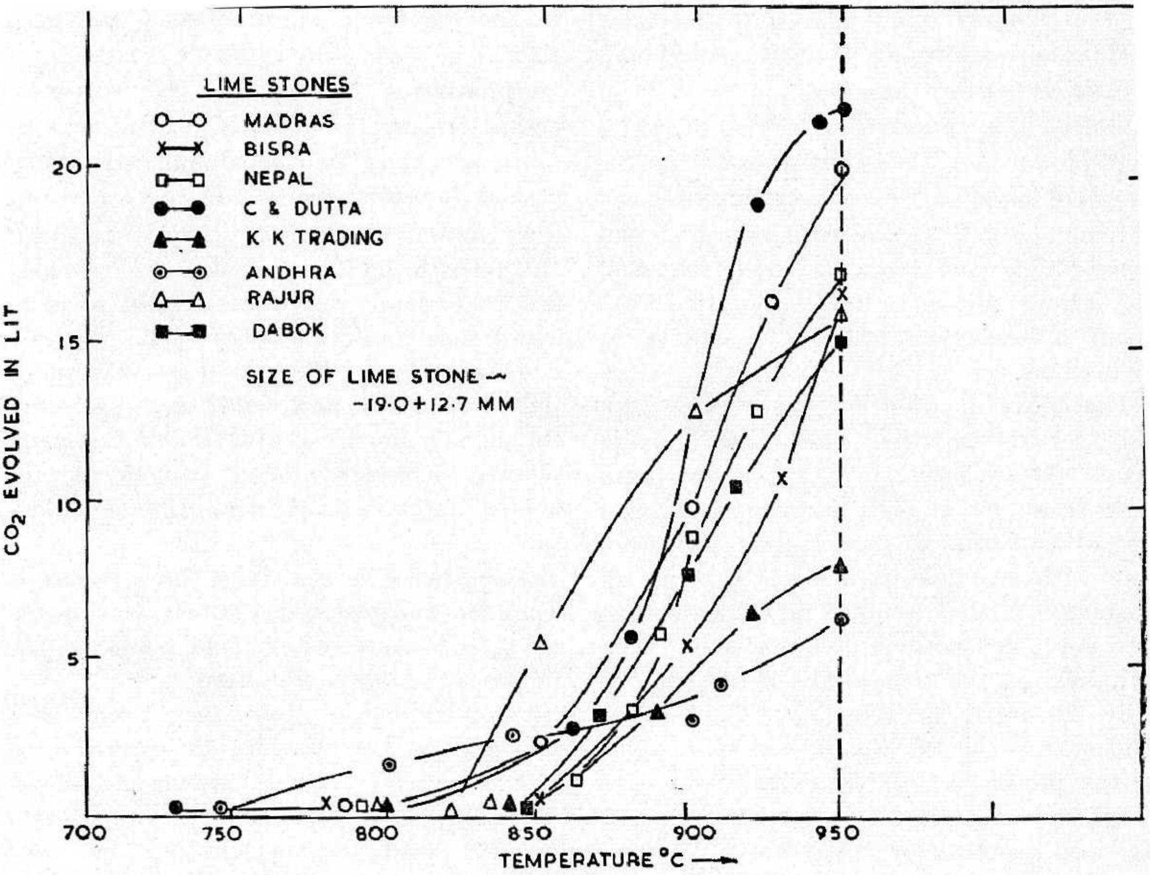


FIG. 67 — DISSOCIATION CHARACTERISTICS OF DIFFERENT SAMPLES OF LIMESTONE

The ore were ground to the standard size −35+75 mesh. The reduction characteristics of the ores were studied and a 95 per cent reduction was obtained by passing the ore twice through the rotary kiln. It was also found that the ores had in general a tendency to agglomorate in the reaction zone of the furnace while coming in contact with the moisture formed by the reduction reaction. This caused the choking of the rotary kiln pipe which necessitated frequent cleaning of the ore in side of the kiln pipe.

The reduction losses of the furnace were studied and it was found to be negligible. There was hardly a difference of 1°C. between the rotary furnace outer surface and the atmosphere. The leaching characteristics and leaching efficiency were also studied. The leaching efficiency in general was found to be 95 per cent approximately. The settling characteristics of both the ores were compared and it was found that the rates of settling in both the cases did not differ appreciably. The recovery of manganese in the leach from the higher grade ore was naturally higher as the amount of ore used for leaching was small and less solution was entrapped in the sludge. But the amount of entrapped pregnant solution was appreciable and a washing arrangement for the recovery of the same was found to be necessary.

The fluctuations of the catholyte *p*H caused by the anolyte leaking from the lower chamber of the cell to the upper one were stopped by modifying the assembly of the wooden diaphragm and the false bottom with additional fittings. An overall C.E. of 63 per cent was found in the cell.

During the period, two tonnes of metal were produced out of which more than 200 kg. were supplied for research activities for development of Ni-free stainless steel and special high and low carbon, ferrous and non-ferrous alloys in the Laboratory and about a tonne was utilized for a trial in steel plant.

The above mentioned types of ores were also tested for the manganese dioxide plant. The graphite and carbon electrodes were tried but though manganese dioxide free of lead was produced, difficulties were met with in making it free of acid by washing. It took about 5 days of vigorous washing to get the dioxide free of acid. The stripping of the deposited dioxide practically presented no difficulty. The current efficiency of the cell was 90 per cent. Out of the produced dioxide about a ton was supplied to outside firms and some dioxide was also supplied for research.

98.0 Pilot Plant for Hot-dip Aluminizing of Steel

Aluminizing of Maleable Cast Iron — Strain clamps, socket eyes, round test bars and other hardware items were aluminized for The Malleable Iron & Steel Castings Co. Private Ltd, Bombay. Eighty-eight samples were aluminized for M/s. Tubes & Malleables, Madras. These included T's, elbows, unions and reducers. Insulator caps for H.T. transmission towers for Kolay Iron & Steel Co., Calcutta, were also aluminized. Samples of water meter bodies were aluminized for Government Precision Instruments Factory, Lucknow.

Aluminizing of Strips for CBRI, Roorkee — Steel strips were aluminized for CBRI for study of the feasibility of employing these in the fabrication of a solar energy unit. For a successful application it was desired to anodize the aluminized steel and then give it a black copper oxide coating.

Aluminizing for EMC — The Electrical Manufacturing Company, Calcutta, sent us some wire and had it aluminized. They wished to go in for ACSR core wire but their material analysed C, 0·45, Si, 0·15; Mn, 0·65; S, 0·05 and P, 0·03. This being low in carbon they were advised to send material as per ASTM B 341-63 T.

Aluminizing of Wire for Fort William — Wires of 0·048 and 0·040 in. dia. were aluminized for the Fort William Company Limited, Konnagar (West Bengal), for intended application in wire rope manufacture.

Aluminizing of Corrosion Test Panels — Some more corrosion test panels were aluminized and supplied for field trials to the Metals Research Committee.

Specification for Pole-Line Hardware — A tentative specification on thickness of coating, preece test, stripping test and salt spray corrosion test for aluminized pole-line hardware was drafted and sent to General Manager, P & T Workshops, Calcutta.

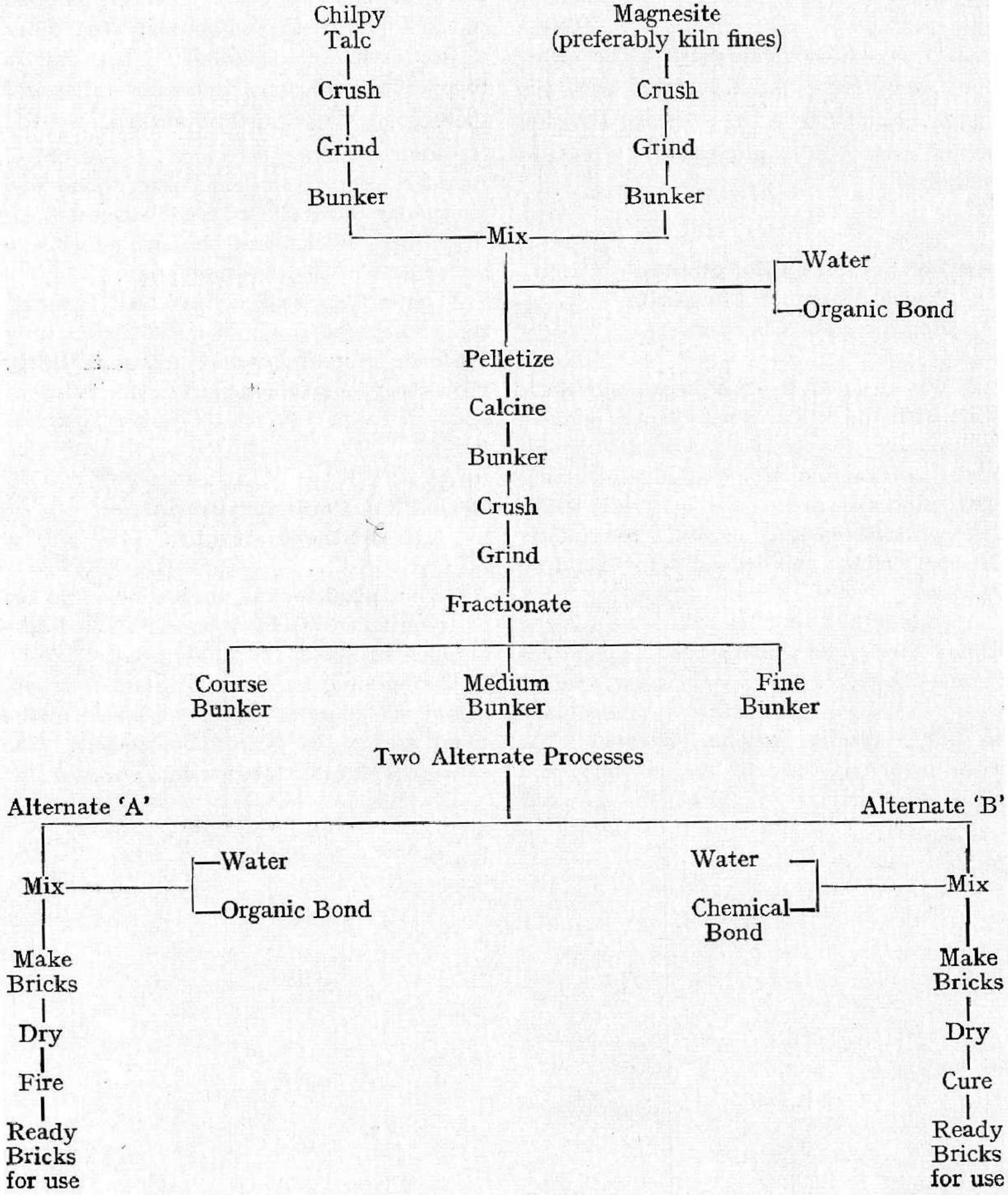
Aluminizing for M/s. Steels Worth — Three hundred and five kilograms of 10, 12 and 14 SWG HB wire was aluminized for barbed wire fencing manufacture and field trials for M/s. Steels Worth Pvt. Ltd, Tinsukia, Assam.

Aluminizing for M/s. K.T. Steel — One hundred kilograms thin 20 to 30 SWG wires were aluminized for K.T. Steel Industries, Bombay. The optimum conditions worked out were 50 fpm at 780°C. with an effective traverse in bath of 2 ft. withdraw at 45° and air cool height of pulley lowered to only 4 ft. The sinker was replaced by light freely moving pulleys.

Window Grills for Digha — A few hundred window grills were aluminized for trials at the Marine Corrosion Research Station at Digha.

Aluminizing at NASCREW — NML staff visited the works of M/s. National Screw &

FLOW-SHEET FOR PRODUCTION OF FORSTERITE BRICKS



Wire Products at Belur for commissioning their small experimental plant and advising on scale up of same to a full scale commercial unit.

Autrometer Filter Pump Parts—The autrometer filter pump parts supplied with the original equipment in Ore Dressing Division having rusted were aluminized to extend their lives.

99.0 Refractories Pilot Plant — Development of Forsterite Regenerator Checkers

It was decided to co-ordinate the NML work with the Bhilai Steel Plant's requirements and the project was reoriented, firstly, to examine the possibility of fitting NML processes to the raw materials available with Bhilai and secondly to provide data on which a full-fledged plant could be designed.

Accordingly a talc sample sent by the Bhilai Steel Plant along with magnesite obtained by them from indigenous sources were examined and their amenability to NML process was investigated. The work involved study of the crushing and pelletizing characteristics of the raw materials, their sintering at various temperatures and production of checker bricks from the sintered pellets thus obtained. Two lines of approach were examined. The first is to produce a chemically bonded brick and the second is to produce a brick by the traditional baking at high temperature. Based on the preliminary work done two alternative flow sheets were suggested to the Bhilai Steel Plant authorities. These are at present under examination.

A patent application was filed covering the technique of chemically bonding forsterite grog to produce forsterite brick. Flow sheet for production of forsterite bricks is given in p. 149.

100.0 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 Laboratory.

The building for the pilot plant is ready. A control laboratory has been added to the plant during the year under review. The procurement of the equipment is in progress.

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 resistant superstrength alloys invariably contain considerable amounts of Ni, Co, W, etc., as alloying additions, in respect of which India lacks in raw material resources. Work was, therefore, undertaken on development of substitute high temperature alloys, utilizing indigenously available materials. With this in view high temperature creep characteristics of Cr-Mn-N type austenitic steels are being studied. As earlier reported encouraging creep test results were obtained with steels having C/N ratio of 1:1. A proper solution treatment temperature was found to be in the range of 1050°-1100°C., in which case the material showed optimum creep strength and rupture ductility.

The work extended further to determine creep properties of the alloys at 700°C. A set of creep curves obtained at 700°C. and for a range of stresses for one of the alloy with lower C content and solution treated at 1075°C. for 1 hour and water quenched are shown in Fig. 68. The minimum creep rates, rupture time and rupture elongation are separately given in Table 96. The rupture elongation is highest at intermediate stress and it appears to be controlled by progress of precipitation during creep testing. Further tests on other alloys in the group are in progress. Metallographic examination of the longitudinal section of the specimens tested under the various stresses revealed, as shown in Fig. 69, that the fracture was intergranular and precipitation processes had progressed to various stages depending upon the rupture time and creep stress.

With a view to study the effect of stress on the ageing behaviour of these alloys, the age-hardening characteristics of the alloys are being studied at 650°, 700° and 750°C. For this purpose specimens were solution treated at 1075°C. for 1 hour followed by ageing for various periods of time extending up to 1000 hours. In case of the alloy with lower C content the initial hardness was low and there was no significant rise in hardness on ageing, whereas in the alloy with higher C there was some increase in hardness with time depending upon the ageing temperature.

Alloys based on Cr-Mn-N austenite composition were made in high frequency

Table 96 — Creep Properties at 700°C.

Sp. No.	Stress kg./mm.^{2}	Rupture time hr	Rupture elongation %	Min. creep-rate hr
L2-1	10	1774	16·5	$3{\cdot}5 \times 10^{-5}$
L2-2	15	585	22·0	10×10^{-5}
L2-3	18	226	11·0	$27{\cdot}7 \times 10^{-5}$

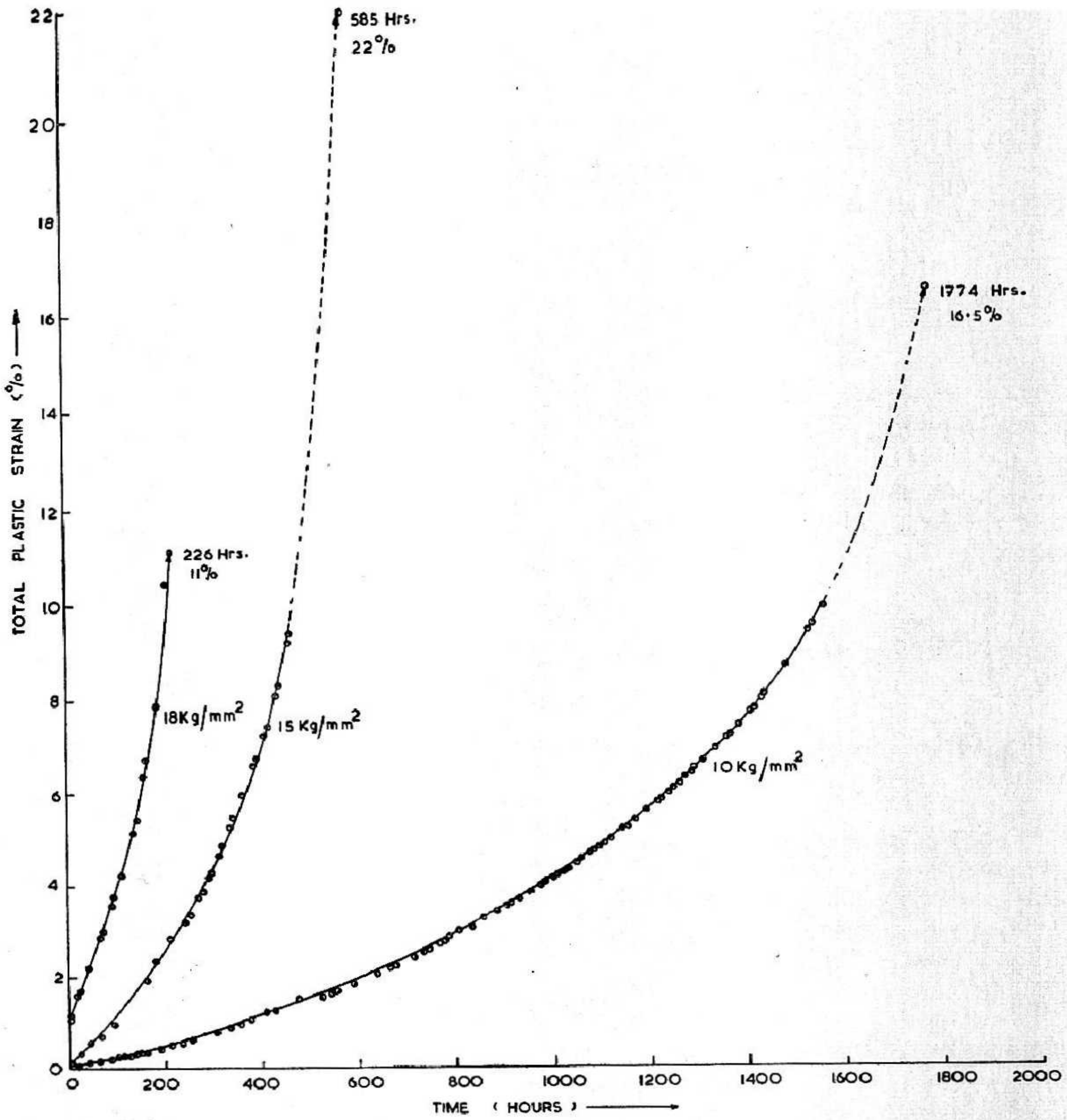
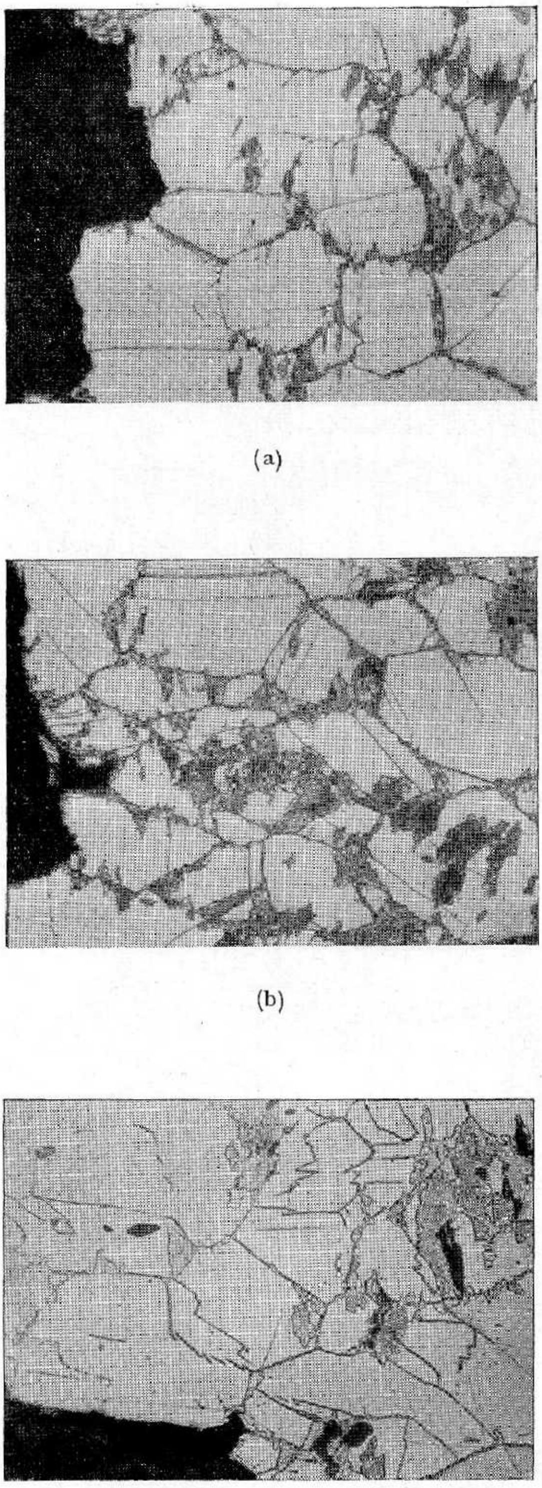


FIG. 68 — A SET OF CREEP CURVES OF LOW C-CR-MN-N AUSTENITIC STEEL AT 700°C. AT VARIOUS STRESS LEVELS

melting furnace with varying amounts of Ti, Al and W in small amounts singly or in combination with the others. The 10 kg. ingots were hot forged to bars of about 1¼ sq. in. size. Studies of their ageing and creep characteristics are in progress.

Besides the work on development of substitute creep resistant alloys as outlined above, it is aimed to help the Indian industries in the evaluation of high temperature creep properties of the conventional high temperature steels, currently produced in the country or planned for production in the near future. It has been necessary because the current specifications and design codes employ high temperature proof stress


(a)

(b)

(c)

FIG. 69 — MICRO-STRUCTURES OF LONGITUDINAL SECTIONS OF CREEP FRACTURED SPECIMENS. (a) L2-1, (b) L2-2, AND (c) L2-3

and long time creep and rupture properties as the design criteria. The problem of collecting high temperature test data has assumed major importance since the demands for indigenous materials for use in high temperature engineering components has increased as a result of the growth of basic heavy engineering industries for the manufacture of boiler, pressure vessels, and steam and gas turbines in the country.

As the long-time high temperature creep testing, lasting for several years, is highly complex, time-consuming and expensive and since metallurgical variables play significant role in the long time high temperature creep behaviour of alloys, it is aimed to have a centralized project on high temperature creep testing, undertaken at National Metallurgical Laboratory in close collaboration with the various organizations concerned with the manufacture, design and/or use of high temperature steels and other alloys.

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, electrodeposition, mineral ore dressing, radio-activation analysis for determination of the minute quantity of elements (10^{-8} to 10^{-12} g.), etc. Radioactive isotopes will also be used for industrial studies like study of processes in blast furnace 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,

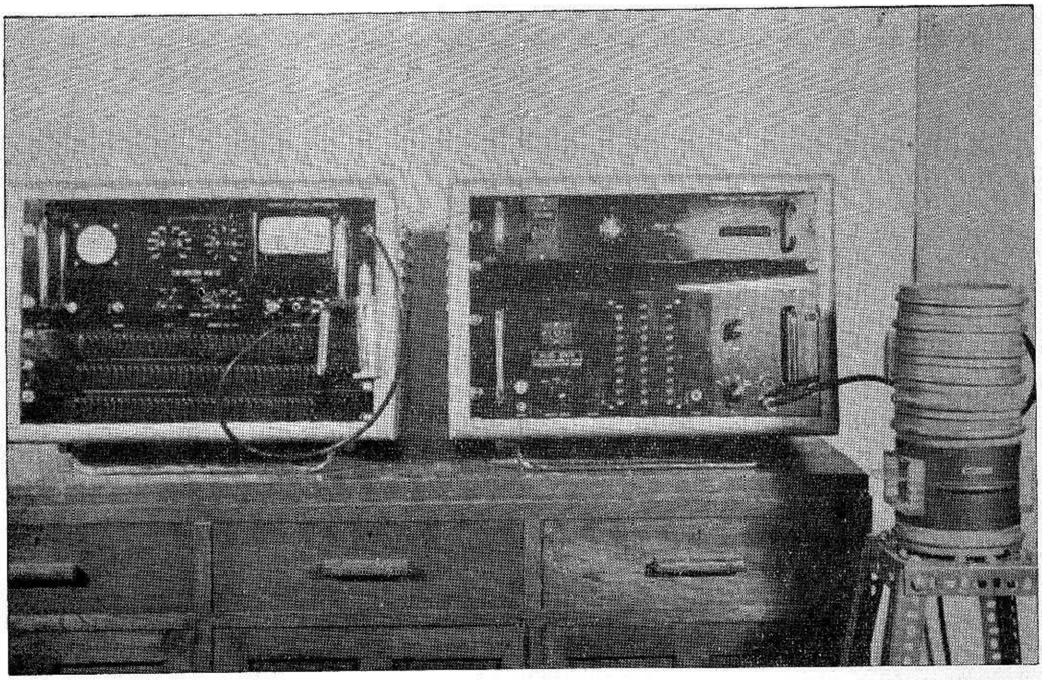


FIG. 70 — CONTAMINATION MONITOR AND SCALER COUPLED WITH TIMER FOR RADIO-ISOTOPE STUDY

Trombay. With the above objectives in view the setting up of the Radio-isotope Laboratory made considerable progress. Following electronic instruments and accessories were set up for the Radio-isotope Laboratory.

Electronic Instruments

1. Preset timer (Type ET 450).
2. Decade scaler (Type DS 325).
3. Contamination monitor (Type CM 167).

Accessories

1. Lead castle (Type C 1700).
2. Geiger tube stand (Type M 1900).
3. Thin wall GM detector (Type I 1010).
4. Aluminium ducting of welded construction for the ventilation system.
5. Centrifugal pump sets for the inlet and exhaust air system of the ventilation plant.
6. End window CM detector (Type I 1030).
7. Hypodermic syringe for remote control pipettor.
8. Iron clad switches.
9. Sample slide assembly.
10. Fume hoods.
11. Omat fibre filters.
12. Direct-on-line air break starters.
13. Belt drive package for the fans and motors of the ventilation system.
14. Necessary connections of the fume hoods, and betagamma boxes to the exhaust air system.
15. Necessary absolute filter box for the ventilation plant.
16. Isotope safe for storage of active samples.
17. Water trap for the ejector system of the glove box.

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 fume hoods, required for the tracer studies in metallurgical research problems, and one isotope safe for the storage of active specimens were fabricated in the Laboratory. A plant room for the inlet air required for the ventilation plant of the Radio-isotope Laboratory had been constructed.

In connection with the setting up of the Radio-isotope Laboratory the following programme of work is in progress.

1. The installation of the proper ventilation plant in the Radio-isotope Laboratory.
2. The fabrication of the necessary waste transport containers for the disposal of the high level active liquid and solid wastes which would result from the tracer studies in metallurgical research problems, in the NML Workshop in consultation with the Atomic Energy Establishment, Trombay.
3. The installation of air conditioning unit for the conditioned air required for the non-active area and the counting room.
4. The painting of the walls of the Radio-isotope Laboratory with high finish plastic emulsion paint.
5. The laying out of linoleum sheet on the floor of the active area of the Radio-isotope Laboratory.
6. Making necessary arrangements for irradiating the specimens for radio-tracer studies, in the Atomic Reactor Piles available with the A.E.E.T.

Standard Metals Development Group

During the year under review the following standard samples were prepared.

Standard Cast Iron Samples — Millings (6·7 kg.) of cast iron sample were obtained 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 analysis is given below:

T.C.	3·34%
C.C.	0·36%
G.C.	2·98%
Si	2·28%
Mn	1·05%
S	0·044%
P	0·045%

Standard Steel Sample — The analysis of the sample was done in the Laboratory and was found to be in agreement with the other co-operating laboratories. The analysis is given below:

Total carbon	0·21%
Silicon	0·089%
Sulphur	0·036%
Phosphorus	0·025%
Manganese	0·58%

During the period, standard cast iron and steel samples were supplied to twenty firms.

SUMMARY OF RESEARCH THESIS SUBMITTED FOR THE AWARD OF DOCTORATE DEGREE BASED ON THE WORK UNDERTAKEN AT THE NATIONAL METALLURGICAL LABORATORY

Studies on the Physico-mechanical Properties of Refractories

Considerable work has been done during the last two decades in U.K. and U.S.A. on the examination of micro-structure and physico-mechanical properties of basic refractories in the context of their service performance in steel-melting furnaces. However, it was felt that a systematic investigation, correlating the technological factors in the production of these refractories with their physico-mechanical properties would go a long way in further improving their quality. The work reported in the thesis was, therefore, undertaken keeping this objective in view. The work was broadly divided into two aspects, namely (1) effect of composition and firing temperature on the constitution, micro-structure and physico-mechanical properties of magnesite and chrome-magnesite refractories, and (2) role of forming pressure on the properties of basic refractories prepared under industrial condition.

The effect of composition and firing temperature were studied on (1) magnesite compositions prepared from four naturally occuring magnesites, and (2) 70: 30 type chrome-magnesite compositions prepared from mixes containing different fraction of a specially processed chrome ore and one of the above magnesites. The CaO/SiO_2 ratio (by weight) of magnesite compositions varied between 0·21 and 1·16. The silica content of the chrome-magnesite compositions varied between 3 and 5 per cen (3 and 6 per cent). The changes in th constitution and micro-structure of tes specimens fired between 1500° and 1700°C were examined by X-ray powder diffraction and microscopical methods, the latter included examination of polished and thin sections under reflected and transmitted lights respectively as also in powder mounts. The completion of reactions between

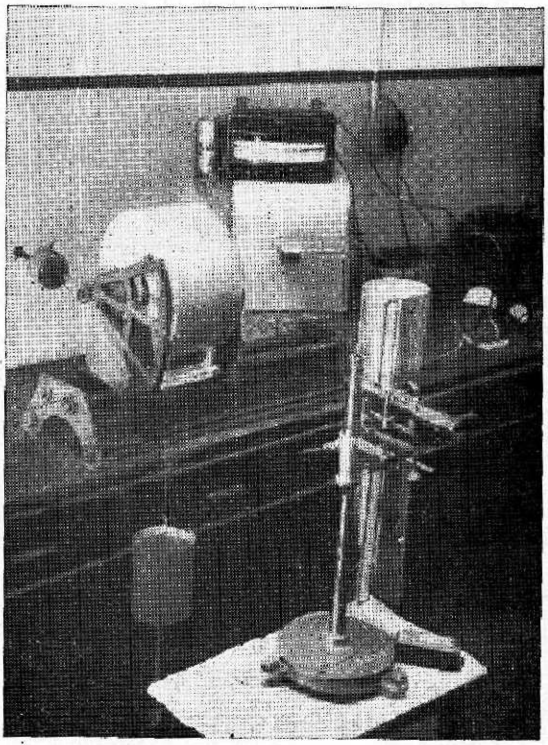
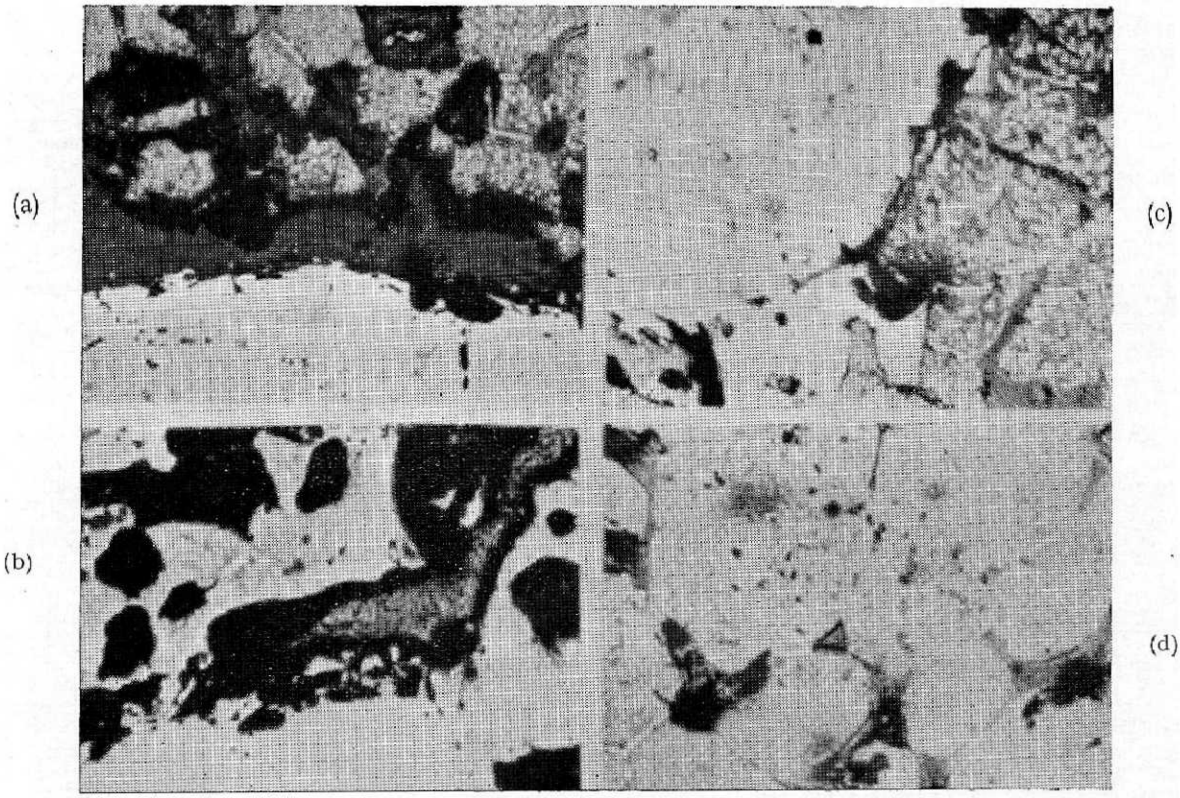


FIG. 71 — APPARATUS FOR TESTING HIGH TEMPERATURE TORSIONAL PROPERTIES OF REFRACTORIES



(a) 1500°C.; gap around the chrome grain
(b) 1600°C.; some direct bond developed between chromite and periclase
(c) 1700°C.; extensive direct bond development between chromite and periclase
(d) 1700°C.; growth of periclase grains in the matrix and relatively low silicate content in the matrix

FIG. 72 — MICRO-STRUCTURE OF CHROME-MAGNESITE TEST SPECIMENS CM3 FIRED TO DIFFERENT TEMPERATURES (REFLECTED LIGHT)

magnesia and chrome spinel in chrome-magnesite test specimens fired to different temperatures was determined by measuring the lattice parameter of the spinel (solid solution by X-ray studies.

The physical properties examined were (1) linear change on firing, (2) apparent porosity and bulk density, (3) cold compression strength, (4) volume change on reheating at 1700°C. (5) thermal shock resistance, and (6) refractories under load. Standard methods were employed for the determination of these properties.

A high temperature torsion apparatus (Fig. 71) first of its kind to be fabricated and used in this country was employed for studying creep and permanent set of magnesite test specimens at 1300°C. As it was considered that the bond-structure of basic refractories might have an influence on their thermal shock resistance as also on the tendency of chrome-magnesite refractories for bursting due to iron oxide attack, these aspects were also investigated. The study on the role of forming pressure on the physico-mechanical properties of basic bricks was carried out on magnesite, magnesite-chrome and chrome-magnesite refractory mixes taken from the normal production run of a basic refractories plant. The test bricks were pressed in hydraulic press under different pressures between 700 and 1100 kg./cm.^{2}. In addition to the physical properties mentioned above, the high temperature load bearing characteristics of the bricks were studied by

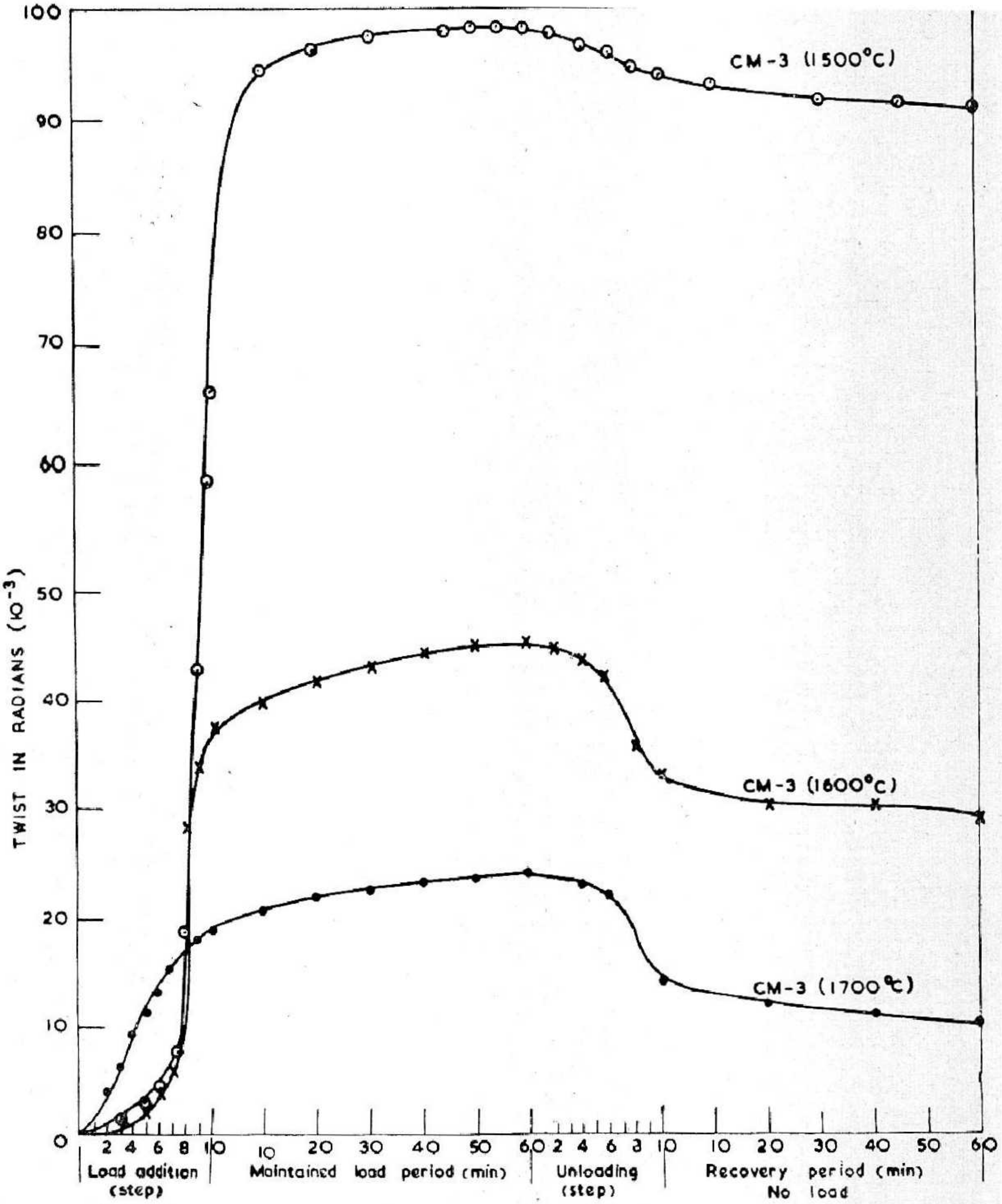
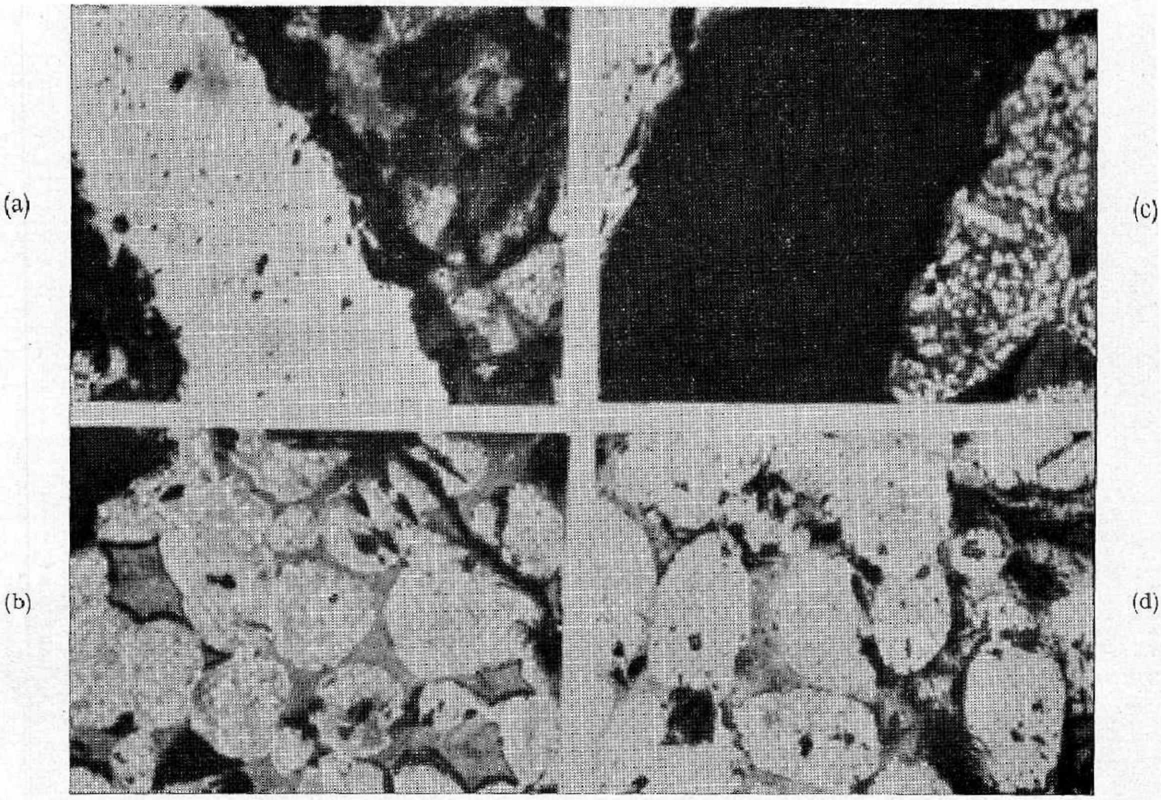


FIG. 73 — SHORT TIME TORSIONAL CREEP AT 1300°C. OF CHROME-MAGNESITE TEST SPECIMENS CM3 (CONTAINING APPROXIMATELY 3 PER CENT SiO_2) FIRED TO 1500°, 1600° AND 1700°C.



(a) 1500°C.; Porous texture; cavity around the chrome grain
(b) 1600°C.; large quantity of silicate in the matrix; rounded periclase grains
(c) 1700°C.; enlarged width of the gap around the chrome grain; well developed rim of rounded periclase
(d) 1700°C.; spherical and elongated periclase grains in the matrix; cracks along the grain boundaries and across the grains; large quantity of silicate

FIG. 74 — MICRO-STRUCTURE OF CHROME-MAGNESITE TEST SPECIMENS CM6 (CONTAINING APPROXIMATELY 6 PER CENT SiO_2) FIRED TO DIFFERENT TEMPERATURES (REFLECTED LIGHT)

determining the time dependent deformation (creep) of the test specimens at 1500°C. (2 hours) under a compressional load of 2 kg./cm.^{2}.

(i) *Effect of Composition of Firing Temperature on the Physico-mechanical Properties of Basic Refractories*

It was found in this study that the changes in the micro-structure of magnesite refractories were closely related to composition, particularly the CaO/SiO_2 ratio, which determines the nature of silicate phase, and to the firing temperature. When the silicate phase was predominant in menticellite and merwinite, periclase grain growth and densification of the matrix took place more readily than when the same was rich in forsterite. In the former case high temperature firing yielded a micro-structure in which periclase grains were rounded and enveloped by a silicate continuum and this resulted in poor resistance to creep in torsion at 1300°C. However, in the presence of large amount of iron oxide in such compositions a compact structure of hexagonal shaped and largely grown periclase grains resulted and this type of structure gave good room

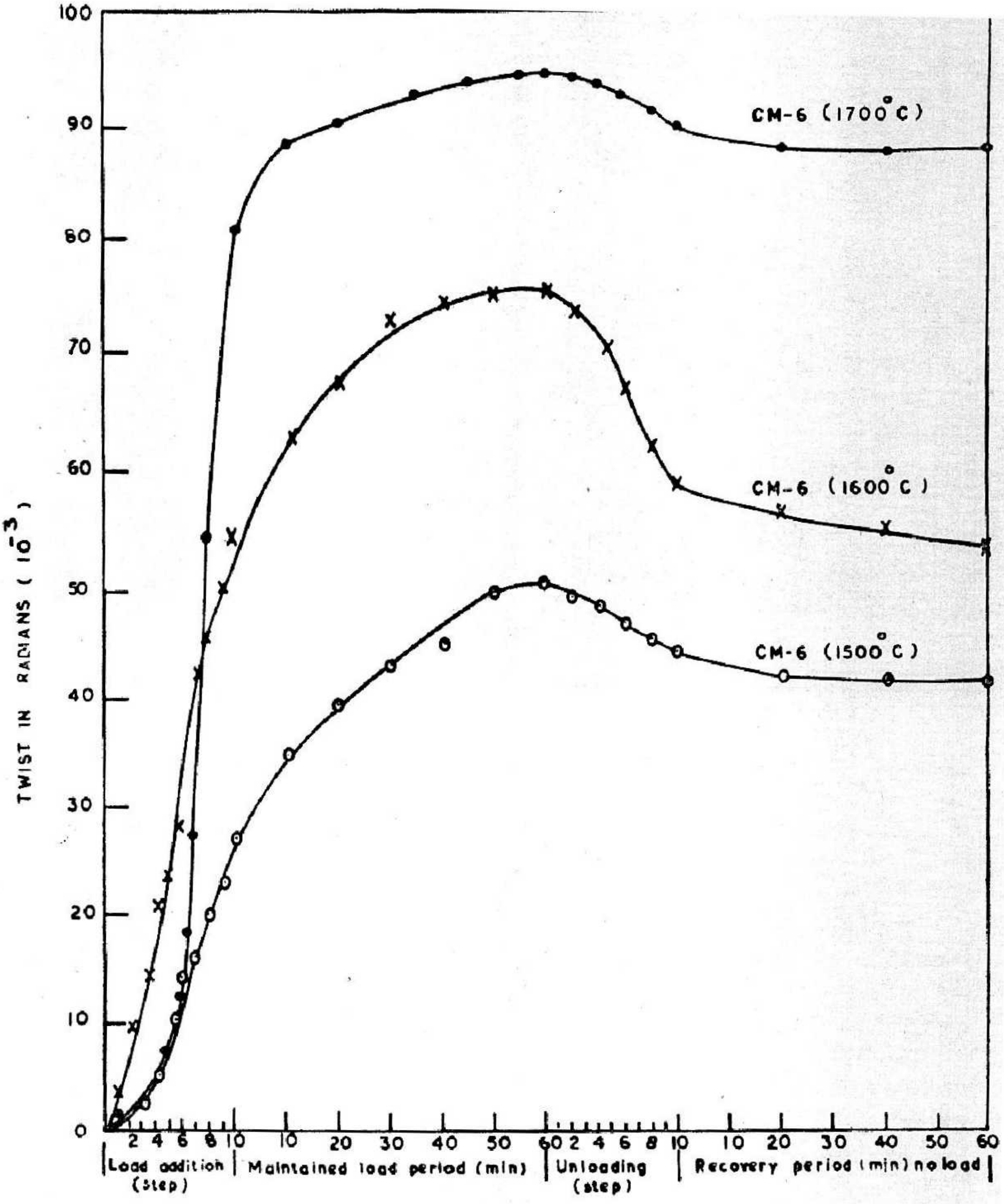


FIG. 75 — SHORT TIME CREEP AT 1300°C. OF CHROME-MAGNESITE TEST SPECIMENS CM6 (CONTAINING APPROXIMATELY 6 PER CENT SiO_2) FIRED TO 1500°, 1600° AND 1700°C.

temperature strength and excellent resistance to creep in torsion at 1300°C. but the refractoriness under load and thermal shock resistance were rather poor. Formation of excessive cleavages in the periclase grains in magnesite refractories resulted in low creep resistance in torsion at 1300°C. It was also found that refractoriness under load of magnesite refractories is influenced not only by the CaO/SiO_2 ratio but also by the relative proportions of periclase and non-periclase phases, a high proportion of periclase in the refractory being advantageous in this respect.

In chrome-magnesite refractories reactions between magnesia and chrome-spinel as also the spatial arrangement of the mineralogical phases, did not reach completion till a firing temperature of 1700°C. In test specimens fired to 1500°C. the microstructure revealed the formation of a gap around the chrome grain thus separating the latter from the matrix. With a silica content of about 3 per cent in the test specimen, an increase in the firing temperature resulted in the gradual closure of this gap and was accompanied by a progressive increase of periclase-chromite and periclase-periclase direct bonds (Fig. 72). Consequently the volume stability at 1700°C. and resistance to creep in torsion at 1300°C. (Fig. 73) of test specimens from this composition showed considerable improvement on firing to high temperature. As the silica content increased above 3 per cent, the beneficial effects of high temperature firing became less significant. When the silica content exceeded 5 per cent an increase in the firing temperature resulted in an increased width of the gap around the chrome grains (Fig. 74) and a weak bond structure in which the periclase chromite direct bond was more or less absent and the matrix consisted of a large silicate continuum enveloping the rounded periclase grains; the resistance to creep in torsion of test specimens from this composition at 1300°C. (Fig. 75) decreased with increasing firing temperature. Thus, this study indicated that while high temperature firing generally improved the bond structure and physico-mechanical properties of chrome-magnesite refractories such improvement was only possible when the silica content did not exceed about 5 per cent. It was also found that the development of direct bonded structure in chrome-magnesite refractories improved their resistance to iron oxide bursting but decreased their thermal shock resistance.

(ii) *The role of forming pressure on the properties of basic refractories prepared under industrial conditions*

The results suggested that the physical properties of basic refractories and their resistance to creep under compressional load at high temperature in particular, showed considerable improvement when the forming pressure exceeded 800 kg./cm.^{2}. In magnesite bricks, however, the refractoriness under load (tested by the rising temperature method) and the thermal shock resistance decreased progressively with increasing forming pressure. In general, forming pressures between 800 and 900 kg./cm.^{2} were considered necessary for the production of dense and creep resistant basic bricks.

ENGINEERING SECTIONS

Design and Mechanical Engineering

PROGRESS ON SOME MAJOR PROJECTS

(i) *Strip Aluminizing Prototype Plant*

A rotary melting furnace for premelting aluminium was received and plans for its installation were worked out. Orders for some items of equipment were placed.

(ii) *Wire Ausforming Unit*

Design of drawing was completed.

(iii) *Synthetic Cryolite Plant*

Extension of a steam line from the existing electrode boiler to the Cryolite Plant Bay was worked out and finalized.

(iv) *Fluidized Bed Reactor*

The design work was completed.

(v) *Baby Cupola*

The design work was completed.

RESEARCH INSTRUMENTS AND EQUIPMENT DESIGNED AND FABRICATED

1. Fume hoods for radioactive tracers were designed and fabrication completed.
2. A perspex model of a counter current extraction system was fabricated.
3. A vacuum sampling accessory for metallic vapours was fabricated.
4. A falling weight apparatus was fabricated.
5. A condenser for the zinc dross distillation unit was designed and fabricated.
6. A circular double punch for X-ray film was designed and fabricated.
7. A rotary induction furnace was designed and specifications drawn up for its manufacture.
8. A boris furnace was designed.
9. A tilt-pouring arrangement was designed.
10. A slag viscosimeter furnace was designed.
11. An extrusion die was made for aluminium fluted sections.
12. Special glass lathe machine burners were manufactured.
13. A vertical travelling furnace was designed and fabricated.
14. Work is in progress on
    (a) design of a stormer type viscosimeter,
    (b) design of an electro-thinning apparatus,
    (c) design of a torque magnetometer,
    (d) fabrication of electrode lifting mechanisms for arc furnace units,
    (e) fabrication of a vacuum distillation unit for magnesium powder.

Electronics Engineering

The salient features of the work done during the period are given below.

(i) *Electronic Potentiostat*

The work on the development of 40 mA output potentiostat was completed. Further work on the development of a high current potentiostat (up to 2 amps.) is in progress. Studies were made for using a saturable core reactor as a control element. A transistorized version of the potentiostat will also be fabricated as soon as some of the imported components are procured.

(ii) Several major maintenance, installation and calibration of instruments were undertaken during the year under review. These include 'Norelco' autrometer, electron microscope, rolling mill control, SRI Controller for multi-specimen creep testing machine, vibrophore, Siemens recorder.

Electrical Engineering

(i) In connection with design of power distribution system, installation and commissioning of several electrical equipment the following major jobs were carried out:

(a) Cable laying and drying of 500 kVA, 6600 Volt/410 V power transformer in Pilot Plant Substation.

(b) Design, fabrication and installation of control panel and other electrical installations for Isotope Laboratory.

(c) Installation of 41·5 kVA capacity silicon-carbide furnace, pneumatic hammer, grinder, drilling machine, surface grinder, 5 ton air conditioning machines, hot mixer, I.D. mixer, blower, etc.

(d) Testing and installation of one multi-specimen creep testing machine.

(e) Installation of some electrical machines and street lighting system of Marine Corrosion Research Station, Digha.

(ii) *Special Electric Furnaces, Designed and Fabricated*

(a) Multizone zig-zag silica tube furnace for 700-750°C. for chlorine heating in connection with Magnesium Chloride Project.

(b) Pt-Rh furnaces for high temperature use.

(c) One split furnace for creep test.

(d) One multi-zone tubular furnace, embedded type, for creep testing machine.

(iii) *Major Fault Shooting and Repairing of Electrical Equipment*

(a) A 6600 volts busbar spout of one O.C.B. in Low Shaft Plant-Substation developed leakage, resulting in earthing of busbar. This caused complete power failure in the plant. Since no spare assembly was available, a defective one was repaired with special technique and care. After H.P. test, power was restored.

(b) Repairing of 125 ton air conditioning seal assembly.

(c) Testing and repairing of O.C.B. 800 amps, 410 volts in the main tech. block substation.

(d) Shooting of the fault and repairing of extrusion press control panel, arc furnace control panel, rectifier and motor generator set.

(iv) *Development Work*

(a) Electrical resistance furnace for creep testing: With a view to develop electrical resistance furnaces suitable for creep research work has been taken up to design and develop furnaces which can attain a constant temperature up to 1100°C. within specified zone and yield extremely long life. Long life of the furnace is the most essential requirement for creep-research. This can only be achieved by constant endeavour in improvement in design and fabrication. Two such furnaces have been fabricated and are under trial in creep testing machines. Their constructional features are outlined below:

1. 73 mm. dia. × 450 mm. length kanthal wire wound embedded furnace for 1100°C. temperature with three zones separately controlled. This furnace has already been tested for 4,500 hours.
2. 82·5 m. dia. × 200 mm. Nichrome wound split furnace embedded with three zones, separately controlled.

(b) *Electroslag Refining Equipment*

Extensive literature survey was carried out in collaboration with General Metallurgy Division to develop suitable electroslag refining equipment. It is proposed to design and develop electroslag refining equipment for eventual indigenous commercial exploitation.

Civil Engineering

The following major civil works were completed and in progress during the period under review.

(i) *Completed*

1. Modification to Cryolite Pilot Plant.
2. Construction of Lift Well at NML.
3. Excavation of Trial Pits at Magnesium Project.
4. Flooring the working area of Cupola, Rotary Furnace and charging areas at L.S.F.P.
5. Dismantling and re-erection of shed over charging equipment of Hot Blast Cupola at L.S.F.P.
6. Modification to the existing fittings, shutters, etc., for Foundry Station, Howrah.
7. Construction of Sintering Plant of M.B.P.P.
8. Construction of plant room for Isotope Laboratory.
9. Protection of area against the wind erosion of M.C.R.S., Digha.
10. Construction of Temporary Sub-station.
11. Construction of Temporary Substation shed for Magnesium Project (Steel Structural portion only).

(ii) *In Progress*

1. Building construction for Strip Aluminizing Pilot Plant at NML.
2. Construction of Store Building at Magnesium Project.
3. Construction of Cooling and Spraying Tank at Magnesium Project.
4. Construction of shed for Dry Grinding Unit at M.B.P.P.
5. Construction of Cement Concrete Road at L.S.F.P.
6. Construction of Substation for Magnesium Project.

INDUSTRIAL LIAISON, OPERATIONAL RESEARCH, INFORMATION AND EXTENSION SERVICES

LIAISON, Information and 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

During the period under review National Metallurgical Laboratory has extended consultancy services to various metallurgical and other industries. Consultancy services thus extended comprised of beneficiation of low grade iron ore from Goalpara, Assam, referred to by Government of Assam, who are exploring the possibility for utilizing the ore for production of pig iron. Beneficiation of Sankaridurg limestone was carried out for use as flux in L-D vessels for Neyveli Salem Steel Plant and about 27 kg. each of green and heat hardened pellets made from limestone concentrate were sent to Rourkela Steel Plant for trials in L-D vessels. Beneficiation of iron ore sample from Kudremukh deposits, Mysore, was done for NMDC and sintering studies were done on iron ore fines from Bellery Hospet area for M/s. Minerals & Metal Trading Corpn. to boost up export of iron ore in international market. Mention can also be made of the pilot plant studies carried out on beneficiation of low grade apatite from Singhbhum, Bihar, for use as manure in agriculture and the report prepared was submitted to Government of Bihar, who are interested to establish a beneficiation plant with technical help from NML. Besides many other work on ore preparation by beneficiation technique were carried out on graphite samples referred to by M/s. Singhania Commercial Co. Ltd, and National Carbon Co. of Calcutta. Beneficiation of manganese ore referred to by M/s. National Carbon Co., limestone sample referred to by M/s. Ashoke Cement Co., magnesite sample referred to by M/s. Dalmia Magnesite Ltd, Nundydroog gold ore referred to by Kolar Gold Mining Undertaking of Government of India, low grade kyanite from Orissa referred to by M/s. S. Lall & Co., removal of mica from Jamuna river sand referred to by National Building Organization and concentration of cobalt from cobalt bearing ferrous manganese ore from Kalahandi, Orissa, referred to by G.S.I., etc., were completed.

Among the work relating to ore preparation referred to NML during the current period mention may be made of the pilot plant trials on beneficiation of fluorspar from Gujarat taken up on behalf of Gujarat Mineral Development Corporation for furnishing detailed investigation project report for establishing and commissioning a 500 tonnes per day beneficiation plant for the Corporation.

Regarding development of refractory materials, studies were completed on dense carbon aggregate as substitute for anthracite coke for production of calcined anthracite coal referred to by M/s. India Carbon Ltd. An interim report was prepared on studies conducted on development of ramming mass and submitted to M/s. New Standard Engg. Co. Ltd. Studies on sintering of Nattam dolomite for use as refractory material was completed for Bokaro Steel Plant.

Report prepared on pilot plant smelting trials conducted in low shaft furnace pilot plant for utilization of blended coke containing Assam coal for iron making was submitted to Dy. Coal Controller, who is interested to utilize the Assam coal for metallurgical purposes. Smelting trials were also conducted in submerged arc smelting furnace for production of high carbon ferrochrome and silico chrome referred to by M/s. Ferro Alloys Corporation Ltd.

Regarding utilization of metallurgical wastes, studies were taken up on recovery of zinc from zinc dross which has accumulated in large quantities in P & T workshops as waste.

In the field of granting industrial licences to private and public enterprises for establishment of new undertakings or considerable expansion of existing units, the National Metallurgical Laboratory was consulted by Government of India and during the period, 76 cases were referred to.

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. Besides, NML is collaborating with Indian Standards Institution in the formation and rationalization of standards, and have conducted and undertaken a number of investigations apart from comments furnished on various draft specifications. Close collaboration is maintained with the defence establishments to meet their needs for various special products as per their rigid specifications. With the Research Design and Standard Organisation of Ministry of Railways, the National Metallurgical Laboratory is in close touch regarding the development of technical 'know-how' relating to production of some specific materials based on indigenous materials.

Industrial Surveys and Collection of Statistical, Economic and Market Data

Statistics relating to latest production and consumption figure of different types of raw materials, ferrous and non-ferrous metals and alloys of India and other countries of the world were collected. Scanning of technical news of the industry and research development in home and overseas press was done.

Technical Aid to Industry

Over five hundred enquiries on different subjects pertaining to process details, raw materials, quality, production, etc., were attended to during the period under review. The majority of the enquiries originated from medium and small scale industries as well as individuals. Detailed technical notes were furnished wherever necessary. About one hundred short-term investigations and specifications were conducted out of which about 52 per cent related to government and semi-government organizations and 48 per cent from private individuals.

Practical Demostrations

Practical demonstrations were arranged for the following licencee firms, during the period under review, to impart practical 'technical know-how' developed and to impart practical training in plant operation, etc.

1. M/s. J. D. Jones & Co. Ltd, Calcutta-1. Production of improved graphite crucibles (clay bonded) IP-62352

2. M/s. Electric Control Gear P. Ltd, Ahmedabad.
   Production of C-free ferro alloys by aluminothermic reactions
3. (a) M/s. Gupta Aluminizing Industries, Kanpur, UP
   (b) M/s. Paramount Iron & Steel Works P. Ltd, Sonepat (Punjab)
   (c) M/s. Navsari Processing Industries P. Ltd, Navsari (Gujarat)
   Hot dip aluminizing of ferrous materials (IP 55289, 57938 & 65230)

Training

Under the Scheme of International Association for Exchange of Students for Technical Experience, Dr. Ing. Hartmut H. Probst from Berlin University, West Germany, has undergone advance training in techniques on Extraction Metallurgy, Electrometallurgy and Physical Chemistry and Ore Dressing in NML.

Under the Scientific and Technical Cooperation Agreement between the Government of India and United Arab Republic, two scientists from UAR completed advanced research training in different specialized research subjects for two years.

Instructional and training facilities were extended to a batch of thirty students of Metallurgy from the I.I.T., Madras, and special lectures were arranged on (1) Manufacture of iron by Low Shaft Furnace, (2) Ore dressing technique for Indian iron and manganese ore, and (3) Modern physico-chemical methods of metallurgical analysis.

Besides, many technical personnel from government and semi-government organizations, engineering colleges, universities, and students from technical institutes and universities were given training in heat-treatment, mechanical testing, refractories, ore dressing techniques, spectrographic analysis, creep testing, etc.

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. Dr. Donald Mclean of National Physical Laboratory, U.K., gave a lecture on 'Creep of Metals'.

Operational Research

A model was developed for determining the maximum permissible limit of MgO and MnO content in the slag, as well as the optimum composition of a slag for its utility as slag cement. The technique of linear programming was suggested and the problem accordingly formulated to achieve the same at the least cost.

NATIONAL METALLURGICAL LABORATORY FOUNDRY STATIONS AND MARINE CORROSION RESEARCH STATION

Foundry Stations

In pursuance of the important role played by the National Metallurgical Laboratory in the field of foundry technology since its inception, and to make available the technical knowledge gained with regard to metals and alloys, foundry raw materials and their testing, to the various small foundries in the country, which otherwise cannot afford such services, recommendation was made for the establishment of foundry stations at various places in India for the development of foundries. The Board of Scientific & Industrial Research and the Governing body of CSIR, accorded their approval for the establishment of four regional centres, one each at Batala, Madras, Howrah and Ahmedabad. The NML Foundry Stations at Batala, Madras and Howrah have already been set up while that at Ahmedabad has made considerable progress.

The NML Foundry Station, Batala, was declared open on 10th April, 1964 and since then this foundry station has been actively engaged in rendering technical service facilities, including on the spot examination of foundry problems, to the vast number of foundries in and around Batala. This service has now been extended to foundries situated at Ludhiana and Amritsar. The Department of Industries of the Government of Punjab in collaboration with the NML Foundry Station has taken up development of selected foundries in the state on modern lines.

Extensive investigation work aimed at exploring the resources in the Northern region with regard to foundry raw materials is under way at the NML Foundry Station, Batala, in collaboration with the Department of Industries of the Government of Punjab. As a first step, the sand deposits in the area are being examined, the collection of samples being done by the Department of Industries. Investigation has so far been completed on nine sand samples and work on further 22 samples is under various stages of investigation.

In addition to the above, the foundry station at Batala has been rendering testing facilities for the analysis of metals and alloys, testing of sand samples, etc., which are essential for quality control and elimination of rejects in the day-to-day working of a foundry.

The NML Foundry Station, Madras, was declared open on 14th December, 1965. It is housed at the Industrial Estate, Guindy, Madras, and has a well-equipped sand testing laboratory and a chemical analysis section provided with up to date instruments for the analysis of metals and alloys, both ferrous and non-ferrous.

The NML Foundry Station, Madras, is taking an actve role in training personnel from industry on the latest methods of sand testing and application of these results to actual foundry practice. The Institute of Indian Foundrymen, in collaboration with the NML Foundry Station, Madras, has arranged a short term course on Foundry Practice for the benefit of foundries in the region.

Investigation on raw materials available in the southern region with a view to find out their suitability for foundry purposes has been taken up in collaboration with the Geological Survey of India, who will collect and supply samples of the raw materials for investigation at the NML Foundry Station. Investigation has been completed so far on four sand samples and several others are at various stages of investigation. The NML Foundry Station, Madras, has been receiving samples of foundry raw materials, metals and alloys for analysis and testing in large numbers from various foundries in the southern region.

The NML Foundry Station, Howrah, has taken a lead over other foundry stations in offering mechanical testing facilities in addition to sand testing and chemical analysis. The mechanical testing section of the foundry station has been equipped with universal testing machine, impact testing machine and hardness testing machine.

The regional survey and investigation of foundry raw materials has been taken up in collaboration with the Geological Survey of India. Considerable progress has already been made in conducting these investigations. Like other foundry stations, the NML Foundry Station, Howrah, has been receiving samples and testing them at its well equipped sand testing and chemical analysis laboratories.

Equipments, apparatus, chemicals, etc., required for the establishment of the NML Foundry Station, Ahmedabad, has been received and staff already posted at Ahmedabad for setting up the laboratory. Action is also under way to set up the fifth foundry station at Agra.

Expansion of the service facilities offered by NML Foundry Stations to include metallographic examination, mechanical testing of metals and alloys, and problems relating to metal melting and casting will be taken up in the second phase regarding which steps are already under way; the mechanical testing facilities having been already provided at NML Foundry Station, Howrah. 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 underdeveloped 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

The National Metallurgical Laboratory has established a Marine Corrosion Research Station at Digha on the coast of Bay of Bengal in West Bengal. A plot of 5 acres of land for this purpose was generously donated by the Government of West Bengal. The Corrosion Research Station has been set up with a view to undertake scientific studies on multiple aspects of corrosion under marine condition, e.g. marine atmospheric corrosion, sea-water corrosion under partially and fully immersed conditions, soil corrosion, cathodic protection, etc.

Keeping in view the requirements of the transport, naval and other organizations, the following long range and short term research projects are being taken up at the Marine Corrosion Research Station.

1. Atmospheric corrosion in marine atmosphere:
   (i) *Outdoor Exposure* — To assess the corrosion of docks, piers, portion of ships above the sea water line, rail roads, other means of inland road transport, structural work of metals and concrete, etc.
   (ii) *Indoor Exposure* — To determine the resistance to corrosion of interior structure, decorative articles, lower portion of vehicles, etc.
2. Corrosion in sea-water under total and alternate immersion conditions with a view to determine the nature of corrosion and to develop protective measures for the portions of ships which are either completely immersed in sea water or near the water line piles and piers of dockyards which lie between maximum and minimum tide level, etc.
3. Corrosion in soil near sea coast for studying the corrosive characteristics of soil near the sea coast with a view to protect structures buried in soil near the sea.
4. Cathodic protection of naval vessels, structures, etc., to determine the optimum conditions of cathodic protection exposed to sea water and to underground pipes; structure for carrying electric power, etc., exposed to the soil near the sea as well as development of aluminium base sacrificial anode.

The National Metallurgical Laboratory has done considerable amount of work on corrosion research and the establishment of the Marine Corrosion Research Station at Digha is another successful step in this direction. Focussing its attention on corrosion problems related mainly to marine conditions, the station has an ambitious research programme and hopes to play an important role in the scientific and industrial field of various aspects of marine corrosion and prevention.

LIBRARY, DOCUMENTATION, TRANSLATION AND REPROGRAPHIC SERVICES

Library and Documentation Service

The Library, during the year under review, attempted to improve the layouts for future growth in terms of acquisition and access to books. In this connection fresh shelving of the books and journals was done followed by binding of periodicals and reconditioning of books.

The documentation work, namely classification of the abstracts and their processing and indexing, arrangement of the Iron & Steel Institute abstract cards according to U.D.C. System, etc., were under way.

Translation Service

Articles in French, German & Russian of direct interest to research 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 them to precisely define the portion of the text of specific interest to them. About 50 technical papers published in French, German and Russian were translated during the period under review.

Reprographic Service

Photostat, reflex prints and micro-films were made of the scientific and technical papers of interest to the research workers. Photo copies of articles were made and supplied to other research institutions at their request. Coloured transparencies, micro-photograph, X-ray photographs, etc., were taken of the various research and investigation projects.

INTERNATIONAL CONFERENCES

33rd International Foundry Congress

FOUNDRY experts from twenty-eight nations of the world met for the first time east of Suez, in India, at the 33rd International Foundry Congress. International Foundry Congress has so far been held annually in different countries, each one of which is highly developed and industrially advanced. The change of venue in essence represents the necessity of economic build-up of developing countries speedily and efficiently which in the long run will be of mutual benefit and rewarding. It is also recognition of the role of the developing countries in the growth and development of industrial technology particularly production metallurgy and foundry techniques of iron and steel and non-ferrous metals and alloys.

The Prime Minister of India, Mrs. Indira Gandhi, expressing similar sentiments in a message to 33rd International Foundry Congress stated ' International Conferences aid the progress of science by providing an opportunity for leaders in various specialized branches of learning to meet and compare notes. The visit of leading scientists and technologists from various parts of the world will be of immense benefit to India as well as the rest of the developing world '.

The Congress was held at Vigyan Bhawan, New Delhi, from 4th to 9th December, 1966. A most striking feature was the participation, for the first time in the history of Congress, by United Nations Team for Industrial Development led by Dr. B. R. Nijhawan. The congress was organized by the Institute of Indian Foundrymen in collaboration with National Metallurgical Laboratory and Government of India.

More than 700 delegates comprising scientists and foundry technologists from Austria, Belgium, Brazil, Bulgaria, Chile, Congo, Czechoslovakia, Costa-Rica, Denmark, Finland, France, Germany, Great Britain, Hungary, India, Iran, Italy, Japan, Nigeria, Netherlands, Poland, Phillipine Switzerland, Sweden, Turkey, U.S.A. and U.S.S.R. took part in the deliberation of the Congress.

Inauguration

The Institute of Indian Foundrymen made extensive and elaborate arrangements for the inaugural and technical sessions. Considering the great industrial and academical significance of this conference to be held for the first time in an Asian capital, Rashtrapati Dr. S. Radhakrishnan, President of Republic of India, graciously consented to inaugurate the Congress sessions. However, due to his indisposition at the last moment, the Vice President of Republic of India, Dr. Zakir Husain, deputized the President and formally inaugurated the 33rd International Foundry Congress. While inaugurating, Dr. Husain said, ' We are today in a great revolution. It is the new industrial one, which provides and transcends all concerns of man. We require many years of peace to master the conservence of this change of values which the industrial revolution has effected '.

Dr. Zakir Husain pointed out that 'Civilization works out a solution to the problem of living in a relatively permanent community at social and technological levels. Civilization is something artificial and man made, the result of making tools — physical and conceptual — of increasing complexity in response to the inevitable stage in human development'.

'In India' Dr. Husain said, 'We had the foundry, industrial and metallurgical, from very ancient times. The 1,500 year old rust-free iron pillar near the Qutab Minar, the huge iron girder at Puri, the iron gates at Somnath and many other such achievement bear testimony to the metallurgical skill of Indian craftsmen. The relics of Mohenjodaro illustrate how the workers were able to cast and forge weapons and tools'. He emphasized that in modern times the efforts of Jamshedji Tata paved the way for modern iron and steel industry. Several units have been set up since independence. The foundry industry has made phenomenal progress. It is today the sustaining key of all industries — cement, sugar, automobile, railways, machine, tools and a host of others. It is very much to be hoped that this technological development will contribute to the cooperative spirit among men, thus paving the way for human solidarity.

Speaking at the inaugural session Mr. W. E. Huber, President of the 33rd International Foundry Congress who incidentally is also managing director of Sulzer Brothers of Switzerland, said that it was indeed a remarkable occasion that for the first time foundry experts from so many different countries had met outside of Europe and

FIG. 76 — DR. ZAKIR HUSSAIN, VICE-PRESIDENT OF INDIA, DELIVERING THE INAUGURAL ADDRESS AT THE 33RD INTERNATIONAL FOUNDRY CONGRESS

North America to discuss problems of the foundry industry.

India had devoted great attention not only to the systematic building up of foundries and plants, but also to vital development and research activities. The National Metallurgical Laboratory in Jamshedpur, with its staff of more than 1,000 and the most up-to-date research facilities, was a significant example of the country's farsighted industrial policy. India could look back on a 3,000 year-old tradition in moulding and casting of metals, and especially interesting from the metallurgical point of view were the alloys used in the bronze statuettes more than 1,000 years old and cast with extraordinary precision.

Mr. Huber then referred to the excellent quality of Indian ore in ancient and medieval times, before dealing with the country's industries to day. Present and future demands for increased productivity in its widest sense could only be met if the metallurgical and technological developments of metal casting kept pace with technical developments in general. It could be said, with justification, that the foundry industry had increased considerably its productivity in recent years. It was characteristic of the advances made in many fields that they were no longer the outcome of mere chance, but the results of intensive research. One could no longer agree with the idea that evaluation of carefully-collected statistical data was the quickest and surest way to perfect a process or raw material.

However, the human aspect was just as important as these technical and material consideration and one could not afford to forget training of a new generation of foundrymen. One could expect satisfaction in this work and a correspondingly high performance only by making constant efforts to improve working facilities and conditions. There was still much to be done in this area.

Finally, Mr. Huber thanked the organizers of the Congress and expressed the hope that it would be a profitable event and that delegates would have an interesting and enjoyable time.

Welcoming delegates to the 33rd Foundry Congress, Dr. B. R. Nihawan, President of the Institute of Indian Foundrymen, and Director, National Metallurgical Laboratory, pointed out that the venue of the International Foundry Congress in preceding years had always been in a highly developed and industrially advanced country and the change of venue this year was thereby a true recognition of the significance, importance and the total necessity of accelerating the economic growth and industrial advancement of developing countries.

Dr. Nijhawan said, ' The Centre of Industrial Development in the United Nations, apart from its enterprising and pioneering work in the field of industrial development of developing countries, has recognized the need and value of actively participating in International Conferences such as the International Foundry Congress, which provide catalytic platforms for the efficient transfer of technical know-how from the highly developed to the developing countries and enable them to mutually understand their problems of economic growth and industrial development. The exchange of technical know-how and interchange of ideas on foundry technology which this congress will undoubtedly promote, will form an important nucleus for the industrial growth and economic development of developing countries and will indeed be valuable, adjudged by any standard national and international'.

Discussing problems confronting the developing countries, Dr. Nijhawan emphasized that the foundry complex related chiefly amongst others, to the procurement of imported capital plant and equipment involving heavy intake of foreign currency, lack of technically trained personnel, and short-comings in meeting the machinery spares and plant accessories indispensable

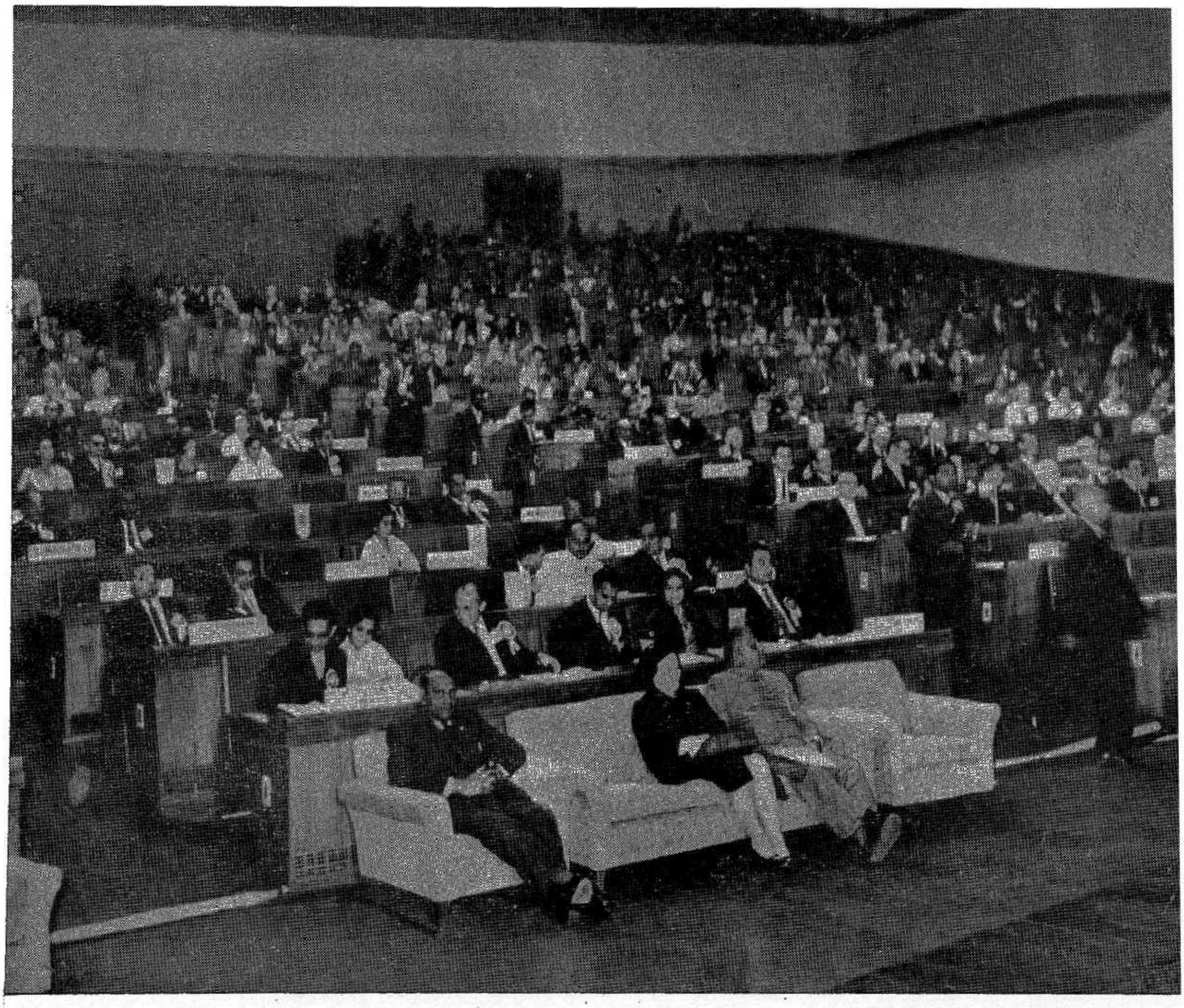


FIG. 77 — A VIEW OF THE DELEGATES AT THE 33RD INTERNATIONAL FOUNDRY CONGRESS

for peak productivity operations of the foundry industry. The developing countries had to choose and accept not essentially the best and most up-to-date technology, but one that would fit in well with the overall pattern of their industrial growth and economic stature and more suited to the background of their specific market trends and consumer needs, fully supported by home skill and indigenous standards of technical proficiency. Latest technological developments were slow to take root in the newly-emerging and developing countries, still slower to find technical and economic acceptance in view of their lean international financial resources and still leaner foreign-exchange reserves. Furthermore, the successful application of a new foundry process in a developing country could not be accepted as axiomatic or automatic; any new process of foundry technique had to 'cut its teeth' over a substantial period before its established success in an industrial scale.

Dr. Nijhawan said that most countries have today realized the importance of well-knit foundry industrial bases to feed the chain reaction growth of secondary and processing engineering industries in tandem forming the backbone of consumer industries. Such integrated industrial growth was the fruit that newly developing countries aspired to grow on home soils instead of importing them as ' canned '. Turning to present Congress, Dr. Nijhawan said ' India is a land of colourful contrasts and offers to visitors an opportunity of seeing centuries old art and architecture side by side with the most modern projects based on science and technology '.

Presiding over the inaugural session of the Congress, Mr. D. Sanjivayya, Union Minister for Industry, Government of India, said, ' India, though a predominantly agricultural and industrially under-developed country till recently, has yet for herself an ambitious, but realistic programme of industrialization. The progress achieved during the three Five Year Plans is heartening and stimulating. The dynamic expansion of engineering industry, more particularly the machine building industries, has cast a heavy burden and demand on the foundry industry, with the result that the foundry industry has become one of the crucial branches of India's economy. He mentioned that the mechanization of integrated foundry plants, automation and closed circuit operations, scientific study and use of foundry moulding science and bonding materials, besides operational control at every stage backed by adequate research efforts are the outstanding features of the growth of the industry in India in the last decade. The advent of the large iron and steel plants, heavy machine tool and machine building projects, foundry and forge units, coal mining machinery and machine tool manufacturers, heavy electrical and heavy boiler plants, fertilizers, chemical, and petroleum and complexes, locomotives, etc., with the collaboration of foreign countries have helped a rapid development of the foundry industry.

Speaking at the session, Mr. M. C. Chagla, Union Minister of External Affairs, Government of India, said, 'The raising of levels of productivity in industry through latest techniques being the central theme of the Congress, it is to be confessed that India lags much behind several other countries in the matter of the level of productivity in industry today. We are fully conscious of the fact that except by means of raising productivity we cannot hope to energize our economy and secure its self-generation. We have, therefore, instituted a multi-pronged and relentless drive to achieve reasonable increases in the levels of productivity and, therefore, have taken to research and developing new techniques. The Congress meeting now being held in a developing country for the first time, will help representatives of the countries of the East to share their experiences with the experts from western countries with a view to broadening their horizons of understanding '.

Sir Jehangir Ghandy, the Chairman of the Reception Committee, was not able to attend the Congress personally due to his indisposition. In his absence Dr. Bharat Ram, Vice-Chairman of the Reception Committee, welcomed the delegates and Mr. R. M. Krishnan, Executive Secretary of the 33rd International Foundry Congress, proposed a vote of thanks.

Award

Mr. W. E. Huber, President of the 33rd International Foundry Congress, was awarded a silver plaque for his outstanding contribution to the development of foundry industry by Indian Institute of Foundrymen. Dr. B. R. Nijhawan, President of Indian Institute of Foundrymen, and Director, NML, received a silver plaque in recognition of his service to the progress and development of foundry industry in India. Messrs Arjan Vaswani of Indian Institute of Foundrymen and R. M. Krishnan of National Metallurgical Laboratory received gold medals in recognition of their valuable contributions in the field of foundry technology and industry and for the meritorious services for organizing this Congress.

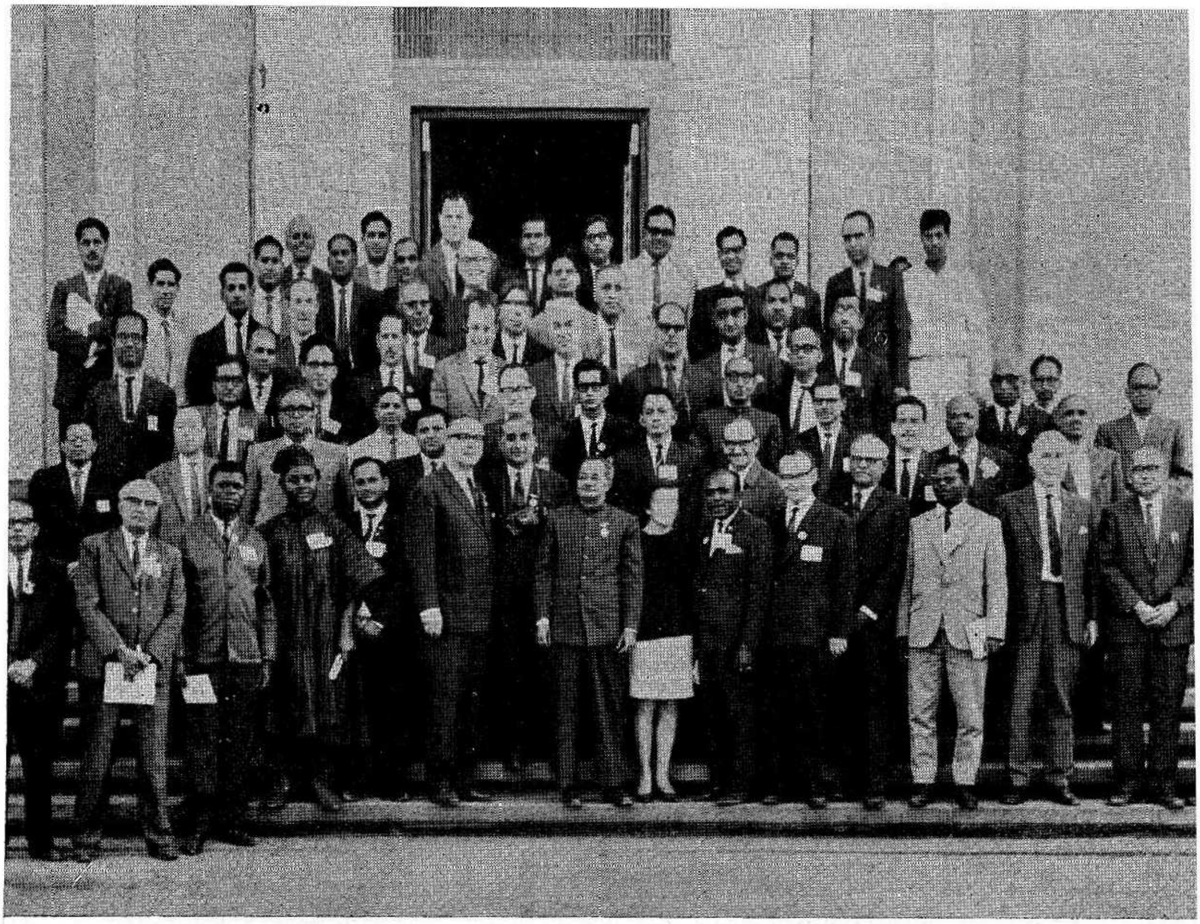


FIG. 78 — GROUP PHOTOGRAPH OF SOME OF THE DELEGATES TO THE 33RD INTERNATIONAL FOUNDRY CONGRESS DURING THEIR VISIT TO THE NATIONAL METALLURGICAL LABORATORY

Technical Sessions

Thirty-four technical papers from foundry experts on their subjects of specialization and seven papers from United Nations Expert Team were presented and discussed in 10 technical sessions in the 33rd International Foundry Congress.

Unprecedentedly large attendance in technical session at the 33rd International Foundry Congress was a matter of general comment amongst foreign delegates. Many of the Chairmen of the technical meeting observed that though they had been attending International Foundry Congress meetings for a long time, it was for the first time in New Delhi that they found a great upsurge of intellectual curiosity among the participants. The floor language of the papers and discussions that followed were simultaneously translated into French, German and English which aroused great enthusiasm amongst the participants in promoting lively discussions which were confined not only during the session but were prolonged even after the conclusion of the official business of the meetings.

Concluding Session

Vigyan Bhavan, the venue of 33rd International Foundry Congress, witnessed the unforgettable scene at the conclusion of the 33rd International Foundry Congress. The general appreciation was that the theme of the congress meeting, namely, technological developments as means to increase productivity in foundry industry, was well discussed and deeper understanding was arrived at.

This became very clear from what Mr. W. E. Huber said at the concluding session.

'The 33rd International Foundry Congress is of exceptional importance to the development of our Committee because the fact that it is being held in India is a proof that the Foundry Industry is prepared to promote technical progress through generous international exchange of technical ideas and experience, and furthermore to make a valuable contribution to the industrialization of aspiring nations.' Mr. Huber pointed out that apart from metallurgical problems and the endeavour to increase productivity, great attention should be devoted to the fundamental questions of metal casting. He thanked the Indian organizing committee for excellent arrangements made for holding the Congress in New Delhi.

Dr. B. R. Nijhawan hoped that at least once in five years the IFC would meet in India and that the U.N. participation in it would become a regular affair. He referred to the problems of under-developed countries and held that bringing them up to the level of the developed countries would in foundry technology also contribute to their well-being.

Mr. Arjan Vaswani, Secretary of the Institute of Indian Foundrymen, the host organization for the 33rd IFC thanked participants and those who helped to make arrangements.

During pre and post congress tours the delegates visited the modern industrial complexes in India and the National Metallurgical Laboratory. They were conducted round the various research divisions and pilot plants of the Laboratory. They were also entertained with classical Indian dance performance by eminent Indian dancers.

Foundry Book Exhibition

During the congress, the Indian National Scientific Documentation Centre (INSDOC) and Institute of Indian Foundrymen jointly organized an exhibition of books and periodicals on foundry technology at the Vigyan Bhavan. A catalogue, listing the exhibits covering about 580 titles published in 18 languages received from 20 countries were brought out. ' Bibliography on Foundry Practice ' and ' Foundry Directory of India ' — the first of its kind, were publications brought out by the INSDOC and IIF on this occasion. The exhibition also displayed photographs of castings and actual intricate cast models.

The IFC Supplement

On the inaugural day of the Congress special supplements were brought out in a masterly fashion by *Economic Times*, *Financial Express*, *The Mail* and the *Indian Express*. Excellent coverage of the day-to-day proceedings was done by leading newspapers of the country. Besides, a special bulletin, *IFC NEWS*, was brought out every day by the Organizing Committee highlighting the important activities of each day of the Session.

PUBLICATIONS

NML Technical Journal

The *NML Technical Journal*, a quarterly, has entered into ninth year of its publication. The journal is maintaining its usual standard as one of the leading journals of the world on metallurgical and allied subjects. The papers published in the journal are abstracted by the world's leading abstracting services.

Monograph on Bentonite

A monograph on Bentonite is under publication by the National Metallurgical Laboratory. The monograph contains the results of investigational work carried out on the indigenous bentonite samples received from various sources in respect of their suitability for use in different types of casting purposes. The monograph also contains results of work on some foreign bentonites conducted at NML on behalf of International Working Group on Bonding Clays. It may be mentioned that a few years before NML published a monograph on *Foundry Moulding Sands of India* which was widely appreciated by foundrymen both in India and abroad. This monograph on 'Bentonite' will also be of immense help to Indian foundrymen.

Brochures

A coloured brochure on the activities of the National Metallurgical Laboratory was printed during the period under review.

Papers Published

During the period under review scientific papers were published and presented as per Appendix I.

Research and Investigation Reports

During the period under review research and investigation reports were prepared. The details are furnished in Appendix II.

GENERAL

DR. B. R. Nijhawan, Director, National Metallurgical Laboratory, took up the assignment of Senior Adviser (Inter-regional) to the United Nations in their Centre for Industrial Development at New York for a period of two years. Dr. T. Banerjee took over as Scientist-in-Charge, National Metallurgical Laboratory.

The Executive Council of the Laboratory after going into details into the activities of the Laboratory has expressed satisfaction on the progress of research and development work. Emphasis has been laid on the projects of immediate industrial importance, import substitution work, etc., and further streamlining of the various projects has been given effect to.

The Laboratory has continued to design and fabricate many special items of equipment needed for research and pilot plant work, besides regular check up and maintenance of the various specialized equipment 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. Following NML staff have been nominated in various special committees:

Dr. T. Banerjee, Scientist-in-Charge, has been nominated as (1) Director, Board of Directors, National Mineral Development Corporation, (2) Alternate Director, Board of Gujarat Mineral Development Corporation, (3) Member, Defence Metallurgical Research Laboratory Advisory Committee, (4) Member, Executive Council of Central Scientific & Research Services Organization, Hindustan Steel Ltd., (5) Member, Bihar Mineral Advisory Council, Government of Bihar, and (6) Council Member, Institute of Indian Foundrymen.

Shri P. I. A. Narayanan, Officer-in-Charge (Ore-dressing), has been nominated to represent NML in the Technical Group set up by the Ministry of Mines & Metals, Government of India, to consider the various aspects of pelletization of iron ore fines in India.

Dr. A. B. Chatterjea, Scientist, has been nominated to represent NML in the Committee on Assessment of Demand formed by Ministry of Mines & Metals, Government of India, to advise the Committee on norms of coking coal consumption in iron and steel industry.

Shri P. P. Bhatnagar, Scientist, has been nominated to represent NML on the reconstituted Development Council for Nonferrous Metal and Alloys of the Ministry of Mines & Metals, Government of India.

Shri G. P. Mathur, Scientist, has been nominated to represent NML on the standing Committee on Modernization, Mechanization and Training of the Ministry of Mines & Metals, Government of India.

Purchase and Stores Sections of the National Metallurgical Laboratory kept up their activity for procurement of capital equipment, raw materials, consumable stores and equipment, etc. The heavy budgetary accounts of the Laboratory were handled with efficiency and speed. Details of the National Metallurgical Laboratory budget expenditure are furnished in this report. Cooperative Banking Society of the National Metallurgical Laboratory continued its good work throughout the year. The cooperative stores is doing exceedingly good job in supplying rationed and other food-stuffs, stationary article, etc., to the staff members.

The National Metallurgical Laboratory Team participated in the Shanti Swarup Bhatnagar Memorial Tournament held at Mysore and was winners in the volley-ball tournament.

Award

Dr. T. Banerjee, Scientist-in-Charge, has been admitted as a Fellow of the Indian Standards Institution.

Dr. S. S. Bhatnagar & Dr. R. Kumar, Scientists, have been awarded the "National Metallurgist's Day Award" for the year 1966, instituted by the Union Ministry of Iron & Steel.

Shri R. M. Krishnan, Scientist, has been elected as Honorary Secretary of the Institute of Indian Foundrymen.

Shri N. Dhananjayan, Scientist, has been awarded the Ph.D. Degree from University of Madras.

Safety First and First Aid Section

The safety measure to be followed in the operation of various plants and equipment was strictly supervised and regular safety classes were held. Minor cases of injury were treated at the First Aid Posts. No fatal accident took place in the Laboratory during the year under review.

PATENTS AND PROCESSES

Patents	*I.P. No.*	*Title*
Filed	108583	Chemically bonded forsterite refractories and method of their preparation (26.12.1966)
	108081	Refractory ramming, plastering and patching mixes consisting of kyanite and method of making and using the same (22.11.1966)
	107982	Stable castable suspensions of non-plastic alumino-silicate materials and methods of making the same (16.11.1966)
	106906	An improved method for removal of iron and upgrading ferruginous chrome ores and other low grade ferruginous ores (3.9.1966)
	105895	A process for the conversion of molten iron of various compositions to different grades of steel by employing air for oxidizing (25.6.1966)
Accepted	96661	A novel method of separating iron from ilmenite and its application in the upgrading of ferruginous ores (11.5.1966)
	96017	Production of high purity iron (12.7.1966)
	94768	An improved device for isolation of dross in molten metallic baths during continuous hot-dip processing of strip or wire (22.4.1966)
	91134	Improvement in or relating to a precision temperature controller for use with electrical resistance furnaces up to 1600°C. (15.6.1966)
Sealed	96661	A novel method of separating iron from ilmenite and its application in the upgrading of ferruginous ores (11.5.1966)
	94769	An improved device for the continuous hot-dip coating of metallic strip and wire (22.6.1966)
	94767	An improved C.I. pot for melting and holding of nonferrous metals in general, and aluminium and zinc in particular (10.11.1966)
	82191	An improved jacketted electrolytic cell for the electrodeposition of metals and metallic oxides in general and MnO_2 in particular (10.5.1966)

Processes

Released for Commercial Exploitation to the following parties during the period :

(a) Hot-dip aluminizing of ferrous materials on following terms and conditions (I.P. No. 55289, 57938 and 65230).

Lumpsum premium ... ... Nil

Royalty	2½% on ex-factory sales price less cost of steel component
Nature	Non-exclusive
Period	14 years

1. M/s. Rainbow Industries, Indore (M.P.)
2. M/s. Hindustan Construction & Commercial Corporation, Calcutta
3. M/s. Gupta Aluminizing Industries, Kanpur
4. Shri Ct. Alagappan, Madras-6
5. M/s. Paramount Iron & Steel Works Pvt. Ltd, Sonepat (Pb.)
6. The General Manager, P & T Workshops, Calcutta (W.B.)
7. M/s. Navsari Processing Inds. Pvt. Ltd, Navsari (Guj.)
8. M/s. Technology Syndicate, Ranchi

(b) Production of ceramic magnets on following terms and conditions:

Lumpsum premium	Rs. 8,000/-
Royalty	2½%
Nature	Non-exclusive
Period	14 years

1. M/s. G. P. Tolani Co., Bombay

(c) Production of thermostatic bi-metals on following terms and conditions:

Lumpsum premium	Rs. 5,000/-
Royalty	2½%
Nature	Non-exclusive
Period	14 years

1. M/s. Cableworks (I) Ltd, Calcutta

(d) Production of C-free ferro-alloys by alumino-thermic reactions on following terms and conditions:

Lumpsum premium	Rs. 7,500/-
Royalty	2½%
Nature	Non-exclusive
Period	14 Years

1. M/s. Jeypore Sugar Co. Ltd, Madras

BUDGET FIGURES OF THE NATIONAL METALLURGICAL LABORATORY FOR THE YEAR 1966-67

			(Figures in lakh of Rupees)
RECURRING			
P-1		Pay of Officers	9·456
P-2		Pay of Establishment	10·729
P-3		Allowances	9·179
P-4		Contingencies	6·588
P-6		Maintenance	0·513
P-7		Chemicals, Apparatus and Equipment for Research	10·605
F.S.S.—		Fellowships and Scholarships	0·680
		Scientist Pool	0·095
			47·845
CAPITAL			
P-5	1.	Works	0·302
	2.	Services	0·572
	3.	Apparatus and Equipment	5·520
	4.	Miscellaneous	1·025
			7·419
PILOT PLANT			
PP-1	(i)	Equipment and Installation	1·859
	(ii)	Building and Services	1·178
PP-2		Provision for Staff	7·050
PP-3		Purchase of Raw Materials	0·679
PP-4		Miscellaneous and Contingencies	3·871
			14·637
LOW-SHAFT FURNACE PROJECT			
PP-1	(i)	Equipment and Installation	0·967
	(ii)	Building and Services	0·130
PP-2		Provision for Staff	3·730
PP-3		Purchase of Raw Materials	1·258
PP-4		Miscellaneous and Contingencies	6·659
			12·744
MAGNESIUM PLANT			
PP-1	(i)	Equipment and Installation	7·098
	(ii)	Building and Services	1·080
PP-2		Provision for Staff	0·641
PP-3		Purchase of Raw Materials	—
PP-4		Miscellaneous and Contingencies	0·193
			9·012
CONSTRUCTION OF STAFF QUARTERS			4·611
		GRAND TOTAL	96·268

APPENDIX I

Scientific Papers Published and Presented

1. Substitution of Scarce Non-ferrous Metals — T. Banerjee, *NML Technical Journal*, VIII (2), (4-9), 1966.
2. A Comparative Study of the A.S.T.M. Thermal Conductivity and the Blakeley-Cobb Apparatus as Instruments for Routine Measurements of Thermal Conductivity of Refractories — H. P. S. Murthy & T. K. Gangadharan, *NML Technical Journal*, VIII (2), (15-19), 1966.
3. Manganese-Carbonate from Low Grade Indian Manganese Ores — B. N. Singh, B. C. Kar & T. Banerjee, *NML Technical Journal*, VIII (2), (20-22), 1966.
4. A High Temperature Graphitizing Furnace for Laboratory Use — H. Singh & B. R. Nijhawan, *NML Technical Journal*, VIII (2), (23-25), 1966.
5. An Electronic Potentiostat for Study of Electrode Reactions — S. K. Sarkar & A. P. Chowdhury, *NML Technical Journal*, VIII (2), (26-28), 1966.
6. Study on the Suitability of Cellulose Materials as Core Binders — T. A. Beck, V. S. Bhandary & P. K. Gupte, *NML Technical Journal*, VIII (3), (4-10), 1966.
7. Development of an Ice Calorimeter for High Temperature Calorimetry — Gangotri Mishra & Rajendra Kumar, *NML Technical Journal*, VIII (3), (11-14), 1966.
8. Failure of a Bucket Wheel Gear Drive Shaft — A Case Study — B. N. Das & B. N. Ghosh, *NML Technical Journal*, VIII (3), (15-18), 1966.
9. Melting and Alloying Practice in Aluminium and Its Alloys — G. G. Nair, V. S. Bhandary & P. K. Gupte, *NML Technical Journal*, VIII (3), (19-28), 1966.
10. Investigations on Granulated Cupola Slag in Cement Making — A. K. Sinhamahapatra & B. C. Kar, *NML Technical Journal*, VIII (3), (29-32), 1966.
11. Recovery of Lead from Scraps — M. C. Sen & T. Banerjee, *NML Technical Journal*, VIII (3), (33-38), 1966.
12. Role of National Metallurgical Laboratory in Corrosion Research — T. Banerjee, *NML Technical Journal*, VIII (4), (5-10), 1966.
13. Interim Report on Atmospheric Corrosion Studies Under Marine Atmosphere — A. K. Dey, A. K. Sinhamahapatra, D. K. Khan, A. N. Mukherjee, Raj Narain, K. P. Mukherjee & T. Banerjee, *NML Technical Journal*, VIII (4), (11-16), 1966.
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15. Chromate Passivation of Aluminium and Copper — A. N. Mukherjee, S. Rao Addank & A. K. Lahiri, *NML Technical Journal*, VIII (4), (22-27), 1966.
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18. Compatibility Relationships in the Quaternary Sub-system $CaAl_2O_4$-$CaAl_4O_7$-$Ca_2Al_2SiO_7$-$MgAl_2O_4$ — M. Ramakrishna Rao, *NML Technical Journal*, IX (1), (4-8), 1967.
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20. Production of Pig Iron from Andhra Pradesh Raw Materials — A. B. Chatterjea, B. R. Nijhawan, J. Goswami, S. K. Biswas, R. Santokh Singh & J. S. Padan, *NML Technical Journal*, IX (1), (16-22), 1967.
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22. Riser Dimensioning — A New Approach for Grey Iron Castings — G. N. Rao & P. R. Prasad, *NML Technical Journal*, IX (1), (27-28), 1967.
23. Shell Moulding with Indigenous Raw Materials — P. R. Sastry, V. S. Bhandary, P. K. Gupte & B. R. Nijhawan, *NML Technical Journal*, IX (1), (29-34), 1967.
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25. Iron Smelting with Non-coking Coals in Low Shaft Furnace — A. B. Chatterjea, B. R. Nijhawan, *Journal of Japan Iron & Steel Institute*, 52 (5), 1966.
26. Recent Trends in Iron and Steel Production in Japan — A. B. Chatterjea, *Journal of Iron & Steel Engg.*, 6 (2), 48-68, 1966.
27. New Iron and Steel Technology in Japan — A. B. Chatterjea, *Economic Times Special Supplement*, 18 April 1966.
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31. Technological Development in Iron Making in Japan — A. B. Chatterjea, *Indian Industries, Annual Number*, 1966, (113-136).
32. Substitute Fuels for Cupola Iron Melting (Condensed Paper) — B. R. Nijhawan, J. Mohan, J. Goswami & A. B. Chatterjea, *Financial Express, Special Supplement*, 5 December 1966.

33. Project Outline for a Malleable Iron Foundry — B. R. Nijhawan, *Indian Foundry Journal, Special Number*, 1966, (114-126).

34. Some Precipitation Reactions in Chromium-Manganese-Nitrogen Stainless Steels on Ageing and Cold Working — J. K. Mukherjee & B. R. Nijhawan, *Journal of Iron & Steel Institute*, Vol. 205, January 1967.

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40. Development of Iron-Aluminium Alloys — S. M. Arora, S. S. Bhatnagar, P. K. Gupte & B. R. Nijhawan, *Transactions of Indian Institute of Metals*, Vol. 19, December 1966, (195-201).

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42. Curie Temperature of Iron-Aluminium and Iron-Silicon Alloys — A. N. Sinha & L. J. Balasundaram, *Transactions of Indian Institute of Metals*, Vol. 20, March 1967, (21-24).

43. Heat Contents and Heat Capacities of Liquid Lead and Liquid Tin — G. Mishra & Rajendra Kumar, *Transactions of Indian Institute of Metals*, Vol. 20, March 1967, (25-28).

44. Some Aspects of Phase Transformation in Iron-Manganese Alloys — S. P. Mukherjee & Rajendra Kumar, *Transactions of Indian Institute of Metals*, Vol. 20, March 1967, (33-40).

45. Heat Contents and Heat Capacities of Liquid Lead-Tin Alloys — G. Mishra & Rajendra Kumar, *Transactions of Indian Institute of Metals*, Vol. 20, March 1967, (49-52).

46. Magnesium Electrolyte — Typical Magnesium Electrolyte with Additives of NaCl, KCl and $CaCl_2$ — T. Matsushime, Tustomo Ito & P. K. Som, *Transaction of Japan Institute of Metals*, 8 (1), 1967.

47. Standard of Fireclay Refractories — H. P. S. Murthy, *Indian Standards Institution Bulletin*, 18 (10), 1966.

48. Reversion in Aluminium — 3·8 Per Cent Copper Alloy — The Effect of Specimen Size, Interrupted Quench and Plastic Deformation — Ved Prakash & B. R. Nijhawan, *Journal of Institute of Metals*, May 1966.

49. Investigation on Some Refractory Clays from Jammu & Kashmir — N. S. Sahota, T. V. Prasad & H. V. Bhaskar Rao, *Transactions of the Indian Ceramic Society*, Vol. XXV (2), April-June, (49-60), 1966.

50. Occurrence and Refractory Properties of Rajasthan Magnesite — P. C. Sen, M. R. K. Rao, Ashimesh Dutta & H. V. Bhaskar Rao, *Transactions of the Indian Ceramic Society*, Vol. XXV (4), October-December, (93-96), 1966.

51. Some Empirical Methods of Evaluating the Suitability of Raw Dolomite for Use in Steel Melting Furnace — P. C. Sen, *Transactions of the Indian Ceramic Society*, Vol. XXV (4), October-December, (107-111), 1966.

52. Formulation of Electroplating Salts: Part I — Alkaline Copper Plating Salt — S. K. Ray, *Indian Journal of Technology*, 4 (8), (245-248), 1966.

53. A Chemical Substitute for Imparting Black Colour to Bidri Wares — S. K. Ray, *Chemical Age of India*, 17 (11), (877-879), 1966.

54. Electroless Copper Plating on Mild Steel Wires — S. K. Ray, *Research & Industry*, 12 (2), (82-84), 1967.

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55. Effect of Interrupted Quench and Specimen Size on the Natural Ageing of an Aluminium-3·8 per cent Copper Alloy — Ved Prakash.

56. Structure and Magnetic Properties of Some Lead-Barium Ferrites — S. K. Bose & Ved Prakash.

57. Significance of Gas Analysis in Cupola Iron Smelting with Substitute Fuels — J. Mohan & A. B. Chatterjea.

58. Study of Kinetics of Hydrogen Reduction of Iron Ores by Electrical Conductivity Method — B. M. Dutta, K. N. Gupta & A. N. Kapoor.

59. Heat Content and Heat Capacity of Lead-Antimony-Tin Ternary Eutectic Alloy in the Liquid State — Gangotri Mishra & Rajendra Kumar.

60. Heat Contents and Heat Capacities of Liquid Lead-Antimony Alloys — Gangotri Misra & Rajendra Kumar.

61. Effect of Titanium on the Formation of Graphite in Cast Iron — Rajendra Kumar & N. K. Das.

62. Metallography of Rapid Solidification — Rajendra Kumar & A. N. Sinha.

63. Magnetism and Crystal Structure — L. J. Balasundaram.

64. Mossabauer Effect and Its Application to Metallurgy — L. J. Balasundaram.

65. Distribution Characteristics of Burden in Low Shaft Furnace — K. N. Gupta & A. B. Chatterjea.

66. Utilization of Blast Furnace Slag as Rail Road Ballast — S. P. Dasgupta, K. N. Srivastava & B. R. Nijhawan.

67. A Model on the Study of Slags and Their Utility as Slag Cement with the Help of Operational Research Technique — C. P. Saxena.

The following papers were presented at the 31st Annual Session of the Indian Ceramic Society (Items 68 to 74).

68. Calcination Characteristics of Petroleum Cokes Available in India — B. Chatterjea, H. P. S. Murthy & N. B. Sircar.

69. The Effect of Certain Manufacturing Variables on the Physical Properties of Alumino-Silicate Refractories — A. V. Subramanyam & H. P. S. Murthy.

70. Phase Equilibrium Relationships in the Binary Joints of the Quaternary System $CaAl_2O_4$-$CaAl_4O_7$-$Ca_2Al_2SiO_7$-$MgAl_2O_4$ — M. Ramakrishna Rao.

71. Phase Equilibrium Relationships in the System $CaAl_2O_4$-$Ca_2Al_2SiO_7$-$MgAl_2O_4$ — M. Ramakrishna Rao.

72. Development of High Alumina Cements from Indigenous Raw Materials — A. K. Bose & T. V. Prasad.

73. Superduty Fireclay Refractories from Jammu & Kashmir Clays — Part I — N. S. Sahota & T. V. Prasad.

74. Kinetics of Thermal Dehydration of Gibbsite — P. C. Sen.

The following papers were presented at the 33rd International Foundry Congress (Items 75 to 77).

75. Effects of Micro-additions of Sulphur, Sodium and Phosphorus to Aluminium-Silicon Eutectic Alloys — G. G. Nair, S. S. Bhatnagar, P. K. Gupte & B. R. Nijhawan.

76. Recent Developments in the Structure of Metals in Liquid State and Their Influence on Founding Properties — Rajendra Kumar.

77. Growth Pattern of Foundry Industry in Developing Countries — B. R. Nijhawan.

78. Role of Different Variables Affecting Atmospheric Corrosion of Steel — S. Rao Addanki, K. P. Mukherjee, A. K. Lahiri & T. Banerjee — Presented at the 3rd International Congress on Metallic Corrosion held at Moscow.

79. Stress Corrosion Cracking of Homogeneous Copper Alloys — A. K. Lahiri & T. Banerjee — Presented at the 3rd International Congress on Metallic Corrosion held at Moscow.

The following Papers were presented at the 54th Session of Indian Science Congress (Items 80 to 82).

80. Recent Developments in the Analysis of Nitrogen in Steel and Alloys — N. G. Banerjee.

81. Thermal Dehydroxylation and Phase Transformation of Bayer's Alumina — P. C. Sen, A. Dutt & H. V. Bhaskar Rao.

82. Problems Encountered in the Development of Industrial Process and Plant Design in India — M. J. Shahani.

APPENDIX II

Scientific Investigations Completed and Reports Prepared

1. Investigation Report on the Utilization of Blended Coke containing Assam Coal for Iron Making — Staff of Low Shaft Furnace Project Authors (I.R. 367/66).
2. Beneficiation and Sintering Studies with a Mixed Sample of Massive and Laminated Iron Ore from Rajhara Mines of Bhilai Steel Plant, Hindustan Steel Ltd. — N. Chakravarty, B. L. Sengupta, M. V. Ranganathan, G. S. Ramakrishna Rao & G. P. Mathur (I.R. 368/66).
3. Use of Zircon Sand in Foundries — V. S. Bhandary, P. K. Gupte & B. R. Nijhawan (I.R. 369/66).
4. Beneficiation of a Low Grade Iron Ore from Goalpara, Assam — S. K. Dhar, S. K. Banerjee & P. I. A. Narayanan (I.R. 370/66).
5. Investigation on Emery Flour — A. K. Bose & T. V. Prasad (I.R. 371/66).
6. Beneficiation of Calcite from Sankaridrug, Madras — K. N. Rakshit, P. V. Raman & P. I. A. Narayanan (I.R. 372/66).
7. Beneficiation of an Iron Ore Sample from Kundremukh Deposits, Mysore — P. V. Raman & P. I. A. Narayanan (I.R. 373/66).
8. Studies on Sintering of Bhawanthpur Dolomite for Its Suitability as a Refractory Material — P. C. Sen & M. R. K. Rao (I.R. 374/66).
9. Moulding Characteristics of Silica Sand from Shankargarh, Central Railway, Supplied by M/s. Southern Alloy Foundries, Pr. Ltd, Madras — V. S. Vijayanarayanan & N. V. Naidu (I.R. 375/66).
10. Beneficiation of a Low Grade Limestone Sample from M/s. Rohtas Industries Ltd, Dalmianagar, Bihar — S. K. Banerjee & P. I. A. Narayanan (I.R. 376/66).
11. Production of High Carbon Ferro-Chrome & Silico-Chrome from Raw Materials supplied by M/s. Ferro Alloys Corpn. Ltd, Garividi — P. P. Bhatnagar, M. P. Menon & B. R. Nijhawan (I.R. 377/66).
12. Beneficiation of Low Grade Kyanite from Magarmohan Kyanite Mines, Pallahara Dt., Orissa — P. K. Sinha & P. I. A. Narayanan (I.R. 378/66).
13. Removal of Mica from Jamuna River Sand — R. Ganesh, M. S. Chopra & P. I. A. Narayanan (I.R. 379/66).

14. Reduction of Insoluble Content of a Selenite Sample from Rajasthan--N. Chakravarty, G. P. Mathur & P. I. A. Narayanan (I.R. 380/66).

15. Investigation Report on the Injection of Naphtha for Iron Smelting — A. B. Chatterjea & B. R. Nijhawan (I.R. 381/66).

16. Moulding Characteristics of Sand No. BTL-2 (Natural Moulding Sand) received from Punjab State Industries Department, Chandigarh — R. C. Arora & Autar Singh (I.R. 382/66).

17. Moulding Characteristics of Kanchanpur Yellow Sand — P. R. Sastry, V. S. Bhandary & P. K. Gupte (I.R. 383/66).

18. Moulding Characteristics of Kanchanpur White Sand — P. R. Sastry, V. S. Bhandary & P. K. Gupte (I.R. 384/66).

19. Moulding Characteristics of Maharashtra Silica Sand (M-S-S-8) — H. P. Singh, V. S. Bhandary & P. K. Gupte (I.R. 385/66).

20. Moulding Characteristics of Maharashtra Silica Sand (M-S-S-5) — H. P. Singh, V. S. Bhandary & P. K. Gupte (I.R. 386/66).

21. Moulding Characteristics of Maharashtra Silica Sand Sample No. SS-7 — S. K. Sinhababu, V. S. Bhandary & P. K. Gupte (I.R. 387/66).

22. Moulding Characteristics of Maharashtra Sand Sample No. SS-1 — R. C. Arora, V. S. Bhandary & P. K. Gupte (I.R. 388/66).

23. Pilot Plant Beneficiation Studies on a Low Grade Mixed Apatite Sample from Singhbhum District, Bihar — M. V. Ranganathan, A. K. Khatry, Joga Singh, G. S. R. K. Rao & P. I. A. Narayanan (I.R. 389/66).

24. Concentration of Cobalt from Cobalt Bearing Ferrous Manganese Ore from Kalahandi, Orissa — P. K. Sinha & P. I. A. Narayanan (I.R. 390/66).

25. Reduction of Silica Content in a Magnesite Sample (No. 2) from Salem, Madras — S. K. Dhar, S. K. Banerjee & P. I. A. Narayanan (I.R. 391/66).

26. Moulding Characteristics of Maharashtra Silica Sand (MSS-3) — R. R. Dash, V. S. Bhandary & P. K. Gupte (I.R. 392/66).

27. Beneficiation of Low Graphite from Palamau, Bihar (I.R. 393/66) — P. V. Raman & P. I. A. Narayanan.

28. Beneficiation of Graphite from National Carbon Co., Calcutta — K. N. Rakshit, P. V. Raman & P. I. A. Narayanan (I.R. 394/66).

29. Reduction of Iron Content in Manganese Ore from M/s. National Carbon Co. Ltd., Calcutta — S. B. Dasgupta, G. P. Mathur & P. I. A. Narayanan (I.R. 395/66).

30. Moulding Characteristics of Sand Sample No. BTL-3 (Natural Moulding Sand) received from Punjab State Industries Department, Chandigarh (I.R. 396/66) — R. C. Arora & Autar Singh.

31. Investigation on Moulding Characteristics of Sand from Tarakeswar, Dist. Hooghly — Amitava Das & Ashimesh Dutt (I.R. 397/66).

32. Moulding Characteristics of Maharashtra Silica Sand (MSS-4) — P. R. Sastry, V. S. Bhandary & P. K. Gupte (I.R. 398/66).

33. Sintering of Iron Ore Fines from Bellary Hospet Area — B. L. Sengupta, S. K. Banerjee & P. I. A. Narayanan (I.R. 399/66).

34. Moulding Characteristics of Silica Sand from Sherthalli, Madras — Savinder Singh, V. S. Bhandary & P. K. Gupte (I.R. 400/66).

35. Moulding Characteristics of Sand No. BTL-4 (Natural Moulding Sand) received from Punjab State Industries Dept., Chandigarh — R. C. Arora, Autar Singh (I.R. 401/66).

36. Moulding Characteristics of Maharashtra Silica Sand (M-S-S-2) — R. R. Das, V. S. Bhandary & P. K. Gupte (I.R. 402/66).

37. Moulding Characteristics of Sand Sample No. GSP-A received from Punjab State Industries Department, Chandigarh — R. C. Arora & Autar Singh (I.R. 403/66).

38. Studies on the Nundydroog Gold Ore with a View to Improving Gold Recovery — P. V. Raman, R. Ganesh, K. Vijayaraghavan & P. I. A. Narayanan (I.R. 404/66).

39. Moulding Characteristics of Sand Sample No. BTL-10 received from Punjab State Industries Department, Chandigarh — R. C. Arora & Autar Singh (I.R. 405/66).

40. Moulding Characteristics of Sand Sample No. BTL-12 (Natural Moulding Sand) received from Punjab State Industries Department, Chandigarh — R. C. Arora & Autar Singh (I.R. 406/67).

41. Laboratory Study of Indian and Foreign Nozzles for Pouring Steel — A. V. Subramanyam & H. P. S. Murthy (R.R. 229/66).

42. Shell Moulding with Indigenous Raw Materials — P. R. Sastry, V. S. Bhandary, P. K. Gupte & B. R. Nijhawan (R.R. 230/66).

43. An Electronic Potentiostat for Study of Electrode Reactions — S. K. Sarkar & A. P. Chowdhury (R.R. 231/66).

44. Aluminizing of ACSR Core Wire — C. A. N. Rao, S. M. Arora, P. K. Gupte & B. R. Nijhawan (R.R. 232/66).

45. A Model on the Study of Slags and Their Utility as Slag Cement with the Help of Operational Research Technique — C. P. Saksena (R.R. 233/66).

46. Heat Content and Heat Capacity of Lead-Antimony-Tin Ternary Eutectic Alloy in the Liquid State — Gangotri Misra & Rajendra Kumar (R. R. 234/66).

47. Heat Contents and Heat Capacities of Liquid Lead-Antimony Alloys — Gangotri Mishra & Rajendra Kumar (R.R. 235/66).

48. Effect of Titanium on the Formation of Graphite Cast Iron — Rajendra Kumar & N. K. Das (R.R. 236/66).

49. Structure of Liquid Aluminium-Copper Alloys — Rajendra Kumar & Manjit Singh (R.R. 237/66).

50. Development of Substitute High Temperature Creep Resistance Alloys — R. Choubey, B. R. Nijhawan, P. K. Gupte, B. N. Das, S. S. Bhatnagar & K. Prasad (R.R. 238/66).

51. Some Effects of Stress Changes on Creep Resistance of Iron-Manganese-Nitrogen Alloys — R. Choubey & B. R. Nijhawan (R.R. 239/66).

52. Selective Chlorination of Iron from Ilmenite with Hydrochloric Acid Gas — C. Sankaran, R. N. Misra & P. P. Bhatnagar (R.R. 240/66).

53. Metallography of Rapid Solidification — Rajendra Kumar & A. N. Sinha (R.R. 241/66).

APPENDIX III

Major Sponsored Projects at the National Metallurgical Laboratory

Sl No.	*Title*	*Sponsor*
1.	Beneficiation of a Low Grade Iron Ore from Goalpara, Assam.	Director of Geology & Mining, Government of Assam
2.	Beneficiation of Calcite from Sankaridrug, Madras.	M/s. Neyveli Salem Steel Plant, Government of Madras
3.	Manufacture of Stable Dolomite Clinker.	M/s. Neyveli Salem Steel Plant, Government of Madras
4.	Beneficiation of an Iron Ore Sample from Kundremukh deposits, Mysore.	M/s. National Mineral Development Corporation Ltd, Faridabad
5.	Studies on Sintering of Bhawanthpur Dolomite for Its Suitability as a Refractory Material.	M/s. Bokaro Steel Ltd, Marafari, Dt. Hazaribagh
6.	Production of High Carbon Ferro-chrome and Silico-chrome.	M/s. Ferro Alloys Corporation Ltd, Garivadi
7.	Pilot Plant Beneficiation Studies on a Low Grade Mixed Apatite Sample from Singhbhum Dt., Bihar.	Commissioner of Mines, Government of Bihar
8.	Sintering of Iron Ore Fines from Bellary Hospet Area.	M/s. Minerals & Metal Trading Corporation of India Ltd, New Delhi
9.	Studies on the Nundydroog Gold Ore with a View to Improving Gold Recovery.	Managing Director, Kolar Gold Mining Undertaking, Kolar.
10.	Beneficiation of a Low Grade Limestone Sample.	M/s. Ashok Cement Co. Ltd., Dalmianagar
11.	Beneficiation of Low Grade Kyanite from Magarmohan Kyanite Mines, Pallahara Dt., Orissa.	M/s. S. Lal & Co. Pvt. Ltd., Orissa
12.	Removal of Mica from Jamuna River Sand.	National Building Organization New Delhi
13.	Reduction of Insoluble Content of a Selenite Sample from Rajasthan.	M/s. Bikaner Gypsum Ltd., Calcutta
14.	Beneficiation of Low-Grade Graphite from Palamau, Bihar.	M/s. Singharia Commercial Co. Ltd., Calcutta.
15.	Beneficiation of Graphite.	M/s. National Carbon Co. Ltd., Calcutta