ANNUAL REPORT

1964-1965

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

CONTENTS

RESEARCH PROJECTS

PILOT PLANTS

ENGINEERING SECTIONS

INDUSTRIAL LIAISON, OPERATIONAL RESEARCH, INFORMATION AND EXTENSION SERVICES

APPENDIX I

APPENDIX II

APPENDIX III

FOREWORD

I HAVE great pleasure in presenting the Report of the Director, National Metallurgical Laboratory, for the year 1964-65.

As will be seen from the Report, the period under review has for the National Metallurgical Laboratory, been of sustained activity. The laboratory has continued to fulfil its obligations to heavy metallurgical complexes, such as the integrated steel plants, non-ferrous projects besides collaborating with the Planning Commission, various Central Ministries of the Government of India, State Governments, different private sector Bodies and Associations. In accordance with the recommendations of the CSIR Reviewing Committee, researches and development work on non-ferrous metallurgical projects, design engineering, etc., were stepped up.

Another important activity of the National Metallurgical Laboratory has been the preparation of Project Reports for heavy industrial complexes, such as for the Bokaro Steel Plant relating to Kiriburu iron ores, their beneficiation and sintering characteristics.

For the Defence and Ordnance establishments of the Government of India, the National Metallurgical Laboratory has done valuable work in respect of supply of strategic alloys, formulation of technical know-how and in completing short-term ad-hoc investigations and long-term research projects well on schedule.

Amongst the various substitution oriented projects underway at the National Metallurgical Laboratory, I may mention the development of technical know-how for the recovery of zinc metal from zinc drosses and wastes on behalf of the Indian Tube Company.

Some new metallurgical processes have been released to the industry during the period under review; such as the abundant and cheap " blue dust " of the Indian iron mines for the production of hard ceramic magnets, an expensive and specialized product.

Work on various projects undertaken on behalf of Railway Research, Designs and Standards Organization and Indian Standards Institution has maintained high tempo of work and progress.

The Government of India some time back set up a Technical Committee under the Chairmanship of Dr. B. R. Nijhawan, Director, National Metallurgical Laboratory and comprising representatives from Hindustan Steel Limited and Tata Iron and Steel Company Limited, to examine the efficient utilization of iron blast furnace capacity in India through suitable preparation of raw materials and by the introduction of proved latest technical innovations. The Technical Committee presented its Report to the Government of India some time back which has greatly appreciated its findings and recommendations that are now finding industrial scale implementation.

The *NML Technical Journal* has now entered into its seventh year of publication and is maintaining its high standard as one of the leading journals in metallurgy. The Proceedings of the Symposium on " Utilization of Metallurgical Wastes " held under the auspices of the National Metallurgical Laboratory in February 1964 have received high acclaim at home and overseas.

In the field of International Co-operation, the National Metallurgical Laboratory is actively co-operating with International bodies like International Working Group on Bonding Clays, International Standards Organization, International Foundry Congress. India will be the venue of 33rd International Foundry Congress and the National Metallurgical Laboratory is actively co-operating with Indian Institute of Foundrymen in holding this Congress. An International Symposium on " Micro-Metallurgy — the Role of Minute Additions to Ferrous and Non-Ferrous Metals and Alloys " was organized by the National Metallurgical Laboratory in early 1965 which was inaugurated by Shri Asoka Mehta, Deputy Chairman, Planning Commission, Government of India.

Dr. B. R. Nijhawan, Director, National Metallurgical Laboratory, has been conferred the Hony. Membership of the Iron and Steel Institute, Japan; and also awarded a Gold Medal for his outstanding contributions made in the field of metallurgy at the Jubilee celebrations of Japanese Iron and Steel Institute; Dr. Nijhawan is the first Indian to receive this coveted and distinguished honour. He has also recently been awarded the coveted Shanti Swarup Bhatnagar Memorial Award in Engineering Sciences by the Council of Scientific & Industrial Research.

The credit for the excellent work the National Metallurgical Laboratory has been doing in various fields of mineral and metal industries, in basic objective researches and pilot plant scale trials undoubtedly goes to Dr. B. R. Nijhawan and his able band of colleagues. I should like to place on record our sincere appreciation of the sustained hard work by the devoted staff of the National Metallurgical Laboratory under Dr. Nijhawan's tireless and able guidance.

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

October 30, 1965

INTRODUCTION

THE story of the National Metallurgical Laboratory during 1964-65 is the story of active consolidation and expansion in multiple fields in the background of completing its Third Five Year Plan Projects often ahead of schedule.

Some major highlights have been the active association of the National Metallurgical Laboratory with the Bokaro Steel Plant Project wherein the Soviet Union and the Indian authorities have entrusted major work relating to the raw-materials such as beneficiation and agglomeration studies on iron ores including the production of super-basic self-fluxing sinters; work relating to development project of National Mineral Development Corporation, such as Khetri and Rakha Copper Projects, the Kiriburu Iron Ore Development Project, the Bailadila Iron Ore Beneficiation and Sintering Project; work relating to the establishment of beneficiation and sintering plants of Dalli Iron Ore Project for Bhilai Steel Plant; the establishment of National Metallurgical Laboratory Foundry Stations; the completion of Marine Corrosion Research Station at Digha, etc.

The Light Naphtha Injection system at the Low-shaft Furnace Pilot Plant of the National Metallurgical Laboratory was completed at a cost of Rs. 3 lakhs and inaugurated by Prof. Humayun Kabir, Union Minister for Petroleum and Chemicals. The entire system indigenously designed, fabricated, assembled and commissioned represents a high degree of team work.

The National Metallurgical Laboratory has maintained its activities on top priority to meet the strategic requirements of Defence and Ordnance Plants. Efforts are being directed to assess the Defence requirements in close co-ordination with Defence authorities including the Defence Metallurgical Research Laboratory at Hyderabad. The National Metallurgical Laboratory is engaged in giving maximum assistance possible to meet the requirements of Defence and Ordnance Establishments by undertaking the production and supplying them essential strategic diverse products in finished or semi-finished state, besides undertaking short-term and long-range applied projects for them.

Apart from the close co-ordination maintained with various industrial organizations in Public and Private Sectors, the National Metallurgical Laboratory is collaborating with various Universities and Higher Technical Institutions in the country. National Metallurgical Laboratory scientists have been deputed to deliver lectures at the University of Roorkee, Bengal Engineering College, Indian Institute of Technology at Kharagpur and Madras, Regional Institute of Technology, etc.

The extension services of the National Metallurgical Laboratory have been further strengthened with the setting up of Foundry Stations. The National Metallurgical Laboratory Foundry Station at Madras has started functioning and those at Howrah and Ahmedabad will go into operation in the very near future. The Marine Corrosion Research Station at Digha has already initiated research work relating to corrosion of metals and alloys under marine environment.

The expansion programme of the Laboratory under the Third Five Year Plan has kept to schedule. The new Central and two Side Wings, fully equipped for advanced researches, were opened; new Foundry

Block was completed and commissioned; construction of radio-active isotope laboratory is in full swing. For the establishment of 250 tons/year Magnesium Semi-commercial Pilot Plant, 7 acres of land are being acquired from the Tata Iron & Steel Company.

Research studies on fundamental research themes have been pursued *pari passu* with applied development and pilot plant projects. Important research themes relating to phase equilibria, X-ray diffraction studies, age-hardening characteristics, structure of liquid metals, gas solubility studies, temper brittleness, etc., are underway which have been reported in the appropriate section of the Report.

Based on these fundamental studies, Theses work on the following subjects is underway for Ph.D. degrees by the National Metallurgical Laboratory staff.

1. Nickel-free austenitic stainless steels.
2. Studies on some mechanical properties of refractories.
3. Thermal properties of liquid metals.
4. Structure of liquid metals.
5. High temperature phase equilibria in refractory mineral system involving magnesia, silica, titania, lime and ferric oxide.
6. Thermal properties of blast furnace raw materials.
7. Study of the phase transformation in electrolytic copper-tinalloys.
8. Beneficiation of beach sand placers of Manavalakurichi, Madras.
9. Structure of liquid aluminium-silicon alloys.
10. Formation and distribution of graphite in cast iron.
11. Pearlitic transformation of austenite in low alloy steels.
12. X-ray metallography of retained austenite and martensite in relation to grain size and residual internal stresses.
13. Corrosion resistance and metallurgical characteristics of substitute stainless steels in relation to chemical composition and heat-treatment.
14. Studies on the structure of electrodeposited manganese.
15. High temperature ceramics coating on metal or high temperature alloys.
16. Viscosities of $CaO\text{-}MgO\text{-}Al_2O_3\text{-}SiO_2$ slags.
17. Phase transformation in alloy steels.
18. Plastic coating on steel.

Senior and Junior Research Fellows take up basic objective research problems, which apart from their intrinsic research training will make them eligible for Ph.D. degree at some of the Indian Universities. The Graduate and Trade Apprentices are given a thorough training in instrumentation techniques, operation of pilot plant, etc., for a period of 1 to 2 years.

The process developed earlier on production of carbon bonded graphite crucible is now under full industrial scale production by Messrs. Patna State Graphite Mining Company. The process on hot-dip aluminizing was licensed to more firms and practical demonstrations were held. Another important process released is the production of copper powder which has been licensed to Indian Copper Corporation, Ghatsila.

During the year under review the laboratory has taken the following Patents:

Patents filed during the period :

1. Indian Patent No. 96661 — A novel method of separating iron from ilmenite and its application in the upgrading of ferruginous ores (24-11-1964).

2. Indian Patent No. 96017 — Production of high purity iron (12-10-1964).

3. Indian Patent No. 94769 — An improved device for the continuous hot-dip coating of metallic strip and wire (20-7-1964).

4. Indian Patent No. 94768 — An improved device for the isolation of dross in molten metallic baths during continuous hot-dip processing of strips or wire (20-7-1964).

5. Indian Patent No. 94767 — An improved cast iron pot for the melting and holding of non-ferrous molten metal in general and aluminium and zinc in particular (20-7-1964).

Patents Accepted During the Period:

1. U.K. Patent No. 989802 — Improvements in or relating to the modification of aluminium coating silicon (19-2-1965).

Patents Sealed During the Period :

1. Indian Patent No. 83968 — A method for reconditioning the coated magnesium powders (29-5-1964).

2. Indian Patent No. 83652 — Improvements in or relating to magnesite refractories (29-5-1964).

3. Indian Patent No. 65610 — Improvements in or relating to the production of chemically bonded metal-clad or unclad basic refractories (4-9-1964).

4. Indian Patent No. 84670 — Improvements in or relating to electrolytic cells (11-9-1964).

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-bonded graphite crucibles).
9. Refractory compositions comprising graphite and alumino-silicate materials and glazes to render such compositions resistant to oxidation (clay bonded graphite crucibles).
10. Production of carbon-free ferro-alloys by alumino-thermic reactions.
11. Production of iron powder for autogenous cutting.
12. Preparation of liquid gold.
13. Production of flux for submerged arc welding.
14. Electroplating of metals on aluminium or its alloys.
15. Chemical polishing of aluminium.
16. Metallization of non-conductors.
17. Brass plating from non-cyanide bath.
18. Production of dense carbon aggregate suitable for being used as base material for carbon products in general and Soderberg paste in particular.
19. Production of electrolytic copper powder.
20. Production of ceramic magnets.

An international Symposium was organized by the National Metallurgical Laboratory from 29th March to 1st April, 1965 on " Micro-Metallurgy — the Role of Minute Additions to Ferrous and Non-Ferrous Metals and Alloys " where the results of NML researches on basic objective themes were presented along with distinguished research contributions from overseas scientists who actively participated in the Symposium deliberations in which 29 papers were presented and discussed in 6 technical sessions. The National Metallurgical Laboratory is now renowned for its annual technical Symposia organized on international scale, the proceedings of which receive equal recognition and acclaim at home and overseas.

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

RESEARCH PROJECTS

1.0 Pilot Plant Studies on Beneficiation and Sintering of a Mixed Iron Ore Sample from (i) Hill No. 1, Bench No. 2 and (ii) Hill No. 2, Bench No. 2 of Kiriburu Iron Ore Mines of the National Mineral Development Corporation

Two samples of iron ore from Kiriburu mines, one from Hill No. 1, Bench No. 2 and the other from Hill No. 2, Bench No. 2 were received for beneficiation and sintering studies. The samples were mixed in equal proportions and the mixed sample assayed Fe, 57·90; SiO_2, 3·0; Al_2O_3, 7·00; P, 0·047; TiO_2, 0·26 and 7·60 per cent loss on ignition and traces of S, CaO and MgO.

Screenability tests with different moisture contents indicated that maximum screening difficulty would be encountered at 7·5 per cent moisture content. Tests were conducted to produce clean, sized products suitable for iron production in the projected Steel Plant at Bokaro and also for interim export. Keeping in view the requirement for Bokaro Steel Plant, the ore after crushing to 2 in. (50·8 mm.) was scrubbed and wet screened at 3/8 in. (9·5 mm.) size. The +3/8 in. (+9·5 mm.) washed lumps assayed Fe, 61·16; SiO_2, 1·37 and Al_2O_3, 5·16 per cent with iron recovery of 62·2 per cent. The rejection of silica and alumina in the slime was 37·5 and 22·1 per cent respectively. The −3/8 in. (−9·5 mm.) classifier sand product constituting 29·9 per cent by weight assayed Fe, 56·24; SiO_2, 3·48 and Al_2O_3, 8·07 per cent and contained 29·0 per cent of the total iron present in the ore. This product being high in insolubles was subjected to jigging after sizing into three fractions; the combined jig concentrate assayed Fe, 59·47; SiO_2, 2·33 and Al_2O_3, 5·97 per cent with an iron recovery of 23·0 per cent. The beneficiated fines would offer distinct metallurgical advantages over unbeneficiated fines for the production of self-fluxing sinter.

Scrubbing the ore after crushing to 4 in. (101·6 mm.) and wet screening to remove the −1/2 in. (−12·7 mm.) fines produced +1/2 in. (+12·7 mm.) lumps assaying Fe, 61·51; SiO_2, 1·43 and Al_2O_3, 5·35 per cent with iron recovery of 62·8 per cent chiefly for export purposes. The rejection of silica and alumina through the slime was 25·5 and 13·8 per cent respectively which was less than the values obtained by identical scrubbing of the ore at 2 in. (50·4 mm.) size. Mere wet screening the ore (without prior scrubbing) after crushing to 4 in. (101·6 mm.) produced +1/2 in. (+12·7 mm.) lumps assaying Fe, 61·10; SiO_2, 1·52 and Al_2O_3, 5·43 per cent with iron recovery of 65·8 per cent. The iron content of the ore lumps was almost similar to that obtained by scrubbing and wet screening it at the same size, but it retained slightly more silica and alumina contents in the lumps. The rejection of silica and alumina in the slime was 23·4 and 12·7 per cent respectively which was slightly lower than in the earlier case. The −1/2 in. (−12·7 mm.) classifier sand product when subjected to jigging after sizing into three fractions, produced a combined jig concentrate assaying Fe, 59·7; SiO_2, 2·59 and Al_2O_3, 5·65 per cent with iron recovery of 24·2 out of 29·5 per cent present in this fraction. The coarse fraction of the classifier sand was already of a fairly good grade and it could bypass the jigging circuit, thus obviating the necessity to jig the

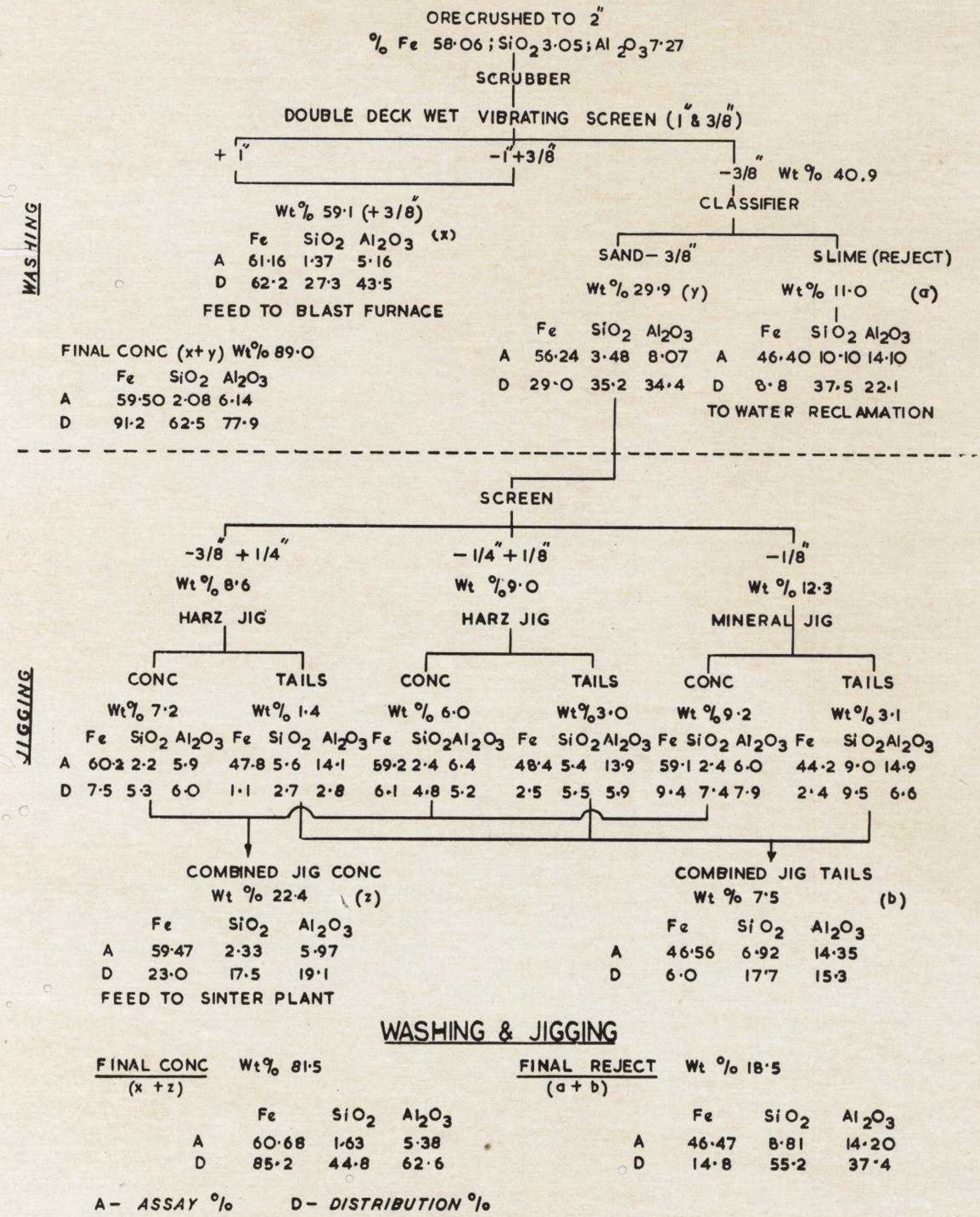


Fig. 1 — Summary of Results of Washing and Jigging of Kiriburu Iron Ore (H_1B_2, H_2B_2) at 2 in. size

entire fines. The results of washing and beneficiation are summarized in flowsheet Figs. 1, 2 and 3. Based on these results a general flowsheet recommended is given in Fig. 4.

The sintering characteristics of −1/2 in. (−12·7 mm.) washed classifier sand, both unjigged and jigged, were studied for investigating fluxed as well as unfluxed sinters. Only unfluxed sinters were made with washed but unjigged fines and it was found that 7 per cent water and 5 per cent coke were the optimum values for producing a strong sinter. In the case of beneficiated (jigged) fines, optimum water, coke and sinter return fines were determined to be 7, 4 and 25 per cent respectively. High grade metallurgical super-basic fluxed sinters up to basicity ratio of 2·0, could be produced from beneficiated fines by requisite additions of limestone. MgO content of the sinters could be increased by the addition of dolomite along with limestone without affecting the sinter quality in any way. Varying amounts of blue dust were added to the sinter mix to study the extent to which it could be incorporated in the sinter mix for making good sinter. Though the sintering rate was slightly lowered with progressive increase in the blue dust/ore fines ratio, it was found that blue dust additions up to 20 per cent with respect to ore fines (blue dust/ore fines ratio 1: 4) yielded a good metallurgical sinter of 1·6 basicity ratio, with a highly improved sinter chemistry. The results further showed that solid fuel, i.e. coke breeze, could be substituted by gaseous fuel (coke oven gas) up to 30 per cent of the total coke needed for effecting proper sintering. The sinters thus produced by such mixed firing technique were of good quality possessing high strength and a very high degree of oxidation when compared to those made by the conventional sintering process employing coke breeze only.

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

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

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

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

(iv) For every one per cent decrease in the alumina content of the ore, the 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 after allowing for depreciation, etc.

1.1 Beneficiation and Sintering Studies with Iron Ore Samples from Bailadila Mines of National Mineral Development Corporation

A 100-tonnes representative sample of run-of-mine ore from Bailadila iron ore mine, collected from Deposit No. 14 was received for beneficiation and sintering studies. The sample as received, assayed Fe, 66·9; SiO_2, 1·5; Al_2O_3, 1·8 and 1·7 per cent loss on ignition.

The ore being of good quality and mostly massive in character, mere wet screening of the ore after crushing to −6 in. (−152·4 mm.) size, without prior scrubbing could

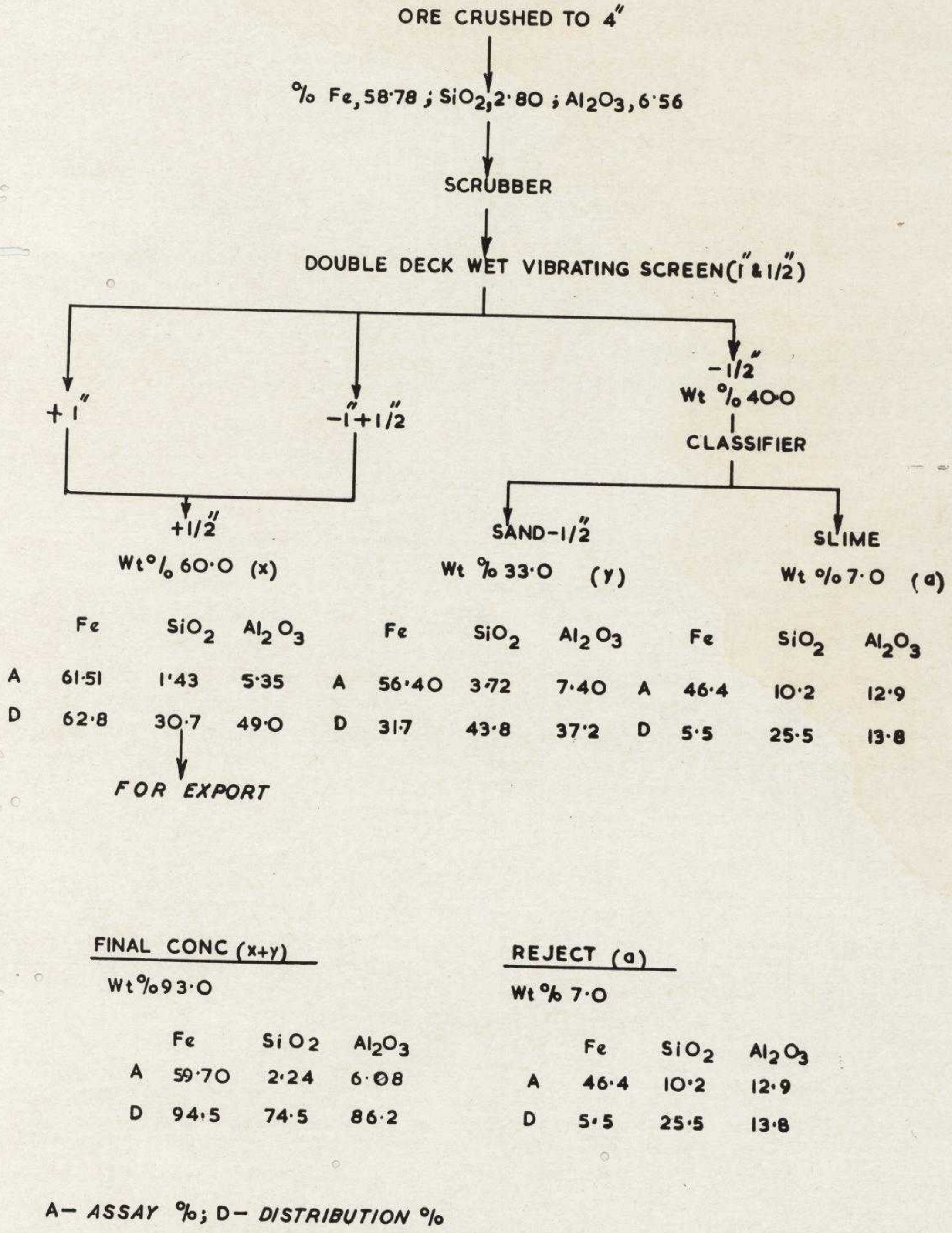
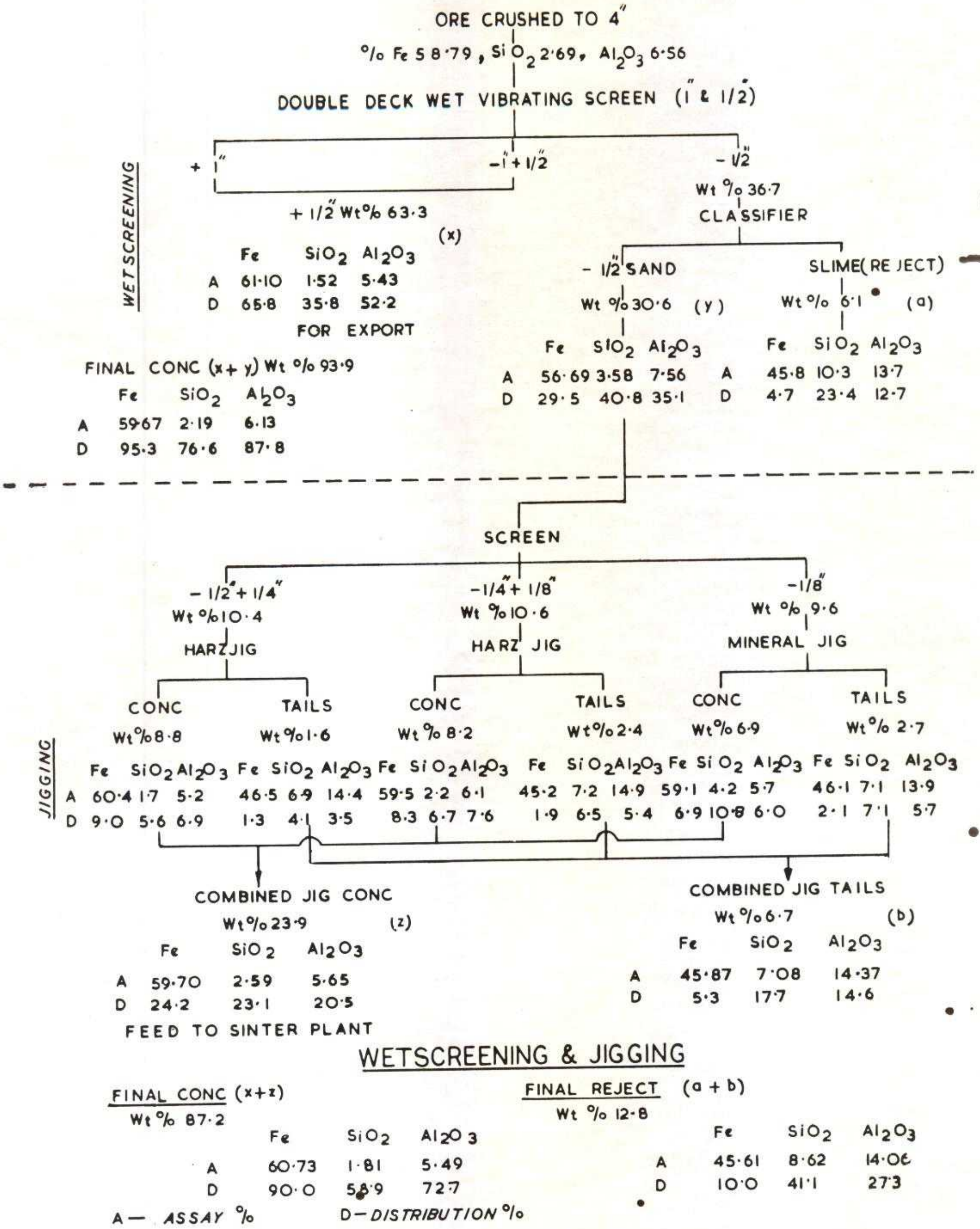


FIG. 2 — SUMMARY OF RESULTS OF WASHING OF KIRIBURU IRON ORE (H_1B_2, H_2B_2) AT 4 IN. SIZE



	Fe	SiO_2	Al_2O_3
A	61·10	1·52	5·43
D	65·8	35·8	52·2

	Fe	SiO_2	Al_2O_3
A	59·67	2·19	6·13
D	95·3	76·6	87·8

	Fe	SiO_2	Al_2O_3
A	56·69	3·58	7·56
D	29·5	40·8	35·1

	Fe	SiO_2	Al_2O_3
A	45·8	10·3	13·7
D	4·7	23·4	12·7

	CONC Wt% 8·8			TAILS Wt% 1·6			CONC Wt% 8·2			TAILS Wt% 2·4			CONC Wt% 6·9			TAILS Wt% 2·7		
	Fe	SiO_2	Al_2O_3	Fe	SiO_2	Al_2O_3	Fe	SiO_2	Al_2O_3	Fe	SiO_2	Al_2O_3	Fe	SiO_2	Al_2O_3	Fe	SiO_2	Al_2O_3
A	60·4	1·7	5·2	46·5	6·9	14·4	59·5	2·2	6·1	45·2	7·2	14·9	59·1	4·2	5·7	46·1	7·1	13·9
D	9·0	5·6	6·9	1·3	4·1	3·5	8·3	6·7	7·6	1·9	6·5	5·4	6·9	10·8	6·0	2·1	7·1	5·7

	Fe	SiO_2	Al_2O_3
A	59·70	2·59	5·65
D	24·2	23·1	20·5

	Fe	SiO_2	Al_2O_3
A	45·87	7·08	14·37
D	5·3	17·7	14·6

	Fe	SiO_2	Al_2O_3
A	60·73	1·81	5·49
D	90·0	58·9	72·7

	Fe	SiO_2	Al_2O_3
A	45·61	8·62	14·06
D	10·0	41·1	27·3

FIG. 3 — SUMMARY OF RESULTS OF WET SCREENING AND JIGGING OF KIRIBURU IRON ORE (H_1B_2, H_2B_2) AT 4 IN. SIZE

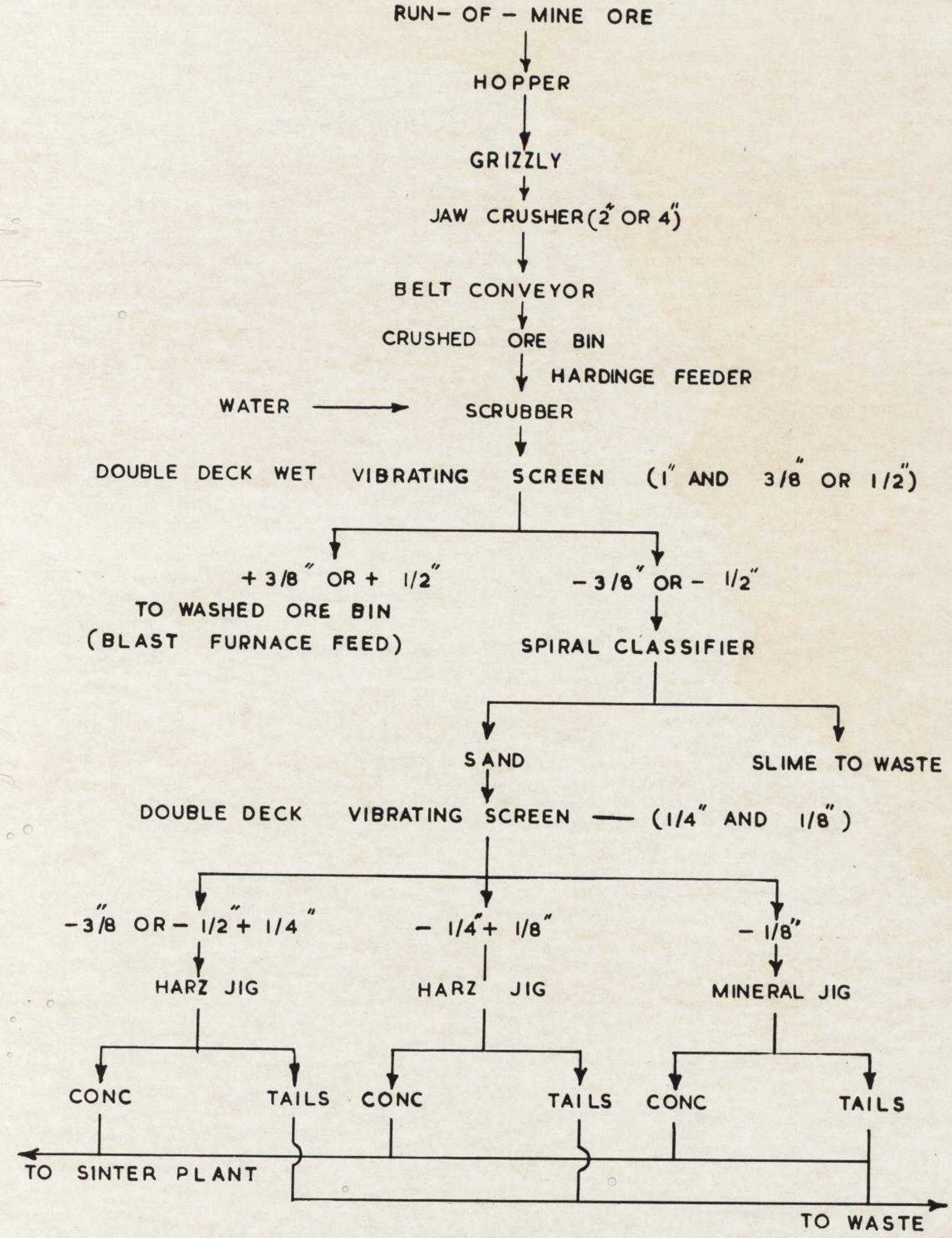


Fig. 4 — General Flowsheet for Beneficiation of Iron Ore from Kiriburu

Table 1 — Economic Evaluation of Beneficiation of Kiriburu Iron Ore

	Crushing to 2″, dry screening & utilization of —3/8″ fines			Crushing to 2″, dry screening & rejection of —3/8″ fines			Crushing to 2″, scrubbing, wet screening & dewatering of —3/8″ fines			Crushing to 2″, scrubbing, wet screening & beneficiation of —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 iron ore	57·9	3·00	7·00	60·56	1·31	5·52	59·5	2·08	6·14	60·68	1·63	5·38
Yield, tonnes/day	14,000 (100%)			8,610 (51·5%)			12,460 (89·0%)			11,410 (81·5%)		
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. 1·453			Rs. 1·349			Rs. 1·972		
(b) Operating cost	Rs. 0·180			Rs. 0·292			Rs. 0·266			Rs. 0·443		
(c) Cost of raw ore as mined	Rs. 5·000			Rs. 8·130			Rs. 5·618			Rs. 6·135		
Total cost per tonne of prepared ore	Rs. 6·074			Rs. 9·875			Rs. 7·232			Rs. 8·550		
Tonnes of ore required per tonne of pig iron	1·664			1·592			1·619			1·588		
Total cost of ore required per tonne of pig iron	Rs. 10·110			Rs. 15·72			Rs. 11·70			Rs. 13·58		
Difference in alumina per cent	—			1·48			0·86			1·62		
Expected saving in production cost per tonne of pig iron in blast furnace iron smelting due to decrease in alumina by beneficiation	—			Rs. 7·40			Rs. 4·30			Rs. 8·10		

yield a clean lumpy fraction (—6+3/8 in. ≡ —152·4+9·5 mm.) 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 washed lumps assayed Fe, 68·47; SiO_2, 0·53 and Al_2O_3, 1·32 per cent. Due to the hard and compact nature of ore and because of the absence of the soft variety of iron ore and clayey matter, slime rejection was only 1·8 per cent by weight. Consequently only 31·5 per cent of silica and 10·8 per cent of alumina could thereby be rejected in slime. The —3/8 in. (—9·5 mm.) classifier sand obtained by wet screening, constituting only 8·9 per cent by weight, assayed Fe, 55·7; SiO_2, 7·0; Al_2O_3, 6·0 per cent and accounted for 7·4 per cent of the total iron content of the ore. Sinters made from this product are not expected to offer any tangible metallurgical advantage due to their high insoluble contents. This product was, therefore, upgraded by jigging. The combined jig concentrate, weighing 6·5 per cent by weight assayed Fe, 66·0; SiO_2, 2·8 and Al_2O_3, 1·9 per cent with an iron recovery of 6·4 per cent. The loss of iron in the jig tailing was only 1·0 per cent with respect to the original metallic values. The summary of results of wet screening and jigging is given in Fig. 5. The flowsheet employed for treating this sample is given in Fig. 6.

The sintering characteristics of the —3/8 in. (—9·5 mm.) washed and beneficiated jig concentrate were studied for producing fluxed and unfluxed sinters. The optimum water, coke and sinter return fines were determined to be 7, 4·5 and 25 per cent respectively in the case of unfluxed sinters. Good metallurgical quality fluxed sinters up to basicity ratio of 2·0 could be produced by suitable additions of limestone. MgO content of the sinters could be increased by the addition of requisite quantity of dolomite along with limestone, without affecting the sinter quality in any way. The addition of freshly prepared burnt lime (2-3 per cent) to the sinter mix along with limestone and dolomite significantly increased the rate of sintering without much affecting the sinter strength, whilst the yield of blast furnace grade sinter was higher. The results further showed that coke breeze for sintering could be substituted by gaseous fuel (coke oven gas) to the extent of 40 per cent of the total coke needed. The sinters produced by such mixed firing technique were of good metallurgical grades and physical strength and also possessed a very high degree of oxidation, when compared with those made by the conventional solid fuel sintering process. The evaluation of the overall economics of beneficiation is given in Table 2 following the parameters referred to earlier.

1.2 Beneficiation and Pelletization of Iron Ore Fines from Messrs Salgaocar E Irmao Ltda, Goa

Beneficiation and pelletization studies were conducted on four samples of iron ore fines received from Messrs Salgaocar E Irmao Ltda, Goa, collected from their Velguem, Surla, Vimson Pale and Sallitho's Pale Mines.

The sample from Velguem mines assayed Fe, 64·3; Al_2O_3, 2·48; SiO_2, 1·98 and L.O.I., 3·64 per cent. Dry screening at 1/2 in. size followed by washing and cyclone treatment of the —1/2 in. fines yielded 75·8 per cent by weight of the beneficiated —1/2 in. product assaying Fe, 65·19; Al_2O_3, 1·56; SiO_2, 1·54 per cent. The sample from Sallitho's Pale mines assayed Fe, 62·94; Al_2O_3, 5·01; SiO_2, 1·43 and L.O.I., 4·75 per cent. Dry screening followed by washing and cyclone treatment of the —1/2 in. fines yielded 65·6 per cent by weight of beneficiated product assaying Fe, 64·88; Al_2O_3, 2·33; SiO_2, 1·14 per cent. The sample obtained by mixing all the four samples assayed Fe, 62·37; SiO_2, 1·5; Al_2O_3, 4·13 per cent. The mixed sample when subjected to washing and cyclone treatment yielded a beneficiated product (—1/2 in.) of weight 70·56 per cent and

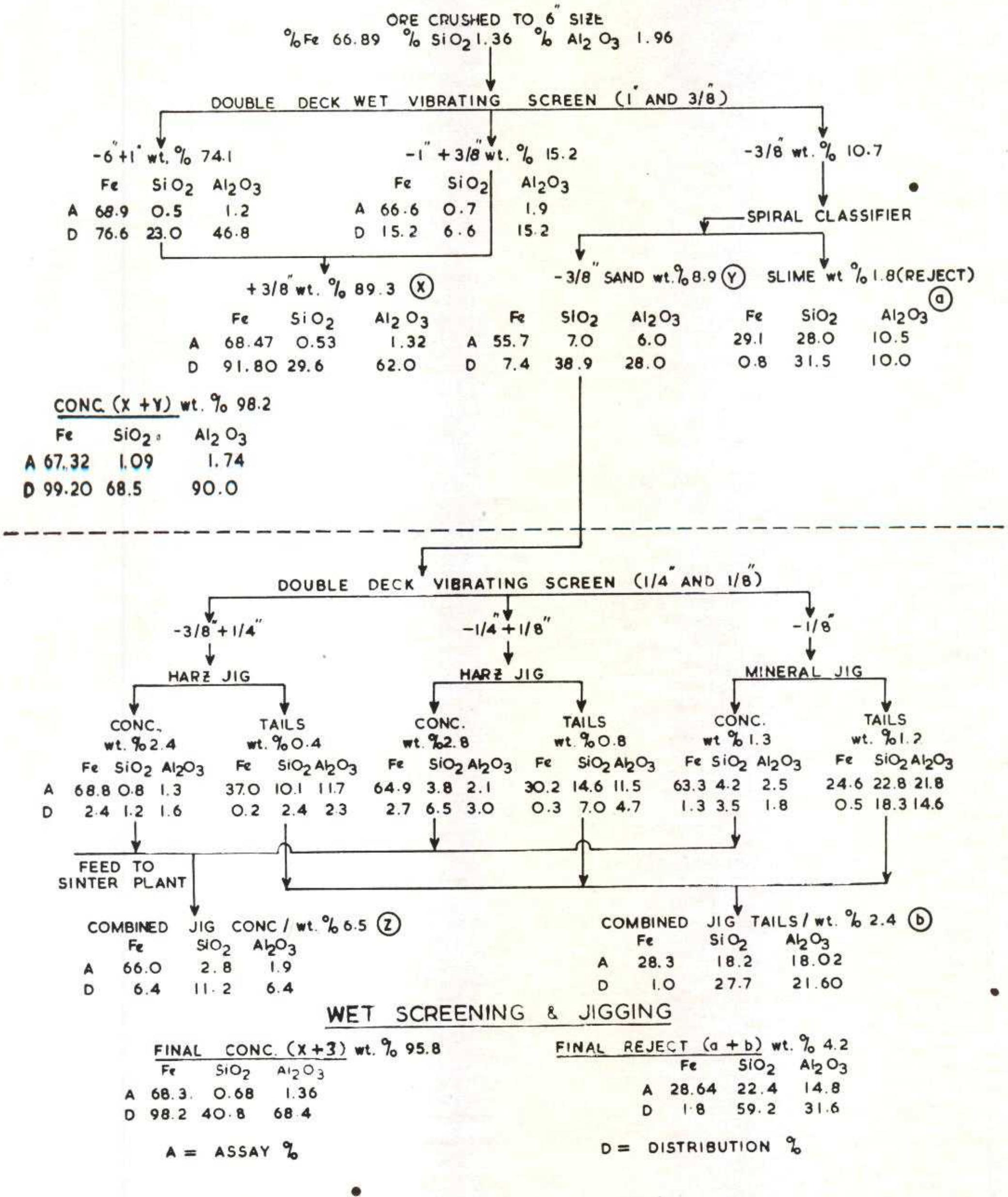


Fig. 5 — Summary of Results of Wet Screening and Jigging of Iron Ore from Bailadila

Table 2 — Economic Evaluation of Beneficiation of Bailadila Iron Ore

	Crushing to −6″, dry screening & utilization of −3/8″ fines %			Crushing to −6″, dry screening & rejection of −3/8″ fines %			Crushing to −6″, wet screening & dewatering of −3/8″ fines %			Crushing to −6″, wet screening & beneficiation of −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 ore:	67·16	1·49	2·06	68·7	0·67	1·5	67·32	1·09	1·74	68·3	0·68	1·36
Yield tonnes/day	14,000 100%			12,572 89·8%			13,748 98·2%			13,412 95·8%		
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. 0·995			Rs. 1·224			Rs. 1·680		
(b) Operating cost	Rs. 0·180			Rs. 0·200			Rs. 0·246			Rs. 0·338		
(c) Cost of raw ore as mined	Rs. 5·000			Rs. 5·569			Rs. 5·091			Rs. 5·219		
Total cost per tonne of prepared ore	Rs. 6·074			Rs. 6·764			Rs. 6·561			Rs. 7·237		
Tonnes of ore required per tonne of pig iron	1·435			1·403			1·432			1·411		
Total cost of the ore required per tonne of pig iron	Rs. 8·718			Rs. 9·488			Rs. 9·396			Rs. 10·210		
Difference in alumina per cent	—			0·56			0·32			0·70		
Expected saving in production cost per tonne of pig iron in blast furnace iron smelting due to decrease in alumina by beneficiation	—			Rs. 2·80			Rs. 1·60			Rs. 3·50		

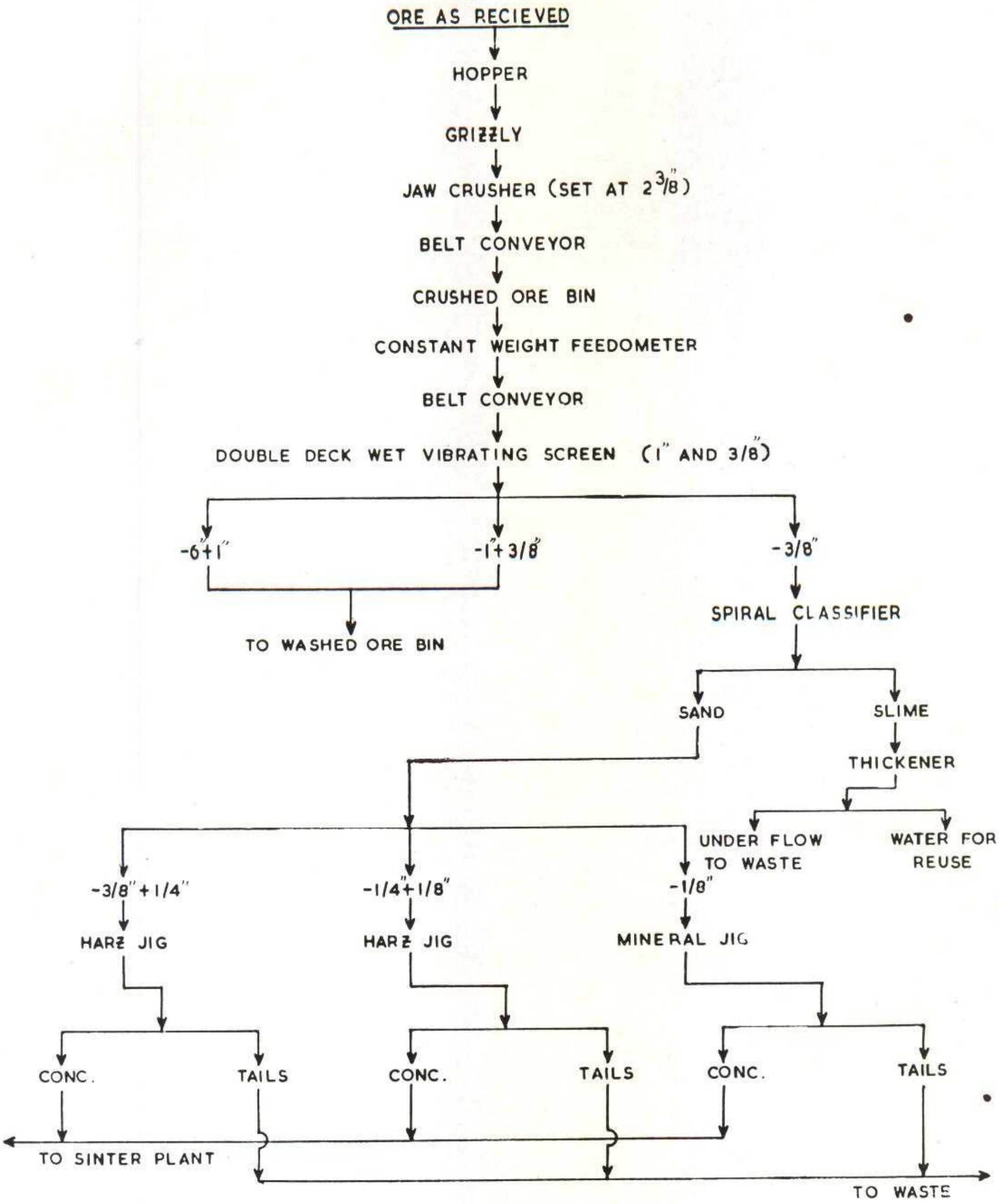


Fig. 6 — Flowsheet Employed in NML for the Beneficiation of Iron Ore from Bailadila

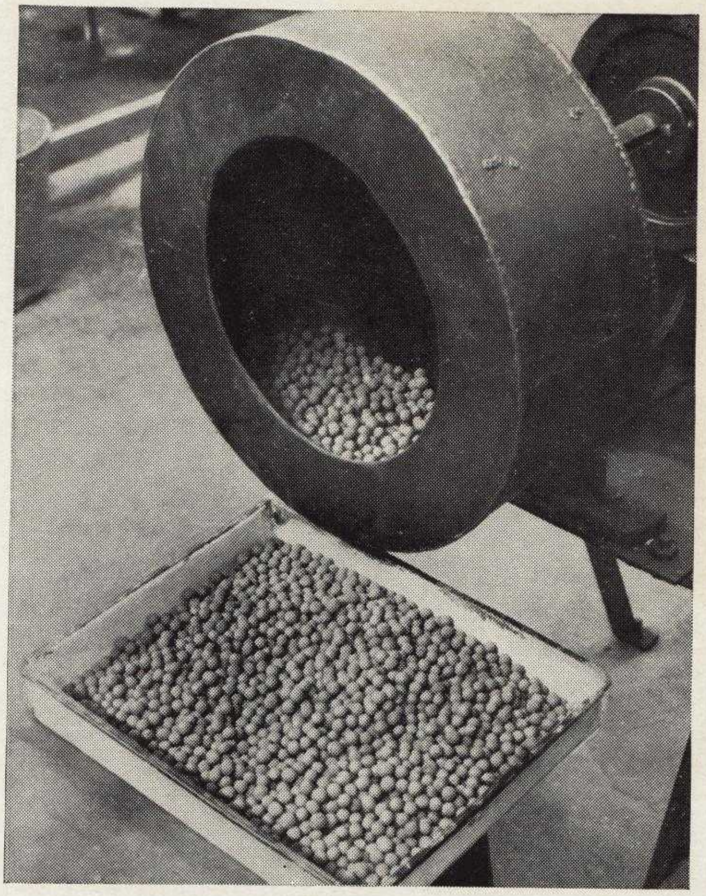


Fig. 7 — A View of the Batch Type Drum Pelletizer, Designed and Fabricated at the National Metallurgical Laboratory

assayed Fe, 63·74; Al_2O_3, 2·84; SiO_2, 1·35 per cent. The washed +1/2 in. ore accounted for 24·35 per cent by weight and assayed Fe, 59·53 per cent. The beneficiated fines when ground to 78·4 per cent −325 mesh yielded green pellets of good strength which could withstand rough handling and when heat-hardened around 1380°C. yielded good grade fired pellets of a grade as high as Fe 66·74 per cent. The quality of pellets was fully satisfactory from the point of view of chemical composition and physical properties. These pellets should make an ideal burden feed for iron smelting. On the basis of the results obtained, the flowsheet given in Fig. 8 has been recommended for treatment of mixed fines.

Tests done with synthetically prepared water of the maximum salinity available at the proposed plant site did not show any adverse effects either in grinding or in pelletizing; in fact, the results were somewhat more favourable. Hot compressive strengths of the heat-hardened pellets indicate that these pellets are not likely to crack or deteriorate in strength when reheated up to 800°C.

1.3 Beneficiation of Specular Hematite Samples from Rajasthan — Sample from Amsiwali, Udaipur Dist.

A sample of low grade specular hematite ore from Amsiwali section Nathara-ki-pal deposits was received from Messrs Kamani Engineering Corporation for batch beneficiation studies. The sample assayed Fe, 49·85; SiO_2, 27·82; Al_2O_3, 0·38; MgO, 0·2; P, 0·02 and S, 0·12 per cent. Microscopic studies indicated that specular hematite was the chief ore mineral while quartz constituted the main gangue. Hematite was free from gangue at about 100 mesh.

Tabling, spiral, high intensity magnetic separation reduction-roast followed by magnetic separation, electrostatic and flotation techniques, were investigated with the following results.

Straight tabling at 65 mesh yielded a concentrate assaying Fe, 67·0 and SiO_2, 2·1 per cent with a recovery of Fe, 86·0 per cent in it. Spiral treatment of a 65 mesh deslimed sample, followed by tabling of the spiral tailing yielded two iron concentrates which when mixed together would yield a combined product assaying Fe, 66·74 per cent with a total iron recovery of 85·6 per cent. Straight high intensity magnetic separation with a —65 mesh deslimed sample, produced an iron concentrate assaying Fe, 62·3 per cent with a recovery of Fe, 85·3 per cent. Reduction roast treatment at 3 mesh size followed by magnetic separation after grinding the reduced material to 65 mesh yielded a magnetic product assaying Fe, 66·73 per cent with a recovery of Fe, 98·2 per cent in it. This method yielded the best results, but its overall economics has to be studied before commercial scale application.

A good grade of iron concentrate was produced when a —65 mesh deslimed sample was subjected to electrostatic separation but iron recovery in the final concentrate was low.

Flotation studies undertaken on a 76 per cent, —200 mesh grind ore indicated that oleic acid flotation, at a *p*H of 7·8 could be successfully employed for producing good iron ore concentrates with high recoveries. The final concentrates assayed Fe, 65·72 and 66·74 per cent with recoveries of Fe, 85·8 and 82·7 per cent respectively. Flotation studies performed in acid circuit and employing petroleum sulphonate as collector for hematite at a *p*H of 4·5 yielded a rougher iron float, which when subjected to cleaning operations yielded a refloat concentrate assaying Fe, 66·4 per cent with a recovery of Fe, 90·3 per cent. This method, if adopted, will need special types of acid resisting flotation cells and other equipment to prevent heavy wear and tear to machinery.

Spiralling followed by tabling of the spiral tails is one of the best methods to produce a good iron concentrate. The advantage of this process is its low capital and operational costs; most of the equipment could well be readily fabricated indigenously. Magnetic separation after reduction roast yielded the best results, but the overall economics of the process will determine its acceptance including its heavy capital cost structure. As the concentrates are proposed to be smelted at Udaipur, the question of utilizing the blast furnace gas as well as Rajasthan lignite for magnetizing reduction roast treatment might be considered in the long run. Although flotation yielded good results, employment of the acid circuit for flotation will need acid resistant materials for the cells, pumps, etc., which will further increase the capital cost whilst the operating costs are also likely to be high. The final choice of the flowsheet to be employed will therefore, depend upon the study of the overall economics based on the above factors. The problem of agglomerating the concentrates to make it suitable for smelting, also needs separate study based on its capital cost structure and operational cost figures.

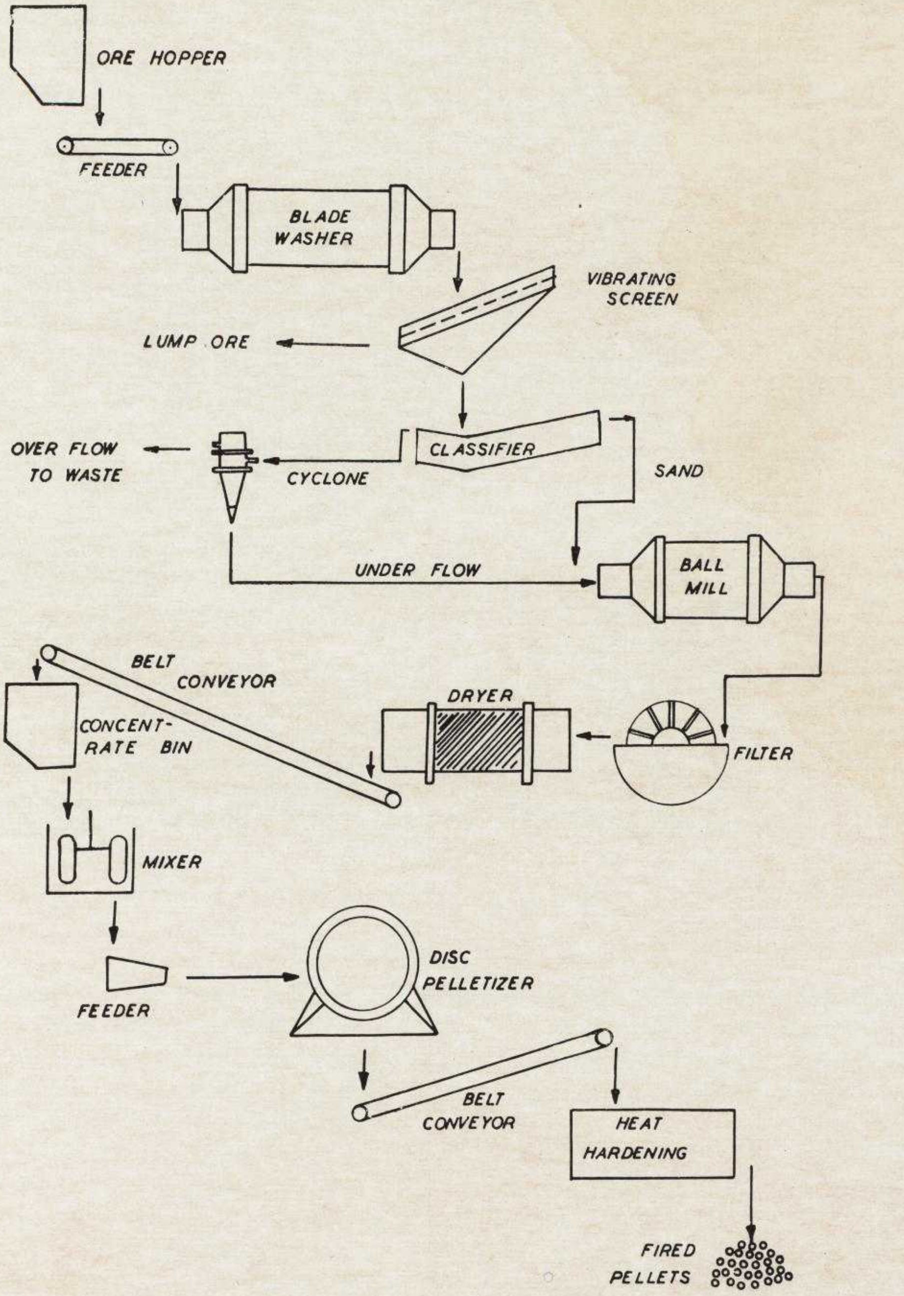


FIG. 8 — FLOWSHEET FOR BENEFICIATION AND PELLETIZING OF MIXED IRON ORE FINES FROM GOA

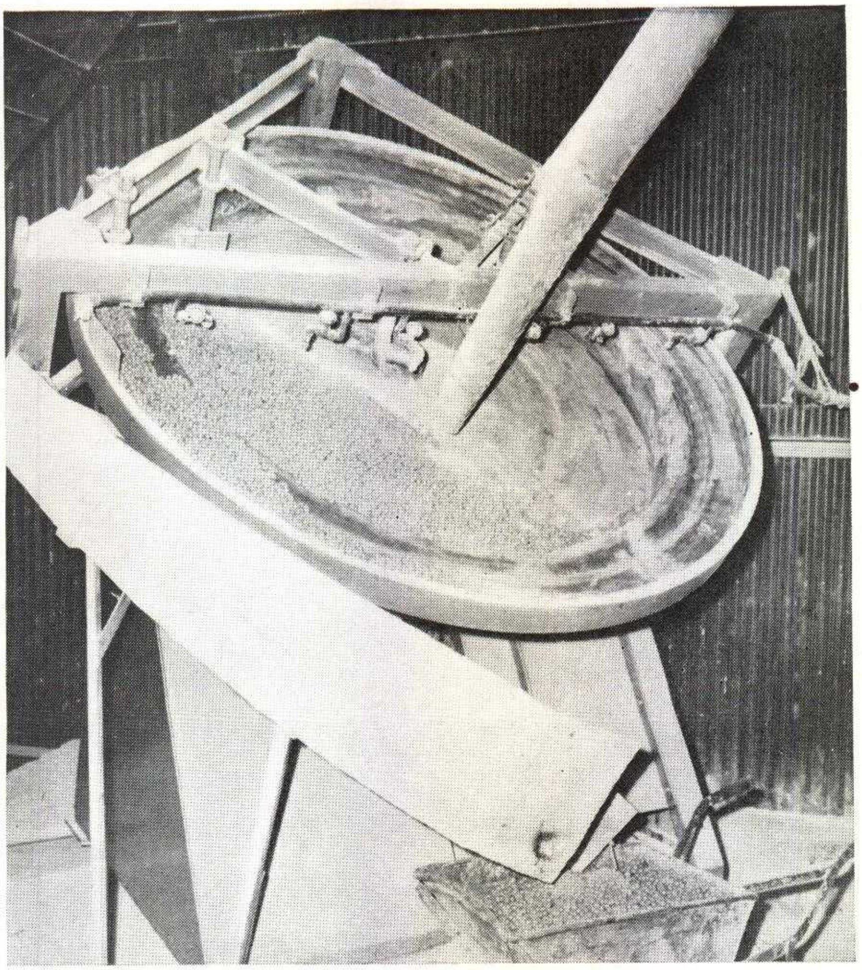


Fig. 9 — A View of the Disc Pelletizer of the Mineral Beneficiation Pilot Plant

1.4 Beneficiation of Specular Hematite Sample from Rajasthan — Sample from Daralmata, Udaipur District

A low grade specular hematite sample assaying Fe, 40·8; SiO_2, 40·37; Al_2O_3, 0·8; MgO, 0·2; P, 0·03 and S, 0·13 per cent from Daralmata section of Nathara-ki-Pal deposits in Rajasthan was received from Messrs Kamani Engineering Corporation for beneficiation studies.

Quartz was the principal gangue in the sample and it was fairly well liberated at −48 mesh size. Straight tabling at −48 mesh after classification yielded a concentrate assaying Fe, 65·8 and SiO_2, 3·8 per cent with a recovery of Fe, 79·4. Combination of Humphrey Spiral and table treatment produced a slightly poorer grade of concentrate assaying Fe, 62·0; SiO_2, 10·2; CaO, 0·31; Al_2O_3, 0·43; P, 0·05 and S, 0·15 per cent with a recovery of Fe, 84·7 per cent in the product.

High intensity magnetic separation of the −48 mesh deslimed ore did not show encouraging results, but wet magnetic separation after magnetizing reduction-roast yielded a high grade concentrate assaying Fe, 64·8 and SiO_2, 8·37 per cent with a recovery of Fe, 90·0 per cent. High tension separation yielded a concentrate of good grade, but metallic recovery was low. Flotation in alkaline circuit after grinding the sample to 78 per cent −200 mesh and desliming and using fatty acid as collector and sodium silicate as depressant produced a rougher float assaying Fe, 63·37 and SiO_2, 9·4 with a recovery of Fe, 64·7 per cent. The recovery improved to Fe, 78·5 per cent for a concentrate grade of Fe, 60·7 and SiO_2, 11·0 per cent by mixing a middling product after cleaning. Flotation in acid circuit using petroleum sulphonate as collector gave a concentrate assaying Fe, 61·1 and SiO_2, 10·87 per cent with a recovery of Fe, 86·1 per cent. One cleaning of the rougher concentrate improved the grade to Fe, 64·07 and SiO_2, 8·13 per cent with a recovery of Fe, 82·3 per cent. The ore was amenable to beneficiation by conventional methods though encouraging results were obtained by (1) gravity, (2) magnetic separation after reduction-roast and (3) flotation. From the point of view of operating costs, gravity method should be the cheapest. It should be readily possible to fabricate spirals and tables in India. Flotation in acid circuit should be avoided if possible, due to the acid-resistant nature of the flotation cells and other equipment needed and heavy wear and tear as well as higher operating costs. Though magnetic separation after reduction-roast treatment had given the best results, the economics of the process will depend on the question of utilization of lignite deposits of Rajasthan and/or the blast furnace gas that may be available from the projected Pig Iron Plant at Udaipur in future. The flowsheet to be employed for beneficiating this ore should be determined after closely studying the local conditions and working out the overall economics of the operations. The concentrate will need to be finally agglomerated before use in blast furnaces and this aspect needs study.

1.5 Studies on the Production of High Basicity Sinters from the Beneficiated Bolani Iron Ore Fines for the Durgapur Steel Plant, Hindustan Steel Ltd.

Following raw materials excepting beneficiated Bolani iron ore fines were received from the Hindustan Steel Ltd., Durgapur, for producing self-fluxing and superfluxing sinter.

Iron Ore Fines

(i) Jig concentrate produced in the National Metallurgical Laboratory Pilot Plant from the washed classifier sand (93·8 mm.) from the Bolani iron ore.

(ii) Fines from the iron ore supplied by the Minerals & Metals Trading Corporation.

(iii) Mill scale.

(iv) Fine iron powder obtained by Hecket Slag Reclamation Process.

(v) Flue dust.

(vi) Coke breeze : The sample of coke breeze was crushed to −8 mesh size.

(vii) Limestone : The limestone sample from Birmitrapur was crushed to −4 mesh size.

(viii) Dolomite : Dolomite sample was crushed to −4 mesh size.

(ix) Burnt lime received from Durgapur was found to contain lumps to fines and was sampled and stored in sealed tins. The material was slaked with the quantity of water usually added, before the sintering ingredients were mixed.

It was observed from the results that good superbasic sinters having basicity ratio $(CaO+MgO)/SiO_2$ even as high as 4·0-4·5 could be obtained with the jig concentrate. The results further showed that the rate of sintering improved with the increase in the basicity ratio of sinter mix.

Addition of fresh burnt lime in the sinter mix appreciably increased the rate of sintering. The sinter strength also improved. The yield of blast furnace sinter was increased by more than 30 per cent when compared to other tests of same basicity. It thus appears that addition of fresh burnt lime would have beneficial effect in sintering of iron ore fines.

Varying amounts of MMTC iron ore fines were introduced with the jig concentrate in the sinter mix. It was observed from the results that though the rate of sintering was slightly lower with the ore fines/MMTC fines ratio of 9: 1 the sintering rate increased with larger amounts of MMTC ore fines even up to 50 per cent of ore mix. Fine iron powder from Hecket scrap also could be used in the sinter mix up to 3 per cent for the utilization of this waste product. Tests conducted at a basicity ratio of 4·0 using MMTC fines and fine iron powder in the sinter mix, yielded a good sinter having high rate of sintering and good strength.

Introduction of mixed firing in sintering for the reduction of coke consumption was very useful in sinter making particularly since coke breeze contained high ash contents. Results of the present tests showed that the amount of coke for sintering could be reduced by 30 per cent if mixed firing is employed. Tests on the weathering properties of sinters indicated that sinters of basicity 1·6 did not disintegrate even after storage for 10 days. Sinters of basicity 3·0 could also be stored without disintegration during the first 3 days but they had a tendency to produce up to 2·8 per cent fines, during the next 7 days.

1.6 Beneficiation and Sintering Studies on Laminated Iron Ore Sample from Dalli Mines of Bhilai Steel Project

A sample of laminated iron ore from Dalli mines was received from Messrs National Mineral Development Corporation for beneficiation and sintering studies at the National Metallurgical Laboratory. The sample as received assayed Fe, 58·3; SiO_2, 4·1 and Al_2O_3, 7·4 per cent with L.O.I., 5·2 per cent.

Screenability tests performed with the sample showed that screenability was minimum when the water content of the sample was 5 per cent. Scrubbing followed by wet screening of the ore crushed to —2 in. (—50·8 mm.) size indicated that a clean sized washed product of +1/2 in. size (+ 12·7 mm.) free from adherent fines, could be obtained for direct charging into the blast furnace and a —1/2 in. size (—12·7 mm.) slime-free classifier sand for sintering. The +1/2 in. (+12·7 mm.) washed lump constituting 60·7 per cent by weight assayed Fe, 61·5; SiO_2, 2·84 and Al_2O_3, 5·84 per cent with a recovery of Fe 62·6 per cent. The slime constituted 5·8 per cent by weight and accounted for 21·0 per cent of the total SiO_2 and 15·1 per cent of the total Al_2O_3 originally present in the ore, for an iron loss of 3·9 per cent only. The —1/2 in. (—12·7 mm.) classifier sand obtained from washing (33·5 per cent by weight) assayed Fe, 58·66 and 10·5 per cent insolubles and contained 33·5 per cent of the total iron content of the ore. Being high in insolubles, this product was subjected to jigging and the —1/2 in. (—12·7 mm.) beneficiated jig concentrate (25·1 per cent by weight) assayed Fe, 63·2 and 6·41 per cent insolubles. The loss of iron in the tailing was about 6·5 per cent only.

Sintering characteristics of the —1/2 in. (—12·7 mm.) beneficiated jig concentrate were also studied for the production of unfluxed as well as fluxed sinters. Good quality fluxed sinters up to a basicity of 2·4 could be produced by suitable additions of limestone. Mixed firing technique employed for sintering indicated that coke could be partially substituted by gaseous fuel (coke oven gas) to the extent of 30 per cent without much affecting the sinter quality. The sinters produced from this had good strength and possessed the desirable degree of oxidation. Weathering tests performed with the fluxed sinters showed that the amount of −1/2 in. (−12·7 mm.) fines generated from sinters during storage for 24 hours varied from 2·1 to 3·8 by weight.

1.7 Pilot Plant Studies on Beneficiation and Sintering of a Sample of Hydroxide Ore from Dalli Mines of Hindustan Steel Ltd.

The sample of hydroxide variety of iron ore received from Messrs National Mineral Development Corporation was also from the Dalli mines of Bhilai Steel Plant, Hindustan Steel Ltd. The sample as received assayed Fe, 49·6; SiO_2, 7·6 and Al_2O_3, 11·3 per cent with L.O.I., 10·2 per cent.

Screenability tests showed that the minimum screenability was around 7·5 per cent moisture. " Dry Screening ", "Wet Screening " " Scrubbing " followed by wet screening tests performed with the sample crushed to 2 in. size (50·8 mm.) showed that the best results were obtained by " washing " (scrubbing followed by wet screening), indicating the necessity of scrubbing the ore prior to wet screening. The +1/2 in. (+12·7 mm.) washed lumps assayed Fe, 52·5; SiO_2, 6·25 and Al_2O_3, 10·2 per cent with a recovery of Fe, 58·7 per cent. Heavy media separation tests performed with this fraction at a medium sp. gr. of 2·9 (galena with water) produced a concentrate assaying Fe, 63·3; SiO_2, 2·1 and Al_2O_3, 2·56 per cent. The −1/2 in. classifier sand obtained from washing at 2 in. size, constituted 35·3 per cent by weight and assayed Fe, 48·2 per cent with 18·64 per cent insolubles and contained 34·2 per cent of the total iron present in the ore. This product was subjected to jigging after sizing into three fractions; the combined jig concentrate assayed Fe, 54·3; SiO_2, 4·8 and Al_2O_3, 8·24 per cent.

Sintering characteristics of the −1/2 in. beneficiated jig concentrate were also studied and it was found that good quality fluxed as well as unfluxed sinters could be produced. Fluxed sinters with basicity up to 1·8 possessed good physical strength and could be made by suitable additions of limestone. It was also noted that by employing the mixed firing technique, 30 per cent of the total amount of coke needed for sintering could be replaced by gaseous fuel (Producer gas), without varying the sinter quality much.

1.8 Pilot Plant Studies on Beneficiation and Sintering of a Sample of Compact Laminated Iron Ore from Dalli Mines of Hindustan Steel Ltd.

A compact laminated iron ore sample was received from Dalli mines of Hindustan Steel Ltd., for pilot plant beneficiation and sintering studies. The sample consisted of massive and compact laminated iron ore. The chemical analysis of the sample as received was Fe, 66·45; SiO_2, 1·94 and Al_2O_3, 2·0 with L.O.I., 1·7 per cent.

Screenability was found to be minimum at 5 per cent moisture. Wet screening of the crushed ore at −2 in. size produced +1/2 in. lumps (+12·7 mm.) constituting 74·6 per cent by weight and assaying Fe, 68·2; SiO_2, 1·1 and Al_2O_3, 1·31 per cent with Fe, 76·3 per cent recovery. The amount of slime produced was very low, i.e. 2 per cent by weight and 23·2 per cent of the total SiO_2 and 14·3 per cent of the total Al_2O_3 had been rejected, for an iron loss of about 1 per cent only. The −1/2 in. (−12·7 mm.) classifier sand was of a fairly good grade assaying Fe, 64·7 with only 6·24 per cent insolubles.

Sintering tests performed with this −1/2 in. (−12·7 mm.) classifier sand indicated that unfluxed sinters of good strength and quality with the optimum of coke, 4·5; water, 7 and return fines, 2·5 per cent could be made. Good quality fluxed sinters with basicity varying from 1·0 to 2·4 were also produced by suitable additions of limestone. Sinters produced by the mixed firing technique with basicity at 1·6 possessed good strength and desirable degree of oxidation. It was also observed that coke could be easily substituted by gaseous fuel to the extent of 30 per cent without much affecting the sinter quality.

1.9 Pilot Plant Investigation on Beneficiation and Sintering Characteristics on a Sample of Massive Iron Ore from Dalli Mines of Hindustan Steel Ltd.

The sample of massive iron ore was received from the Dalli Mines of Hindustan Steel Ltd., for beneficiation and sintering studies. The sample as received was of a high grade with Fe, 66; SiO_2, 2·3 and Al_2O_3, 2·1 with L.O.I., 1·9 per cent and could be used as such in the blast furnace, without further beneficiation. Attempts were made to reduce the insoluble contents of the ore through dry and wet screening tests after crushing the sample to —2 in. size (—50·8 mm.).

Screenability was poorest at 7·5 per cent moisture. Mere " Wet Screening " of the ore crushed to —2 in size (—50·8 mm.) showed that 75·3 per cent by weight of +1/2 in. (+12·7 mm.) lumps assaying Fe, 67·6; SiO_2, 1·22 and Al_2O_3, 1·58 per cent could be obtained with an iron recovery of 77·2 per cent. The slime constituting about 3·3 per cent by wt. rejected 16·6 per cent of the total silica and 9·0 per cent of the total alumina for an iron loss of 2·6 per cent. The —1/2 in. slime from classifier sand, 21·4 per cent by wt., assayed Fe, 62·4 and insolubles, 7·4 per cent. This product being high in Fe content was directly used for sintering studies.

Sintering characteristics of the —1/2 in. (—12·7 mm.) sand from wet screening tests were studied for the production of fluxed as well as unfluxed sinters. In the case of fluxed sinters, the basicity could be varied from 1 to 2·4 by calculated additions of limestone. The sinters made were of good quality and strength. The results of mixed firing tests showed that in the case of fluxed sinters, coke could be successfully substituted by gaseous fuel (Producer gas) to the extent of 20 per cent, without varying the sinter strength and quality much. The unfluxed sinters produced by this technique were brittle and hence had low shatter values. On the other hand, the fluxed sinters had sufficient strength as well as high degree of oxidation.

1.10 Beneficiation and Pelletization of Iron Ore Fines from Redi Mines, Maharashtra

This investigation was taken up at the instance of the Director of Geology and Mines, Maharashtra, to determine whether the high grade fines from Redi Mines of Ratnagiri District could be beneficiated and agglomerated to give a product suitable for export market.

About 45 tonnes of iron ore assaying Fe, 63; SiO_2, 1·5; Al_2O_3, 2·0 and CaO, 0·1 per cent were received from the Redi mines. For pelletization studies, a representative sample of about 2 tons was taken. After dry screening at 3/8 in. size, both the coarse and fine products were washed separately. The —3/8 in. product was scrubbed in a ball mill and the mill discharge was fed into a classifier. The classifier overflow was treated in a cyclone. The mixed classifier sand and the cyclone underflow assaying Fe, 64·4; SiO_2, 1·3 and Al_2O_3, 1·0 per cent was ground to three different sizes in a ball/rod mill for agglomeration studies which has given very satisfactory results.

1.11 Investigation on the Generation of Fines from Kiriburu Iron Ore during Transport and Handling

The Government of India has entered into a long-term contract to supply 2 million tonnes of iron ore per year from Kiriburu

Mines of National Mineral Development Corporation to the Japanese Steel Mills Association. The size specifications of lump ore for export are —8 in. +1/2 in. with a maximum permissible limit of 10 per cent for the —1/2 in. fines delivered at the port of entry in Japan. The ore mined is crushed and dry screened at Kiriburu and sent by rail to Visakhapatnam Port across a distance of about 615 kilometres for export to Japan. During the rail transport, extra ore fines are generated as also due to handling at the sea port and loading into ore-carriers following incidental drops therein.

At the request of National Mineral Development Corporation, the National Metallurgical Laboratory has undertaken investigations to determine the quantity and quality of fines likely to be produced from the screened lumpy ore of —8 in. and —4 in. sizes during rail transit. Keeping in view the ore requirements of indigenous steel plants, tests were also conducted with —2 in. ore.

Two 5-tonnes iron ore samples of —8 in. and —4 in. sizes were examined. Shatter tests were conducted with the —8 in. sample as received as well as after crushing to —4 in. The second sample of 4 in. size was received to determine the —1/2 in. fines produced during transit from Kiriburu to Jamshedpur and for comparison with the fines produced from the 8 in. sample under identical conditions.

The results showed that when the +3/8 in. fractions of —8 in., —4 in. and —2 in. size ore were subjected to two drops each from 6 ft. and 12 ft. heights, the 3/8 in. fines produced constituted 6·5, 5·9 and 5·4 per cent respectively. Similarly, the —1/2 in. fines produced from —8 in, —4 in. and —2 in. sizes constituted 6·9, 6·8 and 5·8 per cent respectively. It was very interesting to observe that when —8 in. +1/2 in. ore was dropped just once from a height of 36 ft., the —1/2 in. fines produced amounted to as much as 7·0 per cent with respect to the feed, which was almost the same as the quantity of fines produced after 4 drops, viz., two each from 6 ft. and 12 ft. heights. Similarly with —4 in. +1/2 in. ore, one drop from 36 ft. and four drops, two each from 6 ft. and 12 ft. heights, produced the same amount of —1/2 in. fines, namely, 6·8 per cent. The quantity of fines produced did not vary much with the feed size for shatter tests. The fines generated during every shatter test, be it on the basis of 6 ft. or 12 ft. drop, amounted to about 1·5 per cent by weight. But nearly four times this quantity was generated by one shatter drop test alone from a height of 36 ft.

Thus, assuming that the fines produced during rail transport between Kiriburu and Visakhapatnam will almost be the same as that produced during road transport between Kiriburu to National Metallurgical Laboratory, it was concluded that during the rail transport of ore from Kiriburu to Visakhapatnam and handling at different transit, loading and unloading points, the amount of —1/2 in. fines likely to be produced from +1/2 in. ore would be of the order of 14-16 per cent, on the basis that there will be only 4 nos. of 6 ft. to 12 ft. drops and one 36 ft. drop. To this amount should be added 6 to 8 per cent of fines already contained in the original "screened ore" leaving Kiriburu. For every additional handling (6 ft. to 12 ft. drop) 1½ per cent by wt. of extra fines should be produced. This amount would further increase if the ore was not as clean as that investigated and contained larger amount of fines adhering to lumps, as would be the case during the rainy monsoon months when the ore would be sticky and if the current dry screening practice is continued. If scrubbing was done to the ore before screening, not only will the lump ore be freed of adherent fines, but also considerable shattering caused during scrubbing itself would further reduce the generation of ore fines during subsequent rail transport. It is the soft and low grade ore that first crumbles during handling and transport, and if prior scrubbing is done, the considerable bulk of the fines normally produced during rail transport and handling, would have been removed in advance by wet screening after prior scrubbing. The advantages of scrubbing are thus clearly brought out.

It would thus be seen that there are no advantages of lowering the top size of shipping ore from 8 in. to 4 in. from the point of view of generation of fines during rail transport and handling. Regarding the grades of fines generated, the fines produced during transit between Kiriburu and Jamshedpur were of the poorest quality, of the softest and lateritic variety. The fines subsequently produced during shatter tests were of better quality indicating that hematite was gradually getting into the ore fines thus generated.

1.12 Study of the Distribution of Hematite in Band I Kanjamalai Magnetite Deposits, Salem District, Madras

Investigation was taken up at the instance of Government of Madras to study the hematite content in their Kanjamalai magnetite ore deposit near Salem, Madras. The problem was referred to by Messrs M. N. Dastur & Co. Pvt. Ltd., who, in the course of their data evaluation for the Project Report for the Salem Steel Plant observed inconsistent iron recoveries during beneficiation tests conducted on samples collected from different zones of the iron ore deposit. The iron recoveries differed with variations in the hematite content reported in the non-magnetic fraction during magnetic separation. It was, therefore, felt that determination of the extent of hematite formation in the ore body will be helpful in planning the ore concentration process and flowsheet for the Salem Steel Plant.

The mineral hematite being an oxidation product of magnetite and the oxidation being dependant upon the atmospheric oxygen, it should be most prominent on the surface of the ore body exposed to atmosphere and should progressively decrease with increasing ore depths. With this aspect in view, samples were collected at regular intervals and from various depths with respect to the ore band from an adit driven into the ore body. The behaviour of the ore in relation to its hematite content was then studied employing microscopic techniques and the results corroborated by chemical and X-ray spectrographic analyses.

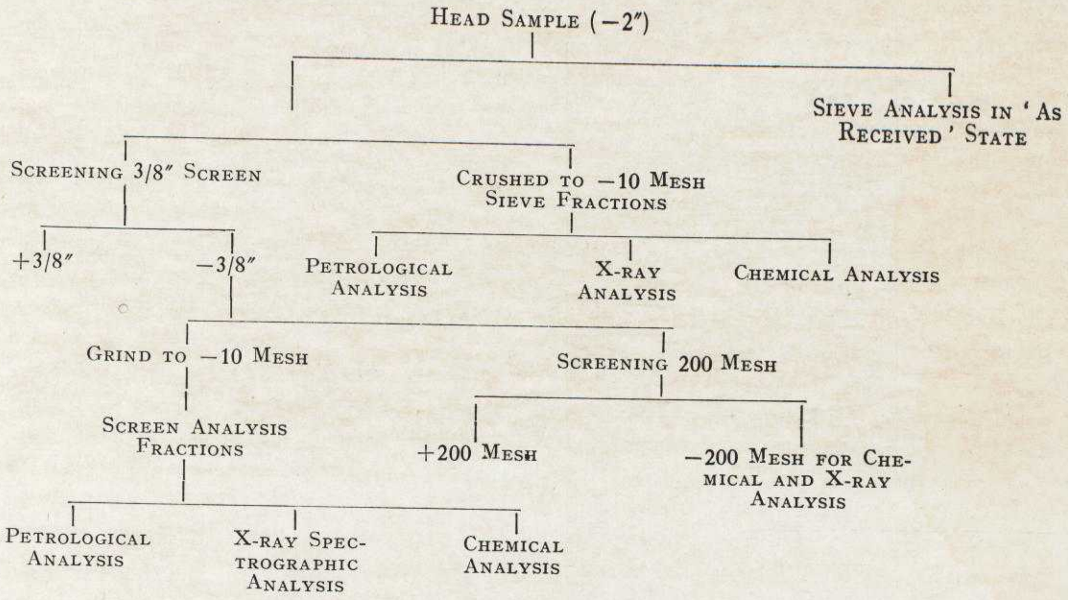
To find out the hematite distribution with respect to various positions in the ore body, it was decided to take up for detailed study the first four samples from P1 to P4 initially from the adit. The last sample P8 which represented the further face of the ore-body, in contact with the chloride-amphibole schist and expected to show mineralogical changes due to contact and regional metamorphism, was also taken up for detailed study after it had been observed that there was negligible oxidation after the first 20-25 metres from the first ore contact.

Each sample was prepared for further detailed study in the manner depicted in the flow-diagram (p. 27).

Petrological studies comprised quantitative estimation of hematite (free and interlocked), magnetite (unaltered and interlocked with hematite) and total gangue in each of the sieve fractions of the head sample as well as in the sieve fractions —3/8 in. (9·5 mm.) sample ground to —[illegible]0 mesh. The distribution of the total magnetite, hematite and gangue slicates in each sample was evaluated and the results were compared with the X-ray spectroscopic and chemical analysis of the head samples as well as the sieve fractions.

The main reason for estimating the hematite in the —3/8 in. (9·5 mm.) material in each sample was to assess the possibility of providing separate treatment facilities for this material (i.e. other than magnetic

Flow-diagram of Sampling



separation) if the hematite tended to concentrate in the fines.

Mineralogical examination and estimations were conducted using petrological and ore-microscopes. Individual sieve fractions and their polished mounts were examined in each case. Norelco's "Autrometer" (Automatic X-ray fluoroscence spectroscope) was used for the estimation of iron in the various samples. The instrumental analyses were supported by wet chemical analysis which also provided the FeO analyses for estimation of Fe_3O_4 in the head samples as well as in some of the sieve fractions.

The results of the comprehensive petrological study of adit samples, supported by X-ray spectrograph and chemical analyses, were summed up as follows :

(1) Hematite present in the ore was of secondary nature formed due to the oxidation of magnetite. This process is called "martitization". It is present along the peripheries and cleavage planes of the magnetite grains. No primary hematite was observed.

(2) Due to more favourable conditions of weathering, the samples from the first few metres (up to 20 metres) of the adit showed higher content of hematite as compared to the samples obtained from the interior of the ore body.

(3) Although the hematite content in the −3/8 in. fractions, of the head samples was more than that in the +3/8 in. fractions, the difference was not very marked. Provision for a separate beneficiation treatment of the −3/8 in. fraction of the ore was, therefore, not necessary.

(4) A study of the mineral contents as estimated from the sieve fractions of various samples crushed to −10 mesh, showed that most of the hematite present was associated with the parent magnetite. This hematite would, therefore, be recovered during magnetic separation and consequently high iron recoveries could be predicted during concentration by this process.

(5) In addition to the major iron minerals, i.e. magnetite and hematite, the iron bearing silicates were also present to variable extents in the ore body. The iron present in these silicates would not be recovered by magnetic separation at low intensities and would, therefore, result in some iron losses in the tailings.

(6) The sulphur contributed by the sulphides was negligible, but possibility of its increase could not be entirely eliminated, especially near the contact zone of the ore body with the amphibolites and chlorite schists.

(7) When compared to the hematite content of an earlier sample received for beneficiation tests in the National Metallurgical Laboratory from Neyveli (Salem Steel Project authorities), i.e. the representative slit sample and float ore sample, a downward gradation of hematite content from float ore (about 20 per cent) to slit sample (about 6 per cent) to P_1 (7 per cent) and P_2 to P_8 (1 per cent and below) was observed. The slit sample had almost the same hematite content as the P_1 sample because of the proximity of both of the samples to atmosphere. Higher content of hematite noticed in the float ore on the other hand was probably due to the larger surface area of magnetite available for oxidation. Efficient utilization of the deposit will, therefore, necessitate the estimation of the ore reserves in each category, so that by careful blending a consistent iron ore feed could be provided to the ore concentration plant.

2.0 Pilot Plant Studies on Beneficiation and Agglomeration of a Low-grade Limestone Sample from Purnapani Quarries, Orissa, for Hindustan Steel Ltd.

A 500-tonnes representative sample of limestone from Purnapani Quarries, Orissa, was received from the Hindustan Steel Ltd. for beneficiation and agglomeration tests on a pilot plant scale. The sample, as received, assayed, CaO, 37·1; SiO_2, 15·3; MgO, 6·5; Al_2O_3, 4·0; Fe_2O_3, 3·1; CO_2, 33·5 per cent.

The sample under investigation was different from the earlier samples from Purnapani, as the gangue in the present sample was more finely disseminated, and as such effective liberation of limestone could be achieved only at about 90 per cent −200 mesh as compared to 75 per cent −200 mesh in the sample investigated earlier.

The beneficiation technique employed consisted of grinding the ore to 90 per cent −200 mesh and separating limestone from quartz by froth flotation using 0·68 −0·77 kg./ton sodium oleate as collector for limestone and 0·46-0·23 kg./ton sodium silicate as depressant for quartz. The flotation circuit included roughing, scavenging, cleaning and recleaning stages so as to produce a concentrate containing 4·5 per cent insolubles.

The flowsheet employed for upgrading Purnapani limestone is given in Fig. 10. This flowsheet which incorporates a bank of scavenger flotation cells, a hydroclassifier and two banks of cleaner flotation cells, has been preferred for the following reasons:

(i) The scavenger circuit was primarily used for floating the interlocked particles which required further grinding.

(ii) For better efficiency, grinding should preferably be done at about 60 per cent solids. As the middlings contained 15 per cent solids, comprising mostly interlocked particles, these had to be sent back to the ball mill for further grinding along with the fresh feed. The middlings had to be thickened, preferably in a thickener or in a hydroclassifier before recycling them through the ball mill.

(iii) As the gangue was finely disseminated, it was difficult to get a high grade concentrate (< 3 per cent insolubles) with good recovery. So two cleanings of the rougher concentrate were performed.

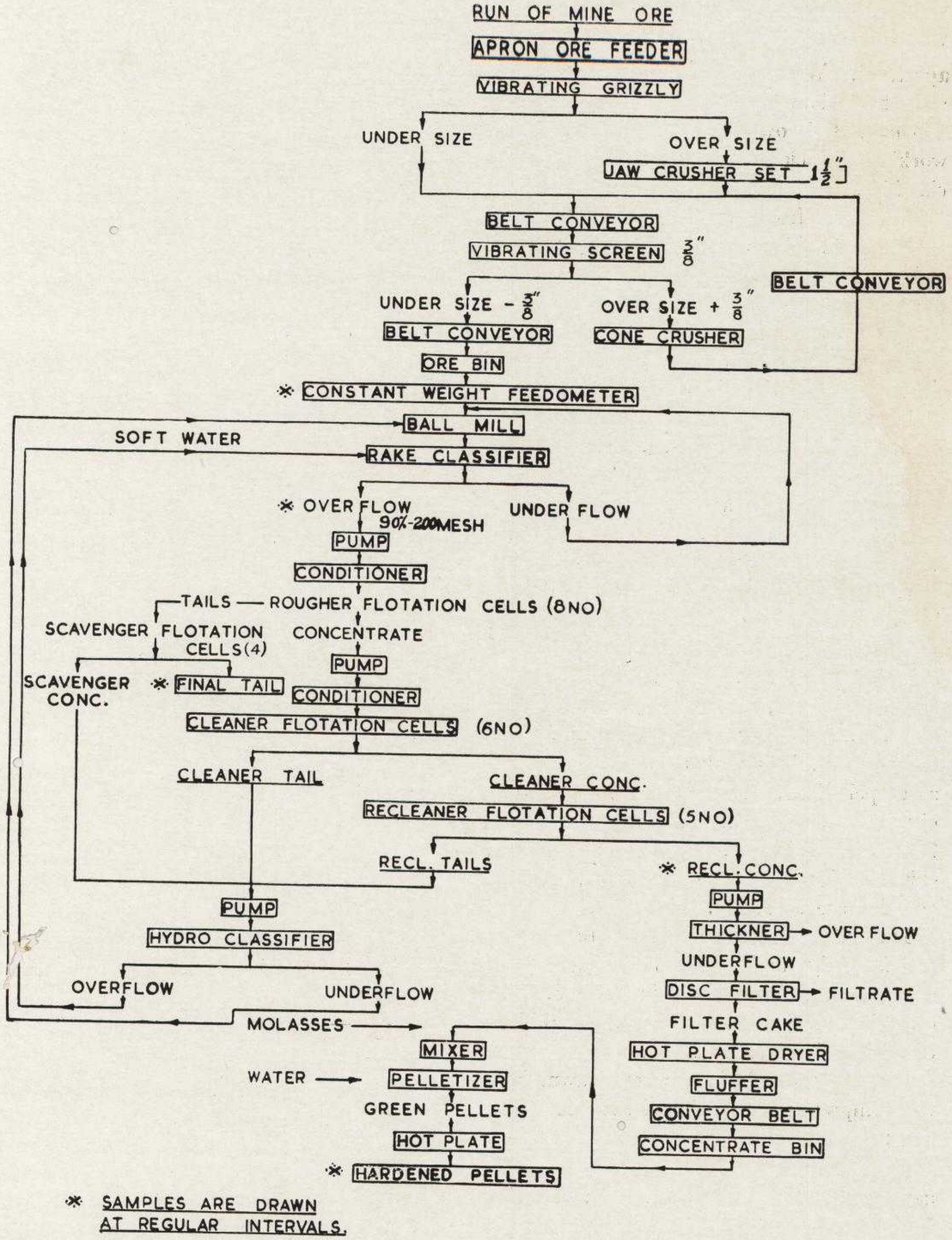


Fig. 10 — Flowsheet for Producing Purnapani Limestone Pellets

The run-of-mine ore was crushed to about 38 mm. size in a jaw crusher and then reduced to about 10 mm. in a cone crusher operating in closed circuit with a vibrating screen (3/8 in. or about 9·4 mm. aperture). With the aid of a constant weight feedometer, the ore was fed at a constant known rate to the ball mill working in closed circuit with a rake classifier.

However, from the actual operation, the rake classifier was found unsuitable for sizing the ball mill discharge containing 85 per cent —200 mesh. Hydrocyclone is the proper equipment for sizing such finely ground solids. The classifier overflow was conditioned with sodium oleate and sodium silicate and the limestone was floated off from the gangue. The concentrate, after two cleanings, was sent to a thickener for dewatering, whereas, the tailings were discarded. Reagent additions were made at different points in the circuit, depending on the quality of froth based on the microscopic examination of the products, obtained during the operation of the pilot plant run. The thickener underflow was filtered, the filter cake after drying to about 7 per cent moisture, was thoroughly mixed with 6 per cent molasses and pelletized. The green pellets thus obtained were heat hardened at about 250°C. for making strong, hardened pellets, suitable for handling, transport, storage and for use as steel-making flux.

The pilot plant was found to operate satisfactorily under the following set of operating conditions given in Table 3. The quantitative flowsheet, under the stabilized conditions is given in Fig. 11.

Following conclusions were drawn from the comprehensive results of the pilot plant run :

(i) Best separation of limestone from the gangue minerals was achieved by flotation from a pulp containing not over 22 per cent solids, 90 per cent of which should pass through 200 mesh.

Table 3 — Operating Data

(a) Feed Rate: 300 to 350 Kg/hr.
(b) *Pulp densities*:
Solids in the ball mill discharge: 44-46 per cent.
solids in the feed for rougher flotation: 20-22 per cent.
solids in the feed for cleaner flotation: 15-17 per cent.
solids in the feed for recleaner flotation: 15-17 per cent.
solids in the primary tails: 5-9 per cent.
solids in the hydroclassifier underflow: 30-35 per cent.
solids in the hydroclassifier overflow: <5 per cent.
Grind: 90 per cent —200 mesh.

(ii) About 0·70-0·77 kg./ton sodium oleate and 0·23 kg./ton sodium silicate were required for achieving the best separation.

(iii) About 86·3 per cent of the total insolubles could be rejected in the tailing, producing a concentrate, constituting 68·6 per cent by weight of the original sample and assaying 4·10 per cent insolubles with an overall recovery of CaO, 87·3 per cent.

(iv) A better grade of concentrate, containing 3·53 per cent insolubles could, however, be obtained at low feed rates, but with a lower recovery of only CaO, 78·4 per cent.

(v) The recovery of CaO could be increased to about 92·3 per cent by employing slightly higher feed rates. But the concentrate assayed 4·56 per cent insolubles.

(vi) The upgraded concentrate was subsequently pelletized using 6 per cent molasses as binder and the hardened pellets, assayed CaO, 44·6 per cent; SiO_2, 3·4 per cent.

2.1 Pilot Plant Studies on Beneficiation of Limestone Samples from Nandini Mines of Bhilai Steel Plant, Hindustan Steel Ltd.

Four limestone samples, viz. (i) run of mine sample, (ii) —1 in. (—25·4 mm.) fines from the old dumps, (iii) currently produced fines and (iv) open hearth flux

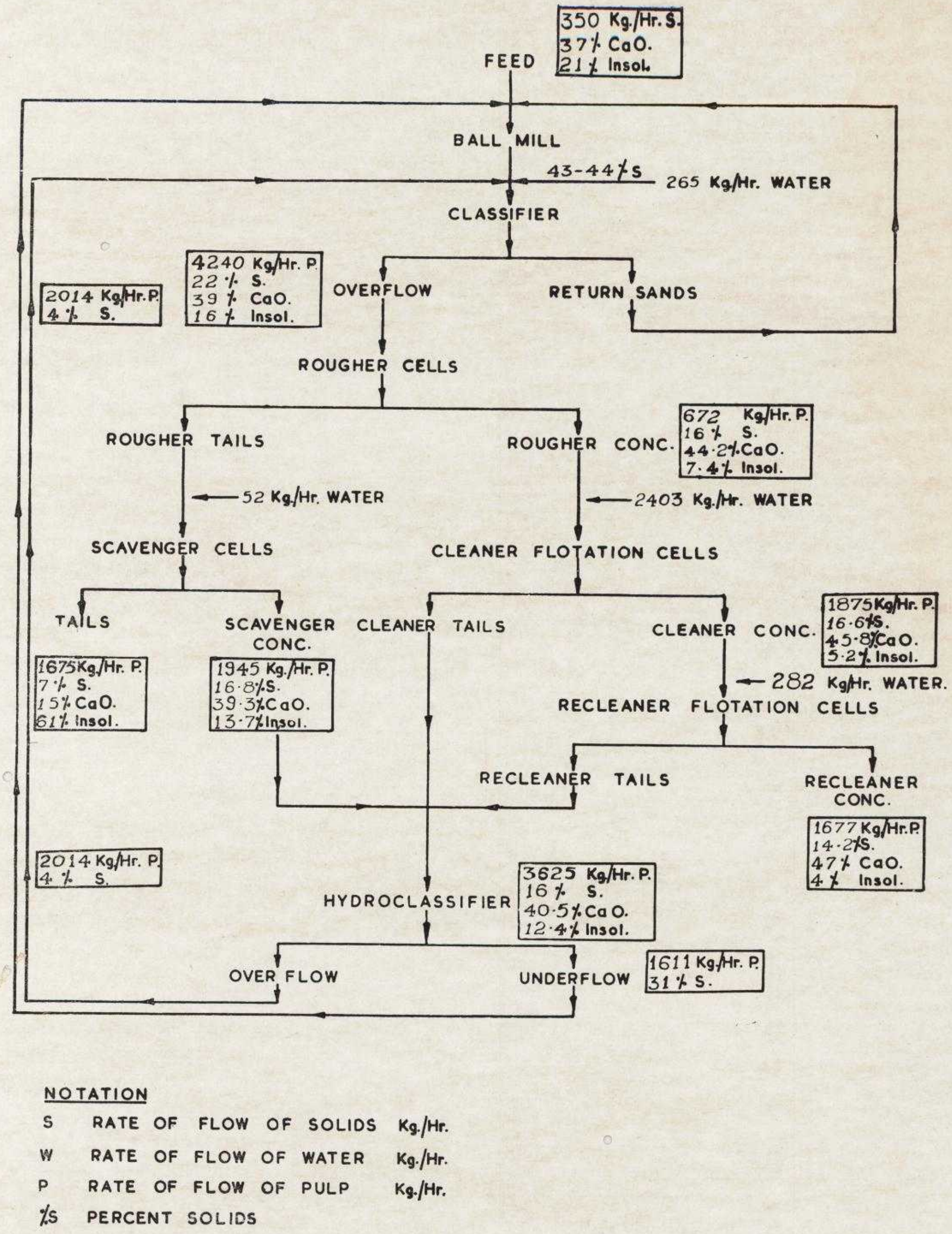


Fig. 11 — Quantitative Flowsheet for Floating Purnapani Limestone

grade limestone, were received from the Nandini Mines of Bhilai Steel Plant. The first three samples were for beneficiation studies to determine suitable processes for removal of clayey matter and ferruginous murrum balls present in limestone. Separation of quartz, intimately associated with limestone, was not required to be attempted.

Samples (i), (ii) and (iii) essentially consisted of crystalline calcite (dolomitic) with quartz in intimate association as the major gangue. A good amount of clayey material in the form of tenacious clay balls were also present. As received, the three samples (i), (ii) and (iii) assayed (i) CaO, 40·7; MgO, 9; Insol., 10·1 per cent (ii) CaO, 36·75; MgO, 8·4; Insol., 12·1 per cent (iii) CaO, 35·82; MgO, 8·1; Insol., 12·25 per cent respectively.

Screenability studies performed with different moisture contents in the samples indicated that screening was least efficient when the moisture contents of the three samples were 5, 7·5 and 7·5 per cent in the case of sample (i), (ii) and (iii) respectively. Crushing and screening flowsheet is furnished in Fig. 12.

Wet screening or scrubbing followed by wet screening of the three samples, could eliminate in the form of slime, most of the clay present. The washed lump fractions namely +1/2 in. (+12·7 mm.) fraction from the three samples were of improved quality, low in insoluble contents, assaying (i) CaO, 41-52; MgO, 7·8; Insol., 7·5 per cent. (ii) CaO, 40·05; MgO, 9·2; Insol., 7·94 per cent. (iii) CaO, 39·97; MgO, 8·65; Insol., 8·4 per cent respectively. Because of their fineness, murrum balls were also not present in this washed coarse fraction. The −1/2 in. (−12·7 mm.) fractions were of poorer grades and also contained most of the ferruginous gangue (murrum balls) present in the sample. Murrum balls being hard, could not be eliminated by scrubbing and washing. Jigging of −1/2 in. (−12·7 mm.) washed fraction could, however, eliminate most of the murrum balls in the heavier fraction. The lighter fraction almost free of murrum balls, assayed CaO, 37·52; insol., 12·34; Fe_2O_3, 1·98 per cent. Flowsheet employed for washing and jigging is given in Fig. 13. Results obtained by scrubbing and wet screening were slightly better than those obtained by wet screening alone. Evidently, this was due to better scrubbing and loosening of clayey coating on limestone lumps as well as breaking up of the clay balls during the process.

2.2 Further Pilot Plant Beneficiation Studies on Limestone from Tisco

A 700 tonnes sample of low grade limestone assaying CaO, 45·9 and SiO_2, 7·1 per cent was received from Tata Iron & Steel Co., Jamshedpur, for upgrading and agglomeration studies on a pilot plant scale and for supplying pellets for trials in the steel furnaces.

By grinding the ore to 80 per cent −200 mesh, and froth-flotation of the limestone using sodium oleate (0·73 kg./ton) and sodium silicate (1·25 kg./ton) about 75 per cent of the entire insolubles could be rejected producing a concentrate constituting 88 per cent by wt. of the original and assaying CaO, 50·3 and SiO_2, 2 per cent with an overall recovery of CaO, 94-95 per cent. The day-to-day results of the pilot plant studies indicated a maximum variation of about 0·5 per cent in the assay of final concentrate. The test results also indicated that the best separation of insolubles was achieved from a pulp containing not over 22 per cent solids, 80 per cent of which pass through 200 mesh. Small additions of pine oil, (0·0227 kg./ton) was found to cut down the collector consumption from 0·73 kg./ton to 0·41 kg./ton for upgrading limestone.

Drop tests with green pellets and crushing load tests on heat-hardened pellets indicated that strong pellets assaying CaO, 47·84 and SiO_2, 1·91 per cent could be obtained from the concentrate containing

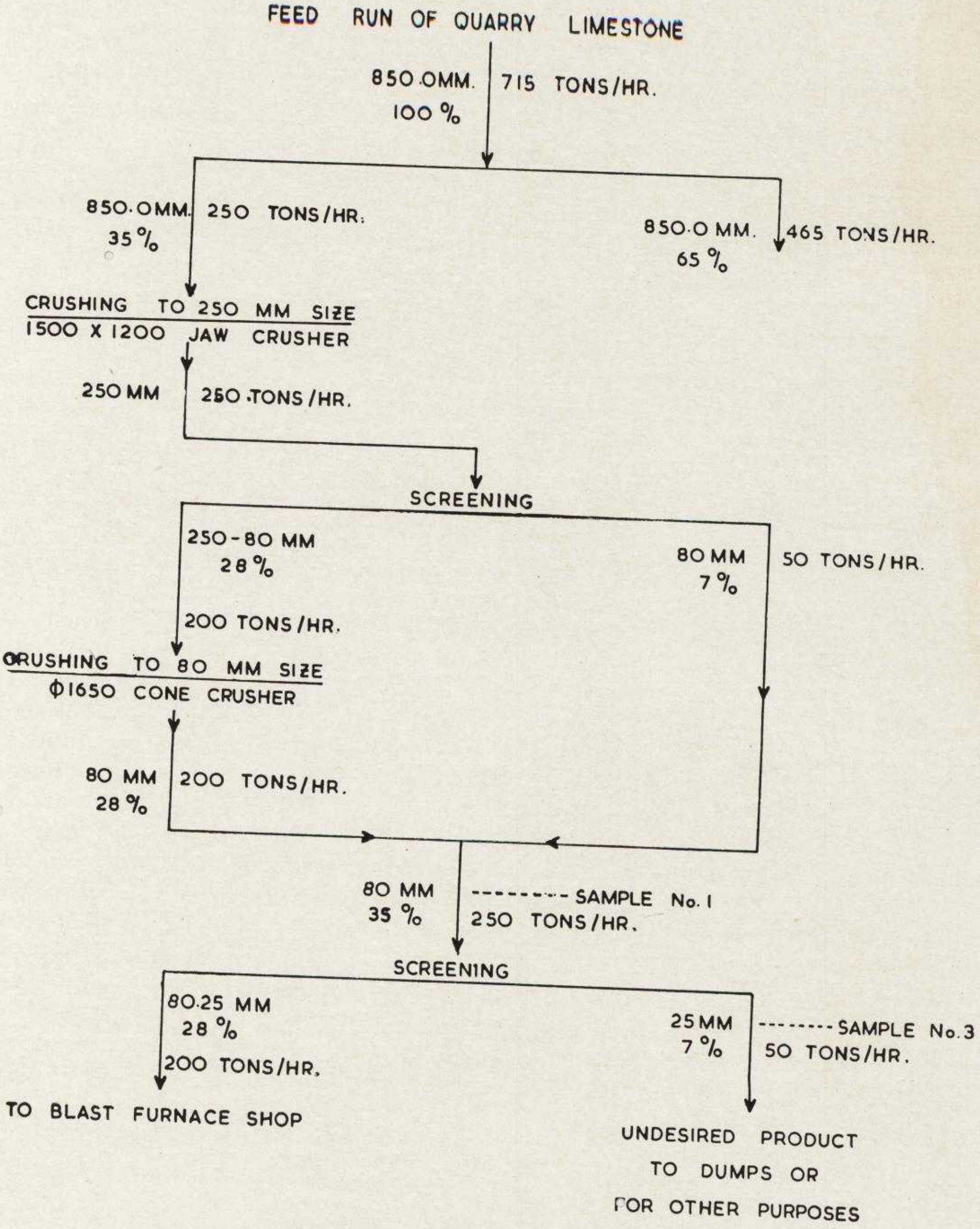


Fig. 12 — Crushing and Screening Flowsheet of Nandini Limestone

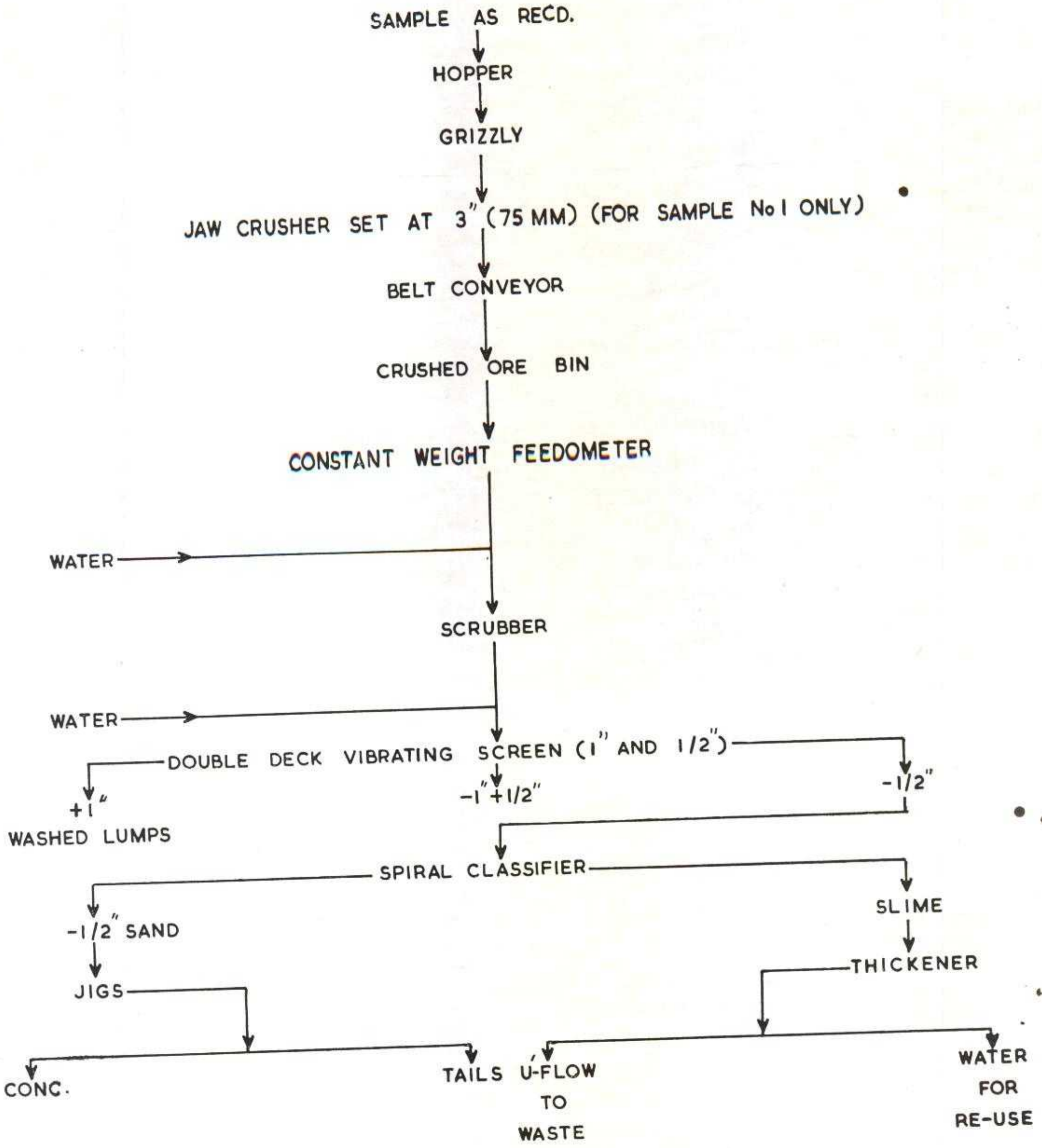


Fig. 13 — Flowsheet Employed in NML for Washing and Jigging the Limestone Sample from Nandini Mines

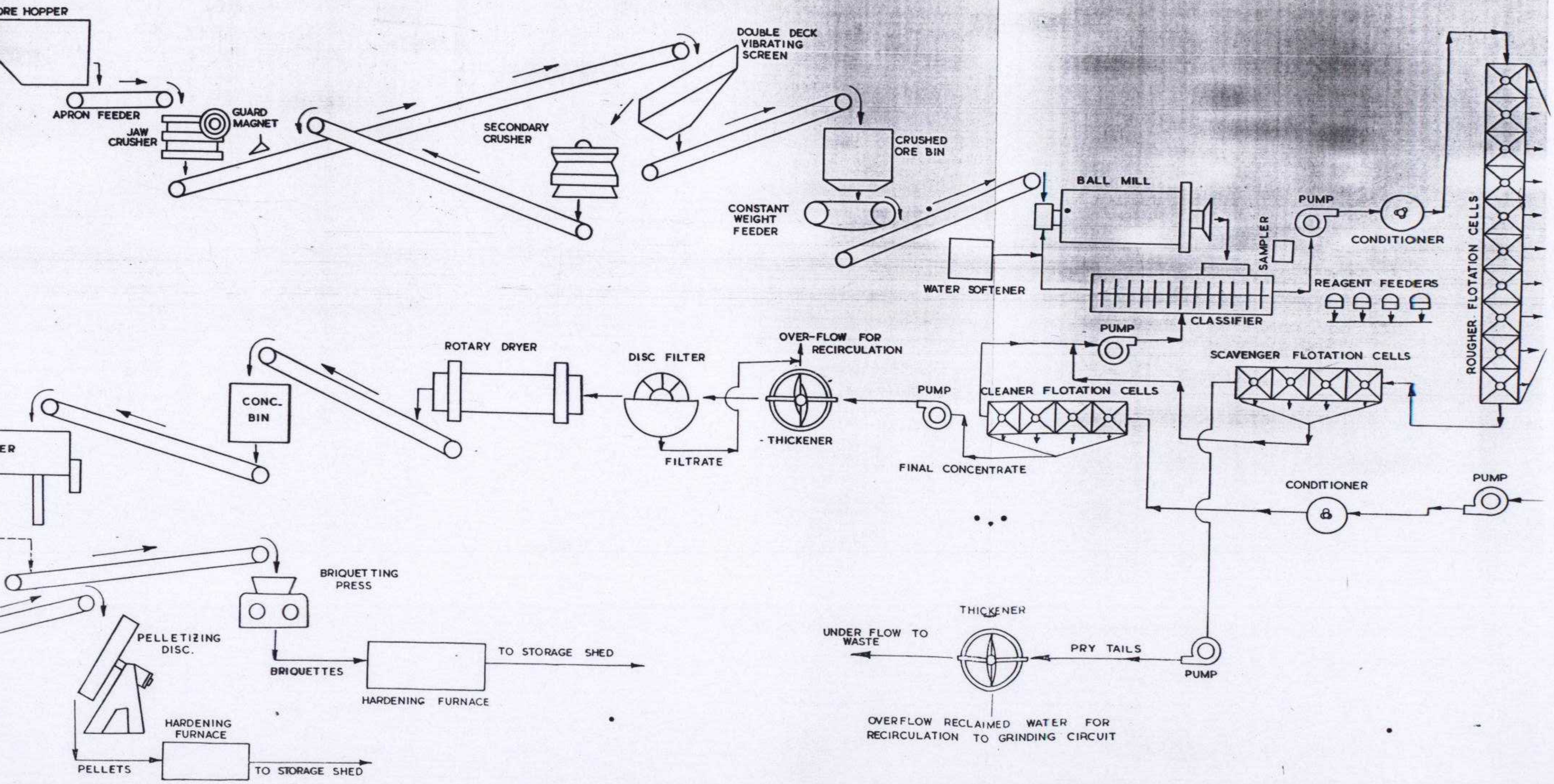


Fig. 14 — Diagrammatic Flowsheet for the Beneficiation of Low Grade Limestone

not over 8·5 per cent moisture and employing 6 per cent molasses as binder.

Based on these pilot plant studies at the National Metallurgical Laboratory flowsheets for setting up a commercial plant, for upgrading 300 tons/day of the limestone are given in Figs. 14 and 15.

2.3 Reduction of Silica Content in a Limestone Sample from Rajpipla, Narmada Project

A sample of low grade limestone from Rajpipla, Narmada Project, Gujarat State, assaying CaO, 36·14; CO_2, 31·52; MgO, 3·20; SiO_2, 17·10; Al_2O_3, 7·31; SO_3, 0·73; Fe_2O_3, 2·98; S, 0·29 and P, 0·03 per cent was received from the Assistant Engineer, Narmada Project, investigation Sub-Division, Rajpipla, for flotation studies to make it suitable for cement production by reducing its silica content.

The net weight of the sample received was 6·30 quintal and it consisted mostly of lumps of 8-1 in. and was free from fines. The gangue in the sample consisted mainly of quartz with a little of feldspar, and micas. The grey coloured fine grained compact carbonates were present in the form of crypto-crystalline material. The gangue and the carbonates were finely disseminated and, therefore, a very fine grinding was necessary for flotation.

Only froth flotation was investigated to separate the gangue from calcite. The effect of feed size, depressant for quartz and collector additions were studied. The optimum grind was found to be of 50-60 minutes grind (finer than —325 mesh). Using 0-90 kg./ton of oleic acid a refloat concentrate would be obtained assaying 12·83 per cent insoluble which constituted 45·2 per cent by weight with a feed employing a 50 minutes grind whereas a conc. of 12·33 per cent insoluble with a weight per cent of 59·5 per cent was obtained with a feed of 60 minutes grind. If 16 per cent insolubles could be tolerated in the limestone for cement manufacture, then the expected yields would be 63 and 74 per cent by employing 50 and 60 minutes grind respectively.

The laboratory scale investigations indicated the technical feasibility of upgrading the low grade limestone from Rajpipla, Narmada Project, Gujarat State for its use in cement manufacture.

3.0 Beneficiation of a Low-grade Manganese Ore from Barajamda Area, Singhbhum District, Bihar

A low grade manganese ore assaying Mn, 30·56; MnO_2, 44·46; Fe, 10·22; SiO_2, 28·32; Al_2O_3, 5·6; P, 0·039 and BaO, 0·4 per cent from Barajamda area, Singhbhum District, was received from Messrs Kharswan Mineral Concern (P) Ltd. for beneficiation studies. Pyrolusite and psilomelane were the chief manganese minerals and quartz and goethite were the principal gangue minerals followed by ochery and clayey material. Most gangue minerals were freed at —35 mesh size. Straight tabling as well as magnetic separation of the sample after grinding of —35 mesh and desliming could reduce the silica content to 6 and 5 per cent respectively but the iron contents were high at 9·2 and 12·74 per cent. Due to high iron content in the sample, the concentrates were low in manganese.

Reduction roast followed by low and high intensity magnetic separation of the reduced product at —35 mesh could reduce the iron content to any desired level, but the silica content in the manganese concentrate was high. Reduction roast followed by tabling at —35 mesh and magnetic separation of the table concentrate yielded a final concentrate assaying Mn, 61·2; Fe, 4·62; SiO_2, 9·5; Al_2O_3, 2·74 and

P, 0·04 per cent with a recovery of Mn. 55·1 per cent. The recovery is expected to improve further if a Mn/Fe ratio of only 7 is aimed at in the concentrate. The concentrate can be used for the production of standard grade ferro-manganese after agglomeration. Reduction roast followed by magnetic separation and cationic flotation of the siliceous gangue after grinding to −65 mesh using starch and Armeen 12D, at a *p*H of 10 produced a concentrate assaying Mn, 53·95; Fe, 7·7 and SiO_2, 7·9 per cent with a recovery of Mn, 63·7 per cent. The chemical composition of the concentrate fully satisfied the specifications for the production of standard grade ferro-manganese.

4.0 Beneficiation of Gypsum Sample from Messrs Bikaner Gypsums Ltd., Rajasthan

A low grade gypsum sample assaying SO_3, 36·06; CaO, 28·9; CO_2, 2·95 and 8·22 per cent was received from Messrs Bikaner Gypsums Ltd., Rajasthan for production of a concentrate suitable for the production of plaster of Paris. Quartz and calcite were the principal gangue minerals followed by chlorite and glauconite. Quartz was liberated at −65 mesh but calcite and glauconite were interlocked with gypsum even at −200 mesh size.

Flotation test performed after grinding the sample to −150 mesh (80·7 percent −200 mesh) yielded a concentrate assaying SO_3, 46·3 (99·3 per cent gypsum) with a recovery of only SO_3, 43·4 per cent. However, most of the gypsum lost in the cleaner tailings may be expected to be recovered by recirculation in actual plant practice. Presence of calcite in the sample necessitated a large number of cleanings thereby lowering the gypsum recovery in the final concentrate. Addition of sodium hydroxide was necessary for good selectivity of gypsum.

4.1 Pilot Plant Studies on a Gypsum Sample from Messrs Bikaner Gypsums Ltd., Rajasthan

Two samples of lowgrade gypsum were received from Messrs Bikaner Gypsums Ltd., Rajasthan for pilot plant flotation tests for producing concentrates suitable for making plaster of Paris. Sample No. 1 assayed SO_3, 36·12 per cent and the principal gangue minerals in it were quartz and calcite followed by chlorite and glauconite. Quartz was liberated at −65 mesh size but calcite and glauconite were interlocked with gypsum even at −200 mesh size. A continuous flotation run was made with about 2 tonnes of the sample after grinding to 80 per cent −200 mesh as indicated in the earlier batch tests and recirculating the refloat tailings; it was found that the concentrate obtained after three cleanings assayed SO_3, 44·4 only (equivalent to 95·5 per cent gypsum) with a recovery of SO_3, 84·7 per cent.

One more cleaning of the above concentrate yielded a final concentrate assaying SO_3, 45·2 per cent (97·2 per cent gypsum) with a recovery of SO_3, 33·6 per cent. Further attempts to improve upon this grade resulted in a product assaying SO_3, 45·5 per cent (97·8 per cent gypsum) with a much lower SO_3 recovery. The pilot plant investigations at the National Metallurgical Laboratory indicated that due to the high calcite content of the sample, it was not possible to produce as high a grade of gypsum concentrate as indicated by batch tests, due to recirculation of the middling products, which is usually not done during batch tests. The concentrate of grade 97·2 per cent gypsum obtained after 4 cleanings was sent to Messrs Bikaner Gypsums Ltd., for marketting trials after calcining. The final concentrate after drying was found to be slightly off-coloured perhaps due to iron contamination during grinding, but was dark grey when wet. The flowsheet of pilot plant test of sample 1 is given in Fig. 16.

The calcite content in sample No. 2, also received from the same source, was even higher than in the first sample. Production of plaster of Paris grade of concentrate from this samples was, therefore, not attempted due to its high calcite content.

5.0 Pilot Plant Studies on Concentration of Economic Minerals from Manavalakurichi Beach Sand, Madras

Two flowsheets were recommended, and are given in Figs. 17 and 18. The flowsheets differ in the initial stage separation only; essentially flowsheet 'A' employs an initial low intensity magnetic separation by X-belt separators followed by a high intensity magnetic separation of the non-magnetics in the same type machine. The second flowsheet, 'B', on the other hand, employs a high intensity induced roll separator in the 1st stage, followed by subsequent separation similar to the flowsheet 'A'. The details of the 2 flowsheets are given below.

Flowsheet 'A'

The sample was screened through a 28 mesh screen (+28 mesh fraction rejected) and subjected to low intensity magnetic separation (abbreviated as LIMS) using cross belt lift type machine, giving 22 per cent wt. as clean ilmenite in the 1st stage, 24 per cent wt. as ilmenite with garnet in the 2nd stage, and rest as non-magnetic product. The 2nd stage magnetic fraction containing 87 per cent ilmenite and 13 per cent garnet was separated by H.T. separation, giving clean ilmenite and garnet.

The non-magnetic fraction from LIMS was about 53 per cent wt. It was subjected to high intensity magnetic separation (HIMS) using induced roll separators in 3 or 4 stages, to take out the remaining ilmenite and garnet along with leucoxene, monazite and (ferruginous) part of magnetic rutile as magnetic product. The non- magnetic product which was about 20 per cent of original feed after this stage, was almost free of ilmenite, monazite and garnet. The HIMS magnetic product was further cleaned by H.T. separation giving 20 per cent wt. as conducting fraction consisting of clean ilmenite and leucoxene (having < 0·15 per cent monazite). If contamination of leucoxene or magnetic rutile was objectionable in the saleable ilmenite, then the H.T. separation could be done for the 3rd and/or 4th stage HIMS magnetic fraction separately. The conducting fraction from this stage contains the bulk of the leucoxene and magnetic rutile. The non-conducting product from H.T. separation HIMS magnetic fraction contains almost all monazite with residual garnet and leucoxene and fine ilmenite. This rough monazite concentrate was further cleaned by cross belt magnetic separator and wet gravity separation to produce clean monazite (±10 per cent wt. % of 96-97 per cent purity).

The non-magnetic fraction obtained after HIMS was about 20 per cent of the feed sand and consisted of zircon, rutile, quartz and sillimanite, etc. It was then subjected to wet gravity concentration by Wilfley table using fresh water. This also cleaned the saline coatings on the grains. The gravity concentrate (about 15 per cent wt. 16-18 per cent rutile content) was subjected to H.T. roughing separation in one or 2 stages with closed circuit. This gave a zircon concentrate and a rough rutile concentrate. The rough rutile product was screened into 3 fractions, +48 mesh, −48+65 mesh and −65 mesh size. The −48+65 and −65 mesh size fractions were subjected to H.T. separation in 2 or 3 stages in closed circuit, producing clean rutile. The zircon rich middlings were recirculated to earlier stages.

Sillimanite may be produced from the last fraction, i.e. table tailings, by flotation. Sillimanite contaminating zircon in the

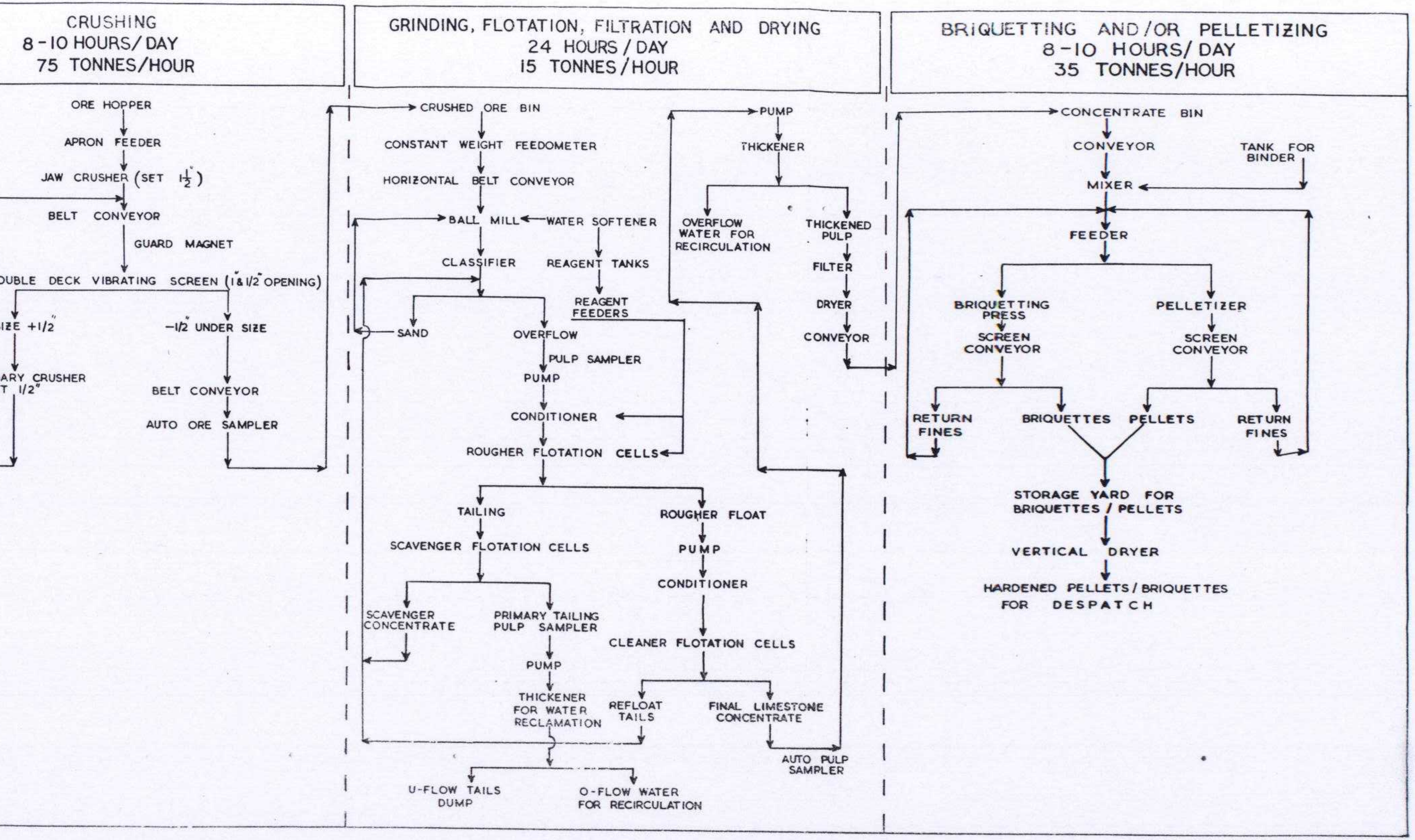


Fig. 15 — Schematic Flowsheet for the Beneficiation of Low-grade Limestone — Capacity 300 tonnes of Raw Materials per Day

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The non-magnetic fraction obtained after HIMS was about 20 per cent of the feed sand and consisted of zircon, rutile, quartz and sillimanite, etc. It was then subjected to wet gravity concentration by Wilfley table using fresh water. This also cleaned the saline coatings on the grains. The gravity concentrate (about 15 per cent wt. 16-18 per cent rutile content) was subjected to H.T. roughing separation in one or 2 stages with closed circuit. This gave a zircon concentrate and a rough rutile concentrate. The rough rutile product was screened into 3 fractions, +48 mesh, −48+65 mesh and −65 mesh size. The −48+65 and −65 mesh size fractions were subjected to H.T. separation in 2 or 3 stages in closed circuit, producing clean rutile. The zircon rich middlings were recirculated to earlier stages.

Sillimanite may be produced from the last fraction, i.e. table tailings, by flotation. Sillimanite contaminating zircon in the

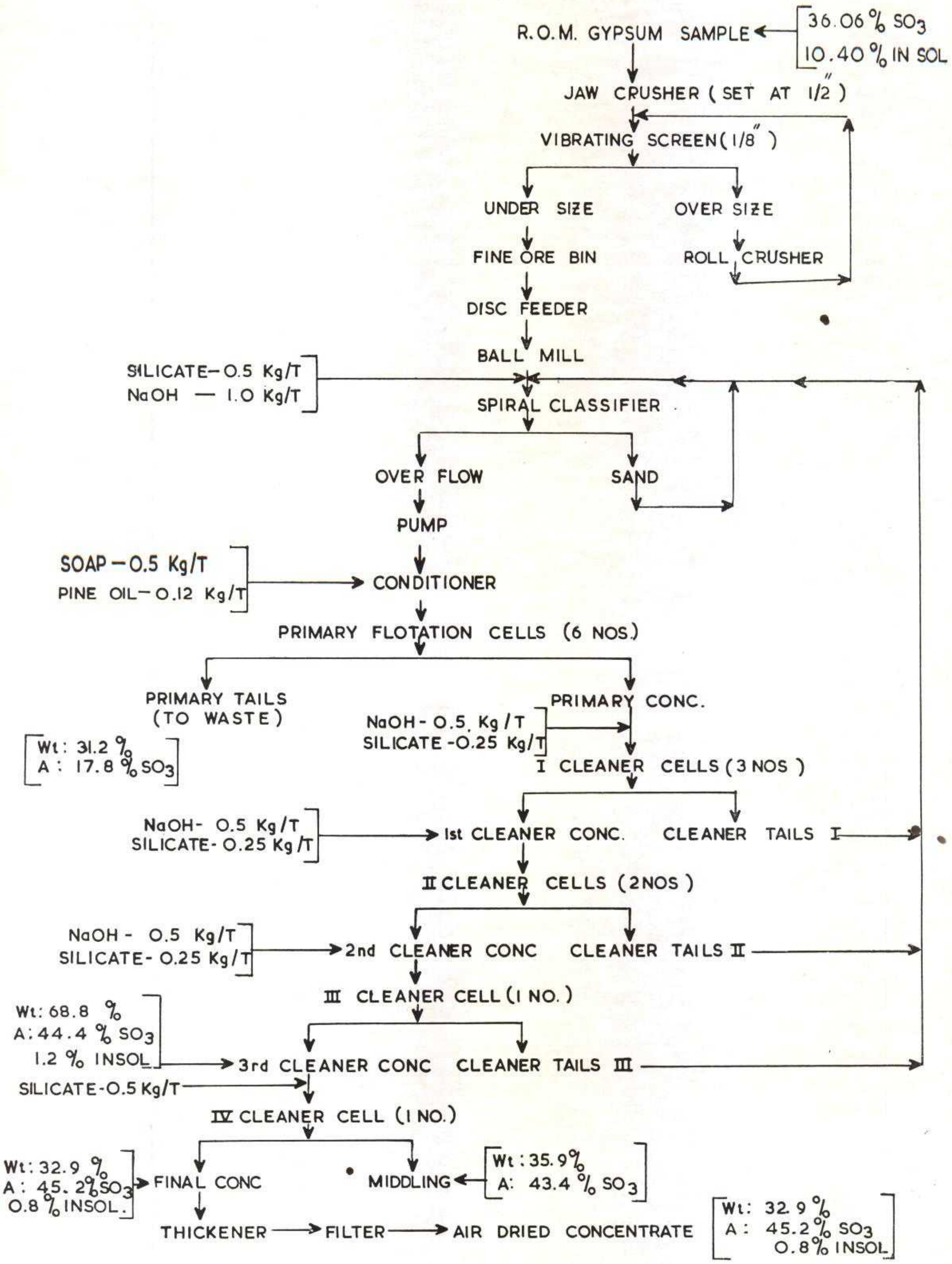


FIG. 16 — FLOWSHEET OF PILOT PLANT TEST FOR GYPSUM SAMPLE No. 1

H.T. non-conducting fraction may be separated by a further gravity cleaning of zircon by wet or dry pneumatic table.

Flowsheet 'B'

According to the second flowsheet, the —28 mesh sand was first subjected to HIMS in induced roll separators in 3 or 4 stages of increasing intensities for taking out all magnetic minerals in one group consisting of about 80 per cent of the bulk weight. The magnetic fraction was then subjected to H.T. separation giving (i) conducting fraction (63 per cent wt.) as clean ilmenite (ii) middling which was recirculated to the fresh feed and (iii) non-conducting fraction consisting of about 17 per cent by weight. The non-conducting fraction contained all the monazite, along with garnet and some leucoxene, ilmenite, etc. In the H.T. separation, some ilmenite and leucoxene were allowed to escape in the non-conducting fraction, so as to ensure a conducting fraction of clean ilmenite (to be free of monazite below $<$ 0·15 per cent).

The non-conducting fraction was then passed through a cross-belt type separator at high excitation (6-8 amp. exciting current while 3·8-4 amp. was needed for ilmenite separation) to remove the garnet and ilmenite as a magnetic product. Garnet was separated from this magnetic product from cross belt by H.T. separation.

Further steps for cleaning of monazite rough concentrate and for treatment of HIMS non-magnetic fraction for production of rutile and zircon were similar to the corresponding stages in flowsheet 'A'. In cases where presence of leucoxene or magnetic rutile may be objectionable, the HIMS magnetic fractions may be collected in 2 groups of corresponding lower and higher intensity magnetic intensities (66 per cent, as magnetics) in 1st and 2nd rolls, and 14 per cent as magnetic by 3rd and 4th roll.. The conducting fraction from 1st group would be the saleable ilmenite product.

6.0 Flotation Studies on Copper Ore Samples from the Khetri Copper Project of National Mineral Development Corporation

Two samples of low-grade copper ore were sent by National Mineral Development Corporation, from (i) Ghatiwalli adit and (ii) from Shaft No. 3, both the samples being from the Khetri mines. Flotation studies were performed to determine the optimum conditions for production of concentrate suitable for the flash smelting process for extraction of copper.

(i) *Sample of Copper Ore from Ghatiwalli Adit*

This sample received from NMDC analysed Cu, 0·99; Fe, 23·1; S, 6·08; SiO_2, 42·38; Al_2O_3, 11·4; MgO, 5·96 per cent, 0·14 dwt./ton Au and 2·26 dwt./ton Ag. It consisted of pyrrhotite, magnetite, chalcopyrite, pyrite and traces of chalcocite. The bulk of the ore was composed of siliceous gangue. Chalcopyrite was mostly free below 150 mesh size.

Optimum conditions were determined for maximum recovery of copper value, using 0·1 lb./T. of potassium ethyl xanthate as collector and 0·074 lb./T. of pine oil as frother during roughing at a *p*H of 7·6.

Rougher flotation at natural *p*H followed by three cleanings of the bulk rougher concentrate at 9·5 *p*H yielded a concentrate assaying Cu, 20·46; Fe, 33·3; S, 30·1; 2·0 dwt./T. Au and 23·6 dwt./T Ag with a recovery of Cu, 82 per cent. The grade of concentrate improved to Cu, 29·72; Fe, 31·1; S, 33·4 per cent 2·2 dwt./T Au and 27·8 dwt./T Ag with a copper recovery of 80·2 per cent, by grinding the bulk rougher concentrate to —200 mesh before cleaning at 9·5 *p*H.

(ii) *Sample from Shaft No.* 3

The sample assayed Cu, 0·79; Fe, 20·16; S, 2·85; SiO_2, 52·82; Al_2O_3, 10·2; Mg, 0·014

APPENDIX – I

RAW SAND	
ILMENITE & LEUCOXENE	66·6
RUTILE	1·8
ZIRCON	10·8
MONAZITE	10·7
GARNET	4·2
QUARTZ & SILLIMANITE	4·5
SHELLS Etc.	0·6

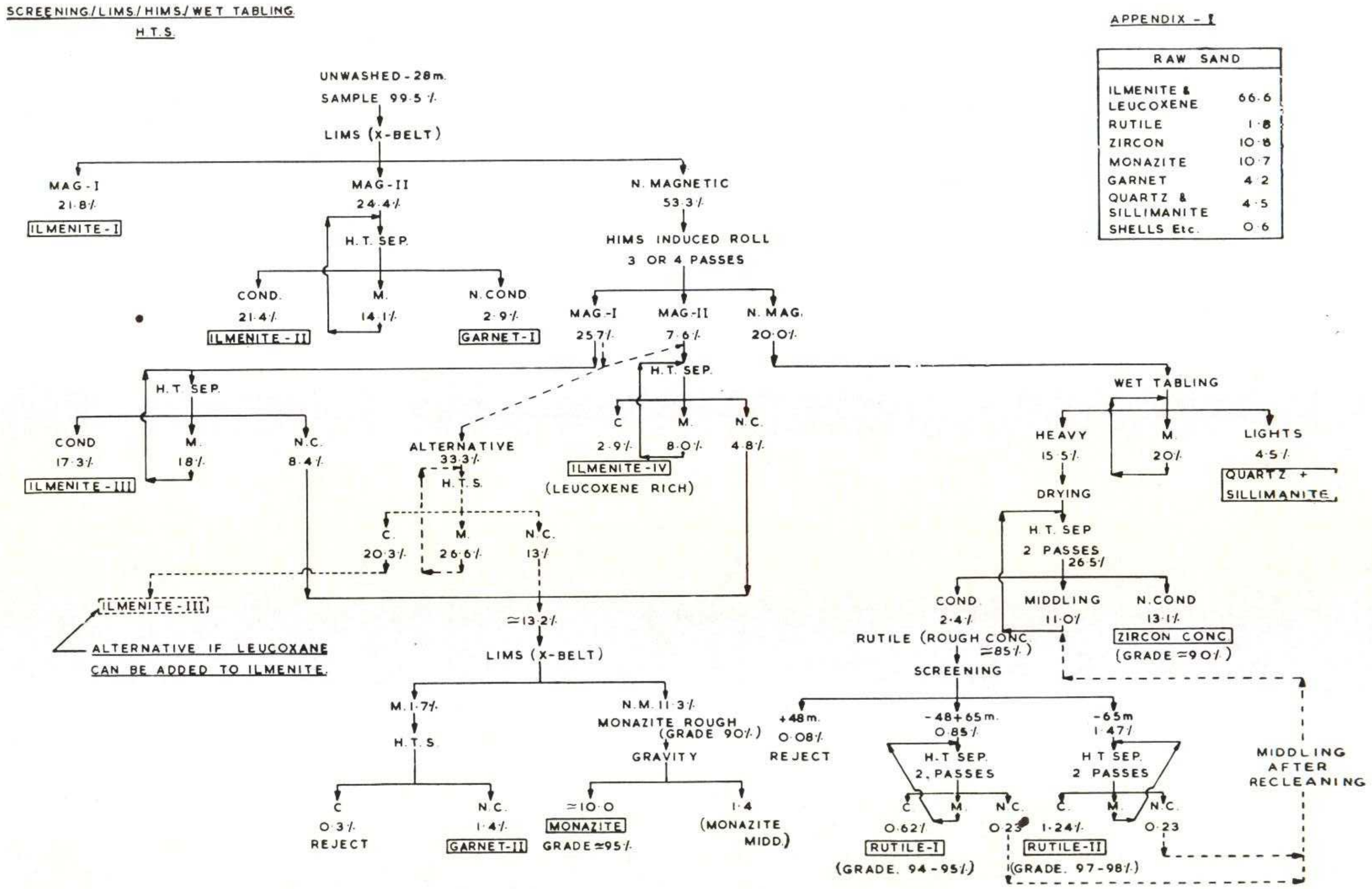


FIG. 17 — MANAVALAKURICHI BEACH WASHING — FLOWSHEET 'A'

APPENDIX - II

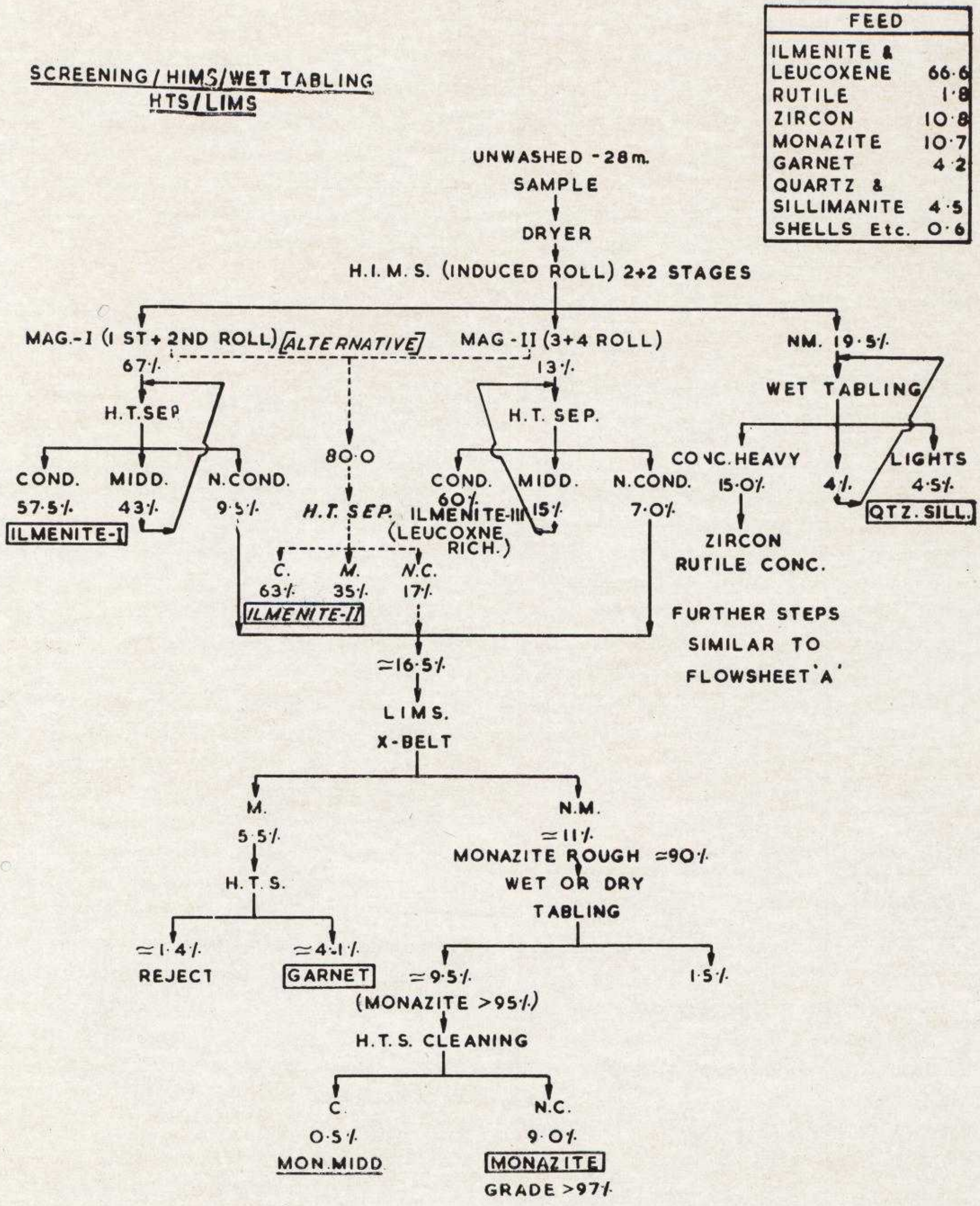


FIG. 18 — MANAVALAKURICHI BEACH WASHING — FLOWSHEET 'B'

per cent, Ca, trace, Au, trace, and 1·65 dwt./T ag. Petrological examination showed that magnetite was the most abundant metallic mineral followed by chalcopyrite, pyrrhotite and pyrite. The country rock consisted chiefly of quartz-chlorite, chalcopyrite was mostly associated with the siliceous gangue. The non-metallic minerals in the order of abundance were quartz, chlorite, ferro-magnesium minerals and biotite. Liberation of chalcopyrite was fairly good below −150 mesh.

6.1 Flotation Studies on Copper Ore from Rakha Mines, Bihar

A copper ore samples assaying Cu, 2·73; Fe, 5·54; S, 3·96; SiO_2, 73·08; Al_2O_3, 10·14; MgO, 0·6; CaO, 1·00; Ni, 0·072 and Mo, 0·02 per cent from Rakha mines, was received from N.M.D.C. for beneficiation studies. Preliminary crushing, grinding sieve analyses of −10 mesh crushed sample were completed. Flotation tests employing feeds of different grinds were conducted and high grades of concentrates with very good recoveries were obtained.

7.0 Washing of Selenite from Messrs Bikaner Gypsums Ltd., Rajasthan

A sample of selenite was received from Messrs Bikaner Gypsums Ltd. for beneficiation studies. The sample analysed SO_3, 45·2; CaO, 31·8; H_2O, 20·6; SiO_2, 0·44; CO_2, 0·48; Al_2O_3, 0·85; and NaCl, 0·5 per cent with traces of MgO and alkalies.

Although the sample was already of a good grade, washing was employed to see the extent to which the purity could be improved. Straight washing of the sample in its " as received " state yielded a washed product of purity 97·7 per cent $CaSO_4$, $2H_2O$ with a recovery of SO_3, 99·2 per cent in it. Washing at 3/8 in. and 10 mesh sizes yielded washed products assaying 45·65 and 46·1 per cent SO_3 equivalent to 98·15 and 99·1 per cent $CaSO_4$, $2H_2O$ respectively with SO_3 recoveries of 97·7 and 91·6 per cent in them. Though the washed products may be considered suitable for manufacture of surgical grade of plaster, the size at which washing is to be done will depend on the purity of final product required.

7.1 Washing of Selenite Sample from Barmer District, Rajasthan

A sample of low grade selenite assaying SO_3, 27·54; CaO, 28·02; MgO, 2·34; SiO_2, 12·28; Al_2O_3, 3·69; CO_2, 4·74 and Alkalies, 1·33 per cent was received from the Director of Mines and Geology, Government of Rajasthan, for beneficiation studies. Petrological examination of the sample indicated that fine clay and calcitic material constituted the principal gangue.

Washing of the sample " as received " after soaking in water for half an hour yielded product assaying SO_3, 45·36 with a recovery of SO_3, 97·2 per cent in it. If, however, the sample was subjected to scrubbing before washing, the grade of the washed product could be improved to SO_3, 45·77 per cent (selenite equivalent 98·2 per cent) but with a reduced recovery of SO_3, 91·8 per cent. Scrubbing of the sample after crushing to 3/8 in. size improved the results still further. The washed product assayed 46·16 per cent (selenite equivalent 99·2 per cent) for recovery of SO_3, 90·1 per cent. A continuous washing test performed with the −3/8 in. crushed sample indicated that a product of grade SO_3, 45·5 per cent (equivalent to 97·8 per cent selenite) and representing a recovery of SO_3, 87·6 per cent could be obtained. This recovery could be slightly improved by recirculating the saturated water reclaimed from the classifier overflow, in the washing circuit; a simple flowsheet for treatment of the sample is given in Fig. 19.

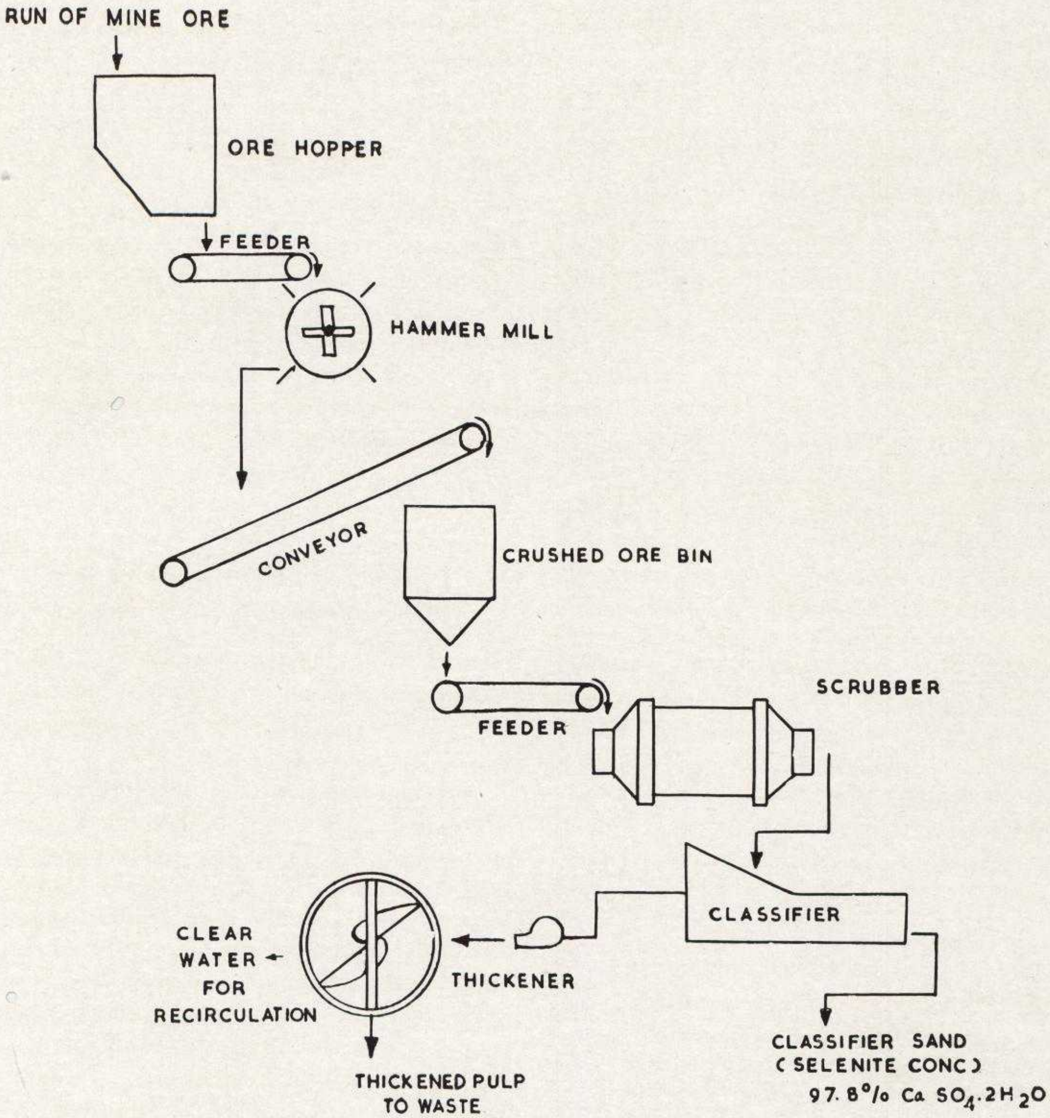


Fig. 19 — Flowsheet for Washing of Selenite from Rajasthan

8.0 Reduction of Silica Content in Magnesite Sample from Salem

A raw magnesite sample assaying MgO, 45·31; CaO, 1·46; CO_2, 48·5; SiO_2, 3·91; Al_2O_3, 0·3; Fe_2O_3, 0·55 and S, 0·12 per cent from Salem, Madras, was received from Messrs Dalmia Magnesite Corporation for reducing its silica content. Quartz, serpentine, felspars, ferro-magnesium minerals and opaque were the principal gangue in the sample. The gangue minerals were liberated at −35 mesh size. Rougher flotation with a feed containing 76 per cent −200 mesh using oleic acid as collector, followed by two cleanings produced a concentrate assaying SiO_2, 1·4 and CaO, 0·26 per cent with a yield of 73·4 per cent by weight. For a weight recovery of about 60 per cent, a concentrate of grade SiO_2, 0·9 per cent could be obtained after three cleanings.

9.0 Beneficiation of Phosphate Rock from Dalbhum Co. Ltd., Calcutta

A sample of low grade phosphate rock was received from Messrs Dalbhum Phosphate Co. Ltd., Calcutta, assaying P_2O_5, 23·26; SiO_2, 19·31; Fe, 11·32; Al_2O_3, 9·15; CO_2, 0·4; F, 2·1; CaO, 30·0 per cent with traces of SO_3 and Ca. The object of the investigation was to determine whether apatite content of the sample could be improved to a grade suitable for super-phosphate manufacture.

Straight flotation of apatite using oleic acid emulsion as collector yielded a rougher float assaying P_2O_5, 41 per cent with a distribution of P_2O_5, 95·5 per cent in it. Variation in grind of flotation feed indicated that a feed containing 76 per cent —200 mesh material would be optimum for best results. Use of sodium silicate as a depressant for gangue improved slightly the grade of the rougher float but the loss of P_2O_5 in the tailing was high.

10.0 Studies on Leaching and Ion Exchange Process for the Improvement of Washed China Clay Sample from Karanjia Quarries, Chaibasa

A sample of washed off-coloured China Clay sample assaying SiO_2, 44·37; Al_2O_3, 37·5; Fe_2O_3, 2·49 and L.O.I., 11·25 per cent from the Karanjia quarries, Chaibasa, was received from Messrs Singhbhum Minerals Co. Ltd., for improving the colour of the clay so as to make it suitable for use in the textile, paper or porcelain industries. Kaolinite and hydro-muscovite were the principal clay minerals present in the sample. The gangue consisted of colloidal ferruginous matter imparting a uniform pink colouration to the clay. Leaching tests followed by a few washings reduced the Fe_2O_3 content from 2·49 to about 2·0 per cent with slight improvement in the colour brightness of the clay. Use of "ZeO-Carb 225" as an ion exchange resin for the elimination of soluble iron from the leached clay pulp reduced the Fe_2O_3 content to about 1·8 per cent with an appreciable improvement in the colour brightness of the clay; this process of colour refinement may find wide applications in the textile and paper industries. However, further large scale tests are necessary to determine the overall economics of the process before the latter can be employed on a commercial scale.

11.0 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 will be made based on these smelting trials. During the period under review the Pilot Plant was operated for the production of high carbon ferro-chrome and silico-chrome.

Different reductants such as Talcher low temperature carbonization coke, Kolsit and Durgapur hard coke were tried.

High carbon ferro-chrome conforming to ISI specification No. 1170/1960 was produced with a typical burden consisting of chromite ore 100 kg., coke 40 kg., quartzite 10 kg. The raw materials chromite ore, coke and quartzite were crushed and sieved to remove −1/8 in. fractions and the average sieve analysed was,

Sieve Analysis in per cent

Size fractions	Chromite	Coke	Quartzite
−1″ + 3/4″	8·51	50·55	48·43
−3/4″ + 1/2″	16·00	8·35	36·48
−1/2″ + 1/4″	59·22	12·50	10·7
−1/4″ + 1/8″	9·22	9·03	2·51
−1/8″	7·05	19·59	1·88

The chemical analyses of chromite and Durgapur coke are given below :

Analysis of chrome ore

	%
Cr_2O_3	51·45
FeO	10·63
Fe_2O_3	4·47
SiO_2	6·5
MgO	17·86
CaO	0·2
Al_2O_3	7·35
P	0·016

Analysis of coke

	%
Fixed carbon	70·9
V.M.	2·18
Moisture	1·52
Ash	25·4

Ash analysis

	%
SiO_2	52·44
Al_2O_3	22·4
Fe_2O_3	11·48
CaO	4·6
MgO	3·6
TiO_2	1·72
S	1·2
P	0·21

After the preliminary heating for two days, the furnace was charged. The furnace operation was quite smooth and tappings were made at intervals of 4 hours. The performance of the furnace is given below.

Production of high carbon ferro-chrome

Total chromite charged	36,700 kg.
Total coke	11,922 kg.
Total quartzite	2,150 kg.
Total power	68,750 kw.
Total quantity of metal obtained	16,401 kg.
Slag/metal ratio	0·9
Power consumption per tonne of ferro-chrome	4193 kWH per tonne

Average alloy analysis

	%
C	65·5
Si	5·04
C	6·6

Average slag analysis

	%
SiO_2	35·3
Al_2O_3	22·18
MgO	30·27
FeO	7·4
Cr_2O_3	4·8

Studies with Talcher Coke

The feasibility of using Talcher low temperature carbonized coke in the production of high carbon ferro-chrome was investigated. The sieve analyse of chromite, Talcher L.T.C. coke and quartzite are given below :

Sieve analysis in weight per cent

Size fractions	Chromite	L.T.C. Coke	Quartzite
+ 1″	3·2	12·3	—
−1″ + 3/4″	9·3	32·4	48·43
−3/4″ + 1/2″	31·6	21·4	36·48
−1/2″ + 1/4″	44·9	14·5	10·7
−1/4″ + 1/8″	5·8	9·2	2·51
−1/8″	5·2	10·2	1·88

Analysis of chromite in per cent

	%
Cr_2O_3	52·40
FeO	15·32
SiO_2	6·26
Al_2O_3	9·48
MgO	15·50
CaO	Trace
P	0·007
S	0·012

Analysis of L.T.C. coke from Talcher

	%
Fixed carbon	68·3
V.M.	7·2
Moisture	5·7
Ash	18·8

Ash analysis

	%
SiO_2	47·3
Al_2O_3	23·4
Fe_2O_3	11·37
CaO	3·46
MgO	3·59
P	0·048

ducing ferro-chrome containing Cr, [illegible], silicon, 4-5 and C, 6-7 per cent with an average power consumption of 4013 KWH per tonne of metal using Durgapur coke were arrived at. This data and performance of the furnace were taken as a starting basis for comparing the performance of Talcher L.T.C. coke in the smelting of high carbon ferro-chrome. The Durgapur coke was gradually replaced by Talcher L.T.C. coke. The performance and operation data are given in Table 4. The performance as well as the composition of the metal produced did not change. However, with using 50 per cent Talcher L.T.C. coke and 50 per cent Durgapur coke, it was found that slag became viscous and extra amounts of quartzite additions were necessitated. It was found that the furnace performance became normal on the addition of extra quartzite. When the entire Durgapur coke was replaced with L.T.C. coke, the excess carbon was kept at 135 per cent of that required theoretically and the quartzite addition was increased from 8 kg. to 10 kg. per 100 kg. of chromite addition. The resulting ferro-alloy analysed Cr, 64·4; Si, 5·8; C, 6·2 per cent with an average [illegible] of metal produced.

11.1 Electrical Conductivity of Ferro-alloy Burden Materials

Electrical conductivity of the burden material plays a vital role during their smelting in the submerged arc electric ferro-alloy furnace. Systematic studies on the electrical resistance offered by the wide range of charge composition used in pilot submerged-arc smelting were taken up and their relationship with other physical properties of the charge mix, were examined.

An apparatus for measurement of resistance of powder mixtures at room temperature was set up. The cell was fabricated out of an ebonite tube with brass ends for electrical connections. The sample is compressed to known bed depths and forms the unknown arm of the Wheatstone's bridge. A stabilized d.c. (12 V.) is passed through the bridge and a reflecting type of galvanometer was used to register the null point. Room temperature resistance measurements were carried out with two coke samples crushed to different particle sizes. Resistivity of the coke samples was found to vary between

Table 4 — Comparison of Performance of Talcher Low Temperature Carbonization Coke with that of Durgapur Nut Coke in the Production of High Carbon Ferro-chrome

% Replacement of coke		nil	25%	50%	75%	100%
Unit Charge: kg	Chromite	100	100	100	100	100
	Coke (Durgapur)	32	24	16	8	nil
	Talcher low temperature carbonization coal	nil	10	20	30	40
	Quartzite	8	8	8	8	10
Per cent of stoichiometric carbon		112	119	125	131	135
Duration of operation in hours		16	21	12	20	20
Feed rate, kg of chromite per hour		181	138	158	130	160
Voltage		50-55	50-55	50-55	50-55	50-55
Power input, kilowatt hours		225-250	225-250	225-250	225-250	225-2 50
Energy consumption, total:		4555	5105	3610	6050	5900
Metal obtained in kg		1135	1473	1082	1478	1299
Slag obtained in kg		1172	1130	838	1221	1513
Slag/metal ratio		1·03	0·77	0·77	0·83	1·16
Power consumption per tonne of metal		4013	4145	3336	4095	4541
Metal Analysis:	Cr %	64·4	63·1	64·1	63·7	64·4
	Si %	4·8	4·5	6·1	6·3	5·8
	C %	6·8	6·9	6·9	6·2	6·2
Slag Analysis:	SiO_2 %	33·5	34·4	30·0	34·4	35·2
	Cr_2O_3 %	4·6	3·4	4·9	4·2	4·6

0·1 and 0·3 ohm/cm.[3] with varying particle sizes and bulk densities. Room temperature resistance measurements were carried out with a sample of metallurgical grade chrome ore and values of resistivity were found to be of the order of megohms.

An experimental unit was also set up for high temperature resistance measurements employing the same bridge arrangements but with a different cell design. The cell was embodied in a furnace having a silica tube as sample holder and copper terminals for electrical connections. Nitrogen atmosphere was maintained in the cell to prevent oxidation of coke samples and copper terminals. The results of high temperature resistance measurements on the coke samples indicated a decrease in resistivity with increase in temperature.

12.0 Extraction of Magnesium Metal Electrolytically from Magnesium Chloride

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

Difficulties were experienced during a set of experiments on the extraction of magnesium metal electrolytically from fused magnesium chloride salt. One of them was that the metal liberated at the cathode could not be directed to the metal collecting pool due to the upward movement of anodic chlorine; second was the development of a hot spot on cell body opposite to the gas burner causing to the failure of the cell pot and change in the composition of the electrolyte due to formation of magnesium oxide during fusion of the charge. These difficulties needed consideration in the design of a suitable cell for further experiments. Accordingly a cell was designed and fabricated with larger dimensions to provide more space in between the anode and cathode. Special arrangement had been devised for the removal of the anode chlorine. To get rid of the hot spots, the gas fired furnace had been modified with suitable baffles inside the furnace to prevent the flame from striking the cell wall directly. A chimney had been provided to the furnace to prevent back firing.

With a view to discard major portion of oxide usually formed during the fusion of the charge, a premelting furnace was set up with a suitable pot for conditioning the molten charge prior to charging in the electrolytic cell. Drying of magnesium chloride salt is being continued for further experiments.

13.0 Electrolytic Recovery of Tin from Tinplate Scrap

Considerable tonnages of tinplate scraps are produced during the manufacture of tinplate and its subsequent fabrication into finished products. Used tin cans also constitute a source of tin scrap. The investigation was taken up on behalf of Messrs Tinplate Co. of India, with a view to recover the tin from tinplate scrap by an electrolytic method.

The electrolysis was carried out in a caustic soda-bath. Stainless steel sheets were used as cathodes and the tinplate scrap, packed in the form of rectangular bundles served the purpose of the anode. Experiments were carried out to determine the optimum conditions for recovery of tin, e.g. concentration of caustic soda, sodium acetate, sodium stannate and sodium carbonate in the electrolyte, temperature of the bath, current density, amount of scrap to be charged, time of detinning and other factors like dissolution and deposition from the same electrolyte, sodium stannate and sodium carbonate build up in the alkaline electrolyte. Fall in cathode C.E. was accompanied by carbonate build up in the electrolyte. Treatment with lime to remove carbonate restored the high value of cathode C.E. even though stannate content remained high. Recovery of metallic tin as cathode deposit had been up to 96·0 per cent under a current desnsity of 13·5 amp/sq. ft. with a current efficiency of 70·8 per cent, purity of the deposited metal being 99·8 per cent, maximum percentage of tin left in the detinned scrap had been 2·5 per cent of the total amount of tin in the coating.

Table 5 shows the results of a few of the experiments.

Table 5 — Results of Experiments on Electrolysis for Recovery of Tin from Tinplate Scrap

1.	Anode — 300 gm. of tinplate scrap pressed into a rectangular bundle measuring 4″ × 3″ × 1″, containing 4·83 gm. of tin.	
2.	Cathode — Two stainless steel plates, measuring 5″ × 3″ × $\frac{1}{16}$″ each	
3.	Time of electrolysis — One hour at 5·1 amp. and 2·1 volts.	
4.	Amount of tin recovered at cathode	4·66 gm.
5.	Amount of tin left undissolved	0·12 ,,
6.	Amount of tin left in the electrolyte (by difference)	0·05 per cent
7.	% tin locked up as stannate	1·0 ,,
8.	%tin left in detinned scrap	2·5 ,,
9.	% tin recovery as metal	96·4 ,,
10.	C.E. wt. cathode	70·8 ,,
11.	Purity of cathode deposit	99·8 ,,

The following will give an approximate idea about the cost of power involved in the process.

For deposition of one kg. of tin, power (D.C.) required is as follows :

$$\frac{5 \times 2{\cdot}1 \times 1000}{4{\cdot}66 \times 1000} \text{ Kwh} = 2{\cdot}25 \text{ KwH}$$

Cost of power (D.C.) for one Kg of tin @ 6 paise per unit = 2·25 × 6 paise.
= 13·5 = 14 paise approx.

For production of one tonne of tin

Cost of power (D.C. only, at 6 paise per Kwh)	Rs. 140·00
Cost of scrap (72 tonnes at Rs. 130/- per tonne)	Rs. 9360·00
	Rs. 9500·00

Cost of chemicals, labour, fixed charges, over head etc. will additionally to be added with data from pilot plant scale trials to be carried out.

14.0 Electro-Slag Melting and Refining

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

Trials on electro-slag remelting were made with medium carbon, C, 1 and Cr, 1·4 per cent ball bearing and high speed steel, manufactured by electric arc and high frequency furnaces. Electrodes were used in the form of 5/8 in. square forged bars. Study was made on the solidification characteristics of the cast ingot. As can be seen from the electro-slag cast macro-structure of plain carbon steel ingot in Fig. 20, the columnar structure-ingotism was eliminated by the process of electro-slag remelting. Further on comparing the structure of the

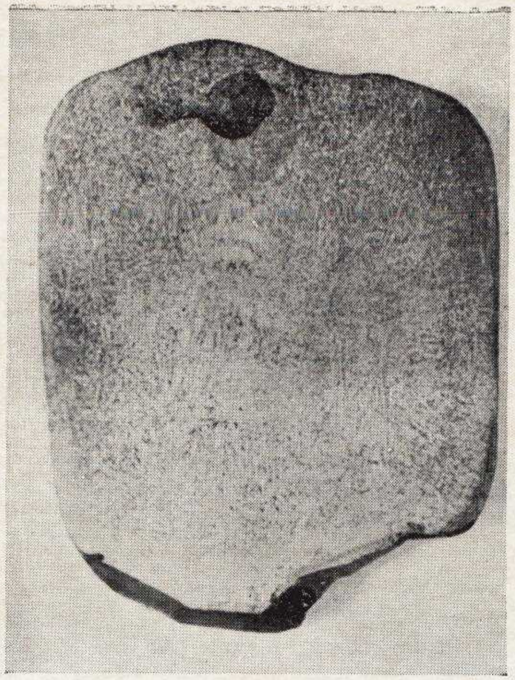
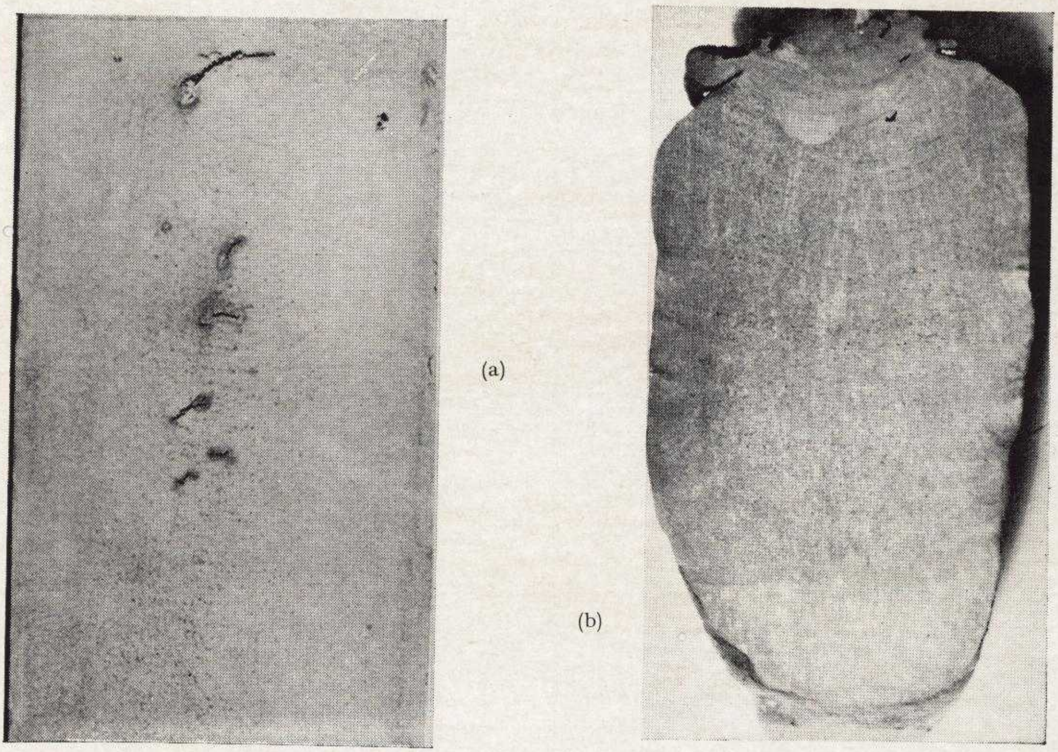


FIG. 20 — LONGITUDINAL SECTION OF ELECTRO-SLAG REMELTED STEEL — 0·4 PER CENT C, MACRO-ETCHED 50 PER CENT HCl



(a)

(b)

FIG. 21 — LONGITUDINAL SECTIONS OF CAST INGOTS OF HIGH SPEED STEEL (18: 4: 1), MACRO-ETCHED 50 PER CENT HCl — (a) HIGH FREQUENCY MELT, (b) ELECTRO-SLAG REMELT

ingot sections of 18:4:1 high speed steel melted by high frequency furnace and electric slag melting method, it was seen that segregation and shrinkage porosity were completely eliminated [Figs. 21 (a) and (b)] Forging of electroslag melted high speed steel showed considerable improvement in forgeability over the conventionally melted one. Micro examination of the structures after about 90 per cent reduction showed a remarkable change in the size and distribution of carbide in favour of electroslag method heat; no axial segregation was marked in the case of electroslag cast. In electroslag refining considerable reduction in the inclusion content was obtained which promoted better impact and fatigue properties. Reduction in the inclusion content was appreciable. Vacuum fusion gas analysis technique showed much less gaseous content in the electroslag refined steels.

15.0 Production of 40 per cent Ferro-titanium by Alumino-thermic Process

The investigation was taken up with a view to establish the optimum conditions to produce 40 per cent ferro-titanium by alumino-thermic process with respect to the amount of energizer aluminium powder, calcined lime, yield of titanium, slag-metal separation, etc.

Metallo-thermic reduction of ilmenite with aluminium powder with potassium chlorate as an energizer yielded ferro-titanium containing 30 per cent titanium and about 7 per cent aluminium. To increase the titanium content in the alloy to 40 per cent, a mixture of ilmenite and rutile was reduced with aluminium and potassium chlorate. Ilmenite analysing TiO_2, 53·75 and Fe, 31 per cent and rutile analysing TiO_2, 90·16 per cent and Fe, 2·88 per cent mixed in the ratio of 10:3 were reduced with theoretical content of aluminium powder with various amounts of emergizer and fluxes like fluorspar and lime. It was observed that potassium chlorate in amounts corresponding to 4 per cent of the weight of ilmenite and rutile together with flux amounting to 20 per cent of the weight of aluminium powder was found necessary for the separation of ferro-titanium. Calcined lime was found to be more effective than fluorspar in separating the ferro-alloy from slag. The addition of ferric oxide in the form of iron ore was found to improve the yield of the alloy. A mixture of ilmenite and rutile in the ratio of 10:5 gave ferro-titanium of the following composition :

Ti %	...	44·35
Fe %	...	38·26
Al %	...	14·29
Si %	...	2·45

15.1 Production of Ferro-boron by Alumino-thermic Process

The investigation was taken up to establish the optimum conditions to produce ferro-boron containing 15 per cent boron by alumino-thermic process with respect to the amount of energizer, aluminium powder, flux, yield of boron, slag/metal ratio, etc.

Boric anhydride was reduced along with iron ore with theoretical amounts of aluminium powder with various amounts of potassium chlorate as energizer. It was observed that potassium chlorate amounting to 6 per cent of the weight of boric anhydride was required for rapid reaction and good separation of metal. The analysis of ferro-boron obtained was :

		Per cent
BX	...	12.7
Al	...	11·3
Fe	...	76·0

15.2 Nitriding of Ferro-alloys

Work was taken up to produce nitrogen bearing ferro-chrome and to study the effect with respect to particle size, temperature, time, grain size, effect of impurities, etc., on maximum absorption of nitrogen.

Ferro-chrome particles of different size ranges were studied at 900°C. and the duration was 6 hours. Nitrogen absorption was found to increase with decrease in particle size. The effect of time for a particle size of 5 mm. was studied at 900°C. at varying periods from 4 hours to 120 hours. The nitrogen absorption steadily increased with time. The metallographic examination revealed that the nitrogen absorption took place along the periphery and the grain boundaries. Further work is in progress to study the effect of grain size, temperature of nitriding.

16.0 Reducibility of Lumpy Iron Ores from Messrs Salgaocar E Irmao Ltda, Goa

Reducibility tests were carried out on four samples of lumpy iron ores from Sallitho, Pale, Surla and Velguem Mines respectively. Sallitho, Pale and Velguem contained about 57 per cent iron and about SiO_2, 1·5 per cent. Surla contained Fe, 58·65 and SiO_2, 2·56 per cent.

Reducibility tests : Experiments were carried out at different temperatures from 500° to 900°C. and at various hydrogen flow rates of 2-6 litres/minute. Optimum conditions for fast reduction were obtained at 800°C. and 6 litres/minute flow rate of hydrogen gas. In order to study the effects of increasing the size of the lumpy iron ores on reduction rates, reducibility trials were carried out on $-63{\cdot}5$ mm.$+44{\cdot}4$ mm. ($-2\frac{1}{2}$ in.$+1\frac{3}{4}$ in.) sized samples of Surla, Pale, Velguem and Sallitho each weighing 300 gm. at 800°C. with hydrogen flowing at 6 litres/minute. Iron ore lump from Surla was more easily reducible than those of Pale, Velguem and Sallitho iron ore lumps had the same reduction characteristics. Experiments were carried out with 50 gm. each from the above sample. It was observed that the reduction characteristics of iron ore lumps of Pale, Velguem and Sallitho fall under one group of less readily reducible than that of Surla ores which is highly reducible.

To assess the reduction characteristics of the four samples, reducibility trials were carried out with other iron ores like Bolani washed ore and Gurumahisani. The time for 90 per cent reduction is given below:

Sl. No.	Sample	Time for 90% reduction in minutes
1	Surla	31
2	Pale	39
3	Velguem	42
4	Sallitho	43
5	Bolani (washed ore)	46
6	Gurumahisani	32

It was found that reducibility of Surla was fully comparable to that of Gurumahisani and that all the four lumpy iron ores are highly reducible than that of the washed Bolani iron ores.

16.1 Reducibility of Specular Hematite Iron Ore Samples of Nathre-ka-Pal Deposits, Rajasthan

Reducibility tests on run-of-mine sample and the beneficiated iron ore from Amsiwali and Daralmata in Rajasthan were carried out to determine the suitability for the pig iron production. Amsiwali and Daralmata iron ores contained Fe, 48·85 and 40·8; SiO_2, 27·82 and 40·37 per cent respectively.

Reducibility Tests

Beneficiation tests to upgrade the iron ores results in finer concentrates of -60 mesh and -48 mesh for Amsiwali and

Daralmata samples respectively. In order to compare the reduction characteristics of the various concentrates as well as with other Indian iron ores, the sample was briquetted for reducibility tests. The concentrates (—60 B.S.S.) obtained after beneficiation of the ore were briquetted and reducibility trials were carried out at different temperature of 500° to 900°C. and at various hydrogen flow rates of 3·0 to 7·0 litres/minute. Optimum conditions for fast reduction were obtained at 800°C. and 4·5 litre/minute flow rate of hydrogen. It was observed that the reduction did not proceed to more than 70 and 80 per cent respectively in the case of lumpy ores of Daralmata and Amsiwali; when the iron ores were crushed and samples were briquetted, the reduction proceeded to completion. Reduction rate was faster for the ore crushed to $-60+200$ mesh than the $-36+60$ mesh crushed ore. Concentrates obtained after beneficiation by various methods indicated that whatever the methods employed, the final concentrates analysed Fe, 66·4-67·54 and SiO_2, 2·1-11 per cent and the reducibility of all the concentrates was similar. Table concentrates obtained after beneficiation were highly reducible, when compared to Kiriburu and Bolani iron ores. The time for 90 per cent reduction is given below :

Sl. No.	Sample	Physical condition	Time for 90 per cent reduction
1.	Table concentrate	—60 + 200 mesh briquetted	30 minutes
2.	Kiriburu iron ore	—60 + 200 mesh briquetted	35 ,,
3.	Bolani (washed)	—60 + 200 mesh briquetted	40 ,,
4.	Bolani (washed)	Lumpy	46 ,,

Briquetted samples of Kiriburu and Bolani retained their shape but Amsiwali and Daralmata samples were found to have disintegrated during reduction.

17.0 Treatment of Ilmenite for 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 ilmenite. Moreover, the production of large quantities of ferrous sulphate poses the disposal problem. Investigations were undertaken on the preferential removal of iron from ilmenite either by roast reduction or chlorination of ilmenite.

Roast Reduction

The ilmenite concentrate from Kerala employed contained small amount of Al_2O_3 and SiO_2, traces of CaO, MgO; and TiO_2, 49·22 per cent, total Fe, 34, Cr_2O_3, 0·07 and V_2O_5, 0·21 per cent.

The method consists in the solid state reduction of ilmenite by carbon at high temperature, whereby most of the iron is reduced to metal and rest of it entered into titaniferous phase. The temperature at 1150°-1200°C. was found optimum for the maximum reduction of iron, i.e. 85 per cent of the total iron into metallic state. At higher temperature the reduced mass started to sinter and some metal carbides formed. Good coalescence of metallic iron grains was observed in the sample heated to 1200°C. for a prolonged period. Reduced ilmenite was quenched in water and by continuous agitation for 16 hours, most of the iron was oxidized to hydrated ferric oxide which was separated by decantation.

Chlorination of Ilmenite with HCl Gas

The ilmenite concentrate employed in the investigation analysed TiO_2, 52·92; FeO, 26·49; Fe_2O_3, 18·3; SiO_2, 0·98; V_2O_5, 0·18 per cent. In initial experiments anhydrous hydrogen chloride gas was passed over the ilmenite sample heated to 800°C.

in a horizontal silica reactor and the reaction products ferrous and ferric chlorides were collected in condensers. Difficulty was encountered in these experiments since the ferrous chloride being less volatile than ferric chloride choked the reactor by condensing in the inner walls as pale brownish crystals. This difficulty was overcome by having a poking mechanism working through a gas tight seal. It was observed that most of the iron was chlorinated with ease but some of the ilmenite was always left behind unaffected being nearer to the walls of the silica tube. Further, sintering effect was observed which prevented free access of hydrogen chloride gas to the bottom layer of ilmenite.

In subsequent experiments, chlorination was carried out in a vertical silica tube, passing the HCl gas from the top. The reaction products were collected continuously at the bottom using a poking mechanism. The ilmenite briquettes were prepared using 1 per cent dextrine as binder. Systematic studies were carried out on the effect of temperature and particle size of ilmenite on the chlorination of iron content of ilmenite and the following results were obtained.

Study of the effect of temperature of chlorination on the removal of iron

Weight of briquettes — 10 gm.
Flow of Hcl gas — 50 cc/minute (298°K)
Time — 3 hours

Temp. °C	*% Fe volatilized*	*Remarks*
600	52·67	No TiO_2 was found to be chlorinated
700	77·66	do
800	94·4	do
850	94·8	do
900	97·3	do
1000	99·84	TiO_2 was found in condensate

Study of the effect of particle size of ilmenite on the removal of iron

Weight of briquettes ... 10 gm.
Flow of HCl gas ... 500 cc/minute
Time ... 3 hours
Temperature ... 800°C.

Average particle diameter in mm.	% Fe volatilized
0·1936	94·27
0·1479	95·3
0·0980	96·03
0·0544	96·9
0·0451	97·85

Further work is in progress to study the effect of various factors such as time, rate of flow of HCl gas, particle size of ore, pressure of HCl gas, pre-reduction and attempts will be made to make the process cyclic, by decomposing the ferric chloride for regenerating the hydrogen chloride.

18.0 Study on Solvent Extraction of Vanadium

Salt roasting of vanadium bearing titaniferrous magnetite followed by aqueous leaching was studied on pilot plant scale. The plant liquor from leaching tanks contained 8 gm. of V_2O_5 per litre and required concentration to 15 gm. V_2O_5/litre for subsequent precipitation of V_2O_5 after adjusting the *p*H to 2. In order to avoid intermediate concentration and to get rid of excessive acid consumption for acidification of the alkaline liquor, work was undertaken on the solvent extraction of vanadium.

A quaternary ammonium base type of anionic liquid exchanger (Tricaprylyl, methyl ammonium chloride) in kerosene was used as the extractant phase. 2-ethyl hexanol was used as a modifier without which the reagent was not soluble in kerosene. The physical make up of the organic phase was studied and optimum ratio of reagent : modifier : kerosene was found to be 1 : 1 : 8 by volume.

Exchange studies were initiated with synthetic solutions of alkali vanadate with or without interfering anions. Both chloride and sulphate reagents were employed, but the sulphate form was found to be more advantageous for extraction of vanadate to the organic phase. Loading of the organic phase with increasing vanadate ions indicated that a minimum reagent/vanadate ratio of 1 : 1 was necessary during loading for efficient vanadium extraction. The exchange equilibria were depended largely on the hydrogen ion concentration of the aqueous phase. On decreasing the acidity from *p*H 3 to 6 the vanadium extraction increased in the organic phase and remained almost unaffected on raising the *p*H to around 7·0. Thereafter, the vanadium extraction slowly decreased with increase in *p*H. Extraction of vanadium was as high as 99·8 per cent at *p*H 6 using a vanadate solution containing 2·65 gms. V./litre.

The exchange reaction was found to be adversely affected on increasing the temperature of reaction and a lower room temperature was found to be most adequate for the purpose. Stripping action of various anions was studied, for regeneration of organic phase and extraction of the vanadate in aqueous phase. The relative order of stripping efficiencies with various anions was followed by

$$SO_4 < \text{Acetate} < Cl < OH < CO_3 < NO_3$$

Reduction of the organic phase by sulphurous acid transferred all the vanadium to the aqueous phase. The stripping action was largely dependent on the type of anion, and was not very much affected by charge of the cation. The slight preference of the cations for stripping was followed by

$$NH_4^+ > Mg^{++} > Ca^{++} > K^+ > Na^+$$

Increasing concentration of stripping reagent helped in better vanadium extraction and use of sodium chloride indicated that the optimum concentration for stripping lies around 4 molar concentrations.

A typical plant liquor containing 2·69 gm. V./litre was tested for vanadium extraction under the conditions arrived at with synthetic solution. It was found that a five stage contact with the organic layer could extract vanadium quantitatively. The loaded organic layer was stripped with 4 M NaCl solution whereby 97·16 per cent recovery was obtained in a single stage. A small counter current exchange system for vanadium recovery from plant liquors is being installed.

19.0 Recovery of Vanadium from Sodium and Calcium Complex Vanadate Salts

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

(i) *Sodium Complex Salts* — The sample of sodium complex salt analysed free alkali (as NaOH), 23·6; PO_4, 12·8; SiO_2, 0·14; Al_2O_3, 30; F, 1·3; V_2O_5, 6·2 and L.O.I., 32 per cent. Leaching with ammonium chloride was attempted in order to dissolve out phosphate so as to leave behind all the vanadium as insoluble ammonium vanadate along with the alumina in the salt. Room temperature leaching with 10 per cent NH_4Cl indicated that both phosphate and vanadate dissolve. Hot water leaching with salt : water feed ratio of 4 : 1 resulted in complete dissolution of phosphate and vanadate, leaving behind alumina in the residue. A typical leach liquor containing 8 gm.

V./litre and 22·7 gm. PO_4 per litre was treated with ammonium chloride at different *p*H and maximum vanadium separation as ammonium vanadate was achieved at *p*H 6. A maximum of 92·9 per cent vanadium recovery was attained on using three times the theoretical amount of ammonium chloride. The ammonium vanadate thus obtained was contaminated with absorbed phosphate and was dissolved in hot water at 70°C. for subsequent reprecipitation with fresh quantity of ammonium chloride. The reprecipitated ammonium vanadate containing V, 42·02 per cent was free of phosphorus and on thermal decomposition yielded pure V_2O_5 with an overall vanadium recovery of 85 per cent.

The possibility of regeneration of ammonium chloride from the final leach liquors was investigated so that ammonium chloride could be crystallized on evaporation. The regenerated product could be used for further precipitation of ammonium vanadate.

(ii) *Calcium Complex Salt* — Sample of calcium complex vanadate salt used analysed V_2O_5, 4·86; P_2O_5, 3·49; CaO, 46·04; Al_2O_3, 9·98; SiO_2, 4·66; F, 0·82; free alkali, 2·32 and L.O.I., 30·29 per cent. Trials were made to convert the vanadium to soluble sodium vanadate and subsequent water leaching. The salt was roasted with soda ash at temperatures from 400-1000°C. and the vanadium recovery increased with increasing temperature till 800°C. beyond which no appreciable change could be observed. The vanadium recovery was found to be almost a linear function of the Na_2O : Salt ratio in the charge and 100 per cent excess of soda ash resulted in 78 per cent vanadium recovery. Both vanadium and phosphorus reacted during roasting and their relative amounts were proportional in the leach liquors. Roasting was almost completed within one hour. Vanadium could be precipitated from the leach liquor as ammonium vanadate by treating with NH_4Cl at *p*H 6.

Acid leaching of the salt was attempted using sulphuric acid and a series of leaching with varying quantities of acid indicated that 20 per cent of H_2SO_4 could leach all the vanadium from 100 gm. of the complex salt. Separation of vanadium from phosphorus is being investigated by liquid ion-exchange method.

20.0 Extraction Characteristics of Alumina from Mysore Bauxites

A sample of bauxite was received from the Director, Department of Mines and Geology, Bangalore to examine its suitability for alumina extraction by Bayer's process. Studies were undertaken to determine the mineralogical analysis and solubility of alumina in alkaline solutions at and above atmospheric pressure.

The bauxite sample analysed Al_2O_3, 59·38; SiO_2, 1·88; Fe_2O_3, 4·60 and TiO_2, 1·68 per cent. Petrographic examination revealed that the ore was soft, fine grained, and oolitic consisting chiefly of amorphous cliachite and minor amounts of crystalline gibbsite. The sample was found to contain Al_2O_3, 52·21 per cent as gibbsite, Al_2O_3, 4·86 per cent as boehmite, Al_2O_3, 1·67 as kaolinite and Al_2O_3, 0·64 per cent as diaspore.

Atmospheric leaching of the bauxite crushed to different sizes indicated that 96·36 per cent alumina can be recovered using −10 mesh (B.S.S.) size of the ore. The ore was leached with varying quantities of alkali with changing Na_2O/Al_2O_3 molar ratios ranging from 1 : 1 to 2·5 : 1 and the best result was obtained at the $Na_2O : Al_2O_3$ of 2 : 1 and higher. With the optimum changing $Na_2O : Al_2O_3$ of 2 : 1, the ore was leached with various concentrations of alkali and it was observed that the alkali concentration should be maintained at 200 gm./1 NaOH or higher for efficient leaching. Leaching was virtually completed in 30 minutes under boiling conditions. Dehydration of the bauxite below 200°C. did not affect the alumina solubility during leaching, whereas significant decrease in alumina solubility was observed on raising the dehydration temperature beyond 200°C. due to mineralogical change in the ore.

Pressure leaching of the ore at 140°C. under a pressure of 10 kg./cm.^{2} was carried out for different periods and a maximum recovery of Al_2O_3, 97·47 per cent was recorded as compared to 96·54 per cent on atmospheric leaching under boiling conditions. The leaching rate did not differ significantly under the two conditions.

20.1 Utilization of Kashmir Bauxite for the Production of Alumina

Bauxite sample received from Kashmir contained high proportions of silica and the alumina occurs as diaspore and clay. The conventional Bayer's process cannot be adopted in this case due to high alumina and soda losses and necessity of high pressures and caustic concentration required to dissolve 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 the conventional Bayer's process or any other suitable treatment consistent with the nature of the bauxite sample under investigation. The bauxite ore sample received analysed as follows :

		Per cent
Al_2O_3	...	55·35
SiO_2	...	22·40
Fe_2O_3	...	5·31
TiO_2	...	2·90
L.O.I.	...	14·56

The sample was subjected to calcination at 950°C. and thereafter leached further at 95°C. with sodium hydroxide for silica removal. The results of the preliminary studies on calcination and leaching are tabulated below :

Period of calcination	Concentration of NaOH in leaching solution per cent	Percentage of silica left in the residue	Percentage of silica removed from the calcine
60 minutes	15	6·22	82·2
	20	4·74	88·1
90 minutes	15	4·06	88·7
	20	5·62	84·8
180 minutes	15	3·47	90·25
	20	3·04	91·80
240 minutes	15	2·53	92·58
	20	2·64	92·78

Most of the silica could be removed by calcination at 950°C. for over 3 hours followed by leaching at 95°C. for an hour at 15 per cent NaOH concentration. Further work is under progress to study the effect of calcination at other temperatures and the desilication characteristics of the calcine in order to effect minimum alumina losses in the leach liquor. The calcined samples were petrologically examined for the changes occuring in the composition due to calcination treatment.

21.0 Reconditioning of Magnesium Powder

Magnesium powder during storage for long periods deteriorates due to the formation of oxide coating on the metallic surface and cannot be used in pyrotechnics. The investigation was taken up at the instance of the Defence Establishments for improving the metallic content to over 90 per cent from powder containing less than 80 per cent metallics.

Experiments were carried out on the utilization of the spent acid obtained after treatment of deteriorated magnesium powder in chromic acid solution. The spent acid on analysis showed the presence of very little of trivalent chromium and all the chromium in the acid was in the hexavalent state. The MgO content of the completely spent chromic acid was found to be 26 per cent. It was presumed that the presence of MgO in the acid was mainly responsible for inhibiting pickling action of chromic acid solution. Possibilities of removing the MgO content by electrolysis were investigated. The spent acid was subjected to electrolysis by separating the anolyte and catholyte chambers via a porous diaphragm. Both the chambers were filled with spent chromic acid and electrolysed using lead electrodes. Samples were collected at different periods from the anolyte chambers and analysed for the CrO_3 and MgO content. It was found that the CrO_3 content gradually increased with a gradual decrease in the MgO content in the anolyte. Calculated amount of oxidized powder based on the CrO_3 content of the electrolysed spent chromic acid solution was treated and analysed for its magnesium content.

Synthetic solutions having same CrO_3: MgO ratio as the samples drawn from the anolyte chambers at different intervals of time were prepared for comparative study by the addition of pure MgO in chromic acid solutions and same amount magnesium powder based on the CrO_3 content was treated as in the case of electrolysed solutions. The results are shown in Table 6.

It was observed that CrO_3 : MgO ratio is an important factor in pickling reaction. With decreasing concentration of MgO in the pickling solution, pickling action improves. MgO had an inhibiting effect on the pickling action of chromic acid.

Table 6 — Effect of CrO_3 : MgO Ratio in the Pickling Solution

CrO_3:MgO in solution	Metallic content of the powder after treatment in electrolysed solution %	Metallic content of the powder after treatment in synthetic solution %
1 : 0·26	85·15	85·15
1 : 0·18	95·92	95·68
1 : 0·09	98·63	98·93
1 : 0·06	99·3	99·3

Six hundred kg. of magnesium powder of Grade V were received from Khamaria, Jabalpur and was reconditioned to the required specification from their original metallic content ranging from 89 to 90 per cent.

It was observed that ordinary softened water was suitable for the treatment and washing of the powder. In view of the suitability of the softened water reconditioning treatment could be considerably speeded up.

22.0 Preparation of Anhydrous Magnesium Chloride

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

The work covered the dehydrations of hydrated magnesium chloride by:

(a) Straight drying method.
(b) Drying under reduced pressure
(c) Drying by fluidized bed technique.
(d) Preparation of the anhydrous salt from magnesia by chlorination method.

Processes at a, b & c underwent special care to prevent oxide and oxychloride formation and thereby a product, $MgCl_2$ containing approximately 20-22 per cent moisture was obtained without undue oxide. Attempts were made to prepare anhydrous magnesium chloride from magnesia by chlorination methods. The magnesia was obtained in the form of fine powder. It was briquetted with coal with $MgCl_2$ solution. Investigations were carried out to make the briquettes sufficiently strong to resist the storage and weather condition and to have furnace stability which meant that the briquettes should not decrepitate into too much finer sizes when subjected to the temperature conditions inside the furnace during chlorination. Following results were obtained :

Composition of the briquettes

MgO	100 gm.
Petroleum Coke	30 gm. (calcined at 1250°C.)
Binder	45-100 cc. of $MgCl_2$ solution of 22 °Be′

Crushing strength of briquettes

Drying temp., °C.	110	170	274	425
Crushing strength lb/sq. in.	155	280	380	15

Attempts were made to increase the crushing strength of the briquettes at a higher temperature.

23.0 Preparation of Synthetic Cryolite

Cryolite is an essential mineral required in steel, ceramic and aluminium production industries. It is also used in insecticides and in the manufacture of enamels and opaque glass. Natural cryolite is not found in India. Its requirements are met entirely by imports. In view of the expanding demand of this material in the country need for its synthetic preparation is imperative, particularly in the context of known extensive deposit of fluorspar in Rajasthan and Madhya Pradesh. Fluorspar is one of the chief raw materials used in the production of synthetic cryolite. The indigenous 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.

The process which was investigated consisted in decomposing the metallurgical grade fluorspar in an aqueous medium with sulphuric acid and boric acid. The reaction was completed at 95°C. in two hours. The reaction products were fluoboric acid and calcium sulphate. The slurry was filtered off to remove insoluble calcium sulphate crystals. The filtered liquor rich in fluoboric acid was neutralized with hydrated alumina and sodium carbonate to form cryolite and boric acid. Cryolite precipitate was filtered out and the spent liquor containing boric acid was reused for leaching fresh fluorspar after the addition of sulphuric acid. Another sample of fluorspar which was also obtained from Rajasthan was investigated.

24.0 Nickel-free Stainless Steels

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

Comprehensive investigations were carried out on the determination of physical, mechanical and high temperature creep properties of the new steels developed. Concurrently, comprehensive work was also done on the industrial scale production of these stainless steels including assessment of production costs, yield value and range of industrial products obtainable under Indian market conditions and requirements. Tests were conducted to determine the low-temperature and sub-zero tensile and impact properties, high-temperature tensile and impact properties, high-temperature tensile strength, weldability, oxidation resistance and ageing characteristics of different compositions of the nickel-free stainless steels.

During the period under review investigations were carried out on the properties of modified 12 per cent chromium stainless steels containing molybdenum, vanadium, cobalt and nitrogen. Chemical compositions of the steels investigated are given in Table 7.

Table 7 — Chemical Composition of Steels Investigated

Heat No.	Cr %	Mn %	C %	Mo %	V %	Co %	N %
T-19	11·44	0·49	0·20	4·66	0·37	0·95	—
T-20	12·38	1·75	0·14	4·54	—	—	—
T-22	12·48	1·49	0·10	3·47	0·25	3·0	—
T-23	12·43	1·43	0·15	4·73	0·25	2·75	0·10

Metalllographic examination of these steels revealed a duplex structure. Fig. 22 shows the tempering characteristics of these steels which indicated considerable secondary hardening. Investigations on the properties of hardenable stainless steel of type AISI-410 containing nitrogen were continued. Corrosion tests were carried out in boiling 10 per cent acetic acid on samples tempered at different temperatures. Electron microscopic studies of the structural changes during tempering of this steel are being carried out.

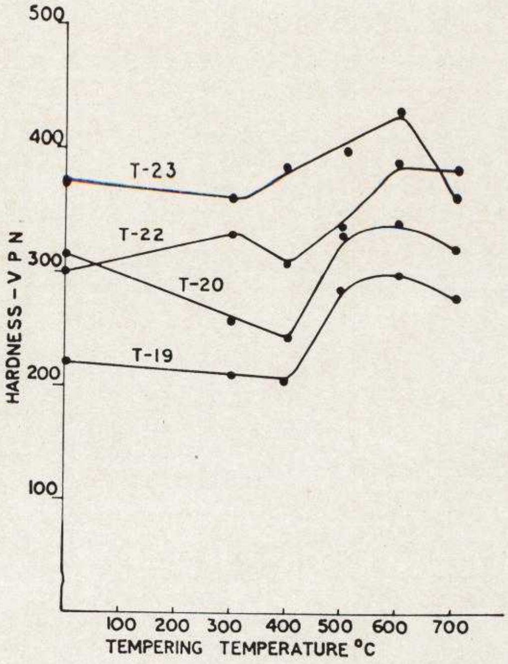


Fig. 22 — Relation Between Hardness and Tempering Temperature of Steel Samples Solution Treated at 1100°C. and Tempered 4 Hours

24.1 Study on Precipitation Characteristics on Cr-Mn-N Stainless Steels

Optimum relationship among the composition, heat-treatment and phase formation in chromium-manganese-nitrogen stainless steels is important in obtaining the comprehensive understanding of the characteristics of these steels.

Study of the structural modifications produced by various heat-treatments of these steels under vacuum was based on the metallographic examination by optical and electron microscopy, electron and X-ray diffraction techniques and chemical analyses (Figs. 23 to 25). Heats were made having 17 per cent chromium, 6 per cent manganese, 0·45 per cent nitrogen and 0·1 per cent carbon contents. The ingots were forged and rolled into sheets which were subsequently solution-treated at 1100°C. Test pieces were cut out from the solution-treated sheets and subjected to heat treatments under vacuum at the holding temperatures of 900°C., 850°C., 800°C, and 700°C. for short periods.

After heat-treatments at desired temperatures the specimens were examined. The results are given in Table 8.

25.0 Effect of Titanium and Niobium in Minute Quantities on Impact Properties of Bainitic Steel

Optimum compostion suggested for uniform bainitic structure as well as for improved low temperature impact properties is C = 0·1; Mn = 0·5; Mo = 0·5; B = 0·002 per cent. The effect of minute quantities of Ti and Nb additions on such a steel was studied.

The compositions studied are given in Table 9.

No particular attention was paid when Ti addition was made to the melt, because the bath had been thoroughly deoxidized and denitrided by ferro-silicon and alu-

Table 8 — X-ray Study of the Cr-Mn-N Stainless Steel 'S'$_1$

Composition %	
Cr	19·49
Mn	11·08
N	0·38
C	0·08

Wire specimens were prepared from the solution-treated and annealed sheets (40 Thou.), and electrolytically polished for x-ray analysis. The extracted residues from these samples were studied by powder diffraction. Filtered cobalt radiation was used in all cases.

Solution treated at 1100°C. — 15 minutes and oil quenched

	dA°	Intensity	Plane hkl	Phase
1.	2·091	V.S.	(111)	gamma
2.	2·033	F	(110)	alpha
3.	1·809	S	(200)	gamma
4.	1·278	S	(220)	gamma
5.	1·175	V.F.	(211)	alpha
6.	1·089	V.S.	(311)	gamma
7.	1·043	S	(222)	gamma

Solution treated and annealed at 800°C. — 4 hr.

	dA°	Intensity	Plane hkl	Phase
1.	2·095	V.S.	(111)	gamma
2.	1·810	S	(200)	gamma
3.	1·278	S	(220)	gamma
4.	1·089	V.S.	(311)	gamma
5.	1·043	S	(222)	gamma

Extracted residue from solution treated and annealed sample at 700°C. — 1½ hours

	dA°	Intensity	Phase
1.	2·422	V.F.	Cr_2N
2.	2·238	m	Fe_2N
3.	2·125	V.S.	Fe_2N
4.	1·640	m	Fe_2N
5.	1·268	m	Fe_2N
6.	1·162	F	Fe_2N
7.	0·934	F	Fe^2N

V.S. denotes very strong
S denotes Strong
m denotes medium
F denotes Faint

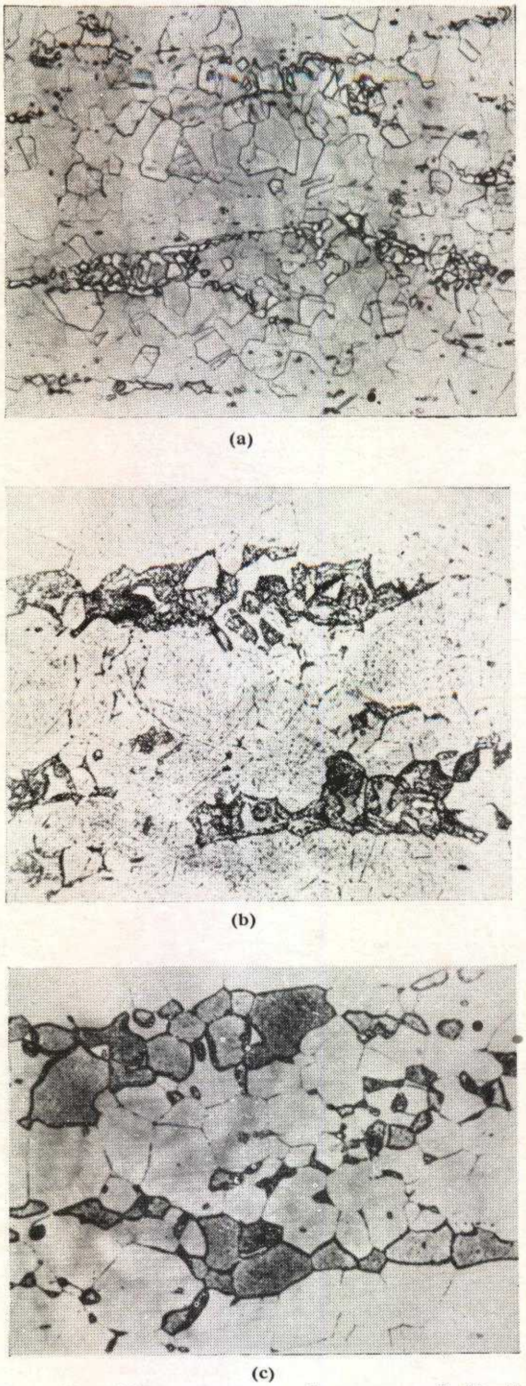

(a)
(b)
(c)

FIG. 23 — METALLOGRAPHIC STUDY OF Cr-Mn-N STAINLESS STEEL S$_1$[(a) 1100°C Solution Treated, Oil Quenched Showing Austenite with δ-Ferrite × 250. (b) Solution Treated and Annealed 880°C for 1½ hr. Formation of δ-Eutectoid from Ferrite Grains and General Precipitation of Cr_2N and Fe_2N × 450. (c) Solution Treated and Annealed 700°C for 1½ hr. Precipitation of δ-Ferrite from Austenite along with Carbide Precipitation at the grain boundary × 450. Picric acid etched]

Table 9 — Composition of Steels Studied

Steel	Compositions wt. per cent							Grain size ASTM number
	C	Mn	Mo	B	Si	Ti	Nb	
D_1	0·08	0·50	0·60	0·0017	0·056	0·008	—	2-3
D_2	0·09	0·57	0·60	0·0010	0·078	0·026	—	4-5
D_3	0·10	0·65	0·60	0·003	0·022	0·032	—	5
D_4	0·09	0·65	0·62	0·002	0·105	0·038	—	7
D_5	0·11	0·65	0·60	0·0018	0·103	—	0·01	4
D_6	0·11	0·60	0·60	0·0020	0·08	—	0·018	5
D_7	0·09	0·65	0·60	0·001	0·07	—	0·034	6-7

FIG. 24 — ELECTRON MICROGRAPH OF STEEL S_1 ANNEALED 700° FOR 1½ HR. SHOWING GRAIN BOUNDARY PRECIPITATION (CARBON REPLICA × 50,000)

minium respectively, before boron addition was made. For Nb addition no special precaution was necessary. The cast ingots were forged to 3/4 in. bars and were normalized at 950°C. The impact specimens were machined from normalized bars. Grain-size measurement by " McQuaid-Ehn Test " showed a decrease in grain size with increasing percentage of Ti or Nb. Metallographic studies showed uniform bainitic structure Tensile Strength was in the range of 45-50 t.s.i. (Fig. 28).

The low temperature impact studies carried out so far, indicated gradual lowering of transition temperature with increasing Nb content when the Nb addition was in between ·01 and ·03 per cent (Fig. 27). Further work on Nb series and the Ti series is in progress. This improvement of low temperature impact properties with additions of Nb is possibly due to either the finer grain size with increasing Nb content in the range indicated or due to the depletion of the interstitial carbon in the matrix owing to the carbide formation or the both.

26.0 Development of Substitute Alloy Steels

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

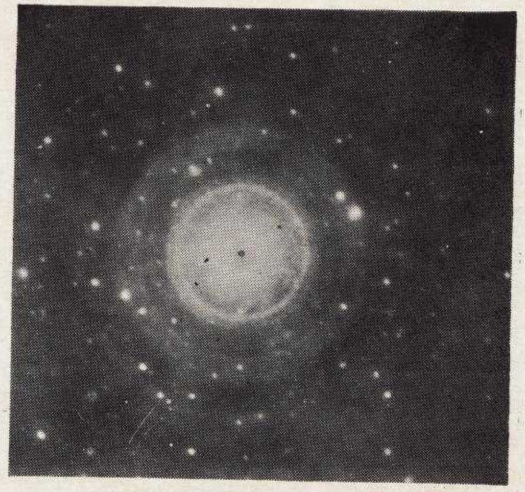


FIG. 25 — ELECTRON DIFFRACTION PATTERN OF THE EXTRACTED RESIDUE OF THE ZONE SHOWN IN FIG. 24

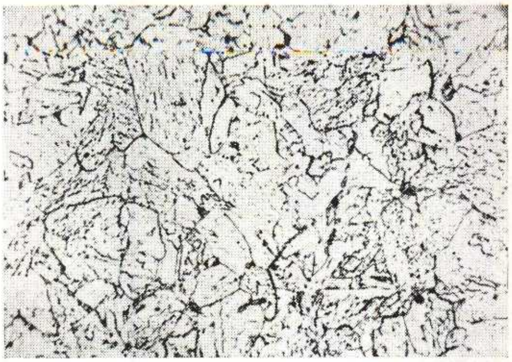


FIG. 26 — MICRO-STRUCTURE OF Nb-TREATED STEEL D. NORMALIZED AT 950°C × 470 NITAL ETCHED

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) *Tool Steels*

Heats of high speed steel were made in 25 lb. basic lined electric induction furnace with the additions of small percentages of rare earth materials to study the effect in relation to cutting efficiency of tool steel in as cast condition. Table 10 gives the chemical composition.

Hardness and other mechanical properties are being studied.

(ii) *Development of Die Steels*

Tempering characteristics of DS_1, DS_2, DS_3 steels of composition given in Table 11 were investigated. Different tempering [illegible] tempering range selected to determine the steel DS_2. The results obtained have been shown in Table 12.

Various austenitizing temperatures were investigated to study the effect of different austenitizing temperature. These steels were tempered at different temperatures. Results obtained are shown in Table 13, which shows quenching temperatures, quenching media, tempering temperatures and tempering time used for each grade of steel.

(iii) *Isothermal Transformation Characteristics of Alloy Steels*

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

Isothermal transformation experiments were carried out on a number of alloy steels, diagrams of which are given in

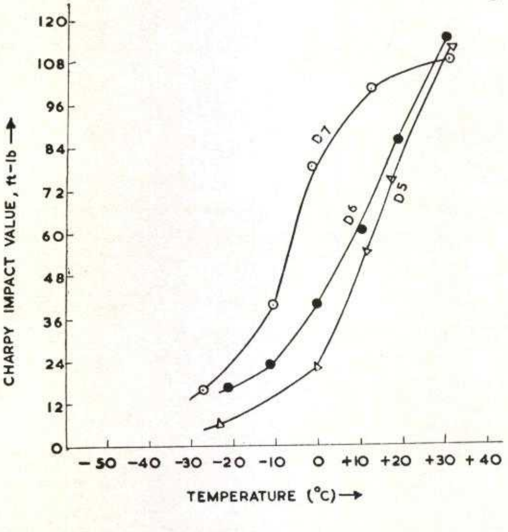


FIG. 27 — ENERGY TRANSITION CURVES FOR NIOBIUM TREATED BAINITIC STEEL

Table 10 — Chemical Composition of High Speed Steel Heats

Steel	C %	Si %	Mn %	Cr %	V %	W %	Ti %	Zr %
H_1	0·57	0·26	0·46	4·94	1·52	17·21	0·02	—
H_2	0·61	0·40	0·72	5·02	1·40	16·41	0·23	—
H_3	0·56	0·26	0·54	5·06	1·52	16·08	0·35	—
H_4	0·60	0·49	0·80	4·43	1·21	16·10	0·45	—
H_5	0·61	0·53	0·53	5·06	1·52	16·41	Trace	Trace

Table 11 — Composition of Heats Made

Steel	C %	Si %	Mn %	Cr %	V %	Ti %	Mo %	W %
DS_1	1·23	0·37	1·03	1·78	0·14	Trace	0·17	0·085
DS_2	0·99	0·51	0·88	1·96	0·11	Trace	—	—
DS_3	1·08	0·44	0·86	0·12	0·31	Trace	—	—

Table 12 — Results of the Experiments done on Steel DS_2

Tempering temperature in °C.	Tempering time		
	½ hour	1 hour	2 hours
			781
150	810	790	622
200	710	671	610
275	651	628	695
350	602	600	578
400	597	596	490
450	530	517	461
500	498	481	403
550	419	409	286
600	322	297	277
660	311	290	

Figs. 28-30. Composition of the steels are given below:

Steels	C %	Si %	Mn %	Cr %	Mo %	Ni %
1.	0·18	0·33	0·63	1·06	0·80	0·09
2.	0·22	0·26	0·68	1·03	0·87	0·08
3.	0·40	0·28	0·55	1·25	0·60	0·20

(iv) *Continuous Cooling Transformation of Alloy Steels*

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

The continuous cooling transformation diagram of the steels investigated are given in Figs. 31 to 33. Composition of the steels are given below :

Steels	C %	Si %	Mn %	Ni %	Cr %	Mo %
4.	0·20	0·23	0·70	0·60	0·50	0·21
5.	0·30	0·21	0·65	0·65	0·60	0·19
6.	0·18	0·33	0·63	0·09	1·06	0·80

27.0 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.

Experimental heats were made with slight modifications in the percentage of alloying elements of the existing low-alloy high tensile steels in use. The main alloying elements of these heats were manganese, chromium, silicon and copper. After analysing the results of these heats, a low carbon heat with carbon in the range of 0·12 to 0·15 suitable percentages of manganese, silicon, copper and vanadium was selected for making heavy tonnage scale heats.

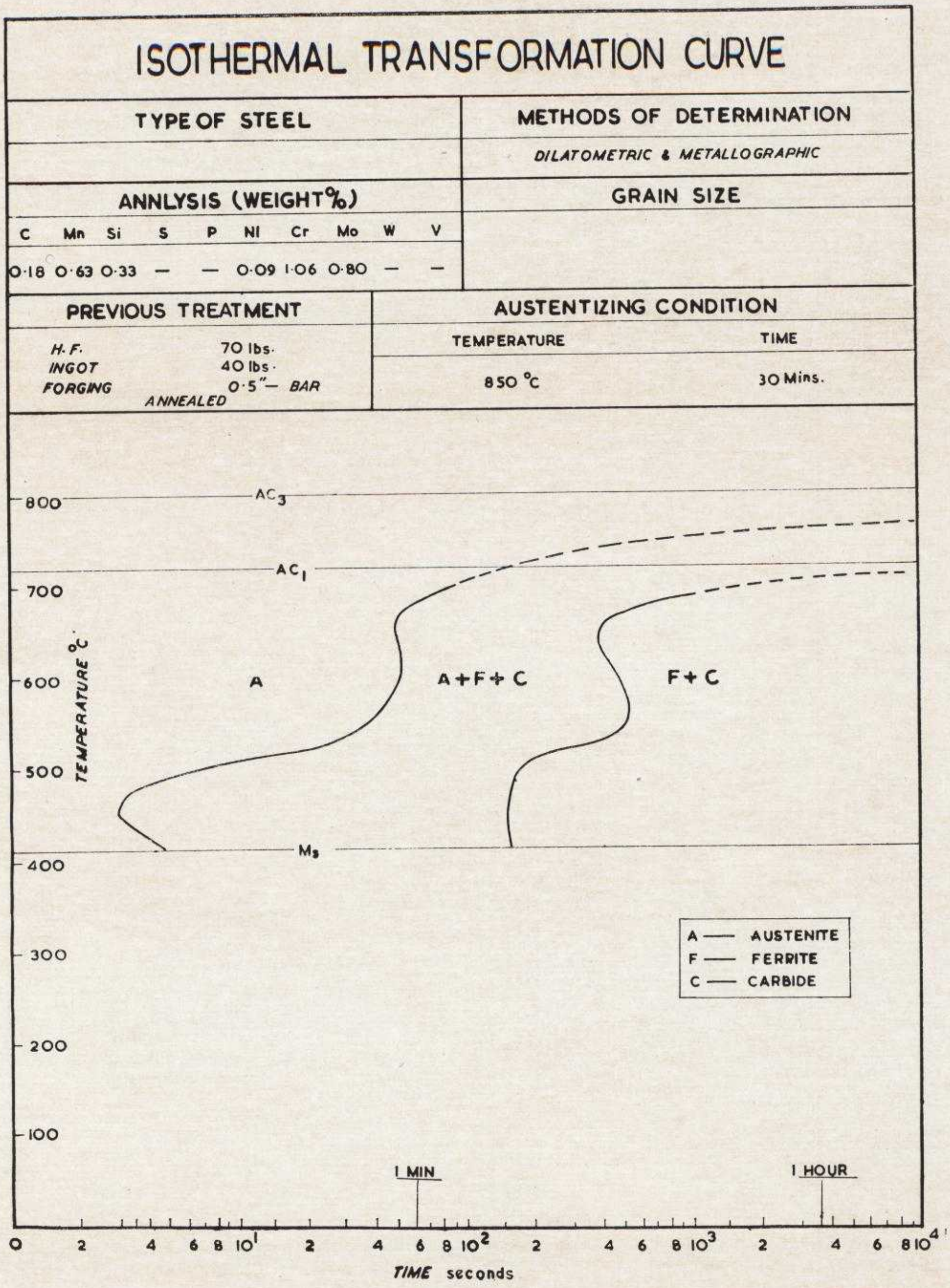
Table 13 — Effect of Austenitizing Temp., Quenching Media on Tempering of Die Steel

Austenitizing Temperature °C.	Quenching medium	Tempering Temperature in °C.	Tempering time in hr.	Hardness (V.P.N.)		
				Steel DS_1	Steel DS_2	Steel DS_3
800	Oil	350	One	520	600	—
800	,,	,,	,,	527	606	513
900	,,	,,	,,	514	575	—
950	,,	,,	,,	614	568	—
1000	,,	,,	,,	—	—	—
1100	,,	,,	,,	—	—	—
800	,,	450	One	518	505	—
850	,,	,,	,,	520	530	451
900	,,	,,	,,	524	540	—
950	,,	,,	,,	533	560	—
1000	,,	,,	,,	538	564	499
1100	,,	,,	,,	505	514	422
800	,,	500	One	490	422	—
850	,,	,,	,,	493	460	418
900	,,	,,	,,	499	476	—
950	,,	,,	,,	507	512	—
1000	,,	,,	,,	512	520	410
1100	,,	,,	,,	—	—	—
800	,,	550	One	397	389	—
850	,,	,,	,,	410	415	392
900	,,	,,	,,	432	436	—
950	,,	,,	,,	448	459	—
1000	,,	,,	,,	499	461	382
1100	,,	,,	,,	500	450	—
800	Oil	600	One	382	270	—
850	,,	,,	,,	389	334	380
900	,,	,,	,,	400	393	—
950	,,	,,	,,	407	400	—
1000	,,	,,	,,	411	403	375
1100	,,	,,	,,	—	—	—
800	,,	650	One	390	277	—
850	,,	,,	,,	313	304	282
900	,,	,,	,,	322	310	—
950	,,	,,	,,	330	313	—
1000	,,	,,	,,	335	318	270
1100	,,	,,	,,	—	—	—

Accordingly some heats were made. Detailed investigation of one of these heats was taken up. Copper content of this heat was slightly higher than that specified. Some of the one ton heats made were however, found to be off grade. Tensile test results and charpy impact values of the heat are given in Table 14.

Test results of normalized specimens showed that the steel possessed the required strength and ductility; its transition temperature was also lower than −60°C.; but its strength in annealed condition was much lower. Following hardness values obtained with different sections also confirmed these results.

Section	Treatment	Hardness value (BHN)
4″ square	As cast	192
2″ square	as forged	212
1″ square	as forged	204
do	normalized	222
½″ square	as forged	219
do	normalized	224
¼″ thick plate	as rolled	250
¼″ thick plate	normalized	244



ISOTHERMAL TRANSFORMATION CURVE

TYPE OF STEEL	METHODS OF DETERMINATION
	DILATOMETRIC & METALLOGRAPHIC

ANNLYSIS (WEIGHT%)

C	Mn	Si	S	P	Ni	Cr	Mo	W	V
0·18	0·63	0·33	—	—	0·09	1·06	0·80	—	—

GRAIN SIZE

PREVIOUS TREATMENT

H. F.	70 lbs.
INGOT	40 lbs.
FORGING	0·5″— BAR
ANNEALED	

AUSTENTIZING CONDITION

TEMPERATURE	TIME
850 °C	30 Mins.

Fig. 28

ISOTHERMAL TRANSFORMATION CURVE

TYPE OF STEEL	METHODS OF DETERMINATION
	DILATOMETRIC & METALLOGRAPHIC

ANALYSIS (WEIGHT %)

C	Mn	Si	S	P	Ni	Cr	Mo	W	V
0·22	0·68	0·26	-	-	0·08	1·03	0·87	-	-

GRAIN SIZE

PREVIOUS TREATMENT

H. F.	70 lbs.
INGOT	40 lbs.
FORGING	0·5" BAR.
ANNEALED	

AUSTENITIZING CONDITION

TEMPERATURE	TIME
850 °C	30 Mins.

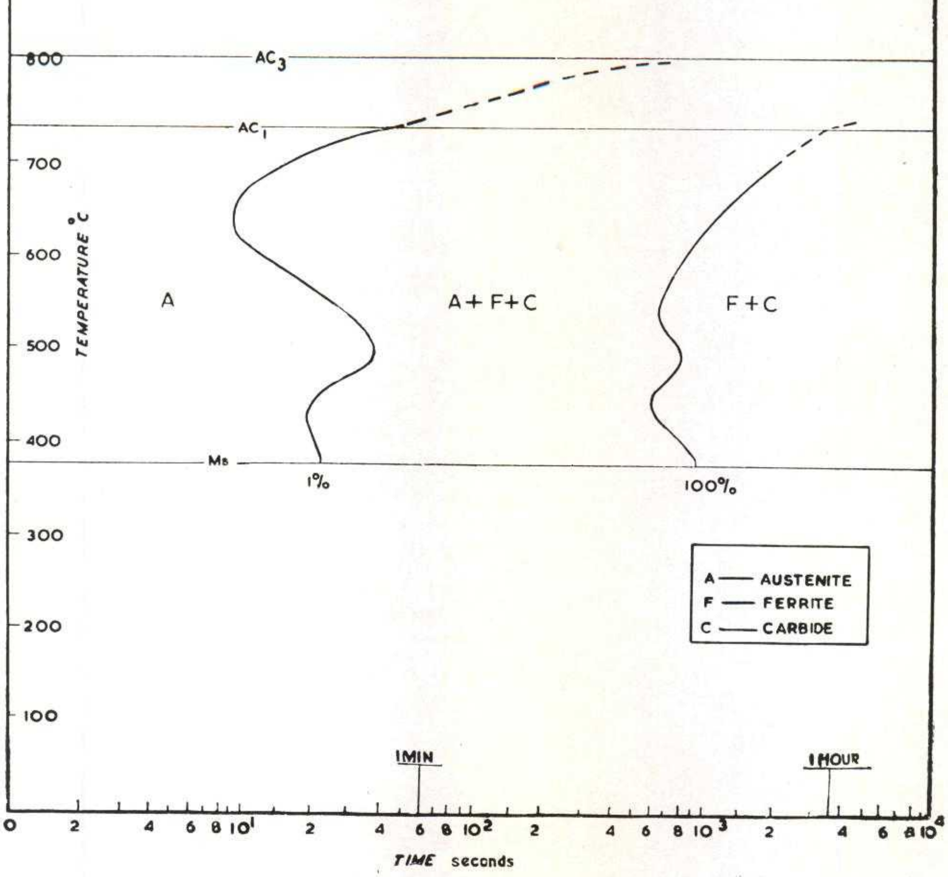


Fig. 29

ISOTHERMAL TRANSFORMATION CURVE

TYPE OF STEEL	METHODS OF DETERMINATION
	DILATOMETRIC & METALLOGRAPHIC

ANALYSIS (WEIGHT %)

C	Mn	Si	S	P	Ni	Cr	Mo	W	V
0·40	0·55	·28	—	—	0·20	1·25	0·60	—	—

GRAIN SIZE

PREVIOUS TREATMENT

H.F.	70 lbs.
INGOT	40 lbs.
FORGING	0·5" BAR.
ANNEALED	

AUSTENITIZING CONDITION

TEMPERATURE	TIME
850°C	30 Mins.

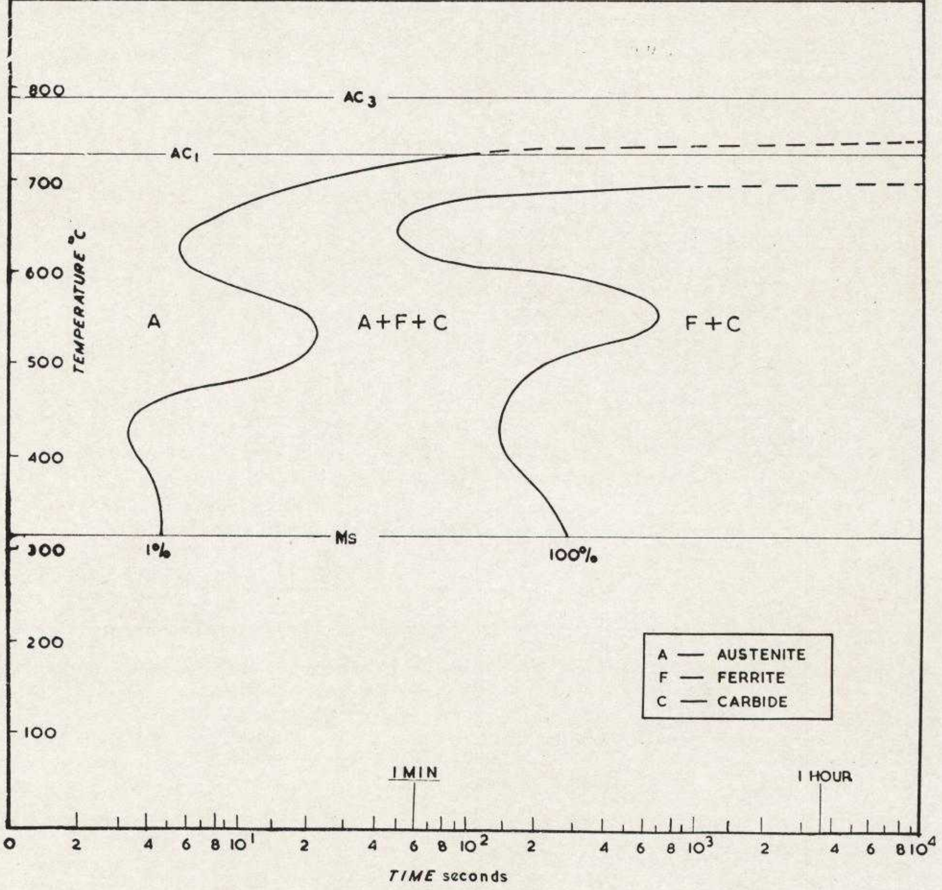


Fig. 30

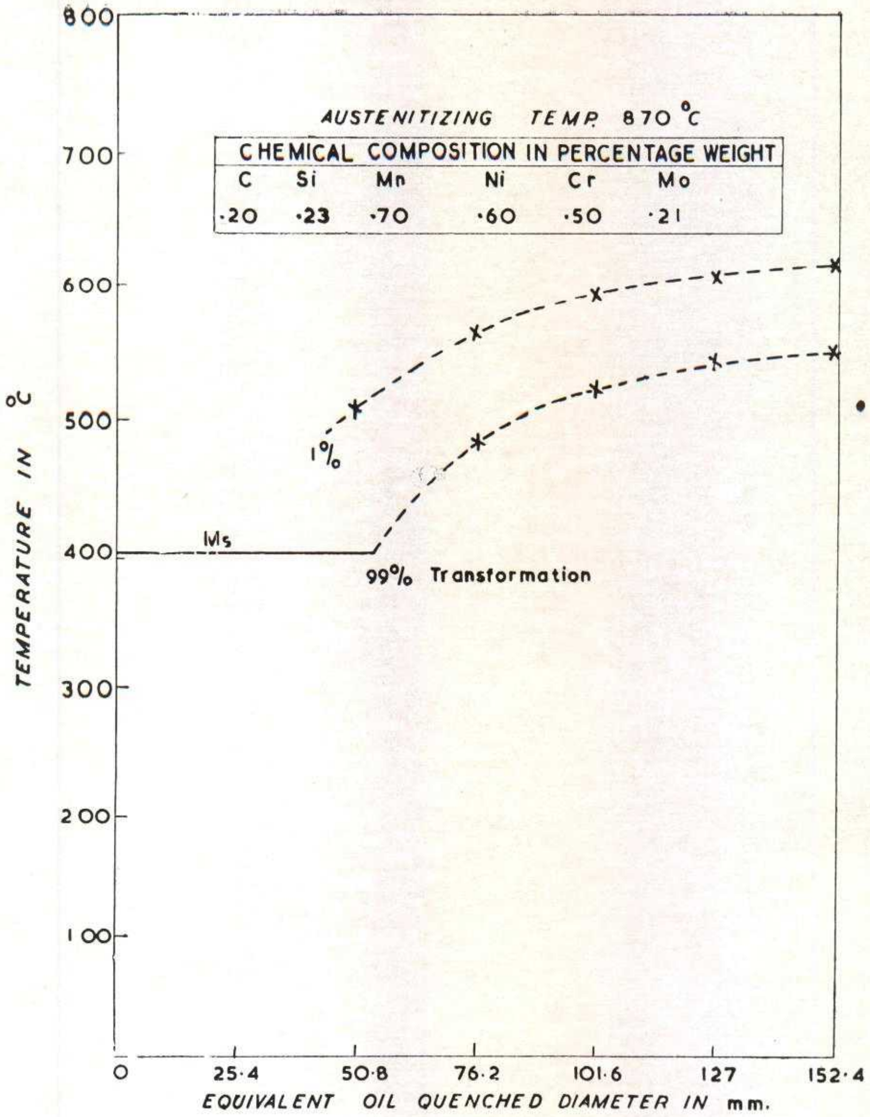


Fig. 31 — Continuous Cooling Transformation Diagram for Steel

Table 14 — Test Results of One of the Tonnage Scale Heats of Low-alloy High Tensile Structural Steels

	Tensile Test Results				Charpy Impact Values (ft. lbs.)				
Heat No.	Max. stress kg/sq. mm.	Yield stress kg/sq. mm.	R.A. %	E % 2″ GL	Room Temp.	0 °C.	−20 °C.	40 °C.	60 °C.
Arc 3*	76·23	—	47·5	25	38	28	22	21	23
**	55·12	42·87	65·5	35	41	—	—	—	—

*Normalized
**Annealed

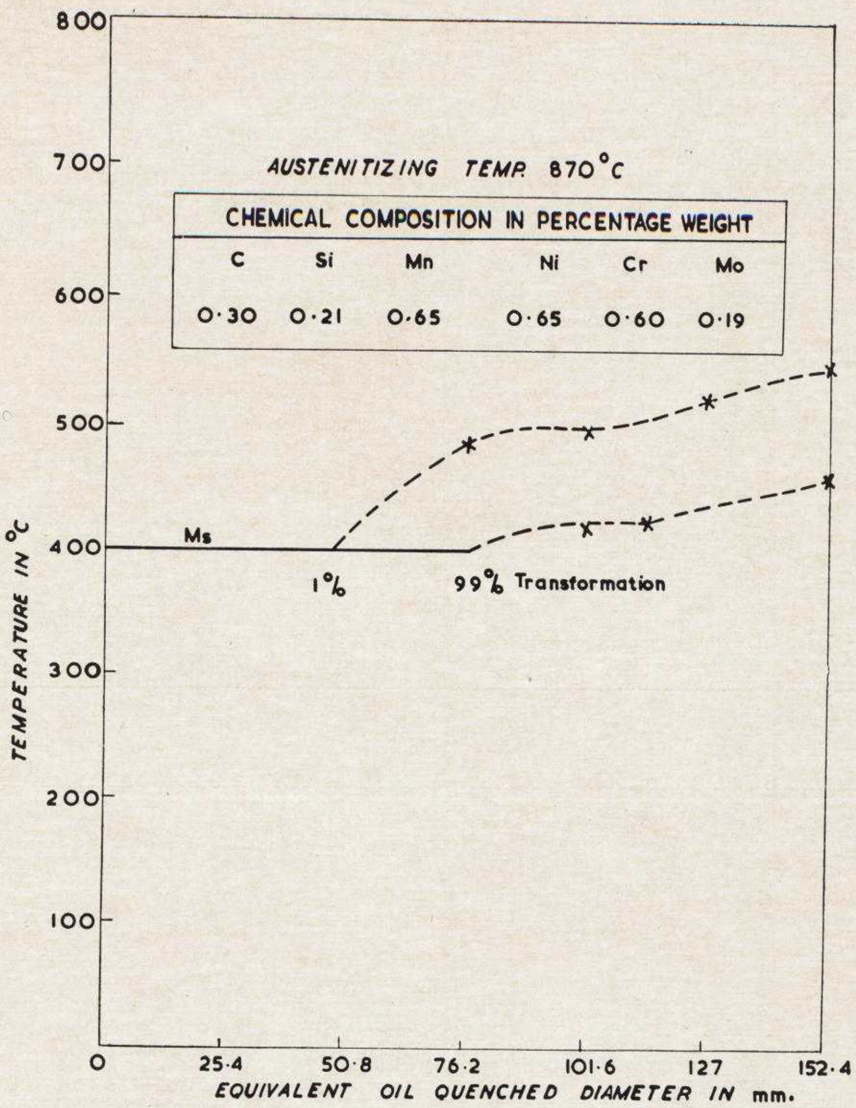


Fig. 32 — Continuous Cooling Transformation Diagram for Steel

Results of Weldability Tests

Tests were carried out according to Reeve's fillet test method (miniature). It was shown that the steel might be on the border line having the hardness of the heat affected zone slightly higher than that accepted for weldable steel. The results of the tests, however, were further checked by other weldability test methods.

Results of Corrosion Tests

Standard salt spray corrosion test of this steel was carried out. Following table gives the comparative result with that of mild steel which was also tested in the same

Material		Rate of Corrosion in gm/dm²
Mild steel	...	0·69, 0·61
Low alloy steel (Arc 3)	...	0·66, 0·64

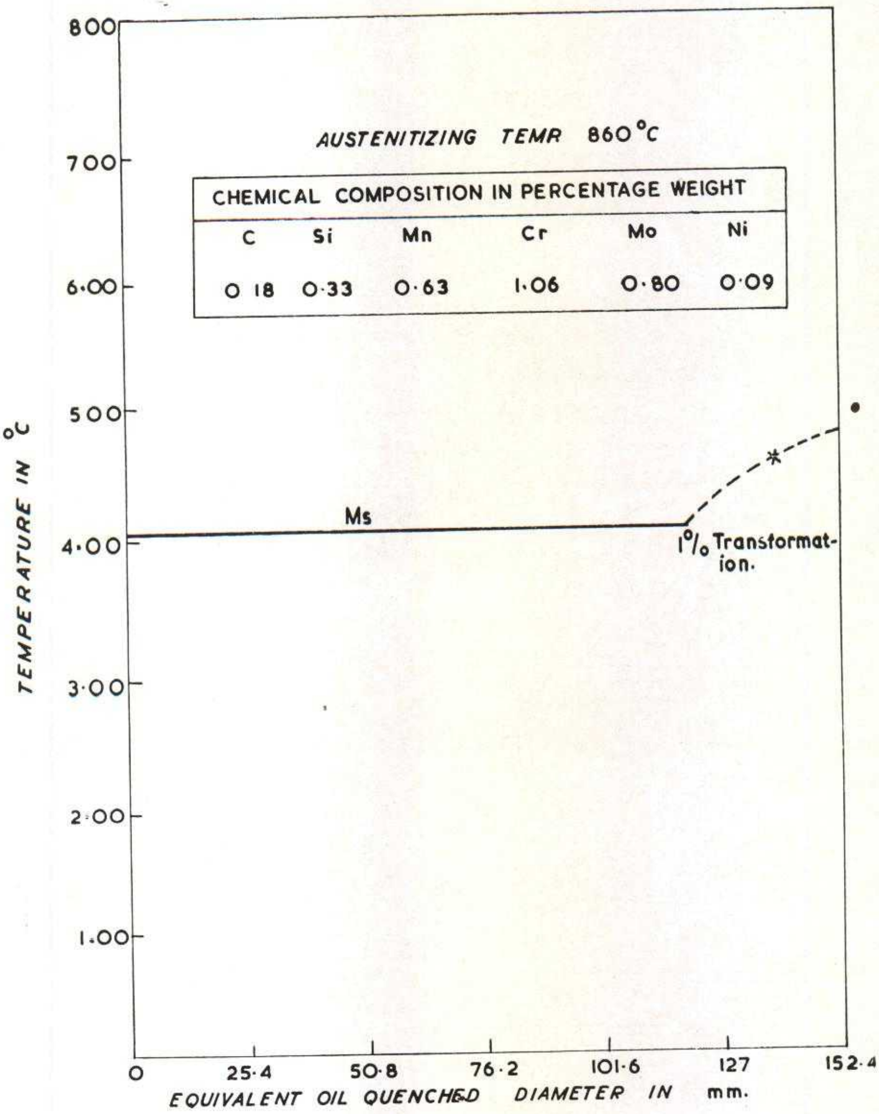


Fig. 33 — Continuous Cooling Transformation Diagram for Steel

way. The atmospheric corrosion tests were also undertaken.

Microscopical Observations

Micro-structure of normalized specimens was ferritic-pearlitic with very small amount of pearlite. The grain size was observed to be moderately fine having ASTM grain size No. 7. The hardness of ferrite base was also measured with the micro hardness tester. The value was found between 165 and 177 V.P.N. This showed that the strength of the steel was mainly due to solid solution hardening of ferrite. As the weldability and accelerated corrosion test results of the ·8 ton heat was not very satisfactory, it was decided to make some additional small experimental heats to modify the composition further. Six 10 kg. heats were made. The test results of these heats are given in Table 15. The weldability tests of the last three heats gave satisfactory results. The sub-zero im-

Table 15 — Test Results of Heats

Heat No.	Max. stress kg/sq. mm.	Yield stress kg/sq. mm.	Elongation % GL 1″	Reduction Area %	Remarks on weldability test
51	84·45	53·47	28	55	—
	54·87				
52	69·90	47·86	28	63	—
53	65·87	41·64	31	55	—
54	49·49	—	—	—	Weldable
41/3A	58·31	40·52	—	—	—
		43·91	37·5	73	Weldable
41/3B	59·66	41·64	34	64	Weldable
		48·43			

pact tests and corrosion tests of these heats are in progress.

Tonnage scale heats were taken up for extending the work on to industrial scale applications.

28.0 Development of Electrical Resistance Alloys for Heating Elements

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

Major work on the development of heating element alloys had already been completed and the process was leased to the industry. During the period under review, the work chiefly related to study of weldability of the alloy developed. For the production of heating element wires, it is often necessary to butt-weld the wire when these break during wire-drawing operation. Welding of the Fe-Cr-Al alloys is generally difficult due to excessive grain-coarsening and also due to oxidation of the constituent elements Cr and Al during the welding operation. This led to formation of weak and brittle weld zone which results in failure of the joint during subsequent wire-drawing operation. To overcome this difficulty, a study was undertaken to evolve a suitable method of butt-welding the alloys. For this purpose, an electric resistance micro-welding machine was employed. Varied conditions of pressure and heating current were used with and without flux.

Under various conditions of welding the wire, specimens with flat as well as tapered ends failed during subsequent drawing showing brittle fracture. Macro- and micro-examinations of the wires were examined. The macro-photograph (Fig. 35) showed heavily oxidized area as having cracks near the surface. These cracks are shown clearly in the photomicrograph (Fig. 36) as due to grain boundary oxidation.

Just under the oxidized zone, the grains were found to be comparatively smaller than those in the adjacent areas, where in turn, the grains were coarse while the parent metal consisted of small grains.

In other experiments, a continous flow of argon gas was applied during the welding operation. The wire ends to be joined were tapered to various degrees. The idea behind this was to control the percentage deformation in the welding zone and to fill up the tapered zone uniformly and

FIG. 34 — THE NEW 2 AND 3 PAISA COINS MADE OF ALUMINIUM-MAGNESIUM ALLOYS DEVELOPED AT NATIONAL METALLURGICAL LABORATORY AND GOVT. OF INDIA MINT, BOMBAY

completely. The pressure and temperature (which was controlled by the current) were adjusted to meet the above requirements. It was seen that neither high current low pressure nor low current high pressure was suitable for successful welding. Optimum current and pressure were needed for a good weld. Several wires welded under optimum condition were drawn successfully for 13 SWG to 19 SWG. A typical microstructure of the wire sample thus welded is given in Fig. 37. The weld zone of the sample showed much smaller oxidized areas with fine grains in the centre of the weld zone and coarser grains on the two sides. The work on this aspect was further pursued to co-relate the micro-structure obtained under different welding conditions with the behaviour of weld during wire drawing. Hot impact tests of the same heats were also conducted at 700°C. with a view to study the grain growth characteristic, but no general conformity was noticed with the grain growth behaviour as found in the welding experiments. Melting technique of electrical resistance alloys will be further perfected including the use of a synthetic slag of MgO-20 per cent+ CaO-45 per cent+Al_2O_3-35 per cent believed to produce sound ingots and improved mechanical properties.

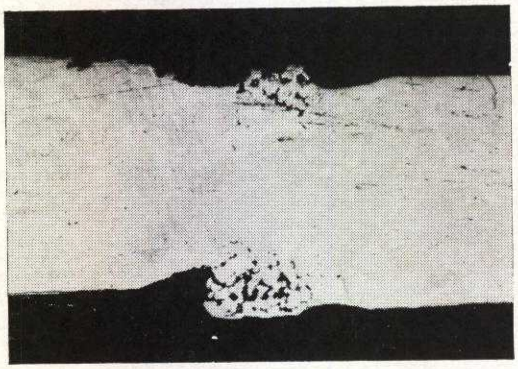


FIG. 35 — MACRO-PHOTOGRAPH OF WIRE SPECIMEN SHOWING OXIDIZED AREA

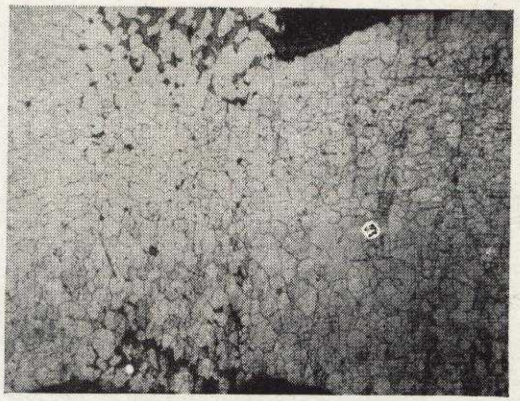


FIG. 36 — PHOTOMICROGRAPH OF WIRE SPECIMEN SHOWING CRACK DUE TO GRAIN BOUNDARY OXIDATION

29.0 Development of Magnetic Materials

(i) *Hard Ferrites*

During the period under review study was made on the magnetic properties of different ferrites in relation to the structure and amount of $BaO.6Fe_2O_3$ phase present. A relative estimation of this phase in ferrites of different " X-compound " content was carried out by X-ray diffractometric analysis. The diffractograms had shown that the intensity of line profiles of all the reflections especially (107), (114) and (203) from the hexagonal phase $BaO.6Fe_2O_3$ changed with change in composition. The relative change in intensity of the reflections was determined by calculating the areas covered by the line profiles after projecting them on a centimeter graph paper. It was found that the maximum area was covered by reflections from ferrite containing 2·65 per cent X, thus showing the presence of maximum quantity of the hexagonal magnetic phase in this composition. This composition had also shown the highest values of coercive force for any sintering time and temperature.

The nature of phases formed during calcination at different temperatures was investigated. The calcination was carried between 500 and 1000°C. and specimens were heated at a linear rate. The density measurements and X-ray diffraction of the calcined product were carried out for identification of the phases. The plot of density, against temperature showed a break at 910°C., which is the temperature for the formation of hexagonal phase. The X-ray diffraction studies revealed the presence of two phases (i) $BaO.6Fe_2O_3$, and (ii) an intermediate phase $Ba_8Fe_8O_{21}$ having fcc structure a — 8·05 Å. The intermediate phase forms between 800° and 900°C. only. Above 900°C. the hexagonal phase is the only structure which is formed.

Work was taken up on strontium ferrite in which strontium will be replaced in part by other alkaline earth oxides and low melting point oxides. A few compositions had been made and sintered up to 1300°C. for different durations. The properties of these sintered ferrites were investigated.

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

Manganese-aluminium intermetallic compound $Mn_{1\cdot 1}Al_{0\cdot 89}$ prepared by the powder metallurgy technique could not be sintered either in vacuum or in hydrogen atmosphere without catastrophic oxidation

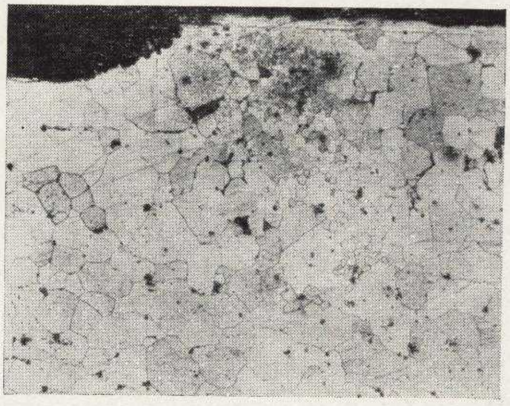


FIG. 37 — A TYPICAL MICROSTRUCTURE OF WELDED SAMPLE

of its compacts. Attempts were, therefore, indirect arc furnace. The alloy prepared could be heat-treated to develop ferromagnetism but could not be used for further mechanical working due to the presence of blow holes throughout the cast rod. The blow holes were formed from evolution of dissolved hydrogen picked up from the electrolytic bath for preparation of electrolytic manganese which was used in the preparation of the alloy. The electrolytic manganese was degassed in vacuum at temperature upto 500°C. and used for making the alloy by melting. The solidified alloy again showed the presence of numerous blow holes. Attempts were made to degass the manganese metal at higher temperatures. To improve the workability of the alloy and stability of the tetragonal ferro-magnetic phases additions of carbon and copper are now being tried.

(iii) *Soft Ferrites*

Work on soft ferrites was taken up to develop a technology and to study the suitability of indigenous raw materials for their manufacture.

Nickel-zinc ferrite rods suitable as radio antenna were prepared by extrusion of a plastic mass prepared from a mixture of nickel, zinc and iron oxides, plasticizers and binders. The extrusion was carried with dies which were designed in the Laboratory. About 10 in. long and 0·25 in. dia. rods could easily be extruded with the press available in the Laboratory. The extruded rods were dried and sintered at and above 1200°C. It was found that though disc rods could be prepared with shorter time of sintering, good magnetic properties were obtained only when the sintering run was 30 hours. Such rods when tested should have hystersis loop equal to that of the imported ones. Experiments are underway to prepare these rods using manganese oxide in place of nickel oxide and iron ore in place of pure

(iv) *Study of Electrical and Magnetic Properties of Some Low-Manganese-Low-Aluminium Steels*

It was shown earlier that steels containing about aluminium, 4; manganese, 1·5 and carbon, 0·03 per cent had very low values of coercive force between 0·2 and 0·5 oersteds and hysteresis loss 100 ergs/c.c./cycle. To exploit their good magnetic characteristics, it was considered necessary to prepare such steels on a fairly big scale to evaluate the possibilities of preparing them commercially. Accordingly, sixty pounds heat containing approximately Al, 3·5 and Mn, 1 per cent was prepared and hot rolled to 0·1 in. thick sheets. The 0·1 in. thick sheets were pickled, degassed and cold rolled to 0·005 in. thickness and annealed at 1100°C. after sealing in a mild steel box. The annealed sheets were tested for watt losses. The sheets showed much higher watt losses than hot rolled 4 per cent silicon sheets. It was found that the sheets had developed minute cracks during cold rolling and these were perhaps the cause of high losses. Arrangements are being made to make another 60 lb. and 1 ton heat for further testing.

30.0 Light Metals and Their Alloys

This project was taken up with a view to develop aluminium based alloys possessing high strength to weight ratio, adequate resistance for stress corrosion, covering the use of rare earth group of elements. Investigations were conducted in different fields of study relating to production of aluminium alloys with high percentages of magnesium, alloying effects with rare earth metals, working characteristics, stress corrosion and ageing characteristics at room and at elevated temperatures. The study of ageing characteristics was extended to conditions where the alloy contained different micro phases produced by the additions of minute quantities of silver and copper.

During the course of these studies, it was observed that additions of misch metal did not cause any significant change in the hardness of alloys on ageing, except in the Al-10 per cent Mg alloy where a small increase was observed. Addition of silver and copper improved the response to age-hardening at 250°C. of Al-7 per cent Mg alloys, the improvement was perceptible at about 24-40 hours after quenching from solution-treated temperature and reached a maximum in 60-100 hours. Further ageing resulted in fall in hardness, ultimately attaining a value somewhat higher than that of the solution treated samples. Changes in tensile strength and 0·1 per cent proof stress were also studied in samples treated under similar conditions or kept for long periods at room temperature. Both tensile strength and proof stress increased tensile strength, copper additions had the opposite effect. Earlier results showed that Al-7-10 per cent Mg alloys could be made workable by the additions of up to 5 per cent of misch metal to the melt. Misch metal additions were tried to find out if it was responsive to age-hardening treatment.

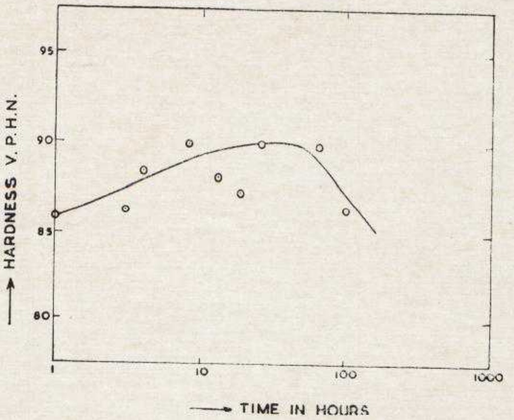


FIG. 38 — CHANGE IN HARDNESS WITH TIME

Solution treated samples prepared from Al-9 Mg-3 per cent misch metal and Al-10 per cent Mg-3 per cent misch metal alloys were aged at 100°, 150° and 200°C. for periods up to 300 hours. No significant change in hardness was observed in the case of the Al-9 per cent Mg alloys. In the Al-10 per cent Mg alloy, however, two hardness peaks were obtained; the first, after 2-3 hours of ageing at 100°C. and the second after about 75 hours ageing; the hardness dropped to the solution treated value after about 200 hours. Ageing at higher temperature gave identical results. Micro-examination of these samples did not show any significant transgranular precipitation after ageing, nor was any clustering of the precipitates, causing loss of hardess observed. Al-(6-7 per cent) Mg alloys containing 0·02 to 0·1 per cent silver were aged, after solution treatment for periods up to 1000 hours at 250°C. Changes in hardness of these samples are shown in Fig. 38. Hardness values were constant at 55-60 VPN for the first 18-20 hours, increasing to a maximum of 80-85 VPN the maximum rising with increasing additions of silver. After the peak value had been attained, the hardness began to fall attaining a value of about 70 VPN after 500 hours' ageing. Micro-structures of these samples in the solution-treated condition and at different stages of ageing were studied. It was observed that precipitation was both intergranular and transgranular. Effects of micro-additions of copper on Al-7 per cent Mg alloy were investigated by adding 0·02 to 0·1 per cent copper to the alloys and observing their response to age hardening at 250°C. for periods up to 1000 hours. Variations in hardness values are shown in Fig. 39. With the addition of copper also, hardness values remained constant at about 60 VPN for the first 20 hours and then rose to a maximum of about 85 VPN. With higher additions of copper to the alloy peak hardness was attained earlier.

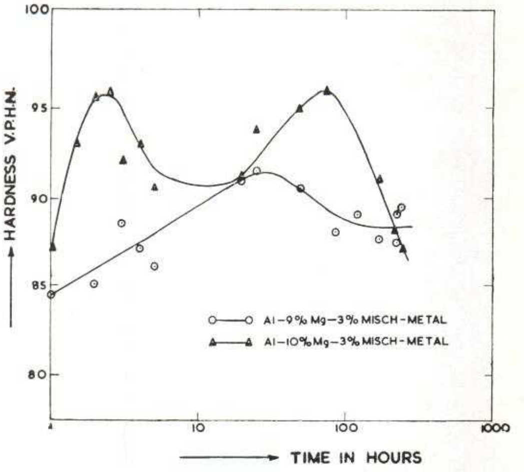


FIG. 39 — VARIATION IN HARDNESS VALUES

30.1 Aluminium-Silicon Alloys

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

A comprehensive study on the micro-structure, mechanical properties and fluidity on an eutectic and hypo-eutectic alloy (Si, 11·7 and Si, 9 per cent-aluminium alloys respectively) was made in their normal and modified conditions. These alloys were treated with the modifying agents like sodium, phosphorus and sulphur separately and in combination with each other. The results were correlated and a report prepared. The effect of the different modifying agents on microstructure, mechanical properties, etc., on certain commercial aluminium base piston alloys are under study. A paper entitled "A new method of modification of Al-Si alloys" was presented at the International Foundry Congress held at Amsterdam in September, 1964. The process is under release for commercial scale exploitation in Europe where it has attracted considerable attention.

30.2 Aluminium Bronzes

The Project was taken up to study the melting and remelting of aluminium bronzes in indirect electric arc furnace with and without fluxes in order to reduce/eliminate the introduction of gases and oxide inclusions in the cast products; and to develop a suitable flux to serve the aforesaid purpose. Further work was undertaken in the production of aluminium-bronze where iron addition was made as hoop iron strips, in place of iron-aluminium alloy. The heats were made in a 17 kW. indirect electric arc furnace and 40 kW direct electric arc furnace. Iron could conveiently be used in place of iron-aluminium alloy. The use of electric furnaces considerably lessened the melting period with consequent ease in attaining high temperature. Heats were made with and without the use of flux (50 : 50 KCl : NaCl) and with the use of CCl_4 in the ingot mould. It was revealed that the use of flux greatly reduced the oxide formation and gave a sound product. Heats were made with iron aiming from 2 to 5·5 per cent, manganese 0·2 to 2·0 per cent and aluminium 9·5 to 10·5 per cent. The effect of chemical composition on mechanical properties is under study and a few microphotographs are given in Figs. 40-43 to illustrate the influence of composition on microstructure.

30.3 Foam Aluminium

Preliminary work was undertaken to develop a simple method of the preparation

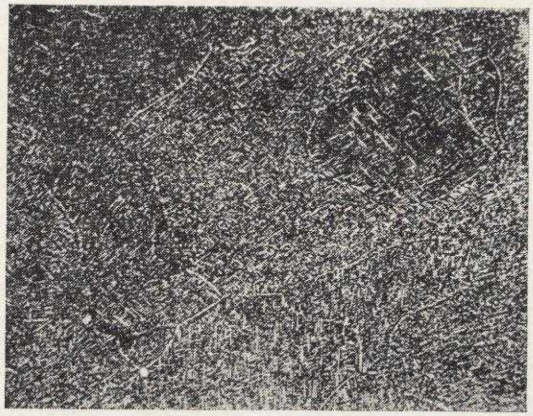


Fig. 40 — Micro-photograph of Heat Having the Composition Al, 10·74, Fe, 1·85, Mn, 0·6 per cent, Cu, Balance

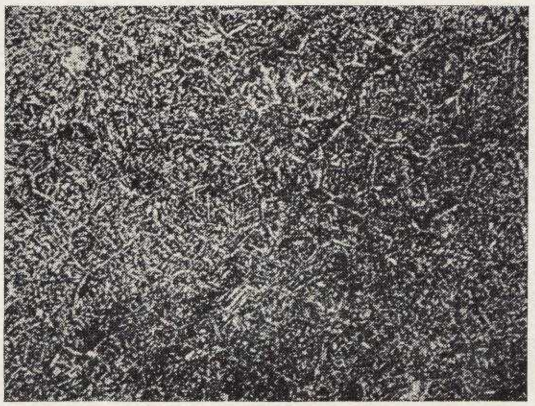


Fig. 41 — Micro-photograph of Heat Having the Composition Al, 10·8, Fe, 4·47, Mn, 0·7 per cent, Cu, Balance

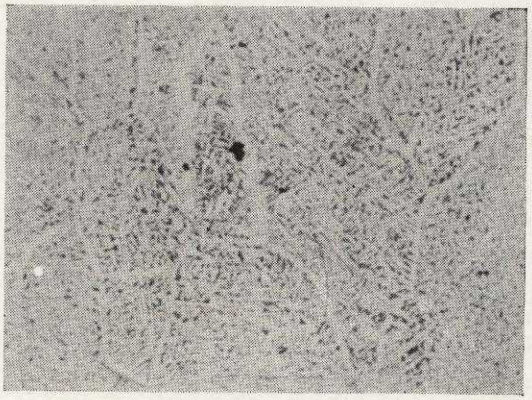


Fig. 42 — Micro-photograph of Heat Having the Composition Al, 10·0, Fe, 2·0, Mn, 0·5 per cent, Cu, Balance

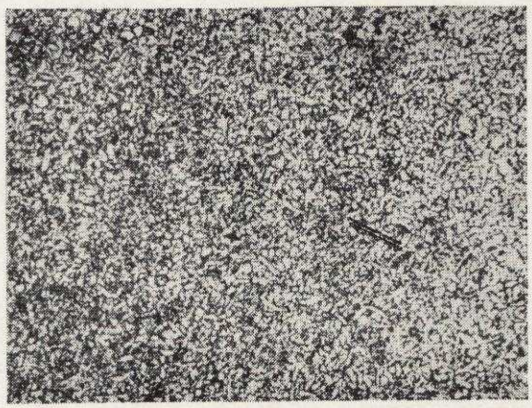


Fig. 43 — Micro-photograph of Heat Having the Composition Al, 9·4, Fe, 4·06, Mn, 1·22 per cent, Cu, Balance

of foam aluminium, using certain foaming agents such as titanium hydride, sodium metaborate, etc. Further work on the subject is in progress.

31.0 Study of the Properties of Indigenous Foundry Moulding Materials

This broad based 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 casting. During the period under review, investigations were completed on twenty sand samples and four bentonite samples. A summary of the work done is given below :

(i) *Godavari River Sand*

Two samples were received from the Director of Geological Survey of India, Andhra Pradesh.

Sample No I: It was a high silica sand having A.F.S. G.F. No. 45·95 and clay content 0·5 per cent. It contained coarse to fine grain sand of sub-angular to sub-rounded in shape. It contained 86·08 per cent silica. The sample essentially consisted of quartz and feldspar along with minor quantities of calcite, hornblende and rutile. Dark opaque minerals were also present in abundance. Sintering range of the sand was 1300°C. Study of the moulding characteristics showed that the sand could be used for non-ferrous and cast iron castings.

Sample No. 2 : It was also a high silica sand having A.F.S. G.F. No. 28·11 and clay content of 1·42 per cent. It contained coarse to fine-grained sand of sub-angular to sub-rounded shape. It contained 86·64 per cent silica. The sample essentially consisted of quartz with minor amounts of feldspar along with calcite, horn-blende, rutile, muscovite and mica. Dark opaque grains were present abundantly. Sintering range of the sand was 1300°C. Study of the moulding characteristics showed that the sand can be used for non-ferrous and cast iron castings after mixing it with fine grained silica sand or silica flour.

(ii) *Glass Sands*

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

(a) *Panchapahra Location* — It was a high silica sand having A.F.S. G.F. No. 51·56 and clay content of 1·66 per cent. It contained 98·98 per cent silica. It contained medium to fine-grained sand of sub-angular to sub-rounded shape. The sample essentially consisted of quartz along with minor amounts of feldspar. Black opaque grains were also present in traces. Sintering range of the sand was above 1350°C. Study of the moulding characteristics showed that the sand could be used for light and medium steel castings.

(b) *Bhainsahi Location* (*upper unit*) — It was also a high silica sand having A.F.S. G.F. No. 56·09 and clay content of 1·0 per cent. It contained 98·86 per cent silica. It contained medium to fine-grained sand of sub-angular to sub-rounded shape. The sample essentially consisted of quartz with small amounts of feldspar. Ferro-magnesian minerals, rutile, muscovite, chlorite were also present. Sintering range of the sand was above 1350°C. Study of the moulding characteristics showed that the sand could be used for light and medium steel castings.

(c) *Manduri Location* — This was a silica sand of A.F.S. grain fineness No. 60·6 and contained 1·5 per cent A.F.S. clay matter. Petrological examination of the sand revealed it to contain quartz with minor amounts of feldspar, traces of calcite and a few opaque grains. The grains were sub-angular to sub-rounded in shape. Its silica content was 97·3 per cent. The sintering range of the sand was above 1350°C. Study of the moulding characteristics showed that the sand could be used in iron and steel foundries.

(d) *Bhandhana Location* — This was a high silica sand of A.F.S. grain fineness No. 64 containing 2·5 per cent A.F.S. clay matter. The shape of the sand grains was sub-angular to sub-rounded. It contained about 98·7 per cent silica. The mineral constituents were quartz in predominence and minor amounts of feldspar, while a few grains had ferruginous coating over them. The sample had a sintering range above 1350°C. The sand was found to be suitable for light and medium steel castings and could be considered for heavy steel castings on blending it with coarser silica sand.

(iii) *Rajendra Silica Sands*

The sand samples were received from Messrs Rajendra Silica Works, Sankargarh, Allahabad.

(a) *Sample No.* 2 — It was a high silica sand having A.F.S. G. F. No. 57·56 and clay content of 1·78 per cent. It contained 96·86 per cent silica. The sand grains were medium to fine in size while their shape was sub-angular to sub-rounded. The sample essentially consisted of quartz with traces of other impurities. Sintering range of the sand was above 1350°C. Study of the moulding characteristics showed that the sand could be used for light and medium castings.

(b) *Sample No.* 4 — It was a high silica sand having 94·8 per cent silica and contained 4·1 per cent of A.F.S. clay grade matter. Its A.F.S. grain fineness No. was 70, and the grains were sub-angular to sub-rounded in shape. The sand mainly contained quartz (95 per cent), little feldspar and traces of other impurities. Sintering range of the sand was above 1350°C. Study of the moulding characteristics showed that the sand was suitable for steel castings of light to medium size. On combination with coarser sand it might be used for moulding in heavy steel castings.

(iv) *Shertallai Silica Sand*

This was received from Geological Survey of India, Kerala. It was a high silica sand of G.F. No. 37 and clay content 0·2 per cent as per A.F.S. Specification. Its silica content was 99·3 per cent, mainly in the form of quartz. Other minerals were feldspar and muscovites. The sand had its sintering range beyond 1350°C. Study of the moulding characteristics showed that the sand could be used in iron and steel foundries. However, for obtaining good surface finish suitable mould washes may have to be applied or mixed with fine grained silica sand in suitable proportions.

(v) *Damodar River Sands*

Two samples were received from Mr. N. G. Chakravorty, Calcutta :

(a) *Sample No.* 1 — This river sand of A.F.S. G.F. No. 46·8 and contained 1·6 per cent A.F.S. clay. Petrologically it consisted of mainly quartz with minor amounts of feldspar, hornblende, muscovite, and calcite. The sand grains had sub-angular to sub-rounded shape. The sand contained silica, 85·5; alumina, 7·4 and iron oxide, 2·7 per cent. Its approximate sintering point was found to be 1250°C.

(b) *Sample No.* 2 — This, also, was a river sand having A.F.S. grain fineness No. 40·9 and contained 0·46 per cent clay matter. Petrologically it consisted of mainly quartz with minor amounts of feldspar and traces of hornblende, muscovite, calcite, etc. The shape of the grains was sub-angular to sub-rounded. It contained silica, 86·6 per cent the rest being alumina, iron oxide and alkalies. Approximate sintering point was determined to be 1300°C.

Both the sand samples, that is, No. 1 & 2 showed that these were not suitable for steel castings in the as-received states. However, they can safely be used in the iron and non-ferrous foundry jobs after addition of fine silica sand.

(vi) *Madras Sands*

Three sand samples were received from Section Officer, P.W.D., Madras to assess their suitability for foundry purposes. All the samples were collected from site No. 9.

(a) *Brown sand* (*Top layer*) — The sand was a coarse grained sand having A.F.S. G.F. No. 46·43 associated with 0·44 per cent A.F.S. clay grade matter. It assayed 94·28 per cent silica with minor amounts of alkalies. The grains were sub-angular to sub-rounded in shape. Mineralogically it contained quartz in predominance with small amounts of feldspar and ferro-magnesium minerals. The sintering temperature was above 1350°C. The sand may be used for steel castings.

(c) *White Sand* — The white sand was a medium grained (G.F. No. 44·30) with an A.F.S. clay content of 0·36 per cent. The sample assayed 96·16 per cent silica along with traces of alkalies in the as-received state. The grains were sub-angular to sub-rounded in shape. Mineralogically it consisted of quartz in predominance with little amounts of feldspar. The sintering range was very high (above 1350°C.). The sand may be made use of in the steel foundries.

(vii) *Hardwar River Sand*

The sand sample was supplied by Messrs Bharat Heavy Electricals (India), Hardwar Ltd. to study its suitability for foundry moulding purposes. The sand was medium to fine grained with an A.F.S. G.F. No. 49·87 and A.F.S. clay content of 0·6 per cent. The sand assayed 92·62 per cent silica along with little amounts of alkalies in the as-received state. The grains were sub-angular to sub-rounded in shape. Mineralogically it consisted of mainly quartz and feldspar along with minor amounts of hornblende, muscovite, chlorite, rutile and garnet both in the as-received and washed states. The sintering range was found to be 1250-1300°C. In view of the high permeability, moderate refractoriness, good green strength properties and three sieve distribution, the sand can be successfully made use of for moulds in cast iron and non-ferrous castings.

(viii) Investigation work on the following sand samples received from Directorate of Geology, U.P. are in progress and the results so far obtained on these are given below :

(a) *Bhainsahi Location* (*Lower Unit*) : *White in Colour* — It was also a high silica sand having A.F.S. G.F. No. 67·4 and clay content of 3·6 per cent. It contained 96·47 per cent silica. It contained fine-grained sand of sub-angular to sub-rounded in shape. The sample largely consisted of quartz with small amounts of feldspar, hornblende, ferro-magnesian minerals and few micaceous flakes.

(b) *Bhainsahi Location* (*Lower unit*) *yellow in colour* — It was also a high silica sand having A.F.S. G.F. No. 72·06 and clay content of 3·76 per cent. It contained 96·82 per cent silica. The sand grains were fine and sub-angular to sub-rounded in shape. The sample largely consisted of quartz with minor amounts of feldspar. Ferro-magnesian minerals, muscovite and dark opaque grains were also present in traces.

(c) *Kanchanpur Location* (*Lower Unit*): *White in Colour* — The sample received from D.G.M., Lucknow, was a high silica sand having A.F.S. G.F. No. 56·9 and contained 1·4 per cent A.F.S. clay. Petrological examination revealed that the sample consisted predominantly of quartz and traces of ferro-magnesian minerals. Shape of the grains was sub-angular to sub-rounded. Sample contained 99·3 per cent silica.

(d) *Kanchanpur Glass Sand* (*Lower Unit*): *Yellow in Colour* — The sample received from D.G.M., Lucknow was a high silica sand having A.F.S. G.F. No. 58·1 contained 1·3 per cent clay. Petrological examination showed it to consist predominantly of quartz with minor amounts of feldspar, traces of calcite and ferro-magnesian minerals. It contained 98·7 per cent silica content. The shape of sand grains sub-angular to sub-rounded.

(e) *Amagonda Location* — The sand sample was medium to fine grained and was associated with 1 per cent A.F.S. Clay content. The sand assayed 98·84 per cent silica, with minor amounts of alumina and alkalies. The grains were sub-angular to sub-rounded in shape. Mineralogically it consisted of quartz with very small amounts of feldspar. Traces of rutile, muscovite and ferro-magnesian minerals were also present. The sintering temperature of the sample was beyond 1350°C. The sand may be successfully used in steel foundry.

(ix) *Preparation of Crude Silica Samples for Use in Foundries and for Glass Making*

Three samples of crude silica rock namely A (ASS), B(ASQ) and C (KTBJ) each weighing about 240 kg. and assaying SiO_2, 94·69; Al_2O_3, 2·53; Fe_2O_3, 0·87; CaO, 0·15; MgO, 0·41 per cent; SiO_2, 91·03; Al_2O_3, 5·5; Fe_2O_3, 0·56; CaO, 0·22; MgO , 0·63 per cent and SiO_2, 88·91; Al_2O_3, 7·45; Fe_2O_3, 0·53; CaO, 0·27; MgO, 0·96 per cent respectively, were received from Messrs Lakshmi Prasad Sao, Chaibasa; for making them suitable for use in foundries and glass making. Preliminary crushing, grinding and sieve analysis are in progress.

(x) *Production of Standard Sized Sand for Foundry Purposes from Beach Sands of Ennore, Madras*

A sample of beach sand from Ennore, Madras, weighing about a tonne was received for determining the best method of producing standard sized foundry sand of size $-30+50$ mesh (A.S.T.M.) (or 28 $+48$ Tyler). The sample was of white quality sand composed mostly of quartz, some feldspar, and minor amounts of zircon, sillimanite, ilmenite and monazite, etc. The heavy minerals in the sand sample constituted 0·5 per cent by weight. The sand as received contained 9·7 per cent $+28$ mesh, 79·3 per cent $-28+48$ mesh and 11 per cent -48 mesh (Tylers) by weight. The $-28+48$ mesh was the size fraction required and, hence, attempts were made to eliminate the coarse fraction by screening at 28 mesh size.

The following separation methods were tried for the sample. (1) Mechanical screening by 28 mesh and 48 mesh screens in vibrating screen. (2) Mechanical screening at 28 mesh followed by tabling for removal of -48 mesh and (3) Tabling only for rejecting the $+28$ and -48 fractions. In the first case, mechanical screening using double deck vibrating screen gave imperfect separation due to blinding especially in the fine sizes and the separation was not efficient.

Screening at 28 mesh to eliminate the $+28$ mesh fraction, followed by tabling of the undersize eliminated most of -48 mesh size sand and produced the desired size fraction of sand. Straight tabling of the sand produced three products, namely coarser fraction, finer fraction and a middling product were collected. The middling contained the sized material required. Hydraulic classification was not attempted due to the presence of different gravity materials including heavy minerals.

Bonding Clays :

Following bentonite samples were investigated during the period under review :

(i) *MP*-33 *& PHG Bentonites*

Samples were received from Ambica Minechem Industries, Bhavnagar, Gujarat. The samples were lumpy in form and yellowish in colour.

(a) *MP*-33 *Bentonite* — The bentonite sample was yellowish in colour and lumpy in form in the as received state. It was associated with moisture content of 11·60 per cent and had a *p*H value of 9·55. Both these values agreed with the specifications for Western bentonites laid down by S.F.S.A. It assayed silica, 42·20 per cent; alumina, 17·57 per cent; iron oxide, 11·55 per cent and total alkali oxides, 7·99 per cent. The ratio of sodium to calcium was 2·586. Its gel swelling index and total base exchange capacity values were respectively found to be 27,14·28 and 80·0 milli-equivalents per 100 gm. of the raw sample. The sintering range of the sample was 950°-1025°C. Petrological examination of the sample revealed it to consist of predominantly montmorillonite and nontronite in traces. Study of the bonding characteristics with high silica Rajmahal sand showed that it developed satisfactory green and dry strengths. The bentonite was a sodium base bentonite and may be made use of as a binder in the steel foundries.

(b) *PHG bentonite* — It was associated with 13·10 per cent moisture which was rather high as compared to the value laid down for the Western bentonites by S.F.S.A., whereas *p*H value of 9·5 was in agreement with the tentative specification for Western bentonites. It had a sodium/calcium ratio of 2·63. Its gel value, swelling index and total base exchange capacity values were 28·5, 14·28 and 73·6 milli-equivalent per 100 gm. respectively. The sintering range of the sample was 1025°-1100°C. The sample assayed 47 per cent silica, 14·21 per cent alumina, 9·96 per cent iron oxides and 7·25 per cent total alkalies. Mineralogically it consisted of predominantly montmorillonites and minor amounts of nontronite. Study of the bonding characteristics with Rajmahal silica sand of G.F. No. 56·7 showed that it developed satisfactory green and dry strength properties. The sample may be satisfactorily used as a binder in iron and steel foundries.

(ii) *B.R. & B.S. Bentonites : (Received from Messrs Hargovindas Shivlal and Company, Bombay)*

These two samples of bentonites are mined in Ghogha and Lakhanka respectively in Gohilwad district of Gujarat State. These are estimated to be available to the extent of a million tons each. Both the samples were light yellow in colour.

(a) *BR Bentonite* — The sample contained only 3·4 per cent moisture in the as-received state which is lower than the specification of SFSA. Its *p*H-value was determined to be 9·15. It showed values of 1·54, 12, 0·181 and 23·4 meq./100 gm. respectively for swelling index, gel value, Na/Ca and total base exchange capacity. On testing its refractoriness, the bentonite showed a sintering range of 1125 to 1175°C. Chemically it contained SiO_2, 48·4; Al_2O_3, 18·17; FeO and Fe_2O_3 5·93 per cent. The oxides of the alkali oxides were 1·05 and 1·26 per cent that of sodium and potassium respectively. The loss on ignition was shown to be 13·26 per cent. Petrological assessment of the sample revealed that it mainly consisted of beidellite which was confirmed by the D.T.A. test. Other minerals were feldspar, quartz and illite in minor quantities. Moulding characteristics of the bentonite in conjunction with the Rajmahal silica sand of G.F. No. 55, showed poor values with regard to green and dry strength values.

(b) *BS Bentonite* — This sample was associated with only 2·1 per cent moisture, much lower than the SFSA specification. Its *p*H value was determined to be 8·8. On testing for swelling index, gel value, Na/Ca and T.B.E. capacity the values of 1·66, 10·5, 0·177 and 21·4 meq./100 gm. respectively were found. Sintering range of the sample was 1125 to 1175°C. Its main chemical constituents were SiO_2, 48·56; Al_2O_3, 15·36; Fe_2O_3 and FeO, 6·40 per cent. Oxides of Mg and Ca were respectively 4·86 and 6·21 per cent. Sodium oxide amounted to 2·42, potassium oxide 0·94 and titanium oxide 0·8 per cent. Loss on ignition was found to be 14·52 per cent. Petrological examination revealed it to be mainly beidellites contaminated with quartz and feldspar followed by mica flakes. The presence of beidellites was also indicated by the D.T.A. test. It developed poor moulding characteristics when bonded with Rajmahal silica sand of G.F. No. 55.

31.1 Particle Size Determination of Indigenous Clays and Their Properties

Particle size of the clay mineral is one of the important factors in developing the bond strength. The clay particles are charged electrostatically and are responsible for the ionic bond, and in a clay sample the binding force for particles of uniform charge density would vary directly with the surface area of the particle. It has, however, been observed that particle size variation of any clay is an inherent function of its sources and the similarity in the grading is suggestive of the similarity of mineral constituents. The above observations, however, do not take into account the exchangeable cations and base exchange capacity which have pronounced effect on the physical properties of clay minerals. Experiments were, therefore, carried out to determine the particle size of the clay minerals and to examine the effect on the physical behaviour of moulding sands bonded with these.

The determination of particle size was carried out by the hydrometer method after treatment of the bentonite slurry with 12·5 per cent sodium hexametaphosphate. The clay particles were then classified as "true clay" measuring 5 microns and less, "silt" having particles in the range of 5-50 microns, "fines" ranging between 50-74 microns and "bondable material" having size greater than 74 microns. Other properties such as, the ratios of Na to Ca, total base exchange capacity and swelling indices of the true clay content of the bentonites were also determined. The mineral composition of the bentonite samples was assessed by petrological examination which was supported by D.T.A. test in most of the cases. To study the physical behaviour of these bentonites, sand mixtures were prepared with 5 per cent of the bentonite and tested for green and dry strength properties.

Correlation of the properties of bentonites with the particle size classification revealed that :

(i) In general, the similarity on the size grading of bentonites indicate similarity in their mineral composition.

(ii) The swelling index values of the bentonites containing approximately pure montmorillonite increased with increasing amount of the clay content. However, the bentonite samples containing predominantly beidellites showed low values of swelling index and did not show any correlation with their true clay contents.

(iii) The total base exchange value of the bentonite samples increased with the increase in the true clay content.

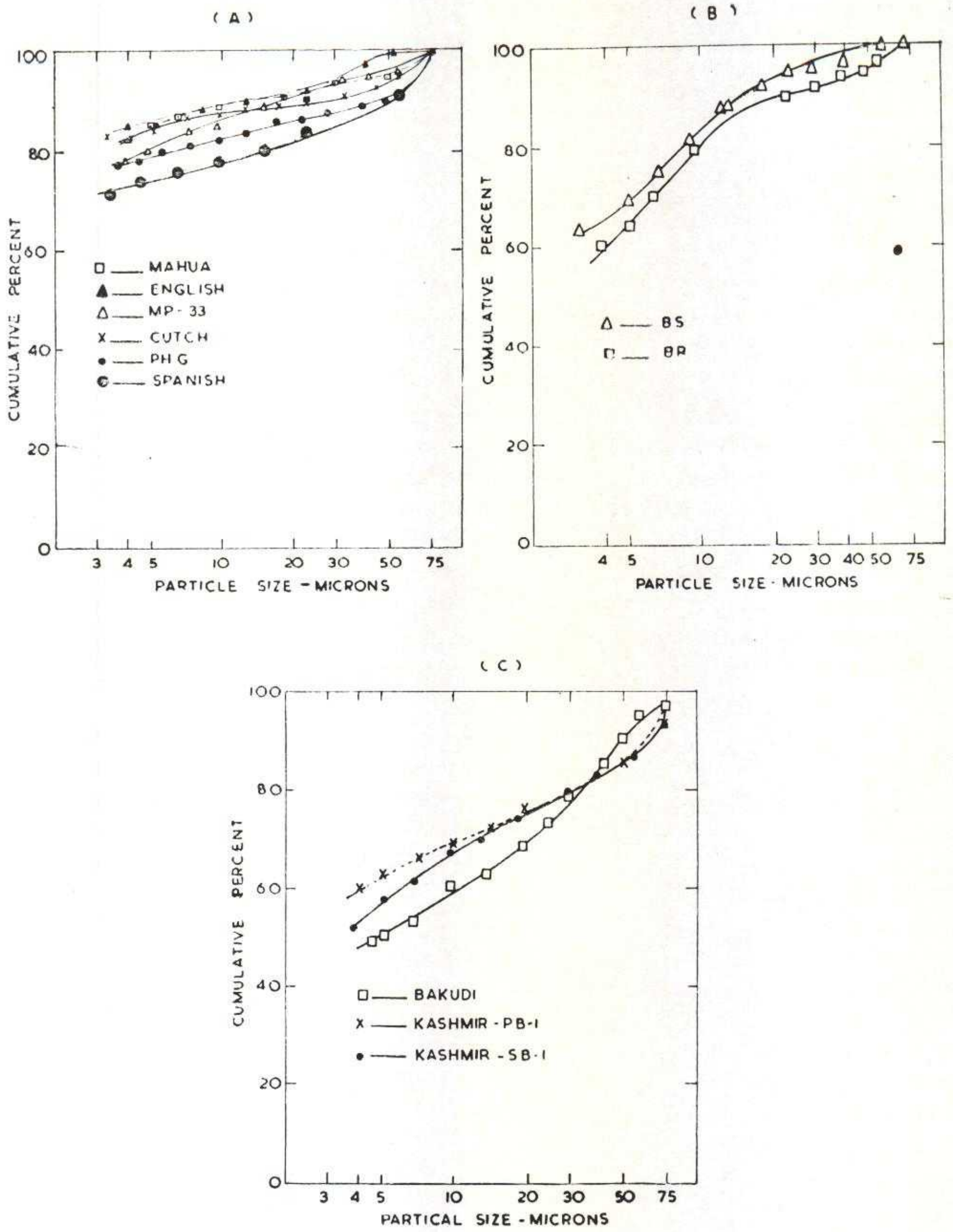


FIG. 44 — CUMULATIVE GRADING CURVES OF BENTONITE SAMPLES

(iv) The true clay content of the bentonite was found to influence the dry strength of the sand mixtures. The higher the clay content of the bentonite, the higher was the dry strength developed. However, such an effect was not observed in the case of green strength values.

31.2 Expansion Characteristics of Indigenous Sands

In the production of quality castings, expansion of the sand grains at elevated temperature is important since due to uncontrolled expansion such defects as rat-tails, scabs, buckles, etc., occur in the castings. In the present study, the expansion characteristics of four typical sands were examined and correlated with those characteristics obtained during actual casting trials. Tests were done on four sands, that is, Rajmahal sand, Manduri sand, Premier sand and Zircon sand. The expansion was measured on thrice rammed sand sample bonded with 5 per cent bentonite and other additives and properly tempered. The size of the specimen was 1/$\frac{1}{8}$ in. × 2 in. which was inserted in the Dieters' thermolab using a fused quartz micrometer activating on a dial scale to indicate the expansion. The study also included the effects of clay moisture and additives such as, coal dust, iron oxide and dextrine. Finally actual casting trials were made in the moulds prepared by the sand mixtures as studied above for expansion tests.

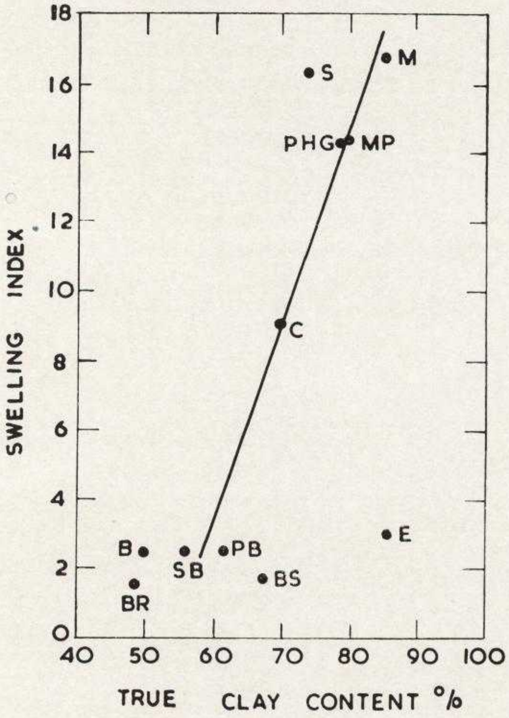


FIG. 45 — EFFECT OF TRUE CLAY CONTENT ON THE SWELLING INDEX OF BENTONITES

On the basis of the test data it was concluded that the study of the expansion characteristics of the moulding sand is important in order to overcome the defects, such as, scabs, rat-tails, buckles etc. The shock-free expansion of the sand can be lowered by cement bonding or by using zircon sand in the sand mixtures. The addition of 2 per cent of coal dust or iron oxide lowered the expansion slightly, while an appreciable decrease in the expansion was obtained in the case of an addition of 6 per cent coal dust only. Increase of moisture increased the expansion slightly at higher temperature only. The different bentonites, viz. Cutch, Mahua and Bakudi, did not affect the expansion to any appreciable degree.

31.3 Shell Moulding

The shell moulding process is considered as one of the greatest technological developments in the foundry industry. It has got several advantages over the conventional sand casting and is expected to become very popular with the Indian Foundries. The advantages of shell moulding process are exceptionally good surface finish and dimensional accuracy, hence, elimination of machining operations, decreased casting weight variation and less cleaning cost.

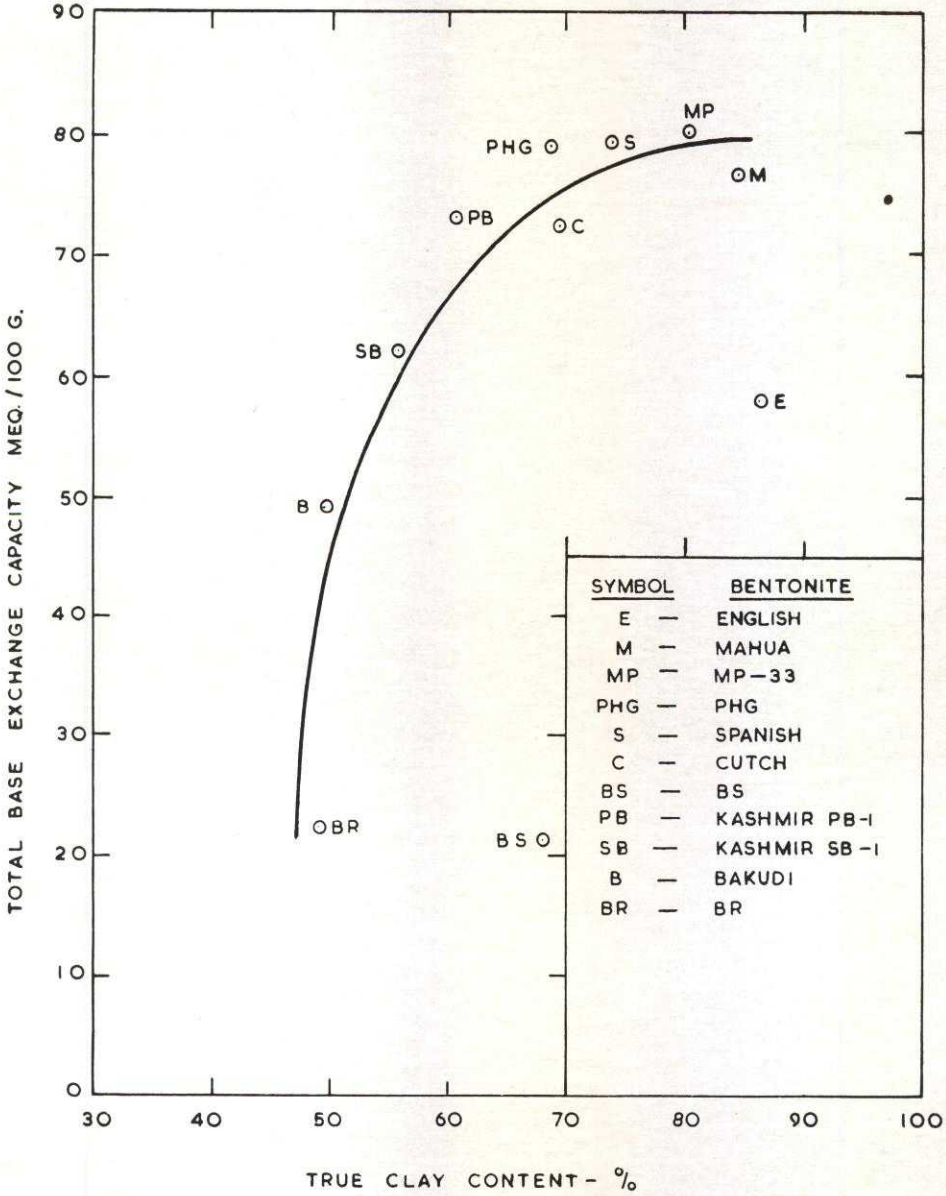


Fig. 46 — Effect of True Clay Content on the Total Base Exchange Capacity of Bentonites

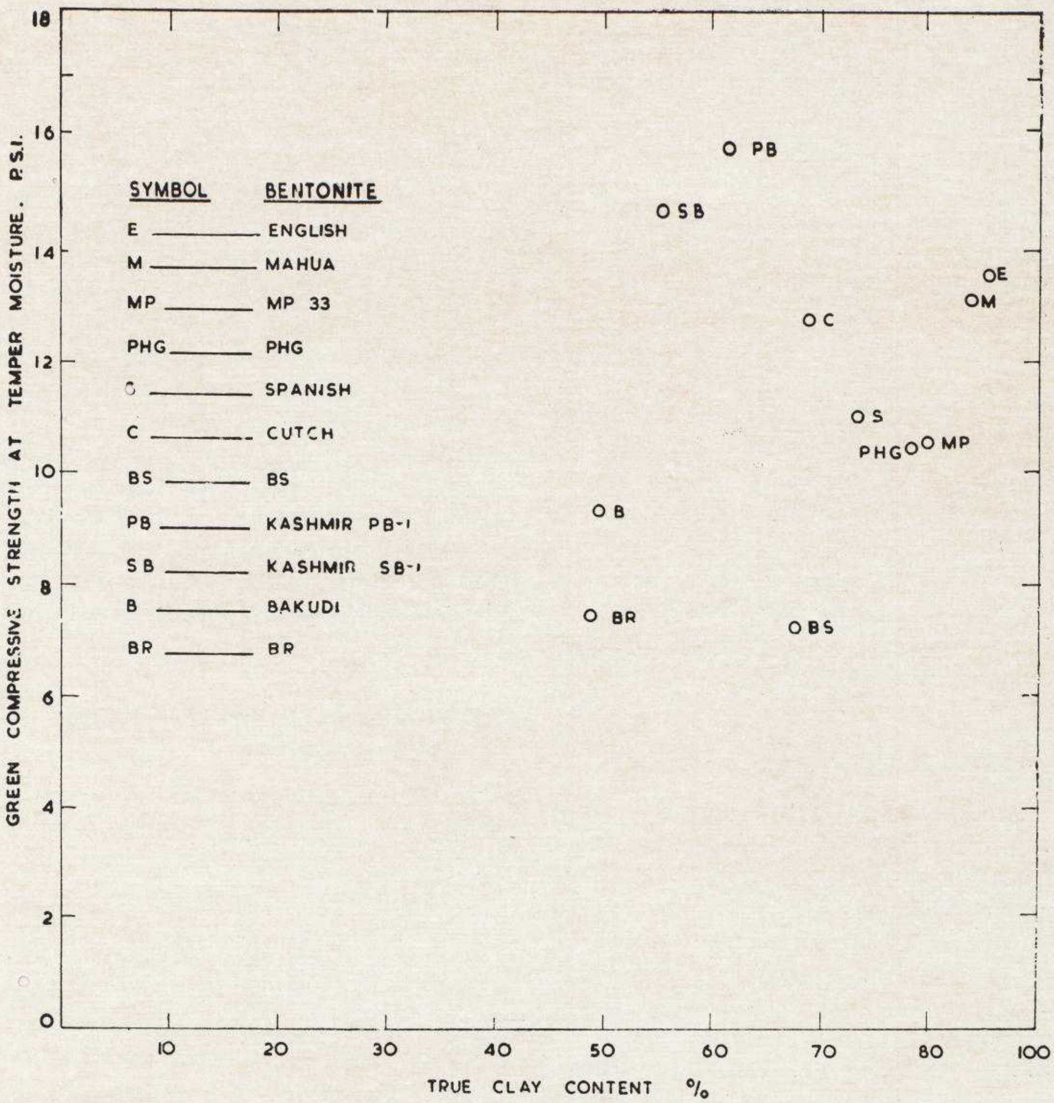


Fig. 47 — Effect of True Clay Content on Green Compressive Strength at Temper Moisture Content

Work was taken up on this project with a view to enumerating the availability of the raw materials such as sand, resin, etc., and study their suitability for this process.

The process consists of mixing a thermosetting resin with a clay free high silica sand having fineness between 90 and 140 G.F. No. which was then applied on a preheated metallic pattern plate. The phenolic type of resin partially thermosetts and builds up coherent sand shell next to the pattern. The thickness of this shell is related to pattern temperature, dwell time on the pattern, etc. Fig. 52 shows a shell made for magnet casting on the Crowing F.6 type shell moulding machine. The cope and drag portion of the shells are then assembled by means of a shell fuser (Fig. 53) and poured.

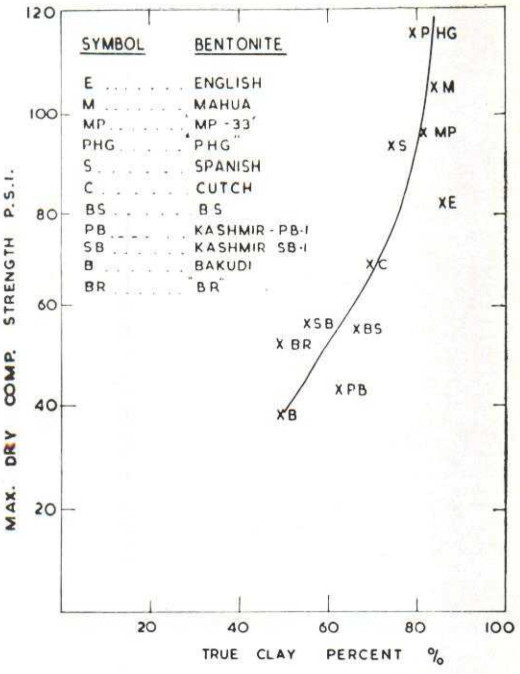


FIG. 48 — EFFECT OF TRUE CLAY CONTENT ON THE DRY COMPRESSIVE STRENGTH OF BENTONITE

31.4 Hot Blast Cupola

A hot blast cupola (having a capacity of 1 ton/hour and an internal diameter 18 in.) has been set up at the National Metallurgical Laboratory. The recuperator attached to the cupola is of radiation type having about 3′-8″ diameter and 34 feet in length. The flue gases from the cupola are mixed with excess of air burnt in the combustion zone of the recuperator and ascend through the central portion to the stack whilst the cold air from the blower descend through the annular space and in the process get pre-heated to about 450°C. The pre-heated air is then led into the tuyères of the cupola. The recuperator and the hot air pipes are insulated by means of glass wool.

32.0 Powder Metallurgy

During the period under review, further work was continued on the preparation of copper powder by electro-deposition using soapnut extract. Preliminary Project Report on the production of electrolytic copper was circulated to the industry for commercial scale exploitation of the process. Investigations carried out during the period have confirmed that soapnut extract has a controlling influence on the characteristics of the copper powder obtained. It is thus possible to obtain powders of controlled apparent density and sieve analysis. The process developed can produce copper powders of any required particle size.

Relating to work on the manufacture of porous bronze bearings it was found that certain necessary additional reagents such as iodide and phosphate simplified the manufacturing conditions and addition of nitrate gave rise to porosity in the bearings. This procedure avoided the necessity of precision control and standardization of the process.

33.0 Production of Iron Powder by Direct Reduction

Investigation into the direct reduction characteristics of iron ore samples received from Messrs Electric Control Gear Mfg. Co., Ahmedabad, Messrs Orient Ltd., Margao, Goa, and Director of Geology and Mining, Government of Maharashtra, Nagpur were carried out and detailed studies were made. The chemical analyses of the iron ores are given in the Table 16:

Iron ore received from Messrs Electric Control Gear Mfg. Co. and Messrs Orient

Table 16 — Chemical Analysis of Iron Ore Samples

Percentage composition	Electric Control Gear Mfg. Co. Ltd., ore	Orient Ltd., ore	Govt. of Maharashtra ore
Fe	0·68	67·20	63·70
SiO_2	1·22	0·96	1·72
Al_2O_3	0·21	0·40	1·62
MnO_2	—	0·15	—
sulphur	Trace	0·12	Trace
Phosphorus	0·01	0·06	0·06

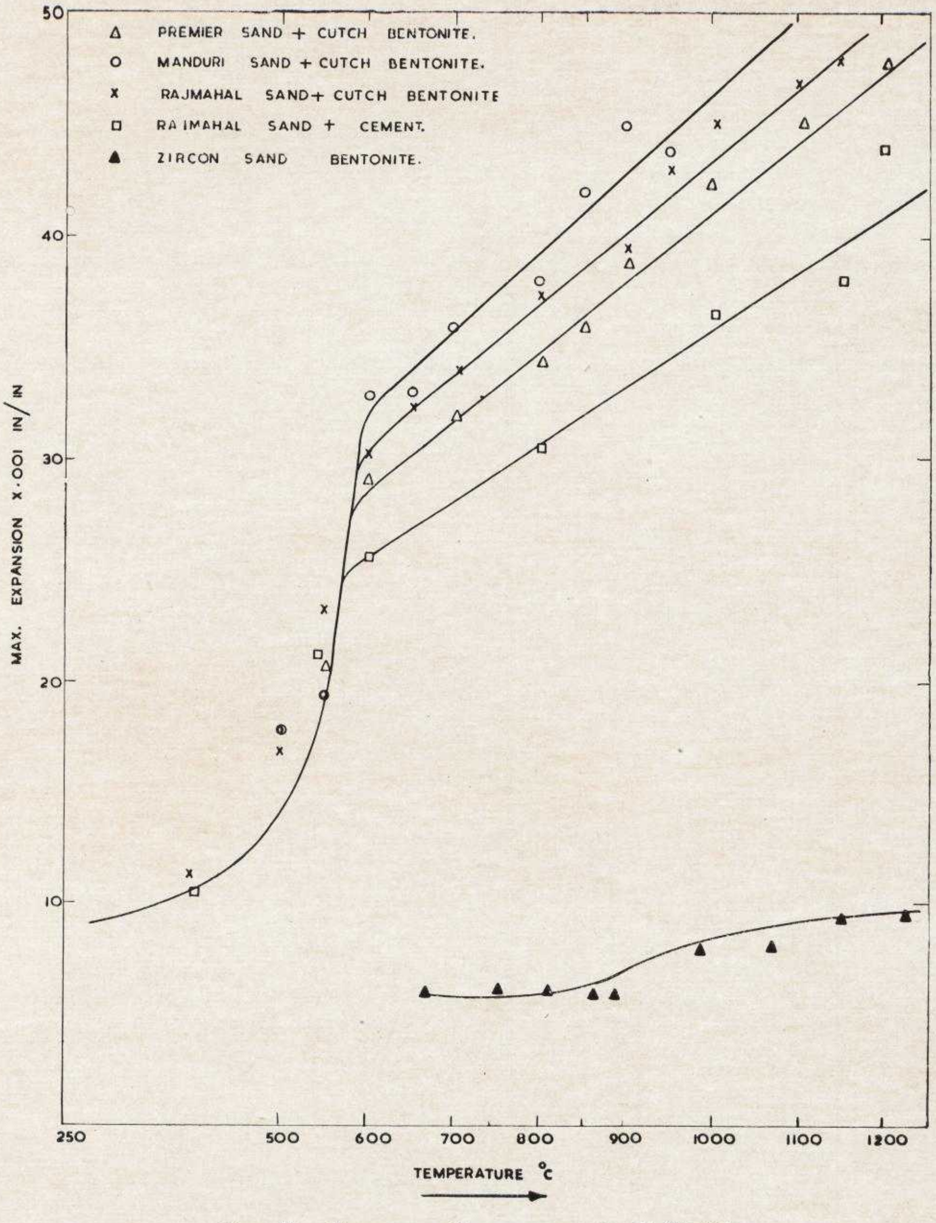


FIG. 49 — EXPANSION CHARACTERISTICS OF SANDS

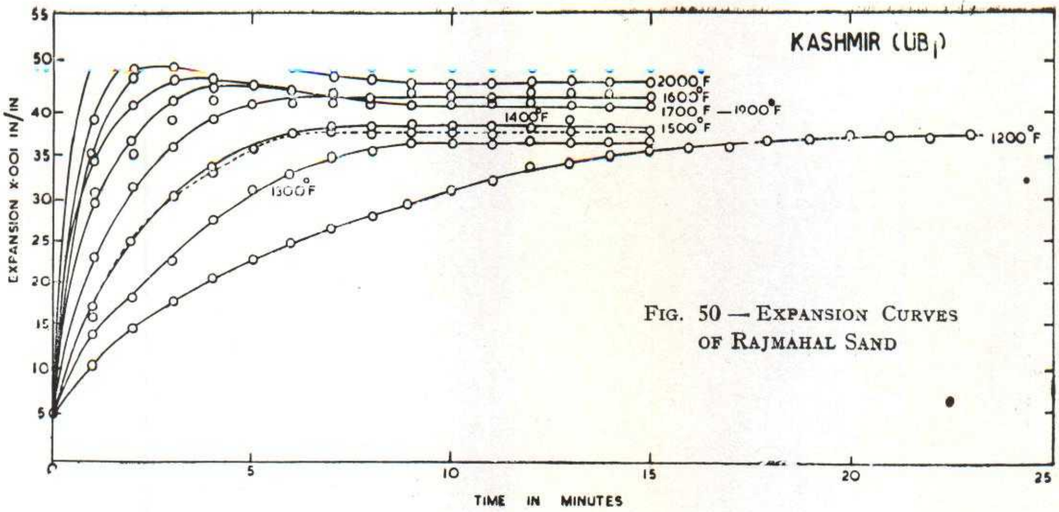


FIG. 50 — EXPANSION CURVES OF RAJMAHAL SAND

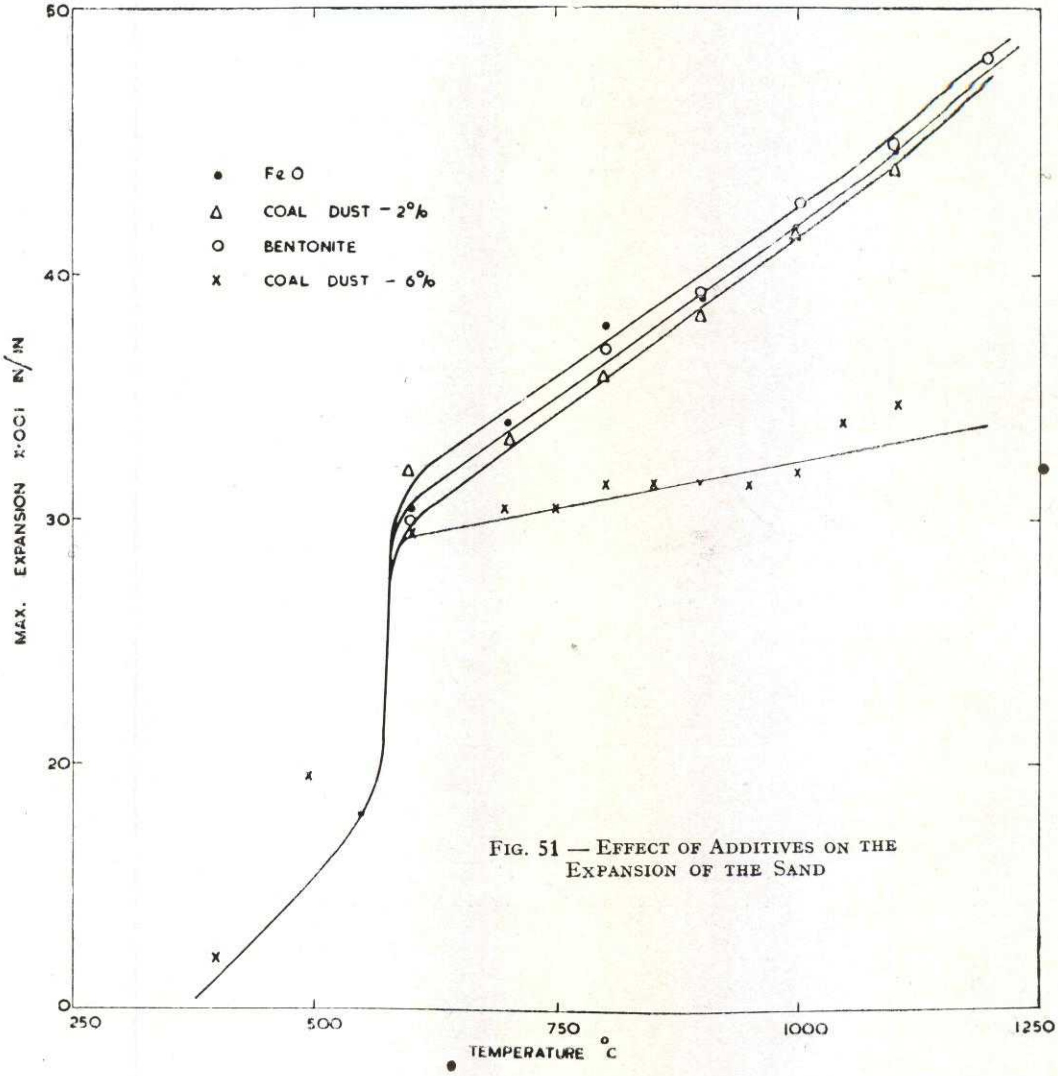


FIG. 51 — EFFECT OF ADDITIVES ON THE EXPANSION OF THE SAND

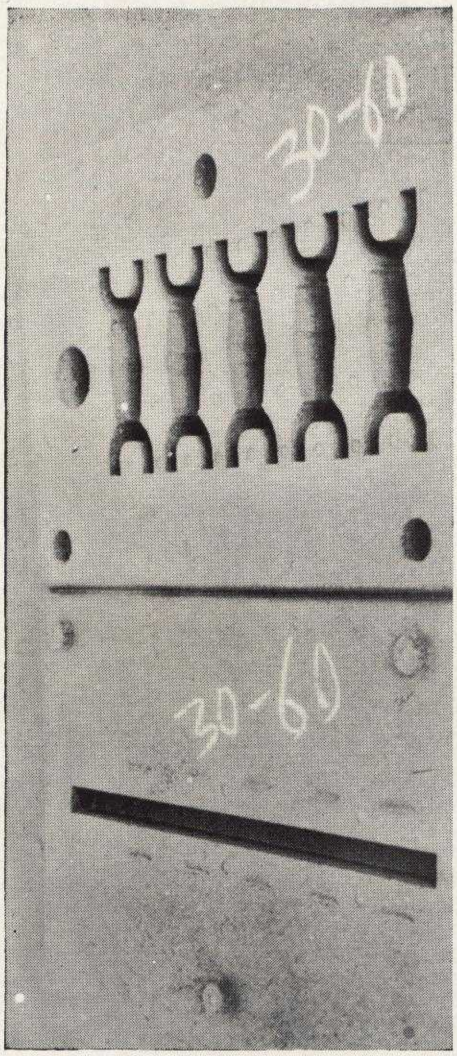


Fig. 52 — Shell Mould Showing Cope and Drag Made for Magnet Casting

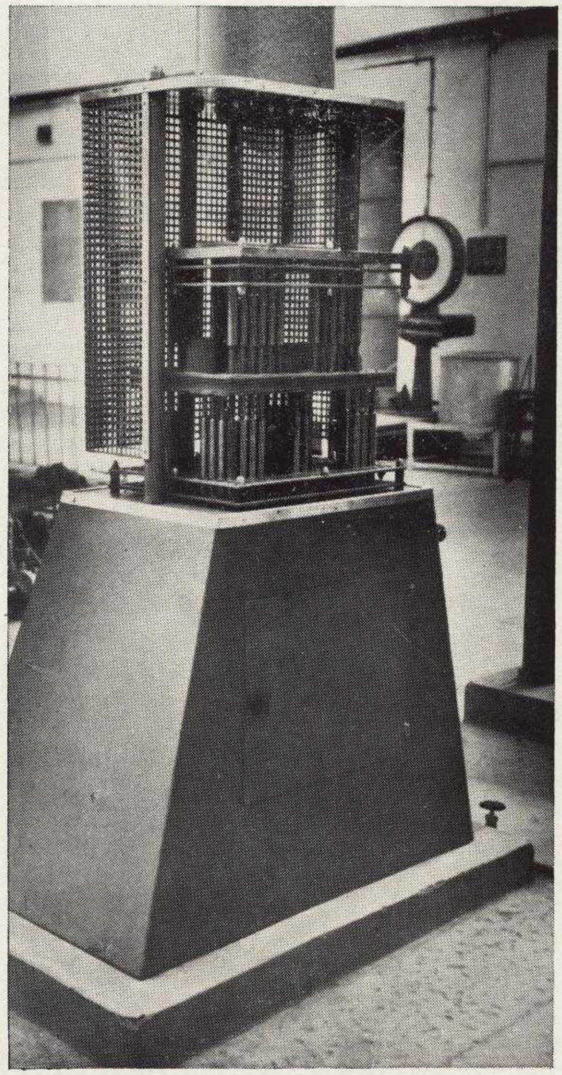


Fig. 53 — Shell Fuser

Table 17 — Direct Reduction Experiments Conducted on Sample from M/s. Electric Control Gear Mfg. Co.

Time	Percentage composition	Temperature °C.				
		850	900	950	1000	1100
6 hr.	Fe	91·72	90·95	93·00	93·43	94·97
	FeO	3·42	5·18	3·96	0·84	0·42
	C	0·54	0·15	0·63	0·58	0·64
8 hr.	Fe	80·93	91·85	91·83	94·54	94·54
	FeO	13·68	8·20	1·71	0·13	0·13
	C	0·23	0·40	0·09	0·93	0·85
10 hr.	Fe	80·05	90·72	—	—	—
	FeO	17·64	5·76	—	—	—
	C	0·23	0·33			
12 hr.	Fe	67·20	90·72			
	FeO	30·60	6·48	—	—	—
	C	0·50	0·35			
16 hr.	Fe	90·42	94·08		93·63	
	FeO	5·40	1·09	—	0·79	—
	C	0·46	0·06		0·76	
24 hr.	Fe		94·54		93·19	
	FeO	—	0·86	—	1·99	—
	C		0·80		0·65	

Ltd. was fine and dark brown in colour. Both these ores were of Goa origin. But the iron ore received from Government of Maharashtra was of —3/4 in. size and had to be crushed. After crushing to —100 mesh, the iron ore had a light brown colour. The ore was packed in paper packets of 1 in. diameter and 6 in. long. The packets were embedded in dry coke breeze of —¼ in. size, inside aluminized steel boxes which were sealed with clay afterwards. Pelletizing of iron ore received from Messrs Orient Ltd. was carried out in a disc type pelletizing machine. The iron ore was crushed to —100 mesh for pelletizing. Two per cent molasses were used as a binder for pelletizing. The pellets had good green and dry strength. Direct reduction studies were also made with these pellets.

Direct reduction experiments were carried out at different temperatures for different lengths of time. The results obtained are given in Tables 17-19.

Table 18 — Direct Reduction Experiments Conducted on Sample from M/s. Orient Ltd.

Time	Percentage Composition	Temperature °C.			
		800	900	1000	1100
6 hr.	Fe		88·49	72·80	92·74
	FeO	—	4·87	21·60	3·54
	C		0·08	0·10	0·[illegible]0
8 hr.	Fe		93·64	96·32	96·77
	FeO	—	0·35	0·56	0·17
	C		0·25	0·65	0·53
12 hr.	Fe		95·43	93·53	—
	FeO	—	2·23	2·16	—
	C		0·68	0·51	—
16 hr.	Fe		91·85	94·52	94·30
	FeO	—	0·90	1·91	3·65
	C		0·61	0·61	0·68
20 hr.	Fe	72·54	96·32	94·08	92·9
	FeO	24·70	0·75	Trace	2·16
	C	0·09	0·90	0·73	0·78
24 hr.	Fe	51·52	92·30	96·32	93·63
		41·76	1·92	0·74	4·91
		0·04	0·55	0·85	0·57

The work on iron ore from Government of Maharashtra showed that the ores were low in gangue and suitable for direct reduction for the production of sponge iron and iron powder. Investigational work had also been taken up on the iron ore pellets made from the ore supplied by Messrs V. M. Salgaocar E Irmao Ltda, Goa, for direct

Table 19 — Direct Reduction Experiments Conducted on Sample from Government of Maharashtra

Time	Percentage composition	Temperature °C. 800	900	1000	1100
6 hr.	Fe	56·27	—	75·26	95·43
	FeO	34·59		18·02	0·80
	C	0·10		0·04	0·25
8 hr.	Fe	—	88·49	94·29	94·24
	FeO		3·24	0·09	0·23
	C		0·30	0·57	0·48
16 hr.	Fe	63·84	—	94·75	95·88
	FeO	29·88	—	0·96	0·59
	C	0.17	—	0.95	0.74
20 hr.	Fe	75·60	96·1		
	FeO	18·72	0·66	—	—
	C	0·05	0·57		
24 hr.	Fe		95·20	93·44	94·97
	FeO	—	2·16	0·81	0·59
	C		0·56	1·11	1·02

reduction studies and its suitability for the production of iron powder.

34.0 Direct Reduction of Iron Ores by Naphtha

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

The method consists in packing the iron ore in the form of powder; or compacts or small lumps of suitable sizes into the reduction chamber, one end of which is connected to the chamber holding the petroleum product through a fuel pump and other end of which is connected to a series of condensing and absorption units so as to remove the water vapours and CO_2 gas. The gases after purification are recirculated to the reduction chamber through the liquid fuel with the help of the fuel pump.

The reduction chamber is heated to the required temperature depending upon the stage of reduction; by heating externally in an electrical furnace. Reduction stages of the iron ores depend upon the quality input of the fuel oil as well as on the time for reduction at the required temperature. The reduced product is cooled to room temperature before opening the system. This reduced product is utilized for (i) making iron powders for autogenous cutting purposes, (ii) as a cold charge for the open hearth furnaces or electric furnace. The iron content of the reduced product is related to the metallic content of the ore itself. Several experiments were performed with the iron ores as given in Table 20. The set up of the apparatus is shown in Fig. 54. The analyses of the products of reduction are given in Table 21.

Complete reduction to metallic iron was observed to take place at 1050°C. and the best reduction was achieved in 1½ hr. time. The rate of flow of the naphtha during the experiment is shown in Fig. 55.

Table 20 — Composition of Iron Ores

% Comp.	Barsua blue dust	Noamundi blue dust
Fe_2O_3	90·8	96·5
SiO_2	2·2	0·92
Al_2O_3	2·8	1·87
CaO	2·23	Trace
MgO	0·31	Trace
Loss on Ignition	—	1·2

Table 21 — Composition of Reduced Products

%	1	2	3	4	5
Fe	80·2	81·55	82·66	84·24	94·04
FeO	8·35	10·31	6·04	—	—
Fe_3O_4	2·2	3·15	1·16	1·31	0·14
Fe_2O_3	Trace	Trace	Trace	—	—

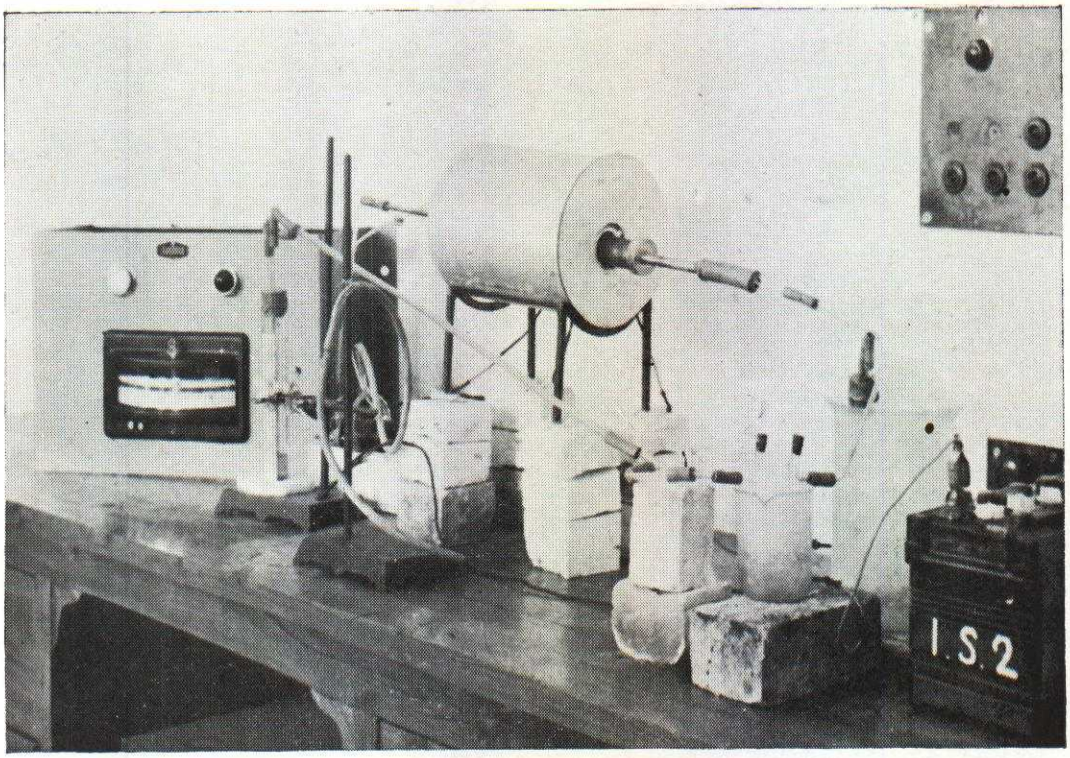


FIG. 54 — SET-UP FOR DIRECT REDUCTION OF IRON ORE BY NAPHTHA

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

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

In the set up (Fig. 56) the gases nitrogen/ or hydrogen are purified of oxygen by passing through alkaline pyrogallol and heated copper turnings and subsequently passing through silica gel, calcium chloride and ascarite, etc. The purified gases are then passed through a furnace which is

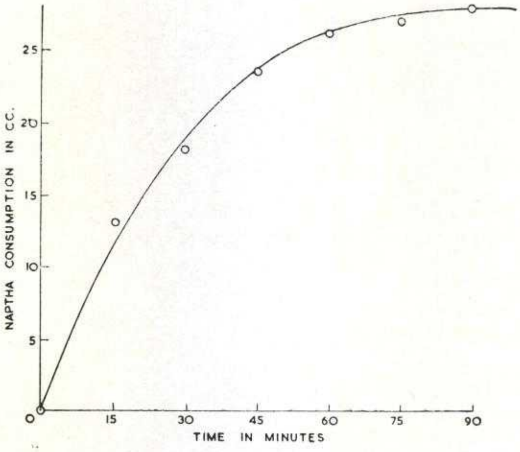


FIG. 55 — CURVE SHOWING NAPHTHA CONSUMPTION

Table 22 — Electrical Resistance of Iron Ores

Sample I	Temp. in °C.	Time in mins.	Resistance in ohms.	% Reduction
Before passing H_2	900	0	2·23	—
After passing H_2	900	180	0·003	94·0
Sample II				
Before passing H_2	900	0	14·99	—
After passing H_2	900	180	0·004	85·3
Sample III				
Before passing H_2	900	0	6·60	—
After passing H_2	900	180	0·010	73·0

maintained at 850-950°C. and holds the iron ore sample by means of copper disc rods. The two ends of the copper rods are connected to Wheatstone Bridge for measuring the electrical resistance of iron ore. Purified hydrogen, when passed through the furnace reacts with the iron ore to form metallic iron and moisture. The electrical resistance was continuously measured till the reduction was completed and no further change in the electrical resistance measurement took place.

Table 22 shows typical results of some of the experiments performed at 900°C. It was found that electrical resistance of the iron ore at about 950°C. was 2-15 ohms and resistance after its complete reduction was only 0·003-0·010 ohm. A notable feature noticed was the change in electrical resistance on heating the samples from room temperature to 950°C. without passing hydrogen. With a nitrogen atmosphere, the resistance at room temperature was about 200,000 ohms and at 950°C., it was only 2-15 ohms.

36.0 Solidification of Cast Iron

Aluminium Cast Iron

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

Heats of aluminium cast iron containing 20-25 per cent aluminium were made. Attempts were made to nodularize the graphite in the cast iron by the addition of misch metal and magnesium. Al-Mg alloy was used for the addition of magnesium. Different methods were used for addition of Al-Mg alloy to the cast iron. The microstructure of these cast irons were studied. Scaling resistance test between 800°-1100°C. were done. Experiments are being conducted to get a suitable composition of aluminium cast iron which will give better heat resistance and mechanical properties.

37.0 Low Residual Soft Iron

The investigation was taken up to produce low residual iron for use as a basic raw material in the manufacture of special alloy steels and super alloys and also for use as a soft magnetic material. Low residual soft iron is used in Defence, Posts & Telegraph, Railways and Telephone industries in India.

Small scale trials were made in a basic lined direct arc electric furnace with manual control of arcing and power input. A double slag (oxidizing and reducing) melting technique was employed, with the difference that two oxidizing slags (instead of one) were made to obtain a low phosphorus content, less than 0·01 per cent and low carbon content less than 0·03 per cent. To achieve this, iron ore additions were made to the melt which was suitably oxygen lanced. After removing

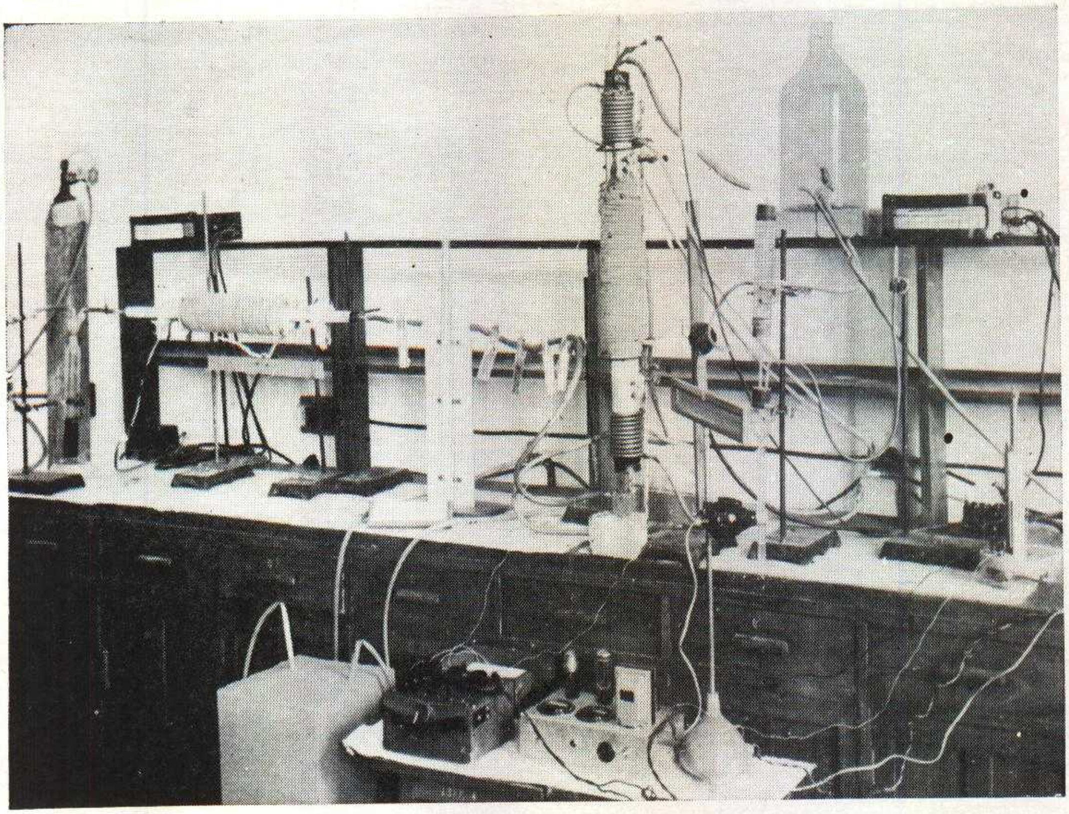


FIG. 56 — SET-UP FOR STUDYING THE KINETICS OF IRON ORE REDUCTION WITH HYDROGEN BY ELECTRICAL CONDUCTIVITY METHOD

the second oxidizing slag, the reducing slag was made with lime, fluorspar and aluminium. The melt was then cast into ingot moulds. The carbon content of melt was less than 0·045 per cent. Carbon content of the melt could further be reduced with automatic electrode control since in manual control it is difficult to maintain the arc without the electrodes impinging on the melt causing carbon pickup thereby. Full scale melting trials were made in an 800 kg. electric arc furnace (Brown Boveri) operated from a 450 kVA. transformer having six different voltage tappings namely of 73, 80, 88, 98, 111 and 127 volts. The initial charge of unsorted plain carbon steel scrap was so adjusted as to give an opening carbon of about 0·3 per cent. Requisite quantity of limestone was charged along with the scrap melting period in order to facilitate elimination of phosphorus which would otherwise be retarded by subsequent high melt refining temperatures, particularly during oxygen lancing. The first oxidizing slag was taken out immediately after scrap melting was completed and a fresh basic and oxidizing slag was made up with limestone, fluorspar and mill scale. After oxygen lancing operation, the second oxidizing slag was run out. At the end of each oxidizing period, bath sample was taken for the determination of carbon and phosphorus contents. The duration of oxygen lancing was adjusted in relation to the carbon content of the first melt sample. In the second melt, sample taken during the second oxidizing slag was run out. At the end of each oxidizing period, bath sample was taken primarily for the determination of carbon and phosphorus contents. The duration of oxygen lancing was adjusted in relation to the carbon content of the first melt sample. In the second melt sample taken during the second oxidizing period, manganese and sulphur contents were also analysed.

After removing the second oxidizing slag, the bath was partially deoxidized with ferro-silicon and a fresh reducing slag was made by the additions of limestone and fluorspar. The bath was held under this slag for about 45 minutes in order to remove sulphur. Slag and the metal were rabbled during this period. Two bath samples were taken during the reducing slag operation, the slag was thereafter run off and a fourth slag was made with limestone and fluorspar alone. Immediately following this, oxygen lancing was done for a short time till all the silicon in the bath had been oxidized as judged by the fumes issuing from the furnace. As soon as this stage was reached, the bath was actively rabbled with aluminium sticks (6 kg./ton) for final deoxidation and the melt was tapped after about 10 minutes. The temperature of the bath was maintained at about 1600°C., the yield of the metal was approximately 80 per cent of the initial metallic scrap charge.

Table 23 — Time Schedule of Heat No. EF77 of Low Residual Iron carried out in 800 kg Electric Arc Furnace

Cast No. EF77			
	Power on	10.50 a.m.	
	Charging complete	12·45 p.m.	
	Complete melt	1·15 p.m.	
I	Slag off (oxidizing)	1·40 p.m.	
II	Slag off	2·28 p.m.	
III	Slag off (reducing)	3·24 p.m.	
	Tapped	4·00 p.m.	
	Total time taken	5·30 hr.	
Basic charge			
	Heavy scrap	500 kg.	
	M.S. pipe	200 kg.	
	Misc. Light Scrap	100 kg.	
Limestone		10 kg.	(with the charge)
Limestone during melting		5 kg.	(12·00 p.m.)
Iron ore		10 kg.	(12.25 p.m.)
Charge melted			1·15 p.m.
Bath sample I			1·30 p.m. (0·13%C, 0·032%S, 0·013%P)
Bath temperature		1550°C.	(1·38 p.m.)
1st slag off (oxidizing)			1·41 p.m.
Slag making mixture:			
	1. Limestone	12 kg.	1·47 p.m.
	2. Fluorspar	1·5 kg.	
	3. Mill scale	2·0 kg.	
Bath sample II for C, S & P			2·21 p.m. (0·08%C., 0·03%S, 0·0038%P)
2nd Slag off (oxidizing)			2·28 p.m.
Ferro-silicon		4 kg.	2·32 p.m.
Slag mixture:			
	1. Limestone	12 kg.	2·33 p.m.
	2. Fluorspar	1·5 kg.	
Bath sample III for C, S & P			3·15 p.m. (0·06%C, 0·013%S, 0·012%P)
Temperature		1600°C.	(3·20 p.m.)
3rd Slag off (reducing)			3·24 p.m.
Slag making mixture:			
	1. Limestone	12 kg.	3·25 p.m.
	2. Fluorspar	1 kg.	
Oxygen, blown at 100 psi			3·33-3·39 (6 mts.)
Aluminium stick		5 kg.	3·43 p.m.
Bath sample IV (C, S & P)			3·48 p.m. (0·03%C, 0·012%S, 0·0082P)
Bath sample V (C, S & P)			3·55 p.m. (0·02%C, 0·012%S, 0·0063%P)
Tapped			4·00 p.m.

Table 24 — Chemical Analyses of Some Low Residual Iron Heats

Heat No.	% Composition						
	C	Si	Mn	S	P	Al	N
EF 77	0·02	0·07	0·053	0·012	0·016	Tr	0·0055
EF 78	0·02	0·047	Tr	0·014	0·006	0·14	0·009
EF 83	0·01	0·03	0·05	0·02	0·0036	0·10	0·0059
EF 84	0·025	0·05	0·082	0·028	0·013	0·10	0·006
EF 87	0·02	0·01	0·04	0·02	0·004	0·10	0·0074
EF 92	0·03	0·12	0·04	0·0095	0·0094	0·210	0·0 53
EF 94	0·03	0·042	0·11	0·019	0·007	Tr	0·0062

Time schedule of a typical heat of low residual iron made in 800 kg. electric arc furnace is given in Table 23.

The process described in Table 23 was modified to some extent in other melts, typical of these alterations are illustrated by Heat Nos. 78, 87 and 92.

Heat No. 78 : The process followed was the same as for Heat No. EF77 given in Table 23 except that prior to final deoxidation of the molten bath with aluminium sticks the slag cover of the bath was removed and a fresh slag was made with fluorspar and limestone.

Heat No. 87 : In this heat, after removing two oxidizing slags, the bath was deoxidized only with aluminium and no ferro-silicon was used. The usual oxygen blow after the reducing slag was abandoned.

Heat No. 92 : The procedure followed was similar to that of heat No. EF 77 except that oxygen blow was curtailed from 6 minutes to one minute only.

Chemical analyses of some of the low residual iron heats made are given in Table 24.

38.0 Gaseous Malleabilization of White Cast Iron

The objective of the investigation is to avoid the packing materials during malleabilization and to study the effect of Cu in different amounts in the reduction of time for the cycle. Trials were carried out under different gaseous medium such as coke-oven, hydrogen, argon and nitrogen at 800°C. and 850°C. for various time periods.

The experimental procedure consists of taking the white iron pieces of approximately 1 in. dia. and heating it in an electric furnace to the desired temperature which is attained in 2-3 hr. and maintained throughout the experiment. The specific gas after purification was allowed to pass at a constant rate. After the desired time, the furnace was switched off and gas flow was stopped. The furnace was then allowed to cool slowly to room temperature. The specimen was then taken out, polished and studied for micro-structure and hardness. Cast iron of the compositions given in Table 25 were studied for their malleabilization characteristics. It was observed that carbon/silicon ratio played an important part. When this ratio was 1 to 3, malleabilizing proceed uniformly with time but with higher ratios the malleabilization process is retarded. This fact is also borne out by the hardness measurements. Fig. 57 gives successive stages of malleabilization with rise in temperature.

39.0 Study of Temper Brittleness

The object of this work is to examine whether ' retrogression ' shown by steels

Table 25 — Composition of Cast Iron Samples

Constituents	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6	Sample 7	Sample 8	Sample 9	Sample 10	Sample 11	Sample 12	Sample 13
C%	2·14	2·12	2·43	2·61	3·33	2·50	2·59	2·47	2·55	3·31	2·7	2·18	2·20
Si%	1·27	1·24	0·32	0·36	0·48	1·18	1·18	0·43	1·33	1·61	1·74	0·86	1·53
Mn%	0·56	0·56	0·37	0·43	0·43	0·55	0·56	0·33	0·56	0·82	0·79	0·66	0·25
S%	0·026	0·03	0·054	0·037	0·014	0·14	0·15	0·07	0·19	0·03	0·031	0·02	0·04
P%	0·186	0·178	0·24	0·19	0·23	0·19	0·18	0·18	0·19	0·31	0·33	0·27	0·18
Cu%	0·03	0·09	0·21	0·61	0·68	1·15	1·17	1·17	1·17	1·23	1·30	1·38	1·4

susceptible to temper brittleness in relation to precipitation which has been put forward to be one of the causes responsible for temper brittleness in steels.

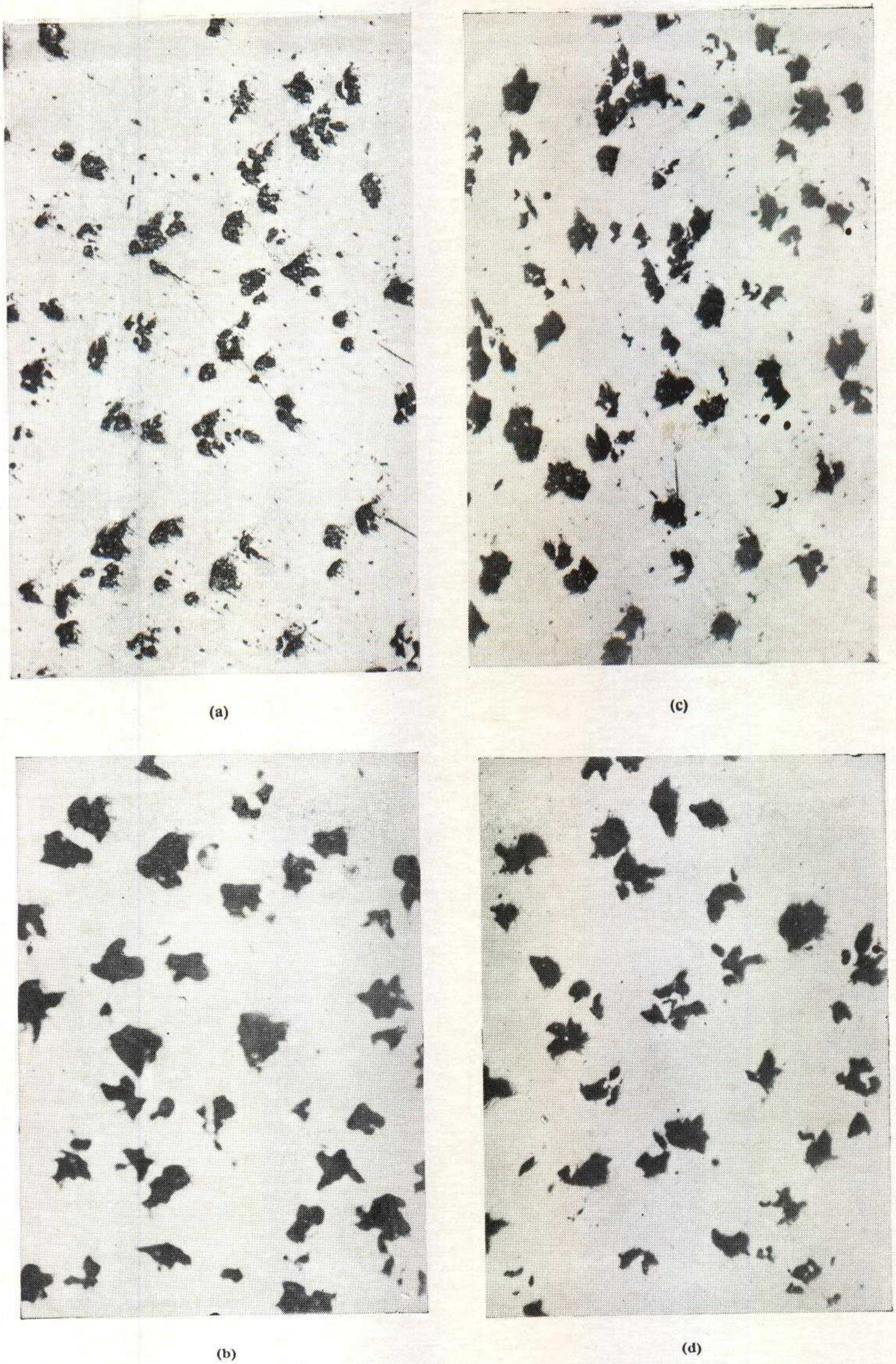
Impact tests were carried out on samples at 400°C. for various periods of time and their transition temperature were determined. From a determination of transition temperatures so far carried out, it appeared that temper brittleness was a precipitation phenomenon. Repeat experiments of impact tests were carried out to check the results obtained earlier. Final report on the subject is under preparation.

40.0 Curie Temperature of Iron-Chromium 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.

Alloys of iron with aluminium and silicon made earlier were re-examined for their curie temperature. The variation of curie temperature of iron on alloying suggests a process of electron absorption by iron at lower percentages of solute followed by dilution of iron, before ordering sets in the alloy. The electron absorption is thought to be due to the filling up of the holes in the electronic states in iron. The process of filling up of the holes is at a rate proportional to the valency of electrons contributed by solute. Addition of more solute once these excess holes are filled up, reduces the curie temperature at a rate suggesting dilution of iron by the solute.

The work has been extended on to the study of cobalt alloys. For this, cobalt and cobalt-copper alloys were melted in the high frequency furnace. The melts made in air were brittle and cracked during subsequent forging and the alloys are being made in vacuum.



(a) (c)

(b) (d)

Fig. 57 — Gaseous Malleabilization of White Cast Iron [(a) Under N_2 atmosphere at 800°C. for 25 hr. (b) Under N_2 atmosphere at 850°C. for 25 hr. (c) Under N_2 atmosphere at 900°C. for 25 hr. (d) Under N_2 atmosphere at 950°C. for 25 hr.]

41.0 Conductivity of Commercial Aluminium and its Alloys

Silicon and titanium normally present in commercial aluminium as dissolved impurities excercise adverse effects on its electrical conductivity. It is well established that the conductivity of a solid solution can be increased if the dissolved impurities could be taken out of the parent lattice. The decrease in the solid solubility of both silicon and titanium with falling temperature presents a possibility of their removal from the lattice of aluminium. In order to bring on the subject a fundamental approach, study was undertaken on changes in conductivity as precipitation progress in commercial aluminium.

In continuation of work on the conductivity of commercial aluminium, the effect of magnesium on the conductivity of commercial aluminium was studied. Two alloys containing 1·0 and 2 per cent magnesium respectively were prepared from commercial aluminium analysing Fe, 1·3-1·4; Si, 0·15; and Ti, 0·01 per cent. Figs. 58 and 59 show the changes in the conductivity of these during ageing at 300, 350 and 400°C. When these results were compared to the earlier results, it was found that (i) the high amount of impurities, particularly Fe, has considerably retarded the precipitation process and thereby no improvement in conductivity; (ii) the conductivity can be improved from about 40 IACS units in the solution treated condition to about 60 in the aged condition after over 50 hours of ageing at about 400°C. as compared with about 3 hours of ageing at 400°C. for alloys having low iron content (about 0·21 per cent).

The effect of boron addition on the conductivity was also determined. Boron was added (less than ·1 per cent) in the form of

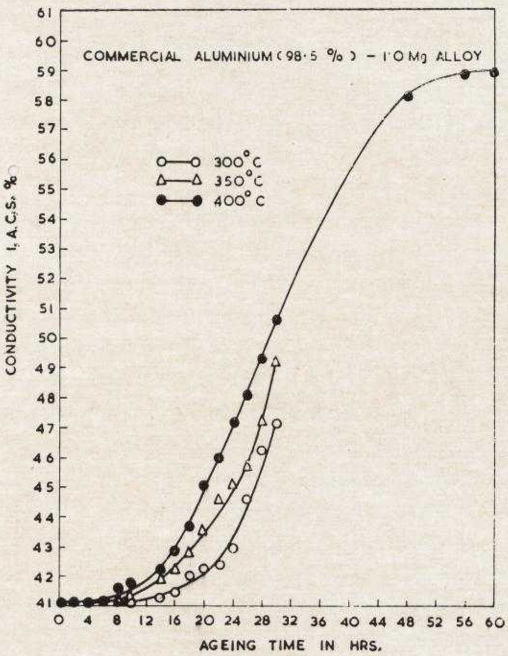


FIG. 58 — CHANGES IN THE CONDUCTIVITY OF Al, 98·5; Mg, 1·0 PER CENT ALLOY DURING AGEING AT 300°, 350° AND 400°C.

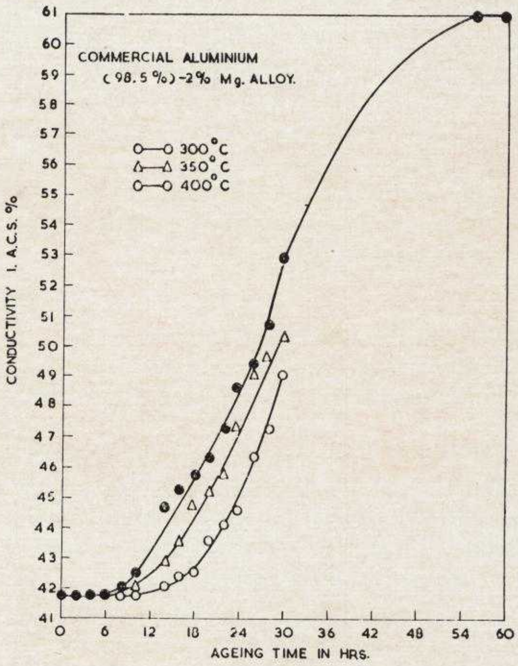


FIG. 59 — CHANGES IN THE CONDUCTIVITY OF Al, 98·5; Mg, 2·0 PER CENT ALLOY DURING AGEING AT 300°, 350° AND 400°C.

boric acid to 99·81, 89·5 per cent Al and also to 98·5 per cent Al. The variations in the conductivity of the as quenched and aged alloys and conductivity improves on ageing, the improvement was not marked in the case of 98·5 per cent Al alloy.

In view of the considerable increase in the industrial production of aluminium in the country, the scope of this project has been widened to collect experimental data on the effects of the impurities on the alloying elements on the conductivity of aluminium. Binary alloys containing 0·25, 0·5, 0·75 and 1·0 per cent each of Mg, Si, Mn and Fe were made and work is now in progress.

42.0 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 :

- (i) Study of interactions between Al-Cu, Al-Cu-Mg, Pb-Sn alloys in the liquid state.
- (ii) Study of the partitioning of magnesium between aluminium and lead.
- (iii) Enthalpy of liquid alloys.

(1) *Study of Interaction Between Al-Cu, Al-Cu-Mg, Al-Si, Pb-Sn and Other Low Melting Alloys in the Liquid State*

The aim and objective of this investigation is to study the distribution and nature of interaction between atoms of the solute in the solvent and the liquid state.

- (i) the effect of solute content on its distribution in the liquid condition.
- (ii) the effect of temperature on its distribution.
- (iii) the effect of centrifugal force on (i) and (ii) above.
- (iv) the effect of ternary additions on the distribution of solute atoms.

(a) *Al-Cu and Al-Cu-Mg alloys* — Work on alloys containing Cu was extended to cover The alloy melts were centrifuged at 700°C. for 1, 3, 5, 7 and 9 hours. The Al-33 per cent Cu eutectic alloy was centrifuged at 600°C. The centrifuging was done under conditions which developed a centrifugal force of 40 and 140 g. Double and even triple runs were made in order to decrease the statistical error. The samples were chemically analysed and metallographically examined. Only the centrifuging introduced a gradient in the distribution of Cu from the outer to the inner end. A typical distribution is shown in Fig. 60 for Al-4 per cent Cu alloy at 700°C.

It was presumed that the copper was present in the form of clusters corresponding to the $CuAl_2$ (Eta phase) composition. On this basis, the cluster size was calculated. Results indicated that the cluster size and interaction between Al-Cu, was dependent upon temperature and composition. Metallographic examination indicated that the grain size of the ingot was fairly coarse at the inner end (nearer the axis of rotation) and became progressively finer at the farther end.

Further work is in progress on Al-4 Cu-1 per cent Mg alloys to study the effect of Mg additions on the distribution of copper.

(b) *Al-Si Alloys* — The Al-Si system is metallurgically important because minute additions of sodium and sulphur and phosphorus can remarkably alter the morphology of the distribution of silicon of the eutectic and hyper-eutectic alloys. Several theories have been put forward to explain the phenomenon but none has yet been universally accepted, largely because of general lack of supporting experimental data. Not much work has been done to elucidate (i) the influence of the temperature of solidification on the morphology of the silicon phase, (ii) effect of variable cooling rates, (iii) effect

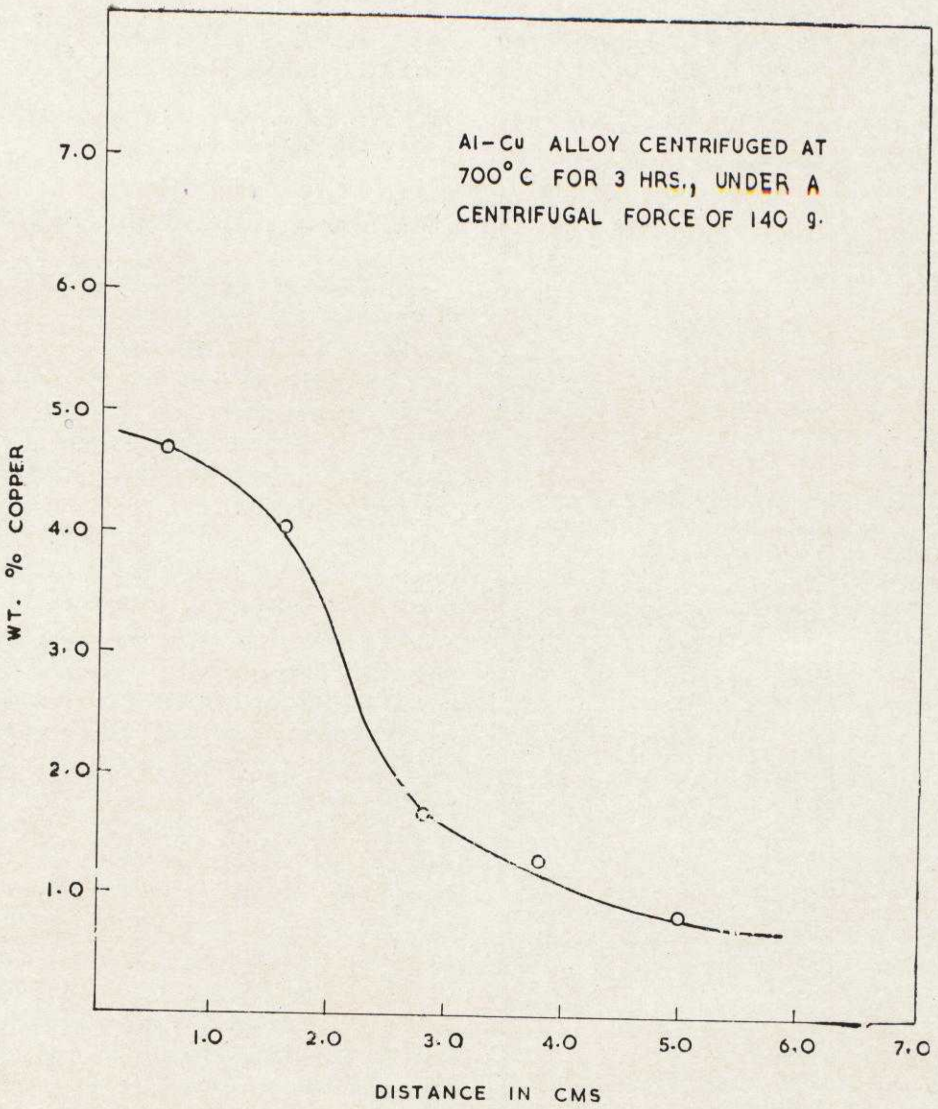


FIG. 60 — DISTRIBUTION OF COPPER AFTER CENTRIFUGING FROM BOTTOM TO TOP OF THE INGOT

of silicon content and modification on the interfacial energies, contact angles, surface tension, and viscosity, etc. In the light of the work done on liquid metals at the National Metallurgical Laboratory, it was felt that modification by sodium may in some respects alter the distribution of silicon in the liquid state.

Experiments are underway with the eutectic Al-Si alloy and hyper-eutectic Al-Si alloy containing 18 per cent Si to determine the size of silicon crystal in the metal as a function of (i) silicon concentration, (ii) temperature and (iii) modification. Any relation between the liquid state and cast structure will also be examined.

(c) *Pb-Sn and Other Low Melting Alloys* — Work on binary alloys of Pb Sn was completed. Some of the results were reported earlier and a full report is under preparation. Work on Pb-Cd eutectic alloy is being taken up.

(2) *The Study of Partitioning of Magnesium Between Al & Pb.*

The study of the distribution of magnesium between aluminium and lead indicated considerable scatter. The entire work is being repeated with modified experimental technique.

(3) *Enthalpy of Liquid Alloys*

Heat content of pure Pb & Sn and their alloys were determined up to a temperature of 900°C. and following conclusions were drawn :

(i) the heat capacity of molten lead continuously decreased with temperature up to 900°C.

(ii) the heat capacity of liquid tin at first decreased with increasing temperature, reached a minimum value at about 450°C. and started to increase above this temperature.

(iii) the heat capacity of eutectic lead-tin alloy decreased, reached a minimum value at about 650°C. and started to increase above this temperature.

(iv) the heat capacity of lead 69·12 per cent-tin 30·88 per cent decreased, reached a minimum value at about 750°C. and started to increase above this temperature.

(v) the heat capacity of lead 22·16 per cent-tin 77·84 per cent decreased, reached a minimum value at about 780°C. and increased above this temperature.

Hypo-eutectic and hyper-eutectic alloys of binary lead-tin alloy of lead and tin were prepared and their heat content was determined at 900°C. Detailed calculations are in progress to draw comprehensive conclusions.

43.0 Effect of Thermal Cycling of Cu-Pb Alloy

Work was undertaken with a view to study the possibility of causing separation of copper from mixed white metal scrap by physical methods. Experiments to cause separation of copper in lead by thermal cycling did not succeed.

44.0 Effect of Titanium on Cast Iron

The characteristic mechanical properties of cast iron are largely determined by (i) the size distribution and shape of graphite flakes/nodules, (ii) the amount combined carbon present either as cementite or as carbides of alloying elements, and (iii) the amount and nature of solutes dissolved in the ferritic matrix.

Ten heats were made analysing: carbon, 3·48; silicon, 1·92; phosphorus, 0·26; sulphur, 0·05; manganese, 0·48 per cent and traces of titanium. The titanium contents of the heats were 0, 0·5, 1, 2, 3, 7 and 10 per cent titanium was added in the form of ferro-titanium in the ladle for all compositions except the 7 and 10 per cent heats. In all cases, heats were cast from 1350°C. into sand moulds of wedge and cylindrical shapes. Examination of the fracture surfaces of the wedge specimens showed that titanium at first exercises a graphitizing effect and that the originally white fracture of the titanium-less cast iron became fully grey at about 2-3 per cent titanium. As the titanium content was increased, titanium carbide formed in preference to graphite; fractured surfaces again become white as shown in Fig. 61 which depicts the fracture surfaces of all the specimens. These observations were confirmed by extracting carbides and graphite from the specimens and subjecting them to X-ray diffraction analysis. Metallographic examination of the specimens was also carried out. The influence of prolonged holding at elevated temperatures on the formation of graphite and titanium carbide was studied. Typical microstructure of the 0·5 per cent Ti as cast structure is shown in Fig. 62. Effect of titanium on the hardness of cast specimens was also studied.

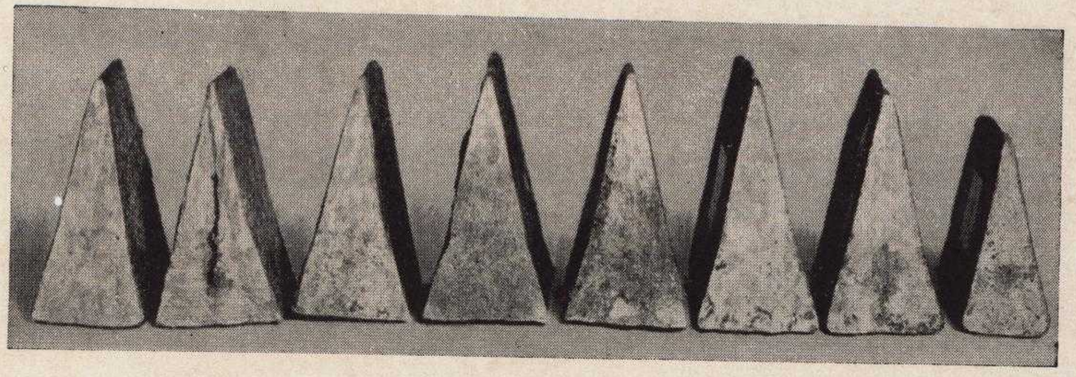


FIG. 61 — SHOWING FRACTURED SURFACE WITH INCREASE OF TITANIUM

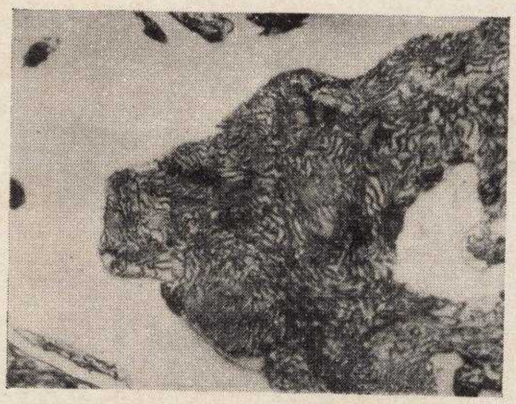


FIG. 62 — 'AS-CAST' MICRO-STRUCTURE OF 0·5 PER CENT TITANIUM CAST IRON

45.0 Mar-ageing of Steels

The conventional process of hardening and toughening steel is based on formation of martensite and subsequent tempering. During tempering, different carbides are precipitated out. The precipitation reactions in an essentially low carbon martensitic matrix can supplement the hardness of martensite, as the softening of martensite by the precipitation of carbides is avoided. Such mar-aged steels retain sufficient toughness in spite of the martensite and subsequent precipitation processes. During mar-ageing, 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.

It was indicated in the earlier report that heats containing Fe-20 per cent Ni with 1, 2, 3, and 4 nominal percentage of aluminium were made. Ageing characteristics of these alloys were determined at 450°, 500° and 600°C., after homogenizing at (i) 900° and (ii) 1100°C. and quenching the austenite first in water at room temperature and then in liquid air in order to transform as much of the austenite retained in the first quench to martensite as possible. The tempering process was followed with the help of hardness measurements. Typical tempering results are shown in Fig. 63. In all cases, the hardness increased during tempering, the increase in hardness being a function of the aluminium content as shown in Fig. 64. Maximum hardness was developed in the alloy with Al, 3 per cent. There was a slight tendency for the hardness to decrease with increasing aluminium content. An increase in the austenitizing temperature from 900° to 1100°C. had practically no effect on the ageing characteristics of the alloy containing 1 or 2 per cent of aluminium but the one with 3 per cent Al indicated that increase in austenitizing temperature had an accelerating effect

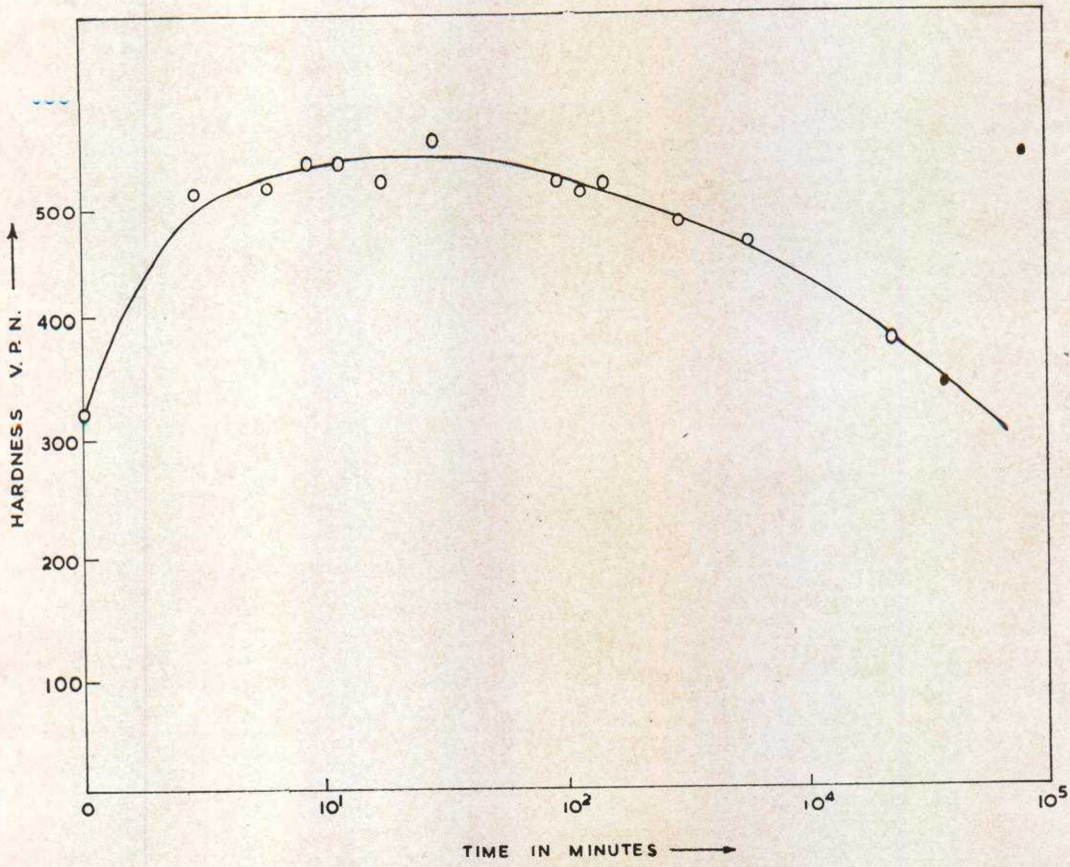


FIG. 63 — AGEING CURVE OF 3 PER CENT Al MAR-AGEING STEEL AGED AT 500°C. AFTER SOAKING AT 900°C.

on the ageing processes at 500°C.; the peak hardness and corresponding degree of softening occur at shorter intervals of time. Experimental observations suggested that during mar-ageing, processes similar to those occurring in the conventional ageing of super-saturated alloys occurred. This was suggested by the fact that the peak hardness was progressively lowered at higher temperatures of tempering and that the peak occurred at shorter times. Attempts to identify the precipitating phase by X-ray techniques had not so far been successful. Based on these results the Fe, 20; Ni, 3 per cent; Al alloy was chosen as a base for the development of alloys with increasing hardness and strength. Alloys containing 1-5 per cent manganese were made and work is now in progress on these alloys.

46.0 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 (i) superheating the melt or (ii) 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 :

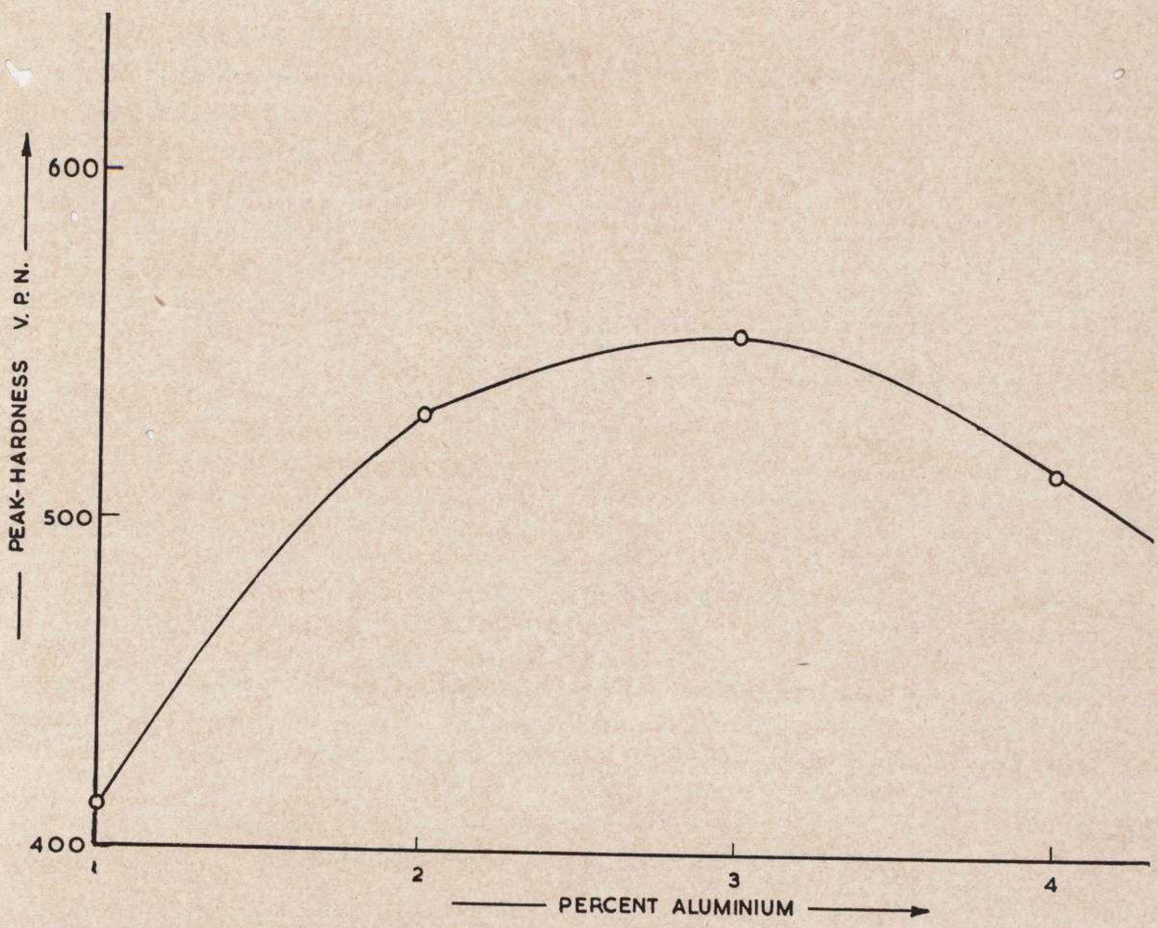


FIG. 64 — PEAK-HARDNESS VS. ALUMINIUM CONTENT ON AGEING

(a) nature of the nuclei 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, and

(c) whether the same mechanism is operative in the two processes of grain refinement.

The effect of aluminium addition was studied on the cast grain size of magnesium. The effect of superheating was also simultaneously studied. In order to avoid any possible influence of flux on the nucleation of solidification, magnesium was melted under argon atmosphere.

47.0 Phase-Transformation in Iron-Manganese System

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

Investigations were carried out on Fe-Mn alloys mentioned in Table 26. Initially an attempt was made to investigate the kinetics of the formation of martensite in these alloys by resistivity measurements.

Table 26 — Composition of Fe-Mn Alloy

	Sample No.			
	1	2	3	4
Nominal Mn content %	5	10	15	20
C	0·07	0·08	0·07	0·08
Mn	4·9	8·7	12·8	17·95
S	0·029	0·037	0·03	0·042
P	0·01	0·01	0·012	0·007

But due to one reason or another, the martensitic transformation could not be successfully traced with this technique in alloys containing Mn, up to 13 per cent. It was then decided to study the dilation characteristics of these alloys with the help of conventional dial-gauge type of dilatometer in order to determine the approximate temperature ranges for the gamma to alpha transformation. Results obtained on the 12·8 per cent Mn alloy are shown in Fig. 65. The dilatometric work indicated that in alloys containing 5-9 per cent Mn the gamma to alpha transformation occurred in the temperature range 400°-300°C. and in those containing 9-13 per cent, the transformation occurred below 300°C. to a mixture of alpha and epsilon phases; in 18 per cent Mn alloy transformation primarily occurs to epsilon phase. It was noted that the temperature of transformation was not progressively lowered with increasing manganese contents as the transformation was completed at a higher temperature in the 18 per cent Mn alloy than in the 13 per cent Mn alloy.

It has been suggested in the literature that there are two possibilities for the decomposition of austenite in Fe-Mn alloys :

fcc—Stacking fault
|
-martensite cph
(paramagnetic)
|
-martensite bcc
(ferromagnetic)

In otherwords, austenite can either decompose directly to ferro-magnetic alpha martensite which is a bcc structure or can decompose into ferro-magnetic alpha phase through the intermediate formation of (i) stacking fault and (ii) paramagnetic epsilon martensite which has a cph crystal structure. It was noted that specimens of the 12·8 per cent Mn alloy which otherwise transformed on cooling to ferro-magnetic alpha martensite no longer did so if these were forged after the austenitizing treatment and transformed into the paramagnetic epsilon martensite instead. Stacking faults introduced in the austenite through plastic deformation, therefore, appeared to promote the formation of epsilon martensite and to suppress the direct transformation of austenite into alpha martensite. Amongst other things, the influences of stacking faults on the formation of epsilon martensite and its subsequent decomposition were investigated.

48.0 Preferred Orientation in Extruded Rods

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

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 incident 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, developed in the extruded rods.

Work was started on duralumin and commercially pure aluminium. Ingots were

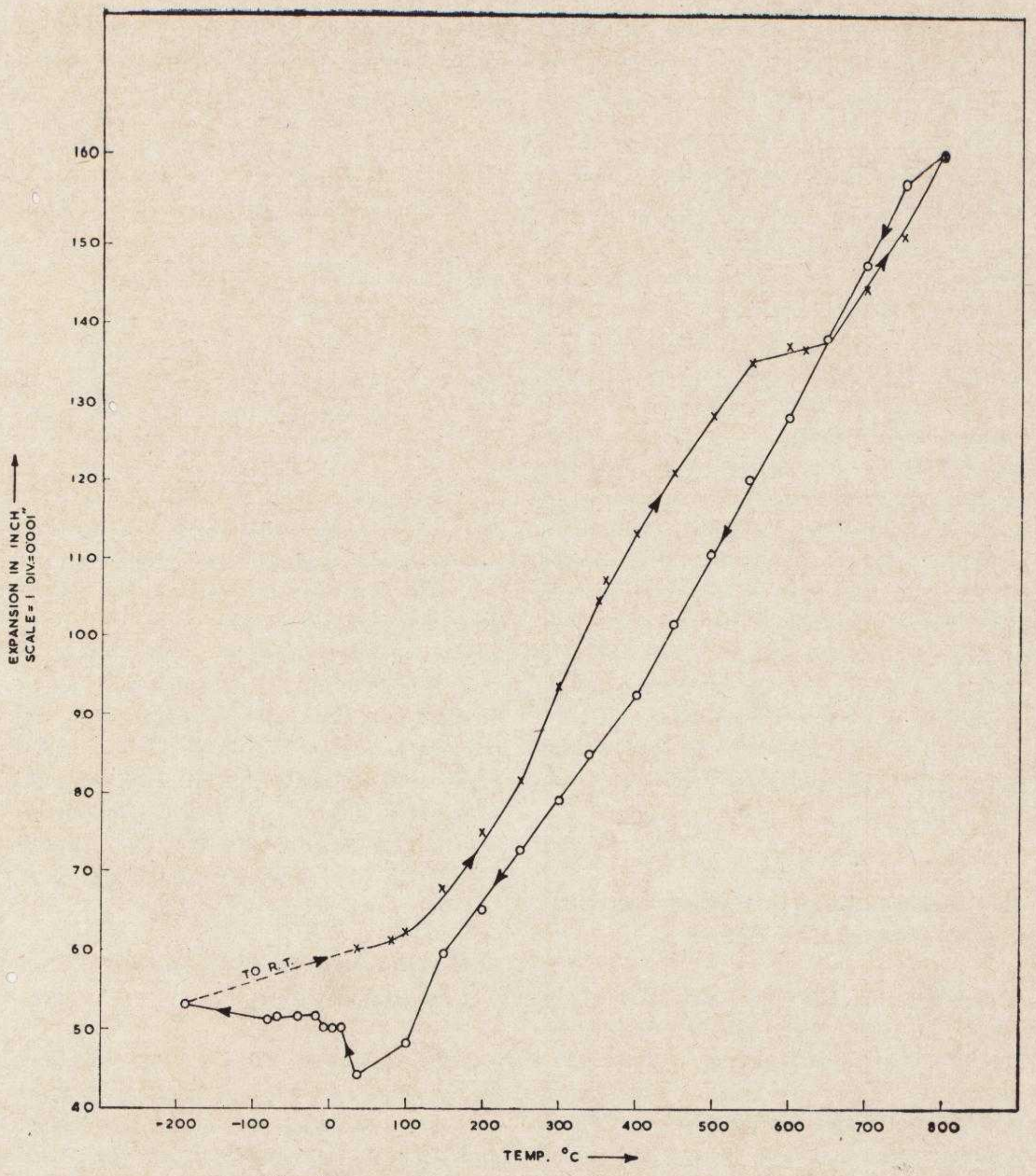


FIG. 65 — DILATION CURVE FOR Fe-13 PER CENT Mn ALLOY

chill cast and machined to 3 in. dia. by 10 in. long extrusion billets, and extruded at 400°C. with exit speed of 10 ft./min. to a rod of 0·752 in. dia. and with exit speed of 5·3 ft./min. to a rod of 0·785 in. dia. respectively. Preferred orientation developed during extrusion in the above specimen rods as a function of the position in the rod was studied.

49.0 Study on the Solubility of Nitrogen in Iron Alloys

Work was taken up to determine the solubility of nitrogen in different binary and complex alloys of iron in the solid state.

An experimental set up of Sievert's type for the determination of solubility of nitrogen in the solid state was fabricated.

This consisted in the purification of nitrogen moisture, arrangements for the introduction of a known quantity of nitrogen gas inside the system where the sample was equilibrated with nitrogen gas measuring burette for determining the rate of absorption of nitrogen. Since the experiments consisted in the accurate determination of the volume of nitrogen at a known pressure and at a constant temperature, it was assured that the system remained leak proof under vacuum for 24 hours by means of appropriate vacuum stop-cocks. The variations in the volume of gas due to change in ambient temperature were minimized by enclosing and circulating the gas measuring burette and the ends of the reaction tube with water maintained at constant temperature by means of a thermostat. The 'hot volume' of the system was found to be 80 ml. at 1000°C. measured at 0°C. and 760 mm. of Hg. Experiments were undertaken with pure iron alloy containing 5 per cent Cr.

50.0 Measurement of Carbon Activity in Iron and its Alloys

Equilibrium study between carbon dissolved in iron and definite mixture of carbon dioxide with carbon monoxide is important in order to get accurate knowledge about the activity of carbon in different phases of iron, heat of solution of carbon in different solid solutions, heat of transformation of the different forms of iron as well as the nature of the crystal structure.

Activity of carbon in austenitic iron at 800°C. and in Fe-Cr alloy at 1000°C. was studied. Transparent quartz reaction tube having a standard joint at its one end of diameter 2·54 cm. was used. The gas mixture was circulated over the laminated pure Fe sample till equilibrium was attained. Experimental results on equilibrium gas pressure relationship (p^2CO/p^{CO_2}) and final are shown below:

S. No.	p^2CO/pCO_2 (in atoms)	Final carbon content % by weight
1.	8·2	0·42
2.	10·2	0·48
3.	11·5	0·57
4.	15·4	0·69
5.	18·1	0·72
6.	19·4	0·81
7.	24·3	0·91

Activity of carbon in austenitic iron at 800°C. was calculated and the activity concentration diagram was prepared.

A few experiments were carried out to determine carbon activity in an Fe-Cr alloy containing Cr, 4 per cent. The alloy was prepared in a vacuum furnace by melting pure iron and chromium and was rolled to 10 thou. thick foils. Initial experiments indicated that it took 36 hours to reach equilibrium at 1000°C. and further studies are being carried out with varying carbon potential of the circulating gas.

51.0 Measurement of Viscosity of Slag System

The attainment of slag/metal equilibria for smelting or refining reactions depends on the viscosities of slags. Systematic and accurate measurements of slag viscosities were initiated both for arriving at adequate slag compositions for industrial processes such as production of ferro-alloys and also to visualize the structure of liquid slags.

A torsional viscosimeter was designed and fabricated in the laboratory. The viscosimeter works on the principle of coaxial cylinders with fixed outer crucible and oscillating inner spindle. The angle of torsion is recorded with the help of a pointer and a fixed circular scale. Arrangements were made to move and position a molybdenum wound furnace holding the crucible at the lower end of the viscosimeter. Care was taken to avoid external vibration such as that due to air draft for the oscillating part of the equipment. The equipment was standardized with the help of calibrating fluids.

52.0 Hydrogen Embrittlement in Steel

Hydrogen is a potential source of hairline cracks, shatter cracks, flaking and embrittlement in steel. In spite of extensive work carried out for the last two decades, the mechanism of embrittlement and the fundamental characteristics and properties of the hydrogen-iron system has been the subject of much research.

The safe level of hydrogen in steel is a matter of considerable argument as several other factors come into play. The purpose of this investigation is to find out the critical value of hydrogen in a particular alloy or steel to which they are susceptible to embrittlement. The work has covered the study of different steels and alloys after introducing hydrogen at different levels by cathodic charging or by thermal charging, i.e. by heating the sample in the atmosphere of hydrogen. Before carrying out the physical tests for hydrogen embrittlement, a study of the diffusion of hydrogen through the iron matrix after cathodic charge was made.

Cathode charge provides hydrogen pick up only on the surface layer. This hydrogen diffuses out of the sample when baked or kept for long time at room temperature. To homogenize the steel sample with hydrogen, the steel piece was plated with cadmium of 0·0003 inch thickness from a cyanide-bath and baked at different temperatures. Cadmium plating was done with the idea to retain the hydrogen picked up during cathodic charge. The steel sample used was a rod of 1/8 in dia. mild steel. From the experimental data, it was observed that when the sample baked at 200°F for 3 hours, 50 per cent of the impregnated hydrogen remained in the sample. Above that temperature, more hydrogen diffused and at 300°F most of the hydrogen was lost. From the graph (Fig. 66) it is evident that cadmium-coated steel can retain more hydrogen than steel without cadmium coating. If coating is non-porous, all the hydrogen cannot diffuse out even at 350°F after prolonged baking. Unless non-pervious coating is given homogenization, it is not perfect, and so attempts are made to improve the cadmium coating from other non-cyanide-bath. After proper homogenization of the test samples, physical test studies will be made for the assessment of hydrogen embrittlement effects.

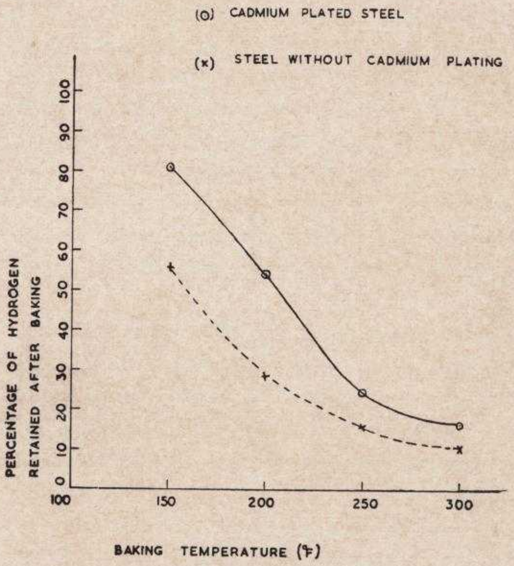


FIG. 66 — DIFFUSION OF HYDROGEN IN CADMIUM PLATED STEEL DURING BAKING

53.0 Development of Thermostatic Bimetals

Thermostatic bimetals are temperature-sensitive elements that are widely used in a great variety of ways for indicating, regulating and controlling temperature, and as thermal relays, circuit-breakers and overload protection devices. The entire requirements of bimetals are met to-day 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. For low expansion alloys Fe-Ni alloy containing 36-46 per cent nickel are the most common choice. Although for some special type of thermo-bimetals ferritic stainless steel or ferritic iron are also used as low expansion component. For high expansion alloys there is a wide variety of choice. Austenitic Fe-Ni-Mn alloys, Fe-Ni-Cr-Mn alloys containing nickel from 19 to 22 per cent, alpha brasses, Al-bronze, silicon bronze, nickel, nickel-silver, monel and lastly, Mn-Cu-Ni alloys, etc., are used. The choice is entirely dictated by the nature of use and the range of operating temperature.

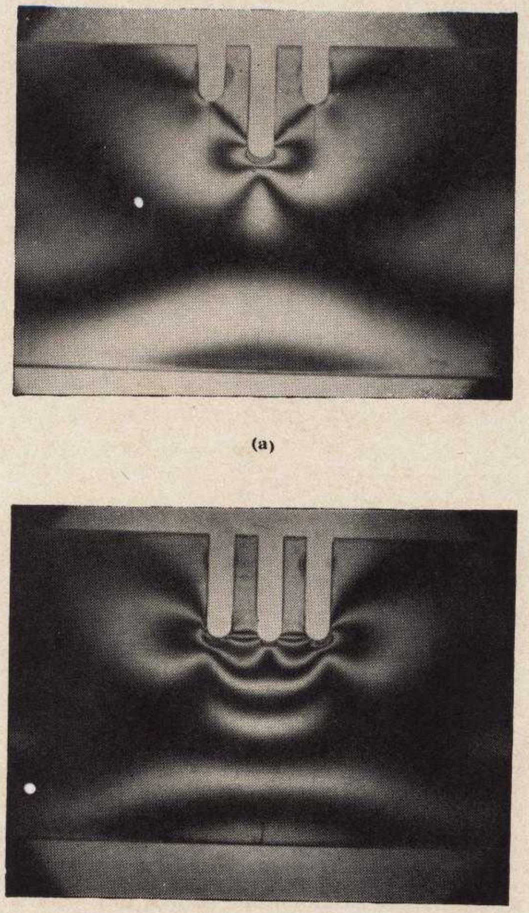
The production of all-ferrous thermo-bimetals (i.e. thermo-bimetals which contain ferrous alloys as both component) has been worked out which includes (i) melting and casting of ingots, (ii) hot working of ingot to slabs, (iii) surface preparations, (iv) bonding of the slabs, (v) hot rolling of the bonded composite to strips, (vi) pickling and cleaning, (vii) cold rolling, (viii) heat treatment, and (ix) testing. Besides, production costs under Indian conditions have been evaluated. The process has been released to industry for commercial exploitation.

Apart from the development of all ferrous thermo-bimetals, thermo-bimetals using non-ferrous alloys were also studied. The details of the production technology of thermo-bimetals with copper base alloys as high expansion component were worked out. Further progress was made for developing the technical know-how for the production of thermo-bimetals with Mn-Cu-Ni alloys as high expansion component. Good progress was made for working out the production technology for the manufacture of composite strips and wire for use as substitute for springs and conductors in the electrical industry.

54.0 Photo-elastic Stress Analysis

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

The evaluation of stress concentration factor was made from the photograph of the isochromatic stress pattern of a beam loaded under pure bending. But there was certain difficulty owing to the inaccurracy in measuring the maximum fringe order at the roof of the notch. So an alternative method of evaluating the stress concentration factor was developed. The load for every ½ fringe order was recorded and the appearance of a new fringe was observed by means of a short-form telescope. A graph was plotted with load against fringe order and from the slope of the st. line graph the stress concentration factor was determined.


(a)

(b)

Fig. 67 — Photograph Showing Variation of Stress Distribution Due to the Change of Relative Depth of the Side Notches [(a) Side Notches are half as deep as the Central Notches. (b) Three Equal Notches.]

Two series of observations were made in beams under pure bending. The variations of stress concentration factor and the per cent relief of stress at the root of a central notch with change of depth and closeness of two symmetrically cut side notches were noted (Fig. 67). Empirical formulae giving per cent relief of stress in terms of two parameters (i.e. relative depth and closeness of the two side notches) were obtained for each series of readings.

55.0 Study on Hot Working of Steel

Work was taken up with a view to study overheating and burning in relation to hot working of steel. Initially a study was made of the tests devised for determining the hot workability of steels, and also various aspects affecting it. Various factors, such as composition, rate and method of heating, furnace conditions and atmosphere and rate of cooling, etc., affecting overheating and burning were reviewed.

A low alloy steel of composition : C, 0·09; Mn, 0·72; Si, 0·27; P, 0·015; Cu, 0·34 and V, 0·12 per cent, was taken up for preliminary work. Two ingots were forged from 4 in. sq. section to 2½ in. sq., cut into four pieces each, 6-8 inches long. These were numbered 1·1, 1·2, 1·3 and 1·4 from first ingot and 2·1, 2·2, 2·3 and 2·4 from the second. These were then forged at varying temperatures as noted in the Table 27.

Table 27 — Number of Reheating and Forging Temperature of Steel Heats

Specimen No.	1·1	1·2	1·3	1·4	2·1	2·2	2·3	2·4
Temperature of working (°C.)	1100	1100	1200	1280	1300	1300	1300	1300
Number of Reheating	1	3	2	2	—	2	4	6

The final size of the forged ingots was 1 in. sq. in all cases. Specimens were pre- [illegible] the forged bars which were value and hardness. The microstructure of the samples were also studied. It was observed that as the time of heating at the forging temperature of 1300°C. increased, the tensile strength, hardness value and the grain size increased progressively whereas the impact value and the percentage of elongation decreased. Further, a clear change in microstructure was observed with the increase of time of heating. The work on interpretation of the changes in the structure is in progress.

56.0 Study on Internal Friction of Metals

The aim of the project is to study the phenomenon of strain ageing, temper brittleness and interactions of interstitial solute atom of nitrogen and carbon in iron through measurement of internal friction. Study of ageing behaviour of Fe-N and Fe-Mn-N alloys was undertaken by means of internal friction technique. A simple low frequency (1-2 cycles per sec.) torsion pendulum apparatus designed and fabricated at the National Metallurgical Laboratory was used. Some minor modifications in the apparatus were made for facilitating handling.

The alloy taken was an Fe-Mn-N alloy of the composition Mn-0·66 and N_2-0·013 per cent (analysed chemically). This was hot forged and finally wire drawn to 19 SWG size for experiments. The specimen length was kept to about 3¾ in. for getting about 1·8 cycle per sec. frequency. Internal friction measurements were made in the solution treated, aged, and sometimes in strained and aged conditions. Care was taken to ensure measurements quickly as possible after each treatment. Solution treatment of the specimens was done in hydrogen atmosphere at 550°C. for one hour after which the specimen was water quenched. Care was taken not to cause handling during setting up in the internal friction apparatus. Ageing was carried out at 46°C. and 100°C. Earlier the ageing was done in a separate oven. But in later experiments, ageing inside the internal friction apparatus itself was adopted to keep other conditions same as far as practicable. Straining was done in a tensile testing machine up to a maximum of about 3·5 per cent strain.

The results of the internal friction measurements on a typical sample solution treated at 550°C. for 1 hour, followed by water-quenching and final ageing at 46°C., showed a continued drop of the peak height with time, up to the ageing time of about one week. The same specimen was then strained by 3·25 per cent. No increase in the height of the peak resulted due to the straining. With further ageing, the height of the peak continued to decrease with time up to about five days under the same ageing treatment as before. A fresh restraining of the sample by the same amount, however, showed an increase in the peak height. Further reageing resulted in the reduction of the peak height again.

Three new heats were made by using low residue iron at different manganese levels while keeping the Mn/N ratio constant with a view to study the effect of manganese on the binding of nitrogen to dislocations.

57.0 Study on the Effect of Nitrogen on the Transition Temperature

An understanding of the contribution of nitrogen alone and in combination with other elements to the embrittlement of steels is important. The effect of nitrogen in combination with manganese on the ductile-to-brittle transition temperature has not been fully clarified yet. Work was undertaken to investigate this aspect of the problem of brittle fracture.

Several experimental heats were made with varying amounts of manganese and nitrogen. Low-residue iron containing C, 0·03 and Si, 0·07 per cent was alloyed with electrolytic manganese. Nirtided electrolytic manganese was used to introduce nitrogen into the bath. A combination of nitrided and unnitrided electrolytic manganese was used to attain various manganese and nitrogen levels. Low-residue iron was deoxidized with silicon before the addition of manganese. Deoxidation was important in view of the fact that low-residue iron charge was used. Residual oxygen would otherwise also contribute to the embrittlement of steel and make the evaluation of the effect of nitrogen very complicated. Moreover, there would be higher loss of manganese due to its interaction with oxygen. Aluminium was not used at any stage for deoxidation because of its affinity for nitrogen. The analysis of heats made so far are given below:

Sl. No.	Mn (%)	Total nitrogen (%)
1	0·35	0·016
2	0·78	0·011
3	0·84	0·012
4	0·45	0·032
5	0·81	0·038
6	0·32	0·052
7	0·47	0·054

C was in all cases less then 0·04 per cent and Al in traces. The gas contents were analysed by the vacuum fusion method. The heats were cast into 2 in. square ingots, conditioned and forged to ½ in. sq. bars. The bars were then normalized and V-notch Charpy impact specimens machined out of them. The specimens were tested at room temperature and at various lower temperatures up to −70°C. in a 264 ft. lb. Olsen impact testing machine. For carrying out low temperature impact tests, the specimens were kept in a petroleum ether-bath which was cooled to the required temperature by the addition of liquid air. The time between taking the specimen out of the bath and its fracturing was less than 3 seconds. The specimens were kept at the test temperature for 15 min. before testing. The values of energy absorbed in fracturing were noted and the percentage crystallinity on the fracture surfaces evaluated.

58.0 Production of Basic Refractories from Indigenous Resources

(i) *Almorah Magnesite*

This investigation was taken up with a view to assess the suitability of magnesite from Almorah region for the manufacture of basic refractories. Earlier work on the magnesite received from Doba and Agargirichchina sector indicated encouraging results. The U.P. State Industrial Development Corporation, who are developing the magnesite mines in the Dewaldhar sector of the Almorah region, with an ultimate objective of setting up a sintering plant, sent magnesite samples from different blocks for assessing the sinterability and quality.

The magnesite, as received, was coarsely crystalline. The chemical analysis as indicated in Table 28 shows that lime content, in contrast to the Agargirichchina sample, was low. On firing to 1600°C., most of the samples showed extensive cracking and some even crumbled when crushed by hand. This was due to the original texture of the ore which was subjected to tectonic crushing. On calcination to 1700°C., the friability decreased and many fired samples

Table 28 — Chemical Analysis of Different Blocks of Magnesite from Dewaldhar Sector

	Block Nos.				
%	III	IV	V	VI	VII
Moisture	0·06	0·06	0·08	0·08	0·02
L.O.I.	50·20	50·04	50·82	51·02	50·2
SiO_2	1·44	1·20	0·84	0·80	1·12
Al_2O_3	2·42	1·90	1·38	1·64	3·08
Fe_2O_3	0·50	1·70	1·56	0·96	0·80
CaO	0·87	0·50	1·08	0·98	0·60

were dense. Addition of mill scale 2 per cent and firing to 1600°C. did not counteract the tendency to friability. Other physical properties of the specimens made from the grog were determined. Experiments were also done to evaluate the suitability of Almorah magnesite for the manufacture of chrome-magnesite refractories.

(ii) *Suitability of Some Dolomite Samples for Use in Steel Plant Refractories*

The investigation was taken up at the instance of Hindustan Steel Ltd. Limestone Development Cell to examine the suitability of dolomite from Bhawanthpur, for its sintering characteristics and physical properties.

The dolomite sample was coarsely crystalline and showed silica, 0·80; magnesia, 21·6; lime, 30·40; alumina and iron oxide combined 0·96 per cent. The sample was calcined to different temperatures from 1500° to 1700°C at 50°C. interval and bulk density and hydration resistance properties were tested. It was found that unless a high temperature of the order of over 1600°C. was used, the bulk density of the calcined material was low and could not be used as such. Similar experiments were carried out giving the test samples a coating of millscale which greatly improved the bulk density and hydration resistance and the material could be used as refractory purposes.

(iii) *Stabilization of Dolomite from Cuddapah and Melur*

This investigation was taken up at the instance of Messrs. Neyveli Salem Lignite Steel Corporation to determine the suitability of two samples of dolomite one from Cuddapah, Andhra Pradesh, and the other from Melur, Madurai, for steel plant refractory. Preliminary calcination experiments showed that both the dolomites contained large amount of siliceous material and the samples after calcination formed dust. The Melur dolomite was found to be not dolomite but limestone and it completely dusted. The Cuddapah dolomite also had a high silica content and partly dusted. Both the dolomites were not suitable for making refractory compositions for steel plants.

59.0 Studies on Carbon Refractories

The objective of this project is to study carbonaceous materials from the points of view of their ultimate utilization as raw materials for the production of carbon products for diverse application in metallurgical and chemical industries.

Work was carried out on: (i) production of dense aggregates from Assam coals, (ii) study on the suitability of Bhilai pitch as a binder and raw material for coke production, (iii) work on the suitability of ' Phillips ' high temperature pitch for low-ash coke product — undertaken at the instance of Phillips Carbon Black Ltd., Calcutta, and (iv) work on the suitability of ' E.I.D. Parry ' carbon nodules for carbon products — undertaken at the instance of E.I.D. Parry Ltd., Madras.

(i) *Study of Assam Coals*

The materials after sampling were passed through jaw and roll crushers to obtain

—10 mesh powders which were further treated in a ball mill to obtain —22, —44, —72 and —100 mesh B.S.S. powders. Sieve analyses, packing densities, proximate analyses, etc., were determined by standard methods. Mix compositions were prepared from these materials using road tar as binder in various proportions by weight. The green compacts were then fired in reducing atmosphere at 1300°C. per pre-determined schedule. After the firing, apparent porosities, bulk densities, apparent specific gravity and crushing strengths of these aggregates were determined and the following conclusions arrived at.

It was possible to make sound carbon aggregates out of Assam coals though their bulk densities and apparent specific gravities are low suggesting considerable intraparticulate porosity. The more important factors affecting the properties of the final compacts were as in the case of petroleum coke, granulometric composition of coal powders, the nature and quantity of binders and heat treating schedules. Introduction of char in these bodies in certain proportions appeared to be beneficial in that it increased the compression strength considerably. Utilization of finer coal and char powders and binder with higher coking value may bring in further improvements.

(ii) *Bhilai Pitch*

In this study, several binders along with Bhilai pitch were used to find out its suitability as a binder in comparison to others. For this purpose, calcined petroleum coke was used to make dense aggregates using road tar, electrode pitch and Bhilai pitch as binder. The green compacts after being fired at 1300°C. were subjected to the physical properties determination, viz. apparent specific gravity, bulk density, crushing strength and electrical resistivity. Table 29 shows the physical properties of compacts from different binders using 16 per cent in each case.

Table 29 — Physical Properties of Compacts Using Different Binders

Binder	Apparent porosity %	Bulk density gm/cc.	Compression strength P.S.I.	Electrical resistivity ohms/cm^3 × 10^{-3}
Road Tar	27·4	1·50	2200	15·8
Electrode pitch	24·3	1·53	3300	18·4
Bhilai Pitch	24·5	1·54	3400	19·7

The important conclusions obtained from this study were as follows:

(a) It was possible to use the Bhilai pitch as a binder in the production of carbon products.

(b) Bhilai pitch needed a defrothing treatment at 180° ± 5°C. in order to render it bubble-free at the mixing temperature.

(c) The mixing and forming temperatures for Bhilai pitch bodies were significantly higher than those for electrode pitch and road tar.

(d) Bhilai pitch bodies suffered less weight-loss during baking than electrode pitch and road tar bodies containing equivalent proportion of binder, indicating increased amount of residual carbon in the case of Bhilai pitch bodies.

(e) The physical properties of compacts made with Bhilai pitch as binder showed marginal superiority in certain respects over those of specimens made with electrode pitch as binder. While the data were insufficient to prove that the Bhilai pitch was superior, these indicate that it was at least as good a binder as electrode pitch.

(iii) *'Philips' Pitch as a Raw Material for High Purity Coke*

it was indicated that it was possible to obtain a calcine from this pitch very similar in electrical and strength properties to calcined petroleum coke available in the market. In order to do this, the pitch had to be first subjected to a thermal pretreatment. The residue from this treatment was a product very similar to uncalcined petroleum coke in texture and other characteristics. Calcination of this residue anaerobically to any present maximum temperature yielded a carbon with desired physical properties. In the preliminary trials, pretreatments were conducted under aerobic conditions at atmospheric pressure. Table 30 gives some relevant data about the product as compared to uncalcined petroleum coke.

The data indicate that the pitch is a good and promising raw material for production of pitch-coke and also dense carbon aggregates. Regarding the use of the pitch as a binder, it was observed that it was not suitable for this purpose because it needed a high mixing temperature at which the pitch lost its workability very rapidly.

(iv) *E.I.D. Parry Carbon Nodules for Carbon Products*

Studies were made on these nodules in order to find their suitability in carbon industries. It was found that these could be used as a carbon black. It might also be possible to use these pellets as granulated

60.0 Study on Some Refractory Clays from Jammu and Kashmir

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

The first part of this investigation which was completed, comprised of mineralogical examination of these clays by differential thermal analysis and studies on their physico-chemical characteristics and refractory properties. The chemical analysis and P.C.E. values of the clays given in Table 31 indicate that the clays are rich in Al_2O_3 content and are highly refractory.

The differential thermograms of the clays are given in Figs. 68 (a & b). An endothermic peak between 600° and 610°C. were observed on the thermograms of clays C_3, C_5, C_6 and C_9 suggesting that they are predominantly kaolinitic in character. Clays BC_4, BC_5 and BC_6 gave thermograms with two endothermic peak

Table 30 — Comparison of Properties of 'Philips' Pitch and Calcined Petroleum Coke

Material	Volatile loss in pretreatment %	Volatile loss in residue at 1300°C.	Calcination temperature °C.	Specific resistivity ohms/cm.^{2}		
				0·21 cm. column	1 cm. column	2 cm. column
Philips pitch	22·3	10·0	1300	0·009	0·016	0·024
Uncalcined petroleum coke	—	11·0	1300	0·021	0·025	0·034

Table 31 — Chemical Analysis and P.C.E. Values of Clays

%	Clays							
	C3	C5	C6	C9	BC4	BC5	BC6	BC9
Moisture	0·32	0·32	0·20	0·16	0·70	0·72	1·02	0·88
L.O.I.	12·88	12·30	13·60	14·88	14·70	14·92	14·54	14·76
SiO_2	43·84	43·48	42·40	44·80	27·48	20·88	35·74	41·22
Al_2O_3	38·31	38·84	39·98	36·80	50·29	58·92	41·96	41·27
Fe_2O_3	1·44	1·76	1·92	1·52	2·40	1·76	4·48	0·88
TiO_2	1·05	1·20	1·70	1·10	1·41	1·42	1·16	0·55
CaO	Tr.	Tr.	Tr.	Tr.	Tr.	Tr.	Tr.	Tr.
MgO	0·18	0·109	0·14	0·54	Tr.	Tr.	Tr.	Tr.
Na_2O	0·80	0·80	0·45	0·32	0·90	0·50	0·05	0·04
K_2O	0·69	1·01	Tr.	0·40	1·60	0·50	0·50	0·55
Flux	4·16	4·88	4·21	3·88	6·31	4·18	6·19	2·02
SiO_2/Al_2O_3	1·145	1·119	1·060	1·214	0·4634	0·3544	0·8517	0·9988
P.C.E. (orton cone)	34	34	34	34	36	36	32½-33	35-36

between 930° and 960°C.; the first endothermic peak appeared at 565°C. in clays BC_4 and BC_5 and at 540°C. in BC_6 whereas the second endothermic peak appeared at about 595°C. in all of them. The temperature of the endothermic and exothermic peaks of clay BC-9 fell within the range of those shown by C-clays. A comparison of the thermograms of BC clays with those of some flint and high alumina clays reported in the literature suggested that the first endothermic peak in clays BC_4, BC_5 and BC_6 was due to the presence of the mineral diaspore (aluminium monohydrate) and the second endothermic peak due to kaolinite. Thus, these clays appeared to be mixtures of kaolinite and diaspore in varying proportions. The relative intensities of the first and second endothermic peaks appeared to bear a good relationship with the relative proportions of diaspore and kaolinite present in them. From the above data on chemical analysis, mineralogical examination by D.T.A. and refractoriness of these clays, it was considered that these belong to a unique variety of high alumina clays of our country and match well with some of the diasporic and burley flint clays found in some parts of U.S.A. and France.

Some of the physico-chemical properties examined for these clays are given in Table 32. Although the dry strength of these clays was not high, these developed good strength on firing to temperatures above 1200°C. All these clays promised potential scope for application in the manufacture of superduty fireclay refractories.

In the second part of this investigation under progress, it is proposed to formulate suitable mixes from these clays for the production of superduty fireclay refractories and study their properties.

61.0 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, from the beach sands of Travancore.

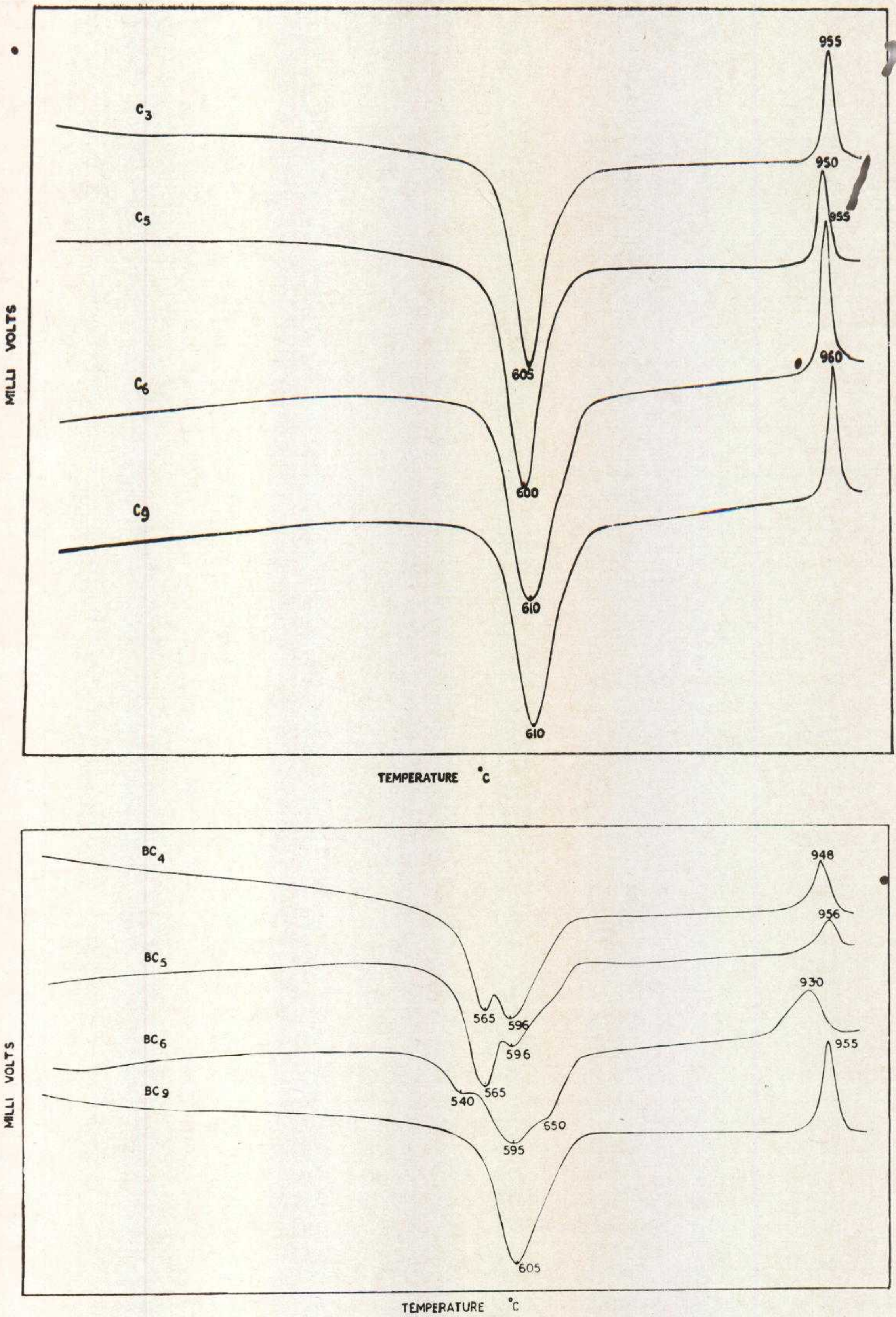


Fig. 68 — D.T.A. Curves [(a) Clay (c), (b) Bauxitic Clays (Bc)]

Table 32 — Physico-Chemical Properties of the Clays

	Clays							
	C3	C5	C6	C7	BC4	BC5	BC6	BC9
Slaking tendency	Started loosening into course grains after 21 secs.	No change	No change	No change	No change	No change	No change	No change
pH	7·14	6·85	6·99	6·95	7·2	7·1	6·9	7·25
Base exchange capacity m.e/100 gm.	4·986	5·318	5·760	5·052	6·356	7·200	10·80	7·030
Water of plasticity %	25·5	30·2	22·1	22·2	21·8	23·8	23·4	21·9
Workability	good	very good	bad	Fair	Fair	Bad	Fair	Fair
*Linear drying shrinkage %	2·5	2·6	2·9	2·7	2·5	2·2	3·0	2·7
Dry modulus of rupture lb/sq. inch	107·4	75·5	66·30	72·96	77·73	58·15	78·32	68·6
*Linear shrinkage % on firing to								
600°C.	0·1	0·3	nil	nil	0·1	+0·3	nil	0·6
1000°C.	0·96	1·4	0·96	0·70	0·74	0·15	1·18	0·95
1200°C.	4·1	4·5	2·5	1·7	2·5	1·7	5·0	2·0
1400°C.	6·0	8·0	6·1	4·57	7·0	3·0	11·5	5·0
1500°C.	14·02	13·8	14·9	11·50	15·36	6·90	16·27	12·83
*App. porosity % on firing to								
1200°C.	23·40	30·60	30·40	25·99	33·79	33·44	36·00	33·36
1300°C.	23·40	27·99	29·07	24·17	32·92	33·50	32·00	28·00
1500°C.	3·20	1·20	2·50	1·70	12·30	25·70	2·60	1·60
*M.O.R. lb/sq. inch. on firing to								
1200	2550	3480	1218	1676	1466	1030	2040	1177
1300	3602	4807	2450	2901	1863	902	4158	2398
1400	5435	6408	3967	3156	4634	1077	6457	2954

*Specimens were prepared from 50: 50 clay grog mixes.
+sign indicates expansion.

Kerala beach sand sillimanite ball milled for 216 hours was taken and the effect of electrolytes like sodium carbonate, sodium silicate and the mixtures of sodium carbonate, sodium silicate on the *p*H and mobility of aqueous suspensions of the sillimanite powder was studied. A Mariott tube was used to measure the mobilities and the time taken for 75 cc. of the suspension to pass through was determined. It was found that with Na_2CO_3 and Na_2SiO_3 as electrolytes, there was decrease in the efflux time of the slips up to a certain limit, after which this time remained constant. Having selected the slip with minimum efflux time and minimum quantity of electrolyte, other properties like total shrinkage and porosity of the cast articles on firing were studied.

61.1 Slip Casting of Sillimanite Tubes

The water content of a casting slip used to make furnace tubes was 40 per cent. The electrolyte used was 0·2 per cent, on the basis of 100 per cent dry powder. The specific gravity of the slip was 1·83 gm/cc. *p*H was 8·65. The fluidity for 100 cc. from 30-130 cc. was 49·5 sec. through the Mariott tube. With this slip, 3 in.

Table 32 Values for pH-fluidity Relationship for Sillimanite Slips Treated with Na C
Tube Was Standardized by Noting the Efflux Time for 75 cc. Distilled Water from 25 cc. to 100 cc. Mark with 160 cc. Pressure Head : It Was 2 seconds

Electrolyte solution	% used for 100 gm. dry sillimanite	Unaged slips		Aged slips	
		*p*H	Mariott tube Fluidity (sec.)	*p*H	Mariott tube fluidity (sec.)
10% Na_2CO_3	0·16	8·4	19	8·45	No flow
	0·18	8·4	19	—	—
	0·2	8·6	10	8·6	17
	0·24	8·8	9·5	8·8	12-24 varying
	0·26	8·8	8·5	8·87	10
	0·28	8·9	8·0	8·9	10
	0·40	9·3	8·7	9·15	settling slip
10% Na_2SiO_3	0·22	9·5	90	—	—
	0·26	9·85	15	—	—
	0·30	10·2	15 (varying)	—	—
	0·34	10·4	10·5	—	—
1:1 mixture of 10% Na_2CO_3 & 10% Na_2SiO_3	0·22	9·35	12·0	9·1	40
	0·23	9·4	8·6	9·2	17
	0·24	9·4	7·8	9·15	16
	0·26	9·5	7·6	9·3	11

O.D. and 9 in. long refractory tubes, open at one end, were made and fired at 1500°C. at roughly 50°C./hr. 3 hr. soaking was given. The resultant tubes had a total shrinkage from mould to fired state of 16 per cent (roughly). The apparent porosity was 6·5 per cent and bulk density was 2·3 gm./cc. These tubes were straight and sound. Further trials to make more impervious tubes with a better control of making conditions were undertaken. Some tubes could be extruded out of ball-milled sillimanite using organic binders in a small-de-airing type of pug mill. Table 33 gives some results of experiments.

62.0 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, Messrs. 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.

Work was carried out (i) to overcome some of the operational difficulties of the erosion test apparatus design of which has been described in the previous annual report and (ii) to study the effect of forming conditions on the properties of refractories made from mixtures of clay and grog.

(i) The erosion test apparatus, as first designed, had difficulty in attaining temperature adequate for steel melting. The flames impinging on the crucible would be diverted downwards rendering the rotating mechanism unduly hot. A larger capacity burner positioned tangentially solved both

Table 34 — Physical Properties of Compacts Prepared

Size of the grog		Clay %	Forming pressure P.S.I.	Water content %	Apparent porosity %		Bulk density gm/cc.		Cold crushing strength P.S.I.		Linear shrinkage %	
					at 1150 °C.	at 1250 °C.	at 1150 °C.	at 1250 °C.	at 1150 °C.	at 1250 °C.	at 1150 °C.	at 1250 °C.
(i) Variation in size of the grog												
Passing 5 mesh sieve		50	500	19	26·9	24·0	1·9	2·00	6815	5179	0·79	1·14
Passing 10 mesh sieve		50	500	19	31·0	26·9	1·85	1·93	3925	3709	—	0·45
Passing 18 mesh sieve		50	500	19	31·0	29·3	1·89	1·88	3559	4380	1·53	0·99
Passing 42 mesh sieve		50	500	19	28·7	27·2	1·90	1·91	5080	7362	2·5	2·59
(ii) Variation in clay/grog proportion												
% of grog												
18 mesh	50	50	500	19	31·0	29·3	1·89	1·88	3559	4380	1·53	0·99
,,	60	40	500	19	26·6	27·1	2·00	1·91	6482	7996	1·00	1·60
,,	70	30	500	19	29·6	29·6	1·88	1·88	2747	4405	2·75	2·44
,,	80	20	500	19	28·1	28·7	1·9	1·88	3109	4105	0·41	—
(iii) Variation in water content of the mix												
18 mesh	50	50	500	17	28·0	27·9	1·87	1·91	4277	5461	3·44	3·5
,,	50	50	500	19	31·0	29·3	1·89	1·88	3559	4380	1·53	0·99
,,	50	50	500	21	28·0	26·9	1·91	1·94	4176	6765	2·77	3·64
(iv) Variation in forming pressure												
18 mesh	50	50	400	19	29·1	27·3	1·88	1·91	4031	4196	1·18	1·49
,,	50	50	500	19	31·0	29·3	1·89	1·88	3559	4380	1·53	0·99
,,	50	50	600	19	28·7	26·05	1·91	2·00	3662	3604	1·76	Irregular
(v) Effect of vacuum												
Without vacuum	50	50	500	19	31·0	29·3	1·89	1·88	3559	4380	1·53	0·99
With vacuum	50	50	500	19	25·9	25·6	1·94	1·95	4867	7675	1·26	1·4

the problems. Arrangements were made for running quantitative tests on certain standard samples and standardize test conditions of the apparatus.

(ii) With Rajhara clay and grog made from the same, different bodies were compounded varying the granulometric composition as well as the particle shape of the grog using 50: 50 clay grog mix. These mixes were homogenized with enough water to render them extrudable. Bars were extruded with and without application of vacuum. These bars were repressed at various pressures and the compacts thus formed were fired to various temperatures. Physical properties were determined before and after firing. Table 34 gives the physical properties of the bodies at firing temperatures of 1150°C. and 1250°C. attained at a rate of 50°C./hr. with 3 hours soaking.

63.0 Investigation of Pyrophyllite

This work was taken up at the instance of Messrs. Assam Sillimanite Co. Two samples of pyrophyllite — one red variety and another white variety — were received. The objective was to study the suitability of these pyrophyllite samples for making refractory compositions.

From a literature survey it was found that pyrophyllite was mainly used in pottery industry. In refractory industry its only use was for making casting pit refractory. Tests were carried out to determine the suitability of this raw material for making stoppers, nozzles and ladle bricks. The chemical analysis is given in Table 35.

The analysis shows that the samples are typical pyrophyllite containing high percentage of iron oxide. The red variety contains about 6·92 per cent Fe_2O_3. Pyrometric cone equivalent of ‘ Cut ’ cone and powdered samples show a value of cone 27-28 (1650°C.) for white pyrophyllite and for red pyrophyllite cut cone showed a value of cone 23 (1586°C.).

Table 35 — Chemical Analysis of Samples

%	White	Red
Moisture	Nil	Nil
Loss	5·11	4·36
SiO_2	63·25	67·2[illegible]
Al_2O_3	27·88	20·[illegible]2
Fe_2O_3	2·9	6·92
TiO_2	0·36	0·32
CaO	0·097	0·12
MgO	0·51	0·57

After crushing the raw material 5 cm. dia. × 5 cm. ht. cylindrical specimens were pressed together with additions of plastic fire clay and fired in gas-fired kiln from 1000°C. to 1400°C. and permanent linear change, bulk density and porosity were determined on the fired specimens. Upto a temperature of firing 1300°C. all the specimens showed about 2 per cent permanent linear expansion. For lower firing temperature, porosity was between 20-25 per cent and bulk density 1·90 to 2·2. For firing at 1400°C. some of the compositions showed negligible shrinkage with consequent lowering down of porosity and increasing in bulk density. A small glass apparatus was rigged up to determine the permeability of the test specimens. Crucibles, about 6 in. × 3 in. dia. were pressed with some composition and fired to conduct a sort of simulative test wherein liquid steel will be poured in these crucibles to see their behaviour.

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

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

Scope

The tests to be carried out are as detailed below:

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

Accordingly, a large number of samples of fireclay and magnesite stoppers and nozzles from different producers and consumers as well as imported were received for study. Chemical analysis of some of the nozzles and stoppers received are given in Table 36. Other properties are under study.

65.0 Study on Refractory Plastics and Castables

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

A plastic refractory is generally a mixture of a graded refractory aggregate and a plastic clay whilst a castable refractory contains a hydraulic setting refractory cement and graded refractory aggregate with some chemical binder being added if necessary. Not only can the mixture be rammed, moulded or cast, but these may also be gunned in position. These refractory specialities are manufactured in our country only to a very limited extent because (i) suitable hydraulic setting cements and binders that can stand high temperature use have, hitherto, not been developed or manufactured in our country and (ii) a systematic formulation of a wide and increasing variety of these mixes suitable for different applications in ferrous

Table 36 — Chemical Analysis of Stoppers and Nozzles

%	B.F.S.	B.F.N.	I.S.N.	I.S.S.	M.N.	M.S.
Moisture	0·05	0·05	0·16	0·12	0·12	0·14
Loss on ignition	0·36	0·15	0·34	0·50	0·20	0·28
SiO_2	52·24	46·92	52·28	55·88	56·64	53·36
Al_2O_3	36·77	42·22	37·48	32·48	36·00	40·36
Fe_2O_3	3·20	3·0	4·96	5·76	3·52	2·56
CaO	1·23	1·10	0·91	0·87	0·22	0·44
MgO	1·03	1·61	1·70	1·90	1·50	1·47
TiO_2	3·53	3·18	1·76	1·60	0·96	0·88

other high temperature furnaces necessitates drawing up acceptable specifications and standard refractory cements resulting in processes for the manufacture of pure calcium-aluminate and high alumina cements from indigenous materials as a first step in this direction. In the present study, it was, therefore, proposed to take up the latter aspect in detail.

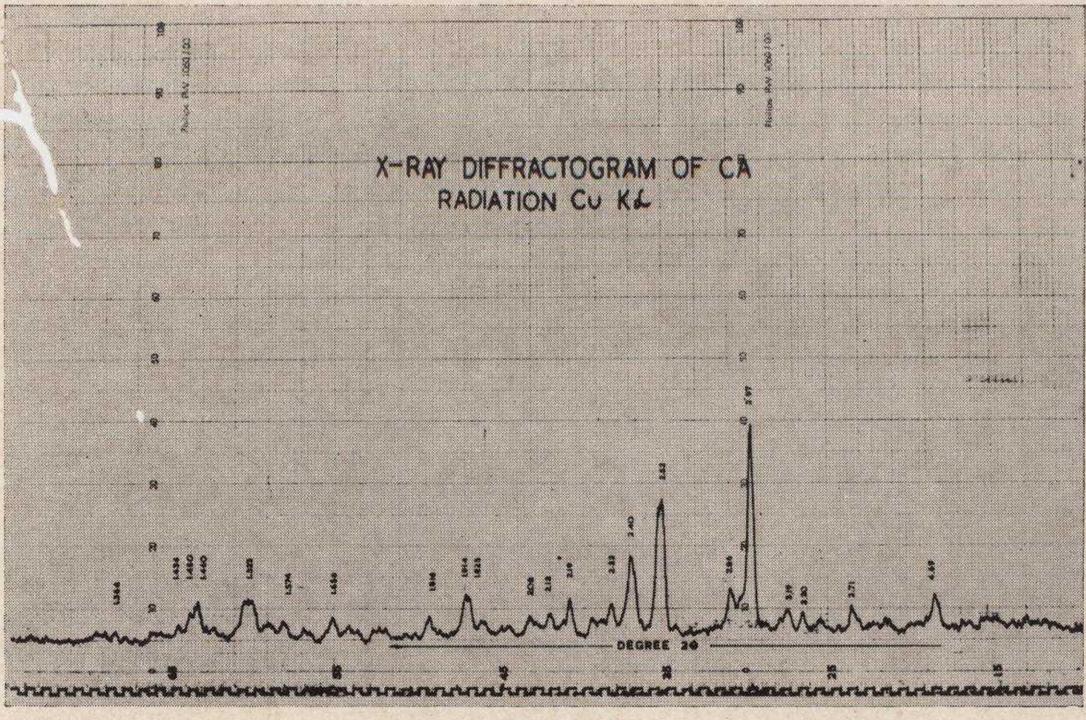
In order to evaluate the quality of the indigenous products, some samples of refractory plastics and castables were collected from different manufacturers of our country with the help of the Indian Standards Institution who are also incidentally interested in drawing up suitable specifications for these products. On the basis of a literature survey carried out on the various testing methods for refractory castables and plastics adapted in some foreign countries, a tentative procedure for testing has been drawn up and a preliminary examination of these samples is under progress. In Table 37 some properties of refractory plastics so far examined are given.

Table 37 — Properties of Plastic Refractories

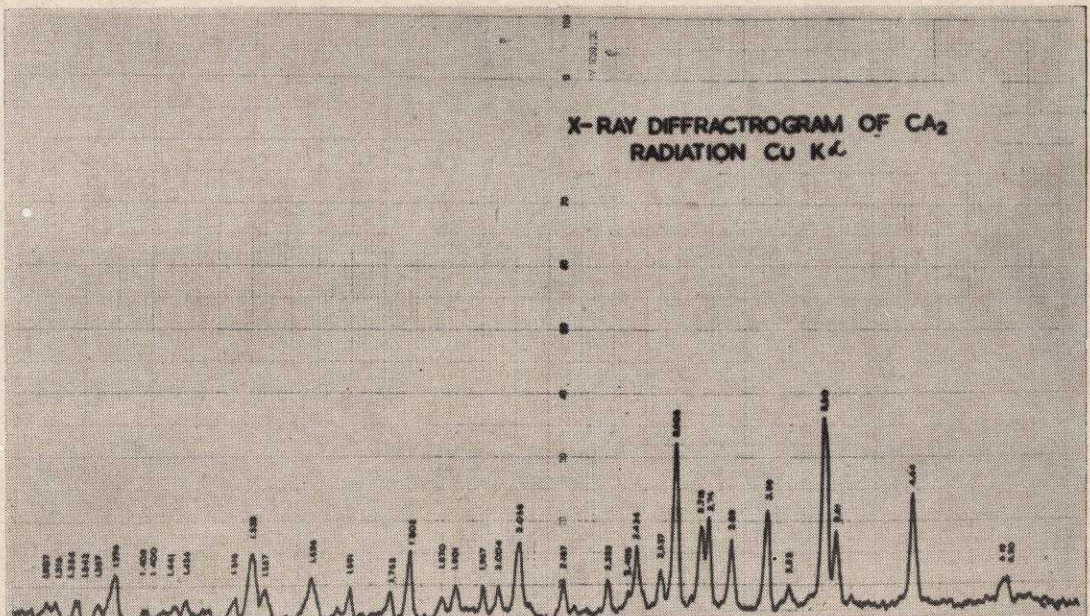
	1	2	3	4	5
Chemical analysis: %					
L.O.I.	2·30	3·80	3·66	1·56	1·88
SiO_2	31·40	36·56	38·80	10·52	57·64
Al_2O_3	55·90	51·20	52·28	66·94	34·76
Fe_2O_3	4·40	5·36	3·36	4·80	1·91
TiO_2	1·14	2·40	1·20	3·20	1·97
CaO	4·67	Tr.	Tr.	12·08	0·88
MgO	Tr.	Tr.	Tr.	0·56	0·43
Alk	—	—	—	—	—
Sieve analysis:					
—5 + 25 B.SS.	40%	58%	28%	63%	55%
—25 + 100 ,,	20%	25%	37%	23%	20%
—100	40%	17%	35%	14%	25%
Moisture	0·36%	0·42%	0·12%	0·21%	0·28%
Workability index	43·9	15·7	16·3	16·0	40
Permanent Linear change (%) on firing to					
100°C.	0·12	—0·52	—0·51	—0·51	0·05
1600°C.	—0·56	3·70	—0·20	—2·60	—1·62
1650°C.	—1·47	2·49	—1·86	—2·90	—1·86
1700°C.	—4·55	—	—3·14	—3·36	—
Transverse Strength ($lb./in^2$) on firing to:					
1650°C.	2060	1312	1998	1380	1875
1500°C.	1890	1155	960	630	1050
1400°C.	1035	1260	900	570	450
1300°C.	580	750	700	450	375
1200°C.	263	750	405	473	240
1100°C.	188	788	338	300	300
100°C.	100	105	109	73	—
Bluk density gm/cc. on firnig to					
1650°C.	2·27	1·92	2·10	2·16	2·04
1400°C.	2·12	1·99	1·99	2·05	2·02
1300°C.	2·07	2·07	1·95	2·07	2·07
1200°C.	2·09	2·07	1·97	2·05	2·09
1100°C.	—	—	—	—	—
App. porosity % on firing to					
1650°C.	22·4	16·5	26·6	17·2	12·0
1400°C.	30·3	21·8	32·8	27·2	22·9
1300°C.	33·5	20·3	35·3	29·6	24·5
1200°C.	35·4	20·1	36·5	28·2	28·0
1100°C.	—	—	—	—	—

66.0 High Temperature Phase Equilibria in Some Refractory Mineral Systems Involving CaO-MgO-Al_2O_3-SiO_2

Studies on the phase equilibria in the four component system $CaO.Al_2O_3$(CA), $CaO.2Al_2O_3$(CA_2), gehlenite and spinel were continued. Equilibrium studies in the solid stare as well as above the solidus were catried out. It was found that the compounds CA, CA_2, gehlenite and spinel were compatible in the solid state. X-ray examination of the fired mixes did not indicate the formation of any solid solution. X-ray diffraction studies on the CA and CA_2 showed close agreement with the results of previous workers (Figs. 69a & 69b).



69(a)



69(b)

FIG. 69 — X-RAY DIFFRACTOGRAM [(a) CA radiation Cu Kα, (b) CA$_2$ radiation Cu Kα]

The melting points of CA and CA_2 were determined by hot-stage microscopy. The binary systems CA_2-spinel CA-spinel have been investigated (Figs. 70 and 71). The system CA_2-spinel forms a binary eutectic at 13·7 per cent spinel at 1700°C. Further work on the system CA-spinel showed that it was not a true binary as CA_2 appears as a phase above the solidus in portions of the system. The systems gehlenite-spinel and gehlenite-CA_2 were checked.

The ternary system CA_2-gehlenite-spinel showed an eutectic at CA_2, 20·5; gehlenite, 58·5 and spinel, 11 per cent at 1514°C. (Fig. 72). The system CA-gehlenite-spinel was not truly ternary as CA_2 appeared as a primary phase in portions of the system. Two piercing points were located in this system (Fig. 75).

Based on the above data, quarternary phase relationships of the system CA-CA_2-gehlenite and spinel were derived (Fig. 76). The quarternary invariant point lay outside the composition tetrahedron. The initial liquid in this quarternary system formed at 1475°C. The above study was of significance in the technology of white calcium-aluminate cements.

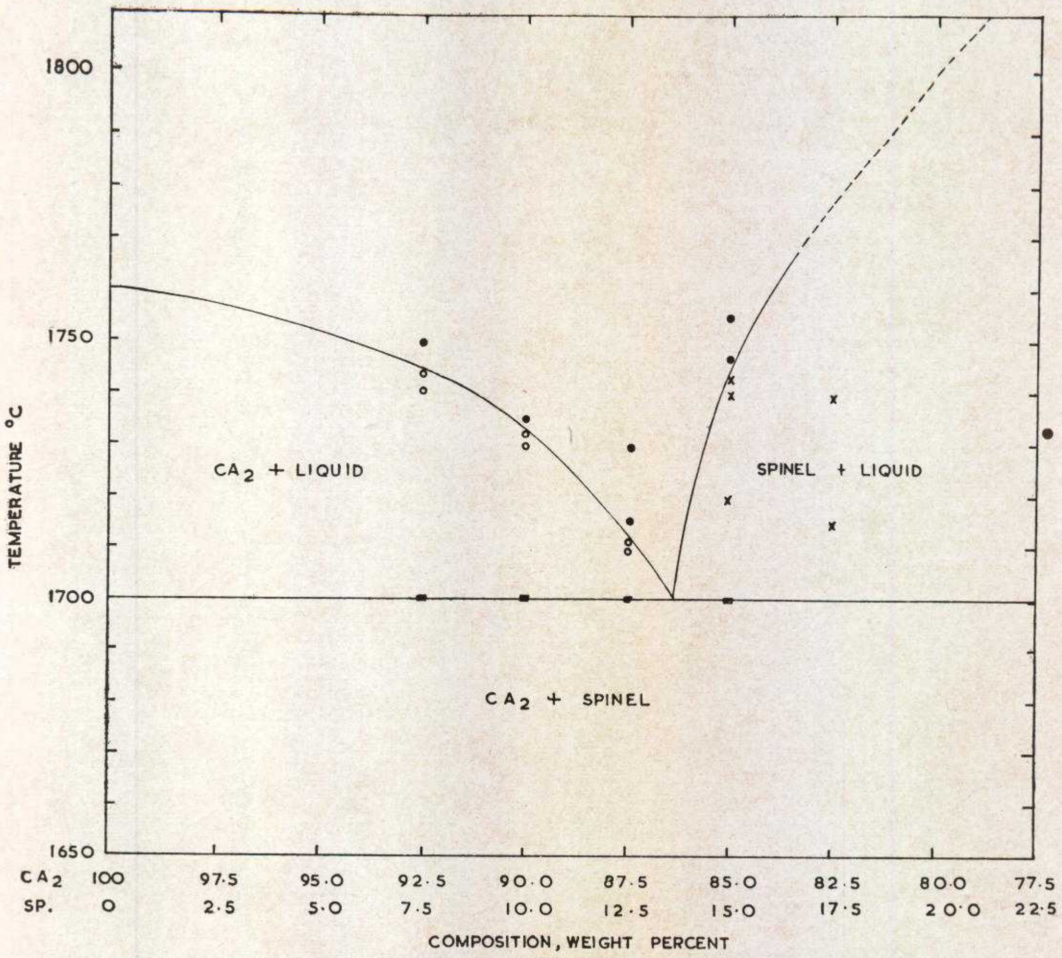


Fig. 70 — Binary System CA_2-spinel

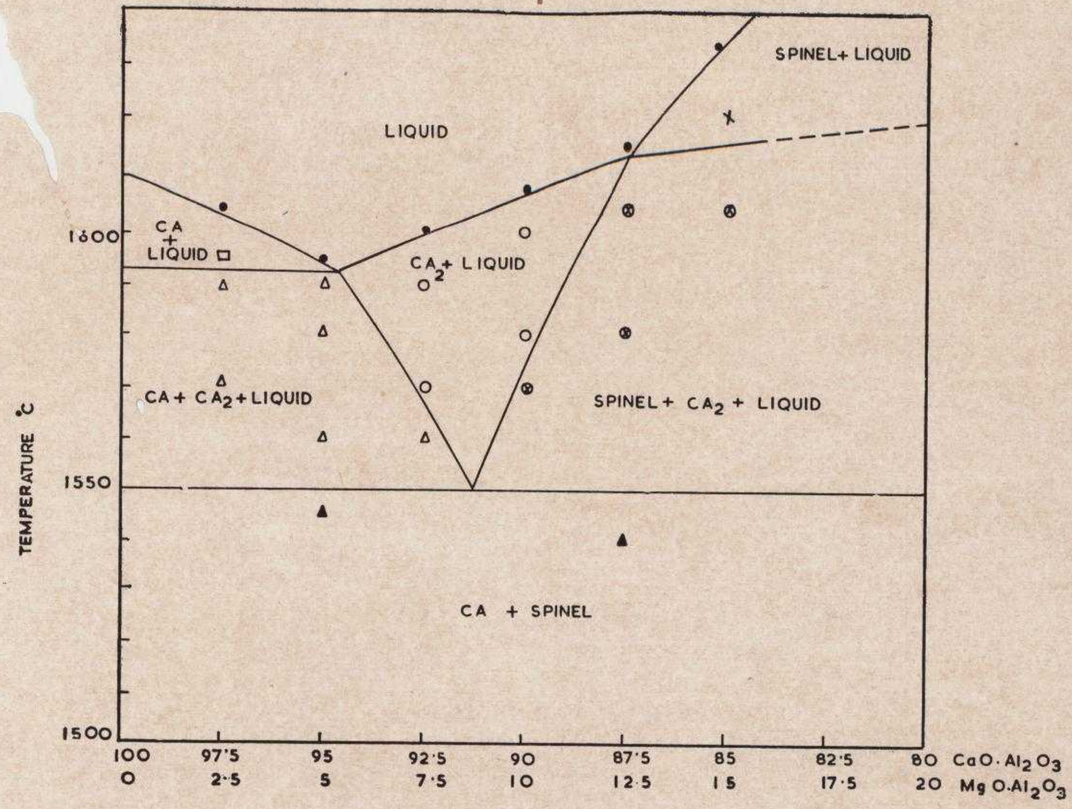


Fig. 71 — Binary System CA-spinel

67.0 Mineralogy of Indian Slags

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

Mineralogical study of three samples of blast furnace slags from Rourkela was completed. The slags are crystalline with a small proportion of glass and contained melilite, a solid solution of gehlenite ($2CaO.Al_2O_3.SiO_2$) and akermenite ($2CaO.MgO.2SiO_2$) as the dominant phase. It occurs as large broad tablets and long laths. Well-developed crystals of spinel were present in considerable amounts. The matrix is composed of fine-grained crystals with or without glass, with refractive index varying above 1·648. It is colourless, deep yellow to deep red and penetrates between the cleavage planes of melilite. The fine crystalline material was found to be merwinite. Accessory opaque minerals occur as dendrites of feathery and bush-like growths and the opaque grains are strewn throughout the slag. Few crystals of pseudowollastonite were detected in the powder mounts. Occasional inclusions of globules of metallic iron were noted. Few more slag samples obtained from the runners and the slag dump are under study.

A preliminary examination of open hearth slags from Rourkela Steel Plant indicated the presence of tricalcium silicate, dicalcium silicate, and deep coloured isotropic phase, probably, FeO-MnO-MgO-solid solution. The L.D. slag was also composed of the mixed oxide phase, together with tricalcium

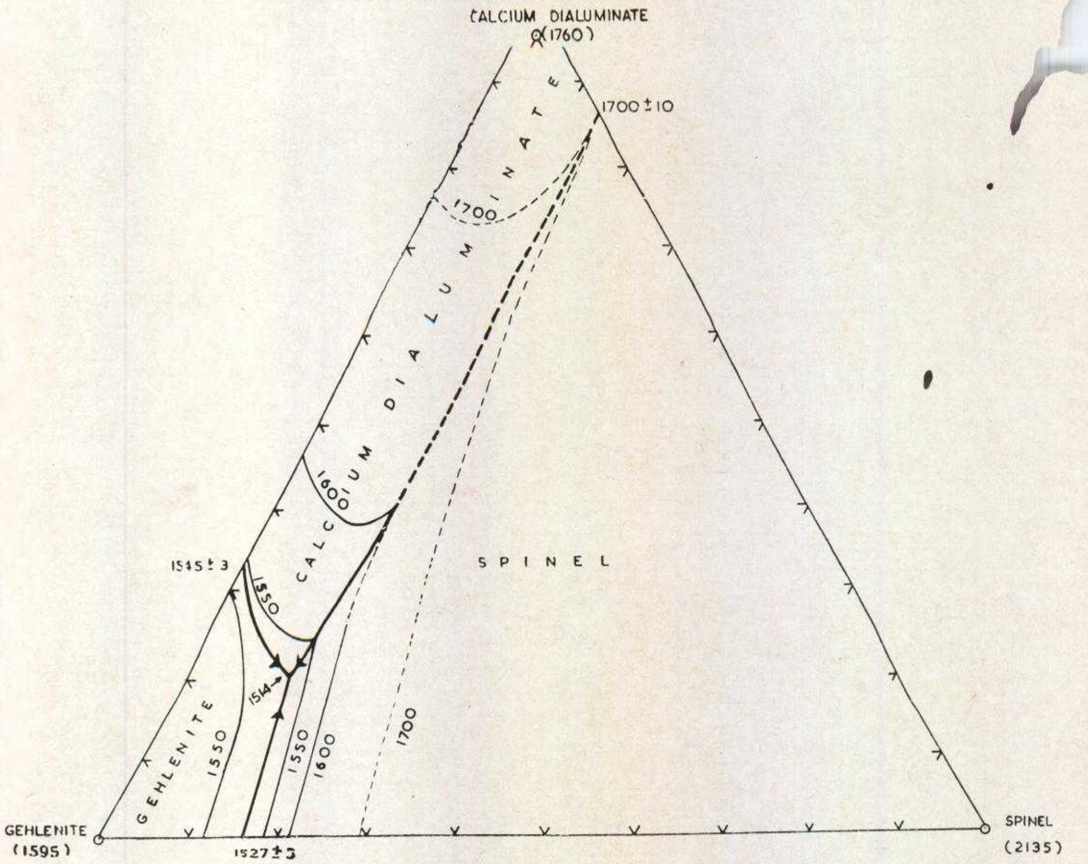


FIG. 72 — PHASE DIAGRAM FOR THE SYSTEM GEHLENITE-SPINEL-CALCIUM DIALUMINATE

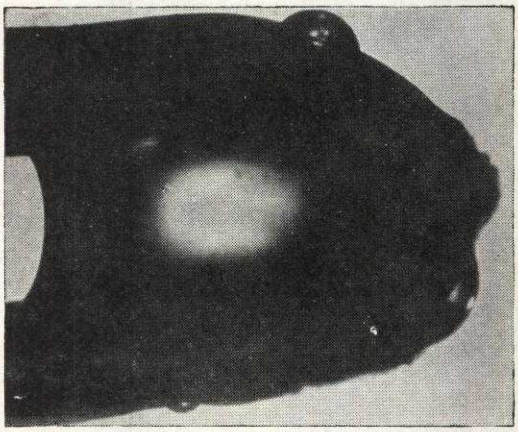


FIG. 73 — PRIMARY OCTAHEDRON OF SPINEL QUENCHED ON THE HOT-STAGE MICROSCOPE (×45)

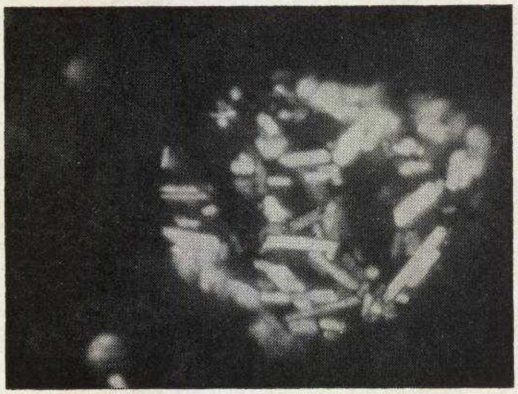
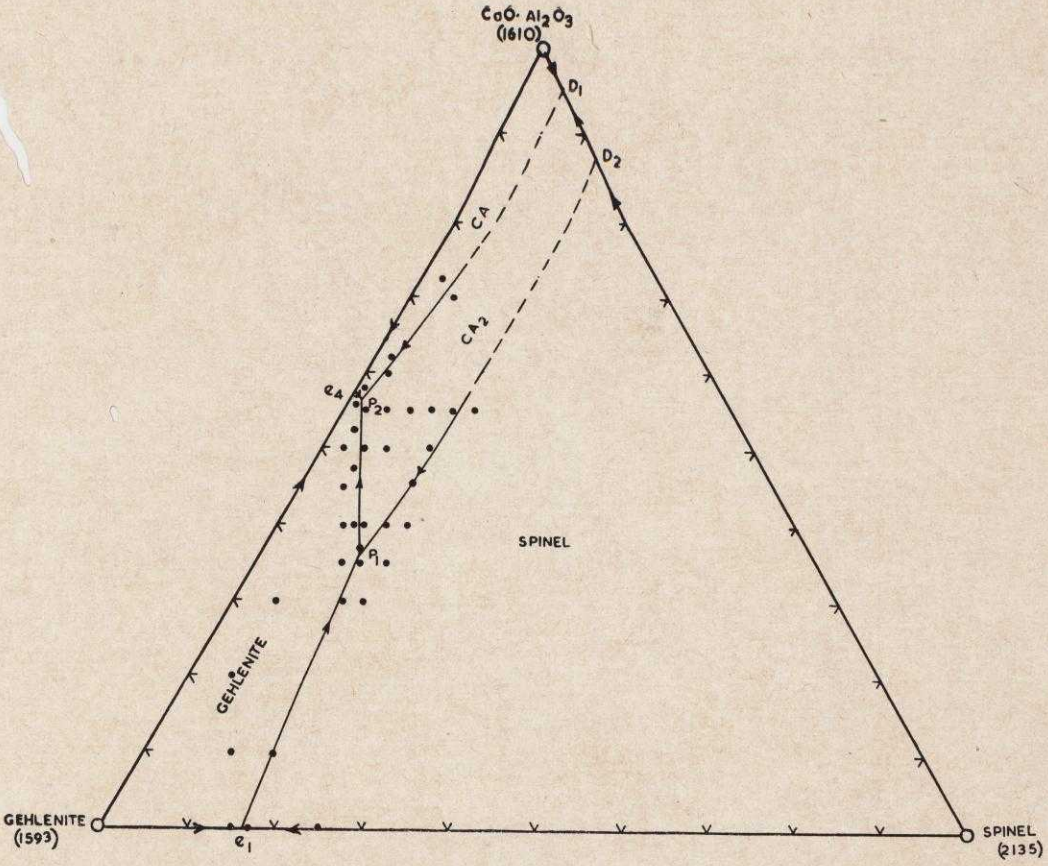


FIG. 74 — PRIMARY CA_2 CRYSTALS IN GLASS



e_1—Geh. 84%, Spinel 16·0%, Temp. 1527°C.±3°C. e_4—Geh. 42·7%, CA 57·3%, Temp. 1512°C. P_1—Geh. 52%, CA 36·8%, Spinel 11·2%, Temp. 1502°C.±3°C. P_2—Geh. 42·4%, CA 56·3%, Spinel 1·3%, Temp. 1496°C.±3°C. D_1—CA 94·6%, Spinel 5·4%, Temp. 1593°C.±3°C. D_2—CA 87·5%, Spinel 12·5%, Temp. 1614°C.±3°C.

Fig. 75 — Ternary System CaO-Al_2O_3-Gehlenite-Spinel Showing Compositions, Phase, Boundaries, Invariant Points and Piercing Points

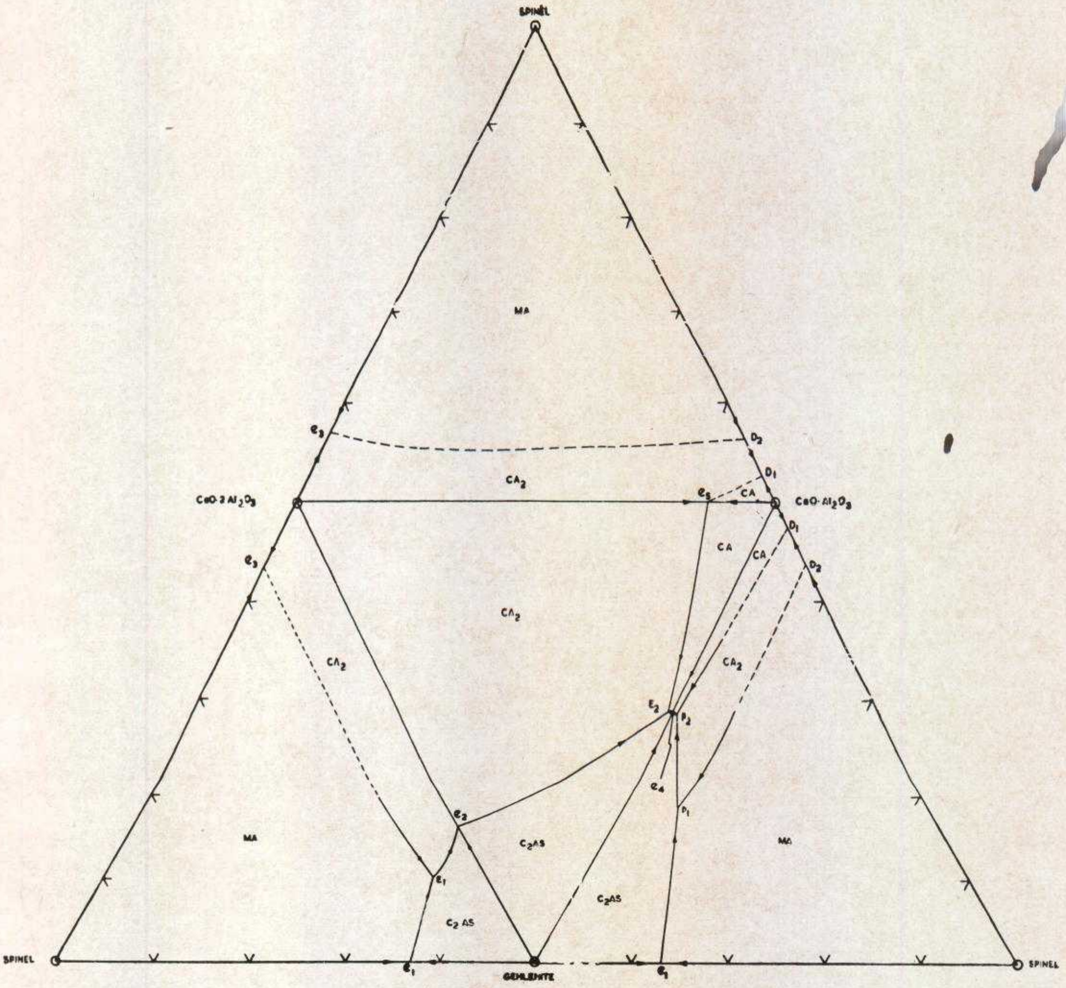
silicate and some free lime. Samples of blast furnace slags and steel-making slags from TISCO were received and these are now under study.

68.0 Studies on Corrosion of Metals and Alloys

Studies on influence of atmospheric pollution, relative humidity, composition of materials and state of exposure on atmospheric corrosion of various metals and alloys were continued at Jamshedpur, an industrial town in Bihar. In addition, tests were started at the Marine Corrosion Research Station, Digha, to have a comparative corrosivity of industrial and marine atmosphere.

(i) *Monthly and Yearly Corrosion Rates of Mild Steel and Zinc*

(a) Four samples each of mild steel and zinc were exposed on the first day of every month both at Jamshedpur and Digha (500 ft. from sea). Two samples of mild steel and zinc were removed at each location after every month and the respective corrosion rate for the particular calendar month was determined. The remaining samples remained exposed to the atmosphere to be removed after the completion



$e_1 = C_2AS$ 84·0%, MA 16·0%, Temp. 1527°C. $e_2 = C_2AS$ 67·5%, CA_2 32·5%, Temp. 1545°C. $e_3 = CA_2$ 86·3%, MA 13·7%, Temp. 1700°C. $e_4 = C_2AS$ 42·7%, CA 57·3%, Temp. 1512°C. $e_5 =$ CA 85·7%, CA_2 14·3%, Temp. 1595°C. $D_1 =$ MA 5·4%, CA 94.6%, Temp. 1593°C. $D_2 =$ MA 12·5%, CA 87·5%. Temp. 1614°C. $P_1 = C_2AS$ 52%, CA 36·8%, MA 11·2%, Temp. 1502°C. $P_2 = C_2AS$ 42·4%, CA 56·3%, MA 1·3%. Temp. 1496°C. $E_1 = C_2AS$ 68·5%, CA_2 20·5%, MA 11·0%, Temp. 1514°C. $E_2 = C_2AS$ 42·7%, CA_2 1·1%, CA 56·2%, Temp. 1500°C.

FIG. 76 — THE QUARTERNARY SYSTEM $CaO.Al_2O_3$(CA)-$CaO.2Al_2O_3$(CA_2)-GEHLENITE-(C_2AS)-SPINEL (MA)

of one year. The monthly corrosion rates at Digha and Jamshedpur varied widely. At Jamshedpur, the corrosion rate was found to depend mainly on the amount of rain fall, the same at Digha was found to be dependent both on humidity and salt content of the atmosphere. Further, the corrosion rate at Digha was found to be, in general, 5-10 times higher than that at Jamshedpur.

Considering the fact that amount of chloride pollution is important in controlling corrosion rate in marine atmosphere, tests were started in February 1965 to determine the difference in corrosion rate between samples exposed at a distance of 50 ft. and 500 ft. from the high tide level. Initial tests showed a profound influence of the distance on corrosion rate.

(b) The long term tests on corrosion resistance properties of copper, nickel, brass, zinc and monel metals are in progress at Jamshedpur. Test panels of the above metals and alloys and also aluminium and aluminium alloys (containing manganese and magnesium) were exposed at Marine Corrosion Research Station, Digha. Samples were removed after 2, 6 and 12 months' exposure, cleaned of all corrosion product, and corrosion rate determined. In general, aluminium and its alloys were found to be most resistant to corrosion.

(c) Studies on the properties of rust formed on mild steel and low alloy steel, initiated in January 1961, were continued at Jamshedpur. A new set of test panel of steel containing small amounts of copper (up to 6 per cent Cu) will be exposed in July '65. Experimental heats of 20 lb. capacity containing varying amounts of copper were made in induction furnace. The cast ingots were forged and then rolled into sheets of 0·1 in. thickness. These were finally cold-rolled to the desired section. Test panels of approximately 100 sq. cm. area were cut. These will be heat treated and then exposed.

(d) Continuous recording of daily changes in atmospheric temperature and relative humidities are being done at both the test sites. Data on rainfall and dew collection are also being kept. Sulphur pollution of the atmosphere at Jamshedpur was determined every month by exposing lead peroxide candle. The chloride pollution at Digha, on the other hand, was determined by exposing wet candles. The atmospheric pollution data for Jamshedpur and Digha have been shown in Fig. 77.

(ii) *Chromate Passivation of Aluminium and Copper Alloys*

(a) Studies on chromate passivation of aluminium and their corrosion resistance under different conditions were made. Studies were also carried out on the effect of solution composition on film formation in two aluminium alloys containing 1 per cent Mn and 1·5 per cent magnesium respectively. 2×2 in. samples of the alloys were treated for various periods in chromate-fluoride-baths. The films were stripped in warm solution of 30 per cent HNO_3 and weight of film formed and metal attacked were determined. For similar conditions of treatment, the film weight on aluminium alloys was found to be more than that on aluminium. The protective properties of the chromate film were evaluated by salt spray test using 5 per cent NaCl solution. The tests carried out for over 200 hours showed that treated samples do not form any corrosion product.

(b) Detailed studies on chromate passivation of copper and brass were taken up to determine the role of solution composition on the protective properties and weight of film formed. Two types of solutions were selected for these studies. The first was based on acidified chromate-chloride solution and the second on chromate-chromic acid solution. The film formed in the first solution was found to be quite thick and brown in colour. On the other hand, the film formed with the second solution was quite thin and was colourless or gave only interfering colours. The protective properties of the film were determined by salt droplet and salt spray test. While the samples without treatment gave a voluminous corrosion product within a few days, the same with treatments showed much improved resistance to corrosion even after a period of 60 days. The protective properties of the film formed in chromate-chromic acid solution were found to be much superior to that of acidified chromate-chloride solution.

(iii) *Fuel Ash Corrosion and Its Prevention*

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

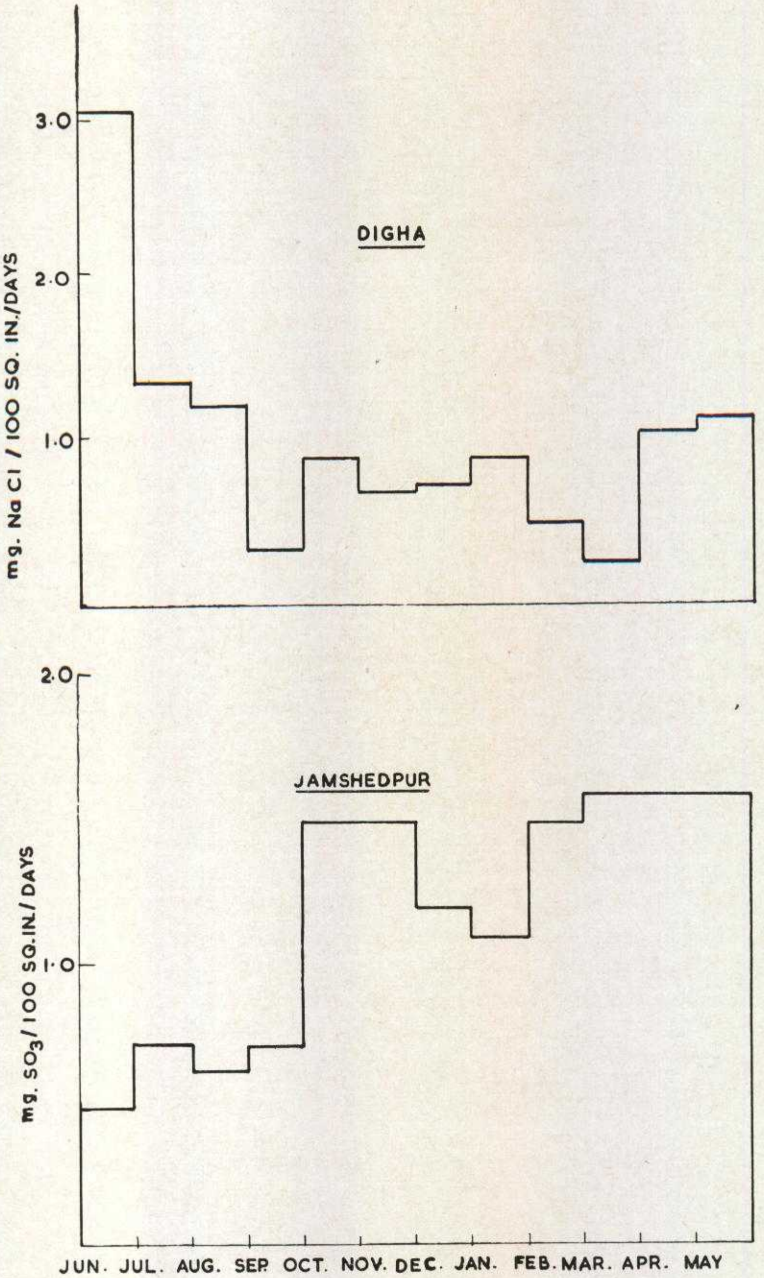


FIG. 77 — ATMOSPHERIC POLLUTION AT JAMSHEDPUR AND DIGHA DURING JUNE 1964 AND MAY 1965

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

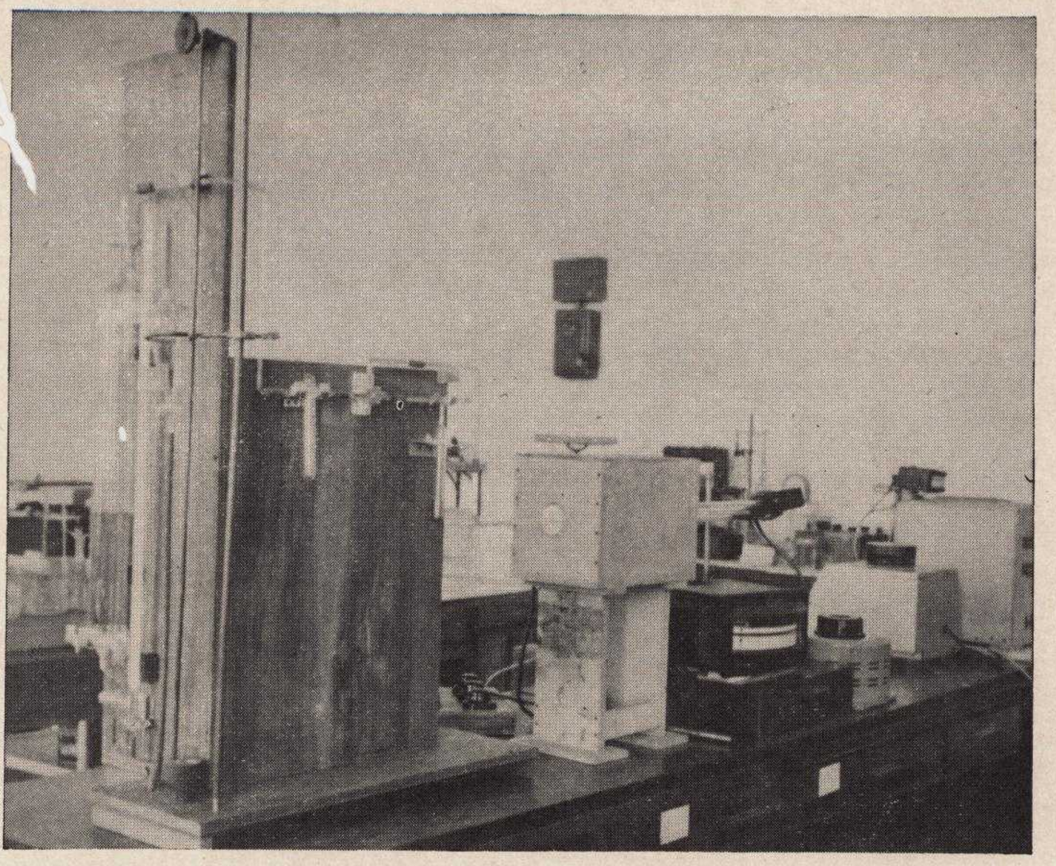


FIG. 78 — APPARATUS SET UP FOR FUEL ASH CORROSION STUDY

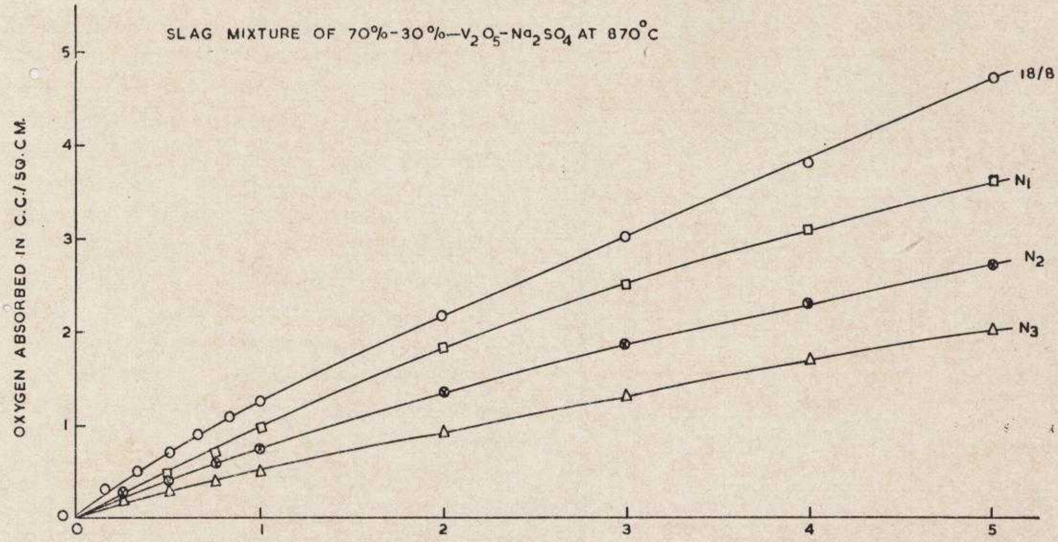


FIG. 79 — RATE OF ABSORPTION OF OXYGEN BY 18/8 STAINLESS STEEL AND THREE NICKEL-FREE STAINLESS STEEL (N_1, N_2 & N_3). HAVING DIFFERENT MANGANESE CONTENT IN 70% V_2O_5-30% Na_2SO_4 SLAG MIXTURE AT 870°C.

The corrosion resistance properties of 18/8 steel and nickel-free austenitic stainless steel, developed in the National Metallurgical Laboratory were determined in a synthetic slag mixture of V_2O_5, 70 and Na_2SO_4, 30 per cent at 870°C. Tests were also carried out to see the effect of varying the ratio of Na_2SO_4 and V_2O_5. The results clearly indicated that replacement of nickel by manganese increased the resistance to Na_2SO_4-V_2O_5 mixtures (Fig. 79). Further, it was found that corrosion rate of all the alloys studied decreased with increase of per cent Na_2SO_4 in the mixture. This will be evident from Fig. 80.

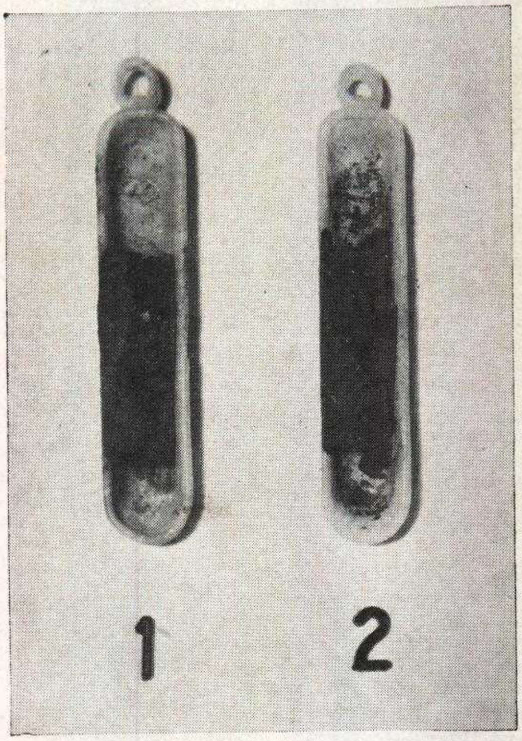


Fig. 80 — 18/8 Stainless Steel Tested at 870°C in (1) 90% V_2O_5-10% Na_2SO_4 and (2) 20% V_2O_5-80% Na_2SO_4 Slag Mixture

(iv) *Stress Corrosion Cracking*

Most alloy systems show susceptibility to stress corrosion cracking under the combined action of tensile stress and some specific corrosive conditions. The object of the investigation is to determine the electro-chemical and metallurgical factors controlling the failure of this type and to evaluate suitable preventive measures for the same.

In the period under review, detailed study on stress corrosion cracking susceptibility of homogeneous copper-manganese alloys containing 8-20 per cent manganese was carried out in copper sulphate/ammonium sulphate solution of varying *p*H. Effect of extent of stress on cracking susceptibility was determined. With increasing manganese content, the alloys were found to become susceptible to cracking at loads below macroscopic yield point indicating that this observation is not unique for copper-zinc alloy, as claimed by many.

(v) *Development of Aluminium Based Alloys for Cathodic Protection*

Sacrificial anodes, based on magnesium and zinc, have been used quite extensively for cathodic protection. India is not yet producing commercially any magnesium and has to depend on import for most of its requirement of zinc. On the other hand, India has abundant resources of aluminium ore and the production of aluminium is increasing rapidly. The investigation was taken up with a view to develop a suitable alloy, based on aluminium, for use as sacrificial anode.

The studies on this problem are being carried out jointly at Digha and Jamshedpur. In the preliminary studies various aluminium alloys were studied to determine the different anode: cathode area ratio which will give adequate protection in sea water. Changes in potential and weight-loss were determined for series of area ratios. Effect of pretreatment of aluminium, to destroy the adherent oxide film, was found necessary to give maximum efficiency. Further tests are in progress to develop suitable alloys to give maximum protection and to determine optimum criteria for cathodic protection.

(vi) *Mechanism of Acid Inhibition and Hydrogen Absorption*

Polar organic compounds are extensively used for inhibition of metal attack during acid pickling and acid attack. In spite of extensive work carried out over the last fifty years, the mechanism of inhibition is still not adequately understood, specially the mode by which it controls the mechanism of hydrogen absorption by the metal. The investigation was taken up to study the various electrochemical and metallurgical factors which control the mechanism of metal dissolution and hydrogen absorption.

Studies on the effect of nitrogen containing organic inhibitors, piperidine and pyrollidine, on metal dissolution and hydrogen absorption of cold-rolled mild steel, were carried out. Effect of annealing on hydrogen pick-up was also studied. Anodic and cathodic polarization, in the presence and absence of inhibitor, were also determined. The results clearly indicated that though these inhibitors were quite effective in reducing metal dissolution, the absorption of hydrogen by the metal was not reduced to the same extent.

69.0 Gases in Metals

Samples of ferrous and non-ferrous metals and alloys were received from different research projects in the Laboratory for the determination of gases in them by vacuum fusion method and by non-reactive carrier gas method. Samples were also received from different firms, laboratories and institutes for the determination of gases in metals and alloys as a regular routine work. The vacuum apparatus designed and fabricated is providing satisfactory and reproducible results.

The problem taken up earlier to study the possibility of determining the nitrogen in solid solution in steel has further progressed. The idea of determining directly the solid soluble nitrogen by an electrochemical method was first put forward by Yu. A. Klyachko and O. D. Larina. They suggested that when a sample is subjected to anodic solution the metal crystal lattice breaks down and gaseous elements present as interstitial atoms become free to get liberated as gas.

The electrolytic cell consists of two one-litre beakers connected at a height of 20 mm. from the bottom by a ground-glass joint 40 mm. wide (Fig. 81). Inside the joint there is flange to which a membrane of double filter paper is fixed. The membrane is tightly pressed so that no electrolyte can leak out of the vessel. The filter paper works as diaphragm and prevents the free exchange of liquid between the beakers. The electrolyte consists of 25 gm. of sodium potassium tartrate and 150 gm. of sodium chloride per litre at a pH of 6·66. So far reproducible results have been obtained. Moreover, the nitrogen collected as gas and the nitrogen in the electrolyte are in agreement with the total amount of nitrogen in the sample. Table 37a gives the results of estimation of nitrogen in solid solution.

70.0 Spectrographic, Polarographic and Other Analytical Work

The following research projects were taken up and completed.

(i) *Determination of Trace Amount of Antimony in Lead-Tin Non-antimonial Solder*

Porous cup technique was adopted for estimation of high amount of antimony up to 10 per cent and oxide arc technique was

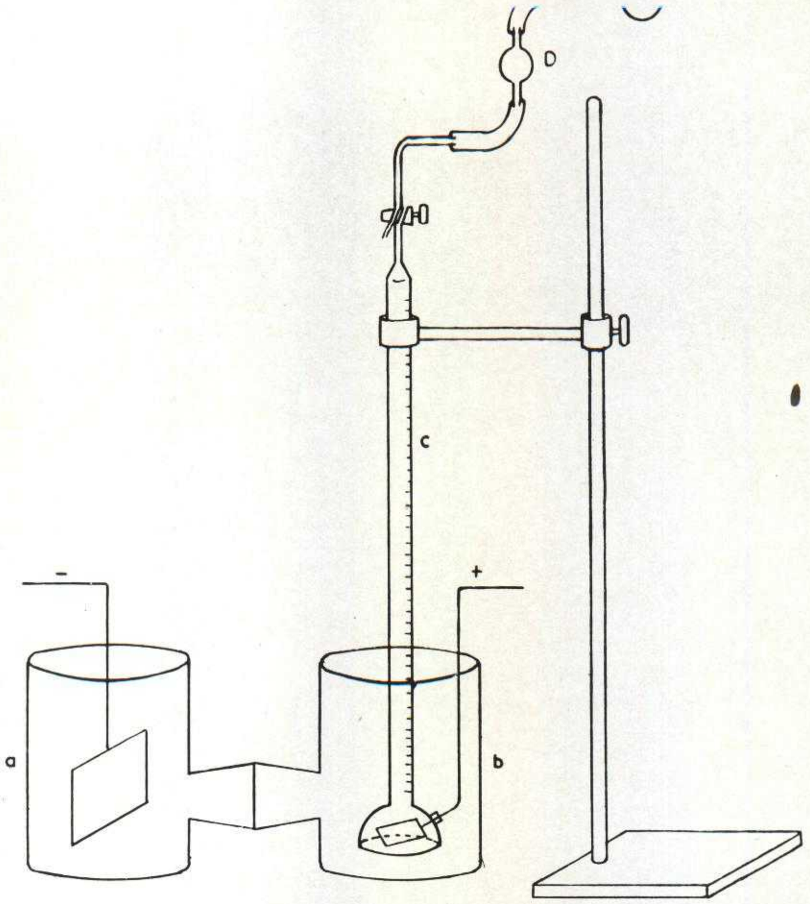


Fig. 81 — Apparatus for Determination of Gases in Metals by Anodic Solution

Table 37a — Estimation of Nitrogen in Solid Solution

Sample	% of N_2 in solid solution by electrolyte	% of Total N_2 by vacuum fusion method	% of nitride N_2 estimated from the electrolyte	Diff. of total N_2 and Nitride N_2
Nitrided steel (0·85% Mn)	0·008	0·036	0·027	0·009
Nitrided steel (0·90% Mn)	0·0070	0·029	0·022	0·007
Nitrided steel (1·1% Mn) (1·1% Mn)	0·0084	0·038	0·0288	0·0092
Low alloy steel (0·62% Cr)	0·002	0·013	0·0115	0·0015
Medium manganese rail steel	0·0022	0·009	0·0066	0·0024

adopted for antimony upto a lower limit of 0·001 per cent.

Analytical accuracy varied from 3 to 6 per cent of the amount present when the concentration of the element to be determined was low. At higher concentration, however, lower values were obtained for both precision and accuracy.

(ii) *Spectrographic Analysis of Beryllium-Copper Alloy*

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

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

71.0 Preparation of Plating Salts

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

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

A process for bright nickel plating which requires no buffiing after plating had been established with organic reagents containing a suitable brightner, viz. naphthalene sulphonic acid, a leveller like ' coumarin ' and an antistress reagent ' saccarin ' together with a sodium salt of a weak organic acid. Studies were undertaken on the influence of each organic reagent in Watt's bath and condition standardized suitable for continuous plating practice. The reagents were added in minute traces in right proportions in nickel plating solution and bright and minor finish of nickel under the rigid control of pH and temperature was obtained on the properly polished and degreased cathode surface. The suitability of such bright platings on production practice was tried on continuous plating performance on much bigger scale in plating vats in light engineering industries. Mechanism of such bright plating of nickel in presence of different organic reagents and the studies of various inclusions and their effects on the physical and mechanical properties of electrodeposited nickel are under investigations.

71.1 Imparting Black Colour to 'Bidri' Wares

At the instance of All India Handicraft Board, New Delhi, a process had been developed for imparting black colour by immersion process to 'Bidri' wares made of zinc with Cu, 4 per cent. The colour at present is usually imparted by an indigenous process and in vogue for long with a particular type of clay available in and around 'Bidri', a small district town in Mysore State.

The chemical substitute containing a mixture of a few chemicals in appropriate proportions, when dissolved, and surface immersed in the solution, the colour imparted on zinc surface varied from blue, grey, black, etc., depending on the composition of the solution and time of immersion. All India Handicraft Board has been furnished with the full details of the process developed in the laboratory and a number of samples were blackened by this process. Some artisans engaged in this craft have also been given training to utilize this synthetic process.

72.0 Analysis of Non-metallic Inclusion in Steels

This investigation was taken up with a view to develop an accurate method for the analysis of inclusions obtained from steels. As the quantity of inclusions was only a few milligrams, semi-micro methods of analysis were adopted.

The following method was found to be suitable for the analysis of the constituents of inclusions. The sample after ignition in platinum crucible was treated with hydrofluoric and sulphuric acids and ignited to determine the SiO_2 content. The ignited residue was fused with potassium bisulphate, extracted with dilute acid and the mixed oxide precipitated, filtered and ignited. Fe_2O_3 was volumetrically determined in the mixed oxide and Al_2O_3 was obtained by difference. From the filtrate, manganese was precipitated with bromine and filtered. The precipitation was dissolved in $FeSO_4$ and H_2SO_4 and determined spectrometrically oxidizing with periodate and measuring in intensity of the permanganate colour at 520 mμ. Calcium and magnesium were determined from the filtrate after separation of manganese by titration with EDTA. The amount of oxygen calculated from the different oxides determined by this method in the inclusions compared favourably with the amount determined by vacuum fusion technique. Typical results are given below:

	SiO_2 %	FeO %	Al_2O_3 %	MnO %	Total oxygen calc. from oxides detd. chemically	By vacuum fusion method
1	0·008	0·054	0·044	0·017	0·040	0·039
2	0·010	0·072	0·030	0·027	0·041	0·039
3	0·012	0·024	0·0086	0·0056	0·014	0·010
4	0·008	0·017	0·010	0·006	0·013	0·010
5	0·039	0·012	0·003	0·005	0·026	0·020
6	0·034	0·016	0·005	0·006	0·025	0·020

72.1 Analysis of Ferro-chrome Slags

For the analysis of the above slag samples received during the production of ferrochrome in this laboratory, necessity was felt for developing a routine method for their complete analysis. The following method was developed and successfully applied for their analysis.

The finely ground sample was treated with sulphuric and hydrochloric acids and sodium sulphate and fumed for fifteen minutes. After cooling, it was digested with hydrochloric acid (1: 1), filtered and the residue ignited and hydrofluorized for silica. The residue left after hydrofluorization was fused with bisulphate, dissolved with hot acidulated water and mixed oxides were twice precipitated with ammonia, washed, ignited and weighed. From the mixed oxides iron and chromium were determined volumetrically and aluminium was obtained by difference. From an aliquot of the filtrates after separation of mixed oxides, calcium was determined volumetrically after precipitation as oxalate and from another aliquot calcium to magnesium was obtained volumetrically with EDTA. Some results are given below:

%	1	2	3	4
SiO_2	35·68	32·96	31·88	27·08
Al_2O_3	23·15	20·36	24·89	28·19
Fe_2O_3	1·92	3·20	4·00	2·24
Cr_2O_3	2·61	6·04	6·19	3·73
CaO	0·90	0·81	0·98	0·98
MgO	36·02	37·31	37·31	38·20

73.0 Recovery of Metallic Zinc from Zinc Ashes from Messrs Indian Tube Co. (1953) Ltd., Jamshedpur

A sample of 160 kg. of zinc ash was received from Messrs Indian Tube Co. Ltd., Jamshedpur, for the recovery of metallic zinc by physical methods without resorting to any pyro-metallurgical treatments. The sample was reported to have been collected from their galvanizing line.

The ash in the ' as received ' state ranged from 6 in. down to fines. Metallic zinc was present in odd shapes and globules, much of which was free from extraneous ash even at $\frac{1}{2}$ in. size. The extraneous material was reddish grey to yellow in colour.

Preliminary Hand-picking: The ash, as received, was screened into 3 fractions — $+\frac{1}{2}$ in., $-\frac{1}{2}$ in. +3 mesh and −3 mesh — to facilitate hand-picking of metallic zinc as well as further processing. The first fraction was hand-picked and the rejects crushed to $-\frac{1}{2}$ in. size. The $-\frac{1}{2}$ in. +3 mesh fraction was mixed with the same size fraction obtained earlier and the mixed product was subjected to hand-picking. The rejects from this operation were crushed to −3 mesh size, mixed with the rest of the −3 mesh material. This constituted the starting material for further processing. By hand-picking of the $+\frac{1}{2}$ in. and $-\frac{1}{2}$ in. and $-\frac{1}{2}$ in. +3 mesh fractions, 13·6 per cent by wt. of zinc could thus be recovered.

A representative sample (10 kg.) of the −3 mesh fraction was jigged in the laboratory Denver Mineral Jig using $\frac{1}{8}$ in. stroke. Some results are shown in Table 37b.

Table 37b — Jigging Results

Product	Weight	Assay, % Total zinc	Assay, % Metallic zinc	Distribution, % Total zinc	Distribution, % Metallic zinc
Concentrate	45·4	98·0	96·5 (Calc.)	56·2	96·5
Tailing	43·2	66·4	3·6	34·8	3·4
Slime	11·4	65·6	2·8	9·0	0·1
Head (calc.)	100·0	82·5	45·4	100·0	100·0
Feed	100·0	82·5	45·4	100·0	100·0

The results indicated that 96·5 per cent of metallic zinc could be recovered which amounted to about 45·4 per cent on weight basis w.r.t. the −3 mesh material. The jig concentrate and the hand-picked metal-

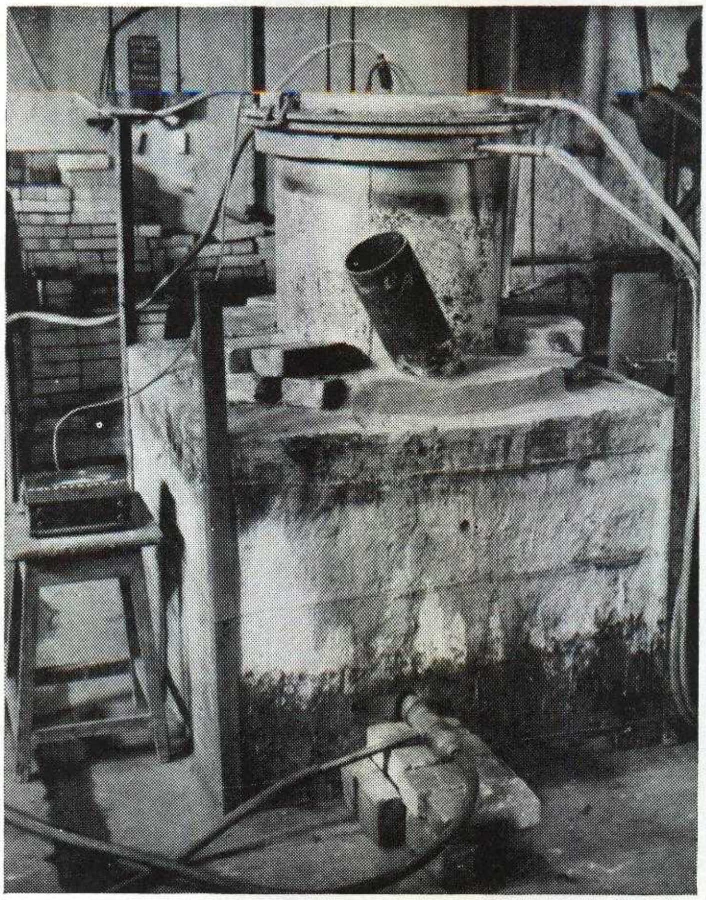


FIG. 82 — VACUUM DISTILLATION UNIT FOR THE RECOVERY OF ZINC FROM DROSS, DESIGNED AND FABRICATED AT THE NATIONAL METALLURGICAL LABORATORY

lics from the −3 mesh fraction together constituting 52·8 per cent by weight of original ash sample had to be melted in a suitable furnace to recover the metallic zinc. It is expected that the loss of metallic zinc values due to contact with water and air would be low in this treatment compared to wet grinding and screening. The total recovery of metallic zinc by hand-picking and jigging would be 97·5 per cent and the weight recovery would be 52·8 per cent of the original. Zinc in the combined form, representing about 35 per cent of the total zinc was lost in the jig tailing and same can be recovered only by some chemical process.

73.1 Treatment of Zinc Wastes from the Galvanizing Bath

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, for every ton of zinc consumed in galvanizing about 0·24 ton is lost in the form of zinc dross and, on an average, for every ton of zinc dross produced about 0·8 ton blowings, 0·5 ton skimmings and 20·7 ton ashes are obtained. The investigation aims at the recovery of the metallic value from the various products for further use in the galvanizing bath.

Zinc Dross — Based on vacuum distillation of zinc in a tubular furnace, a mild steel vessel with ribbed type of condenser was designed and fabricated at the National Metallurgical Laboratory. The vessel has a capacity to handle 10 kg. of dross and was provided with radiation baffle to protect the rubber gasket and to improve the efficiency of distillation. Trials were carried out with 6 kg. charge of zinc dross at 600°C. for 3 hours duration. The condenser was cooled by means of compressed air. The metallic recoveries were poor due to inadequate cooling effect by air. Experiments were carried out with 6 kg. of charge at different temperature and periods with water cooling system for the condenser. Some results are recorded below:

No. of expt.	Temperature of distillation °C.	Duration of distillation	% Metallic recovery
1	550	3	75
2	600	3	80
3	600	3	83
4	650	1	50
5	650	2	83
6	650	3	85

Further work was undertaken to study the distillation rate for longer periods. A new pot was fabricated. It was found that the distillation rate increased with

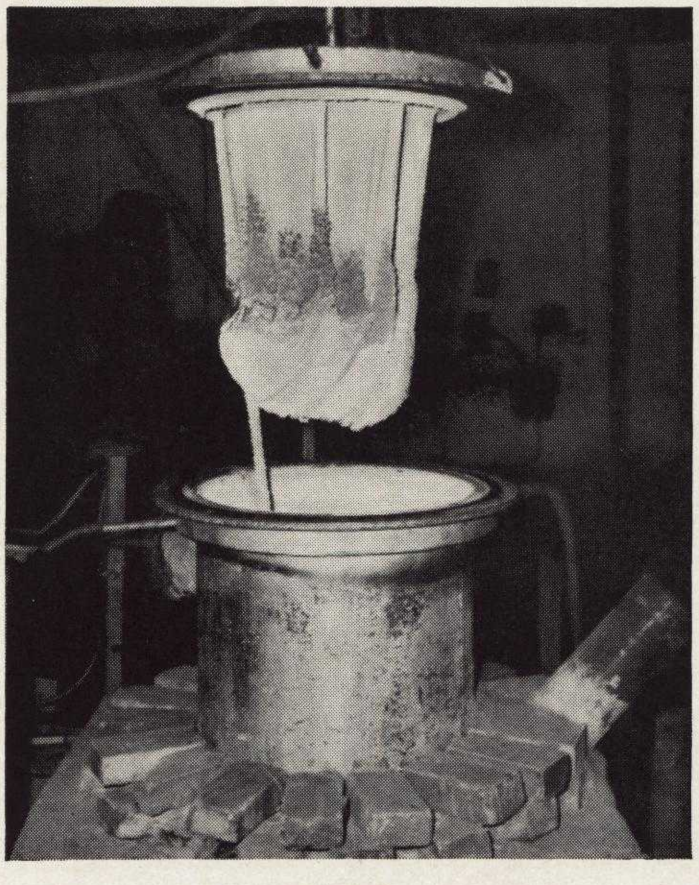


FIG. 83 — ZINC, RECOVERED FROM DROSS, DEPOSITED ON THE CONDENSER OF VACUUM DISTILLATION UNIT

higher temperatures of distillation, but higher temperatures tend to reduce pot life. Based on the experiments, a larger unit having a capacity to distil 500 kg. of dross per batch is being fabricated.

Treatment of Zinc Blowing

Acid treatment of zinc blowing fines to remove the oxide coating was not very successful and further experiments were carried out to melt the coarser fraction of the blowings in a molten pool of zinc. Zinc recoveries obtained varied from 25 to 45 per cent.

Since the melting of blowings did not yield encouraging results, vacuum distillation was tried. The condensate obtained by vacuum distillation was not massive and was found contaminated with oxide particles. The oxide particles were carried mechanically to condensing zone. Even the condensate obtained on slower distillation rates at 450°C. was also contaminated with oxide particles. Briquetting of blowings helped in overcoming the oxide contamination and massive deposit was obtained.

Based on the small-scale trials, briquettes of larger size employing zinc blowings as received were treated in the distillation pot. About 4 kg. of blowings were pressed into briquettes, rectangular in shape, of size 4×2×1 in. The results of the trials carried out at 650°C. for different periods with various binders at low pressures of 0·1 mm. of mercury are recorded in Table 37c.

Table 37c — Results of Trials on Treatment of Zinc Blowings

Expt.	Binder	Distillation period (hours)	% Metallic zinc recovered	Remarks
1.	Dextrine	2	63	Briquettes disintegrated completely after reaction
2.	Dextrine	3	69	do
3.	Sodium silicate	2	65	No disintegration
4.	Sodium silicate	3	72	do
5.	Bentonite	3	75	do

PILOT PLANTS

74.0 Low Shaft Furnace Pilot Plant Project

Incorporation of fuel oil/naphtha injection system to the 15 tonnes/day Low Shaft Furnace Pilot Plant of the National Metallurgical Laboratory was inaugurated by Hon'ble Shri Humayun Kabir, Union Minister of Petroleum and Chemicals on the 5th February, 1965. The entire plant equipment was jointly designed by the National Metallurgical Laboratory and an Indian firm and wholly and indigenously fabricated without spending any foreign exchange. Extensive trials are under way with either fuel oil or naphtha injection with simultaneous enrichment of the blast with oxygen. Due to the limitations of the compensation of endothermal heat requirements for the dissociation of injected fuels by raising the hot blast temperature, the enrichment of the blast with oxygen is a metallurgical necessity. The effect of different degrees of oxygen enrichment of the air blast on the smelting efficiency and particularly fuel economy and productivity has been exhaustively studied. The preliminary results are encouraging. It is considered that the N.M.L. is pioneer in the field of direct injection of liquid naphtha into the hearth of the iron smelting furnace, results of which will enable utilization of surplus naphtha in iron making.

Layout of Oxygen Supply System

About 950 meter long 50 mm. bore heavy steel pipe was laid from the works of Messrs Indian Oxygen Ltd. to the Low Shaft Furnace Pilot Plant of the National Metallurgical Laboratory for the supply of industrial oxygen at the rate of 280 m.^{3}/hr. at a maximum working pressure of 12·65 kg./cm.^{2}. The pipe line was designed on the basis of estimated weekly consumption of 1,5000 m.^{3}. The oxygen for enrichment purposes is injected at appropriate pressure to the hot blast main through a specially designed stainless steel injector placed tangentially at an angle of 30° to the direction of blast stream. In order to prevent high temperature oxidation of metallic recuperator elements, the oxygen is injected after heating the blast. Smelting trials with oxygen enriched blast are described later.

Installation of Fuel Oil and Light Naphtha Injection Systems in Low Shaft Furnace Pilot Plant

The introduction of auxiliary fuels in the blast furnace for iron smelting has gained significant importance and almost universal acceptance during the last decade.

In India, light naphtha, a byproduct of the petroleum refining industry, is reported to be surplus. Besides, the Government of India has reduced the excise duty on this product to only 5 per cent for encouraging its use for iron making purposes. It is considered that the injection of light naphtha may be of considerable industrial significance and it is with these objectives that trials on the injection of light naphtha were planned and conducted at the Low Shaft Furnace Pilot Plant of the National Metallurgical Laboratory. The Light Naphtha Injection System at the Low Shaft Furnace Pilot Plant was completed at a cost of about Rs. 3 lakhs.

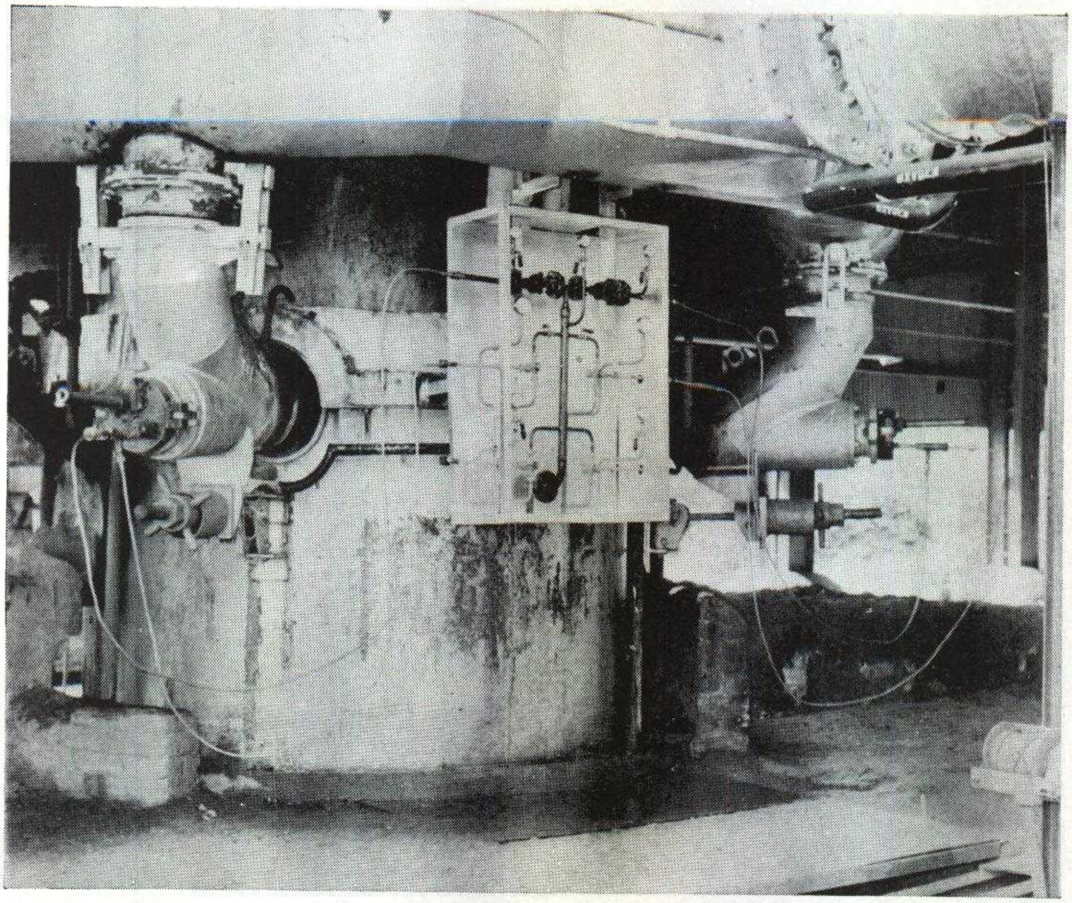


FIG. 84 — INSTALLATION FOR LIGHT NAPHTHA INJECTION THROUGH THE TUYERES OF THE LOW SHAFT FURNACE PILOT PLANT

Reference to technical literature shows that liquid petroleum naphtha has not so far been used for direct injection into the hearth of an iron blast furnace. The National Metallurgical Laboratory will be pioneering a research programme for the injection of a highly inflammable liquid petroleum product in its Low Shaft Furnace Pilot Plant.

Naphtha is a cyclic hydro-carbon with a carbon to hydrogen ratio of 5: 1 in comparison to 7·5: 1 for furnace oil. The heat value of naphtha is 11,110 kcal./kg. in comparison with 10,175 kcal./kg. of the furnace oil. Comparatively naphtha has a higher hydrogen content than the furnace oil which means that endothermal decomposition of naphtha will release a higher volume of hydrogen in the bosh with respect to carbon monoxide than would be the case of injection of furnace oil. Furthermore, for compensation, the blast will have to be heated to a higher temperature for the same injection rate. Fluidity of naphtha is much higher than that of furnace oil which eliminates prior heating and filtration arrangements. It can be atomized easily but as it is much more volatile and inflammable, the installation of the naphtha injection system should have ample audio-visual safety devices and controls so that the operation does not become hazardous or dangerous to the plant operators. Naphtha contains only 0·5 per cent sulphur, against 3·0 per cent sulphur in furnace oil. The introduction of naphtha, therefore, will not create any difficulty of desulphurization of iron smelted in the furnace.

For furnace injection, light naphtha is stored in a 12,500 litre capacity underground storage tank provided with vent hole and an indicator for the quantity in the tank. Naphtha in the tank is subjected to a pressure of 1 kg./cm.^{2} for its transfer from the underground storage tank to a 225 litre capacity service tank installed above the ground in the naphtha pump room. The service tank has also been provided with a relief valve so that the pressure in excess of 1 kg./cm.^{2} can be automatically released. The service tank is adequately provided with pressure gauge and pressure switches. From the service tank, the naphtha flows into two duplex gear feed pumps coupled individually to 1 H.P., 1,440 r.p.m. motors which are expected to supply 100 litres of naphtha per hour at a pressure of 27 kg./cm^2. The total amount of light naphtha passing through the 18 mm. pipeline is determined by the flow indicator. The pumps for the naphtha have been housed in a room containing no electrical circuit therein and coupled to the electric motors located in a separate room with the shaft passing through an opening on the wall properly

Fig. 85 — Hon'ble Prof. Humayun Kabir, Union Minister for Petroleum & Chemicals, Inaugurating the Light Naphtha Injection System at the Low Shaft Furnace Pilot Plant

enclosed in asbestos packings. The entire naphtha pipeline is welded, lagged and is laid underground up to the control cubicle in the Control Room, where it is divided into four 6 mm. diameter pipes each having a control valve and a pressure indicator to control the rate of flow of naphtha through individual lances placed in the four tuyeres of the Low Shaft Furnace. A three-way air purging cock has been provided to facilitate the storage of naphtha and its purging with compressed air. The lances have been provided with a self-closing coupling to prevent possible ignition of naphtha in the supply pipeline at the time of withdrawal of the naphtha lances. A schematic diagram of the injection system is shown in Fig. 86.

The naphtha injection lances are made out of two concentric stainless steel tubes, the inner tube carrying naphtha at a pressure of 18 kg./cm². at the rate of 6-12 litres per hour. The space between the inner tube and outer tube is for passing

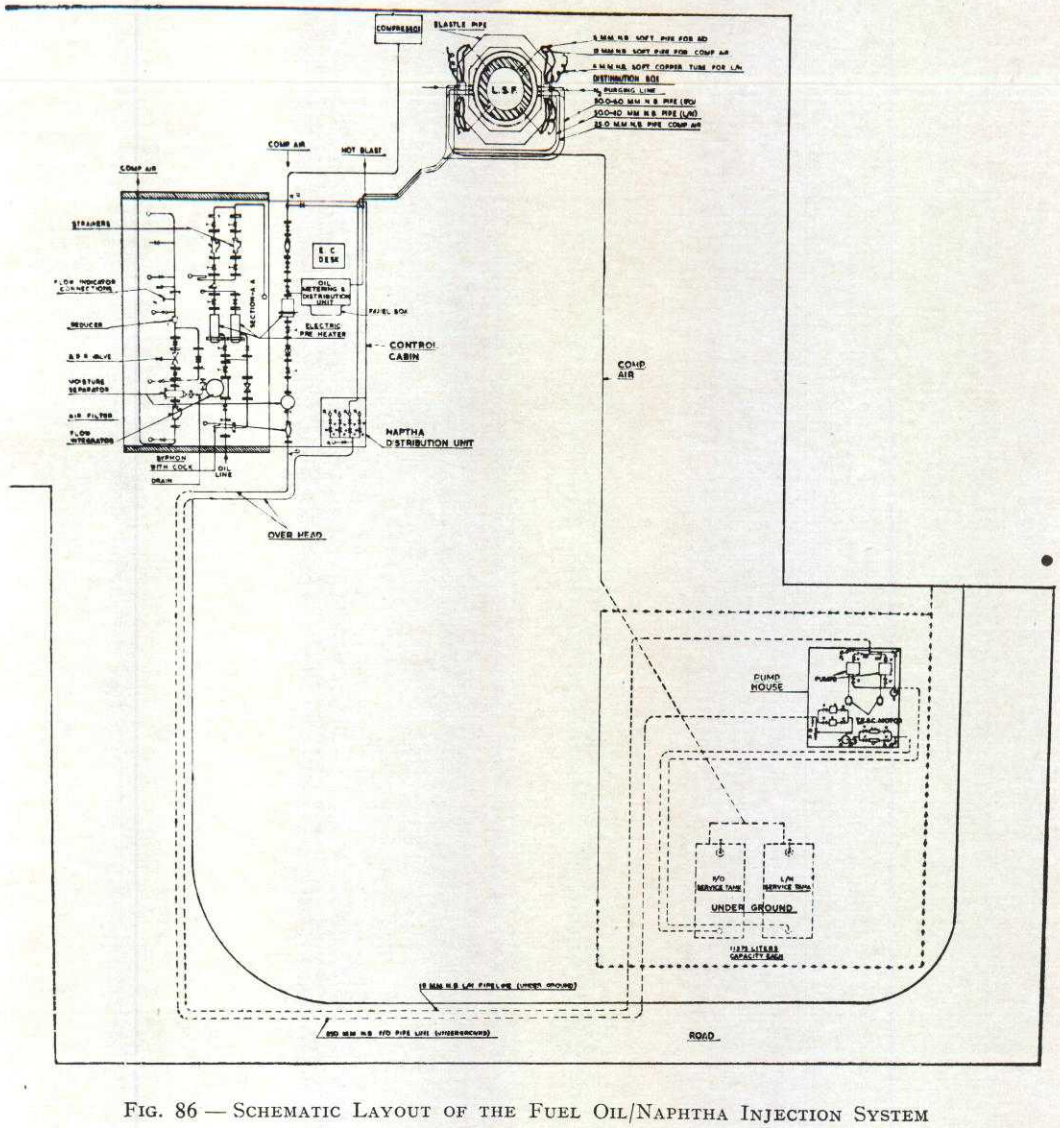


Fig. 86 — Schematic Layout of the Fuel Oil/Naphtha Injection System

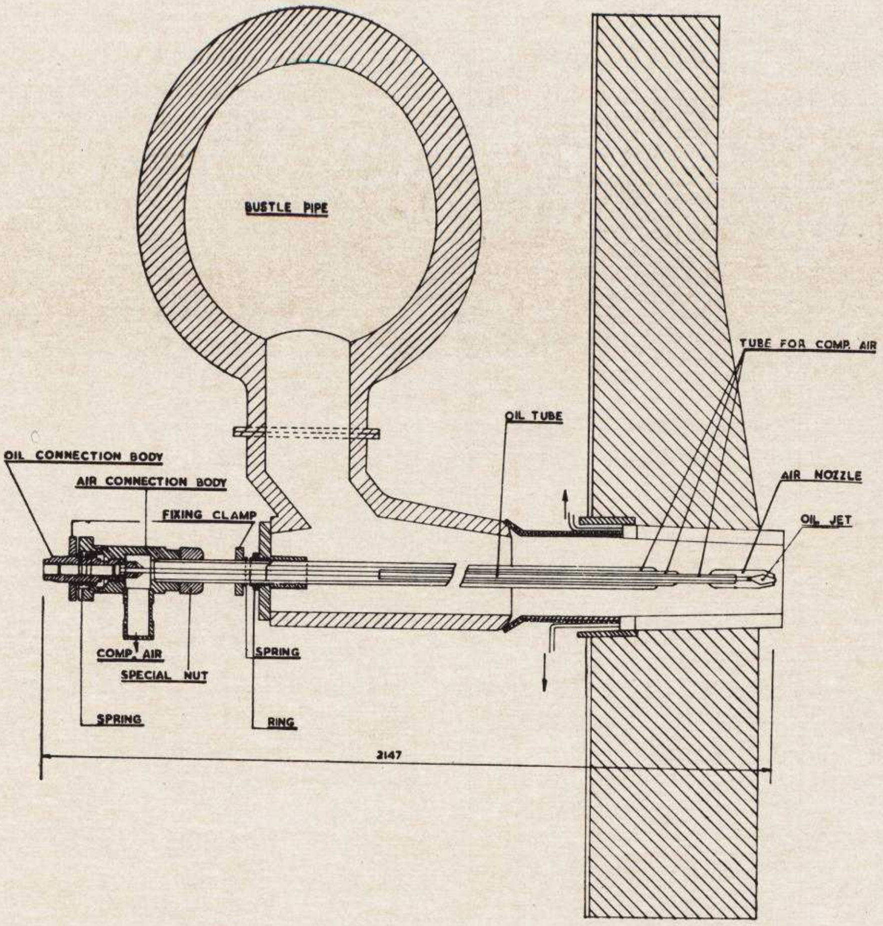


FIG. 87 — INJECTOR LANCE ASSEMBLY THROUGH THE TUYERES

compressed air at a pressure of 3 kg./cm.^{2} which, besides cooling the naphtha lance, admits the requisite amount of air for the atomization of naphtha through a suitably designed jet system. The naphtha is kept at high pressure to prevent its vaporization either in the lance or in the supply pipeline system. The lance assembly in the tuyere is shown in Fig. 87.

The requisite compressed air is supplied by a compressor operated with a 7·5 H.P. motor having a capacity to supply 0·6 m.^{3} at a pressure of 14 kg./cm.^{2} It is provided with a 0·33 m.^{3} reservoir.

Necessary alarms have been provided in the system to indicate failures due to the stoppage of circulation of naphtha in the lances, burning of the lances, choking of the lances and failure of the compressed air lines. These alarms are operated by limit switches which indicate high or low oil level in the service tank, high or low oil pressure before oil measuring and distribution unit if any, in the case of failure of the oil relief valve or the feed pumping set, or locking of the rotating wheel of the oil flowmeter, or even complete clogging of the oil strainers. Alarms operated by pressure switches indicate too high or too low oil pressure at lances, if any, in the case of failure of the air pressure regulator or the compressor.

The oil-cum-naphtha injection system including oxygen enrichment of the blast will provide due facilities at the National Metallurgical Laboratory to improve the scale. These trials will not only be truly pioneering in character but of interest to the steel industry in India and overseas.

Programme of Research and Development Work

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

Phase I

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

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

3. Iron ores from Chapra, Antribeharipur in Mahindergarh, Punjab, with nut coke or non-coking coals.

4. Iron ores from Anantpur, Warangal, etc., with non-coking coals (and low-temperature carbonized coke made thereof) from Kothagudem, Yellandu, etc., in Andhra Pradesh.

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

6. Iron ores from Nathara-ki-Pal near Udaipur and Morinja (Chomu-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(1)].

Phase II

from iron ore fines limestone and non-coking slack coals.

Phase III

1. Utilization of lignites after its high temperature carbonization for iron smelting.

2. Utilization of Salem magnetite after its beneficiation and agglomeration by pelletizing or briquetting in conjunction with carbonized coke.

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 iron production.

Phase V

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

Phase VI

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

Extensive investigations Phase I(1) to (6) and Phase II have already been completed and relevant feasibility reports were issued. Apart from the variation in the physical and chemical characteristics of raw materials, trials were conducted under additionally imposed test conditions keeping the variables within practicable limits. The investigations conducted during the period under review were:

Twenty-fifth Campaign

Smelting trials were conducted with iron ore fines (Orissa), low temperature carbonized coke (C.F.R.I.) made out of non-coking coals from Bihar and blended fluxes; possibilities of raising the blast temperature by suitable modifications were also investigated.

Twenty-sixth Campaign

Smelting trials were conducted with iron ore fines (Orissa), low temperature carbonized coke made from totally non-coking Singareni coal of Andhra Pradesh and blended fluxes with the object of assessing the effects of wind volume on smelting characteristics and iron productivity.

Twenty-seventh Campaign

Smelting trials were conducted with small lumpy iron ore from Orissa (B. Patnaik), low temperature carbonized coke made from Talcher non-coking coal (C.F.R.I.) and blended fluxes, with the object of ascertaining the possibility of employing this variety of non-metallurgical fuel in existing commercial low shaft blast furnace (Barbil) or for the proposed industrial plant (Talcher).

Twenty-eighth Campaign

Smelting trials were conducted with blended iron ores from Orissa (B. Patnaik and Orissa Minerals), low temperature carbonized coke (C.F.R.I.) and (R.R.L.) and blended fluxes with a view to study the actual coke rate with two different ores of widely different reducibility characteristics employing hydrogen gas as reductant.

Twenty-ninth Campaign

Smelting trials were conducted with iron ore fines (Orissa Minerals and Bolani), low temperature carbonized coke (C.F.R.I.), nut coke and blended fluxes with the object of evaluating the effects of oxygen enrichment of the air blast and size grading of iron ore (Bolani) on the smelting characteristics.

Thirtieth Campaign

Smelting trials were conducted with a burden consisting of blended ores (Orissa Mineral, Bolani, N. V. and B. Patnaik), low temperature carbonized coke (C.F.R.I.), blended fluxes with the object of studying the effect of auxiliary fuel injection on the smelting characteristics.

Details of Furnace Campaigns

Twenty-fifth Campaign

In this campaign smelting trials were conducted with an ore burden composed of a mixture of iron ore fines (Orissa Minerals), low temperature carbonized coke made from non-coking coals (C.F.R.I.) and blended fluxes. As it was decided to conduct smelting trials with injection of auxiliary fuels, it was a necessity to find out the possibilities of attaining a maximum hot blast temperature by suitable modifications in the recuperator. Since, it was not possible to incorporate major modifications in the existing metallic fin-tube recuperator, it was considered that appropriate lagging of the mild steel interconnecting chambers by slag wool and thereby reducing the heat-loss by radiation might improve the efficiency of the recuperator. The object of this compaign was to obtain maximum hot blast temperature and thereby to study the effect of hot blast temperature on the smelting characteristics. The chemical and screen analysis of raw materials used in this trial are given in Tables 38 and 39.

Table 38 — Chemical Analyses of Raw Materials

Moisture %	Ash %	Volatile matter %	F.C. %
8·5	25·9	4·1	61·5

1. Proximate analysis of low-temperature carbonized coke (CFRI)

2. Analysis of Coke ash

SiO_2 %	MgO %	Al_2O_3 %	CaO %	Fe %	S %	P %
53·8	1·88	28·0	5·68	4·6	0·23	0·08

3. Analysis of iron ore (Orissa Minerals)

Fe %	SiO_2 %	Al_2O_3 %	S %	P %
64·14	3·28	4·57	0·01	0·02

4. Analysis of Fluxes

Flux and location	CaO %	MgO %	SiO_2 %	Al_2O_3 %	S %	Fe %
Limestone (Madras)	54·31	1·0	0·88	1·2	0·027	—
Dolomite (Andhra Pradesh)	32·20	25·0	0·3	0·56	0·55	N.D.

Table 39 — Screen Analysis of Raw Materials Employed

Raw materials	+50 mm %	−50 mm +25 mm %	−25 mm +12·5 mm %	−12·5 mm +6·0 mm %	−6 mm +3 mm %	−3 mm %
Low temperature carbonized coke	21·5	47·9	21·9	4·8	1·5	2·4
Iron ore (Orissa Minerals)	—	—	—	16·0	24·7	59·3
Limestone (Madras)	—	22·5	27·8	17·4	18·3	14·0
Dolomite (Assam)	—	—	56·9	30·5	8·5	4·1

The furnace was operated with the following conditions:

Tuyere diameter	75 mm.
Hot blast wind volume	2200-3000 Nm³/hr.
Hot blast wind pressure	1500-2100 mm.WG.
Hot blast temperature	440-660°C.
Top gas temperature	220-460°C.

During the trial, it was found that the temperature of the hot blast temperatures could be raised to a maximum of 650-660°C. with the optimum matching of purified furnace gas and combustion air fed in the burner of the recuperator. But the continuous maintenance of hot blast temperature at this level constantly was impracticable and the temperature frequently dropped to 450°C. due to various reasons. The lagging of the other connecting chambers were also carried out, but these modifications could not raise the hot blast temperature significantly. The slag basicity CaO/SiO_2 was maintained at a calculated figure of between 1·2 and 1·3 while in practice it varied between 0·9-1·15. Pig iron and slag analyses are given in Tables 40 and 41.

From the data obtained in this campaign and earlier observations, it was noticed that the alteration of hot blast temperature in the low range of 400-600°C. adversely affected the fuel rate and analyses of data indicated that 200°C. rise in the blast temperature from 400-600°C. could lower the coke rate by 200 kg./tonne of pig iron.

Table 40 — Pig Iron Analysis

C %	Si %	S %	Mn %	P %
2·5-3·1	2·5-3·8	0·03-0·09	0·2-0·4	0·4-0·5

Table 41 — Analysis of Slag

CaO %	SiO_2 %	FeO %	Al_2O_3 %	MgO %
36-40	32-34	0·5-1·5	20-24·5	4-6

Twenty-sixth Campaign

Based on the earlier observations of wind volume on productivity under otherwise

Table 42 — Chemical Analyses of Raw Materials

1. Proximate analysis of low temperature carbonized coke (Kolsit)

Moisture %	Ash %	Volatile matter %	F.C. %
2·4	19·3	9·2	69·1

2. Ash analysis of low temperature carbonized coke (Kolsit)

SiO_2 %	MgO %	Al_2O_3 %	Fe %	CaO %	P %	S %
66·3	2·2	23·3	4·2	0·7	0·028	0·06

3. Analysis of iron ore (B. Patnaik)

Fe %	SiO_2 %	Al_2O_3 %	S %	P %
63·2	2·88	5·6	—	0·05

4. Analysis of iron ore (Orissa Minerals)

Fe %	SiO_2 %	Al_2O_3 %	S %	P %
64·14	3·28	4·57	0·01	0·02

5. Analysis of fluxes

Flux and location	CaO %	MgO %	SiO_2 %	Al_2O_3 %	S %	Fe %
Limestone (Madras)	54·31	1·0	0·88	1·2	0·027	—
Dolomite (Assam)	31·3	20·7	0·4	—	0·6	—

6. Analysis of low temperature carbonized coke (C.F.R.I.)

Moisture %	Ash %	Volatile matter %	F.C. %
8·5	25·9	4·1	61·5

7. Analysis of coke ash

SiO_2 %	MgO %	Al_2O_3 %	CaO %	Fe %	S %	P %
53·8	1·88	28·0	5·68	4·6	0·23	0·08

identical operational conditions, smelting trials were conducted with a burden com- and Orissa Minerals), low temperature carbonized coke (C.F.R.I. and Kolsit, R.R.L.), and blended fluxes.

The burden was calculated with a basicity ratio $CaO/SiO_2 = (P_1) = 1{\cdot}25$ and dolomite was added to obtain 8-10 per cent MgO in the slag. Besides, addition of Mn ore was progressively increased to obtain 1·0-1·2 per cent manganese in the metal. Incidentally manganese recovery in iron smelting in low shaft furnace was ascertained.

The chemical and screen analyses of raw-materials used in this trial are given in Tables 42 and 43.

The furnace was operated under the following conditions:

Tuyere diameter	75 mm.
Average blast volume	2100-2600 Nm.^{3}/hr.
Average blast pressure	1600-2000 mm.WG.
Hot blast temperature	500-550°C.
Top gas temperature	300-350°C.

The pig iron produced in this campaign analysed:

C %	Si %	S %	Mn %	P %
2·5-3·0	2·5-3·5	0·03-0·08	0·4-1·2	0·4

The slag analysed,

CaO	SiO_2	FeO	Al_2O_3	MgO
32-34	36-39	1·5-3·5	20-22	6-8

The pig iron produced was associated with average slag volume of 1·5-1·8. The top gases analysed:

CO %	CO_2 %	CH_4 %	H_2 %	O_2 %	N_2 %	Average CO/CO_2 ratio
24-26	3-3·5	3·5-4·5	nil-0·8	1·5-1·7	64-66	7-8

Although increase in manganese ore addition raised the manganese contents of the pig iron, the recovery was exceedingly poor particularly for higher manganese contents. Within the changes in the wind volume, the productivity slightly increased at higher wind rate without altering the coke rate significantly.

Twenty-seventh Campaign

In order to find out the industrial possibilities of iron smelting with low temperature carbonized coke made from wholly non-coking Talcher coal, smelting trials were conducted with iron ore (Orissa — B. Patnaik), low temperature carbonized coke made from Talcher coal (C.F.R.I.) with blended fluxes. Chemical and screen analyses of raw-materials used in this trial are given in Tables 44 and 45.

Table 43 — Screen Analysis of Raw Materials Employed

Raw materials	+50·8 mm %	−50·8 mm +24·4 mm %	−25·4 mm +12·7 mm %	−12·7 mm +6·35 mm %	−6·35 mm +3·175 mm %	−3·175 mm %
Low temperature carbonized coke (C.F.R.I.)	14·04	65·27	18·20	1·90	0·30	0·29
Low temperature carbonized coke (Kolsit-R.R.L.) −½″ screened	54·0	45·6	0·4	—	—	—
Iron ore (Orissa Minerals)	—	—	2·76	16·22	27·00	54·02
Iron ore (B. Patnaik)	—	—	5·4	60·2	34·4	—
Limestone (Madras)	—	73·91	23·81	1·47	0·27	0·54

Table 44 — Chemical Analyses of Raw Materials

1. Proximate analysis of low temperature carbonized coke (CFRI)

Moisture %	Ash %	Volatile matter %	F.C. %
8·5	25·9	4·1	61·50

2. Analysis of coke ash (C.F.R.I.)

SiO_2 %	MgO %	Al_2O_3 %	CaO %	Fe %	S %	P %
53·8	1·88	28·0	5·68	4·6	0·28	0·08

3. Proximate analysis of low temperature carbonized coke made from Talcher coal

Moisture %	Ash %	Volatile matter %	F.C. %	S %
5·4	16·9	8·6	69·1	0·30

4. Analysis of coke ash (Talcher)

SiO_2 %	MgO %	Al_2O_3 %	CaO %	Fe %	P %
65·36	Tr.	26·6	3·26	4·46	0·085

5. Analysis of fluxes

Flux and location	CaO %	MgO %	SiO_2 %	Al_2O_3 %	S %	Fe %
Limestone (Madras)	54·31	1·0	0·88	1·2	0·027	—
Dolomite (Assam)	31·3	20·7	0·4	—	0·6	—

Table 45 — Screen Analysis of Raw Materials Employed

Raw materials	+50·8 mm %	−50·8 mm +25·4 mm %	−25·4 mm +12·7 mm %	−12·7 mm +6·35 mm %	−6·35 mm +4·175 mm %	−3·175 mm %
Low temperature carbonized coke Talcher	—	40·3	50·2	6·5	1·5	1·5
Nut coke (TISCO)	—	44·2	49·5	3·2	1·8	1·3
Iron ore (B. Patnaik)	—	—	5·4	60·2	34·4	—
Limestone (Madras)	—	94·3	4·2	1·5	—	—
Dolomite (Assam)	—	—	70·4	27·2	2·4	—

The furnace was operated under the following conditions:

Tuyere diameter	75 mm.
Average blast volume	2400-2550 $Nm.^3$/hr.
Average blast pressure	1400-1600 mm.WG.
Hot blast temperature	450-550°C.
Top gas temperature	360-380°C.

Pig iron produced analysed:

C %	Si %	S %	P %	Mn %
2·8-3·0	2·15-3·7	0·07-0·08	0·13-0·14	0·25-0·35

The composition of slag was

CaO %	SiO_2 %	Al_2O_3 %	MgO %	FeO %
33-36	32-34	21-22	6-8	0·6-1·4

The average slag volume was between 1·2-1·5 tonnes/tonne of pig iron. The slag basicity $CaO/SiO_2 = P_1$ was maintained at 1·0. The top gas had CO/CO_2 ratio of 6·8 and the gas analysed:

CO %	CO_2 %	CH_4 %	H_2 %	O_2 %	N_2 %
25-30	3-3·5	4-4·8	nil-0·7	1·0	65-66·5

The low temperature carbonized coke made from other varieties of non-coking coals at the C.F.R.I. was gradually replaced by Talcher coke. The progressive replacement of low temperature carbonized coke (C.F.R.I.), which hitherto had been used extensively for trials by the Talcher coke did not reveal any conspicuous changes in smelting characteristics. It was concluded that this fuel can be employed for iron smelting in industrial low shaft furnace. It may be mentioned that recent trials in 100 tonnes/day low shaft furnace blast furnace at Barbil were also highly satisfactory.

Twenty-eighth Campaign

With the object of assessing the coke rate of two iron ores of widely different reducibility characteristics, as determined in the laboratory employing H_2 gas as reductant, smelting trials were conducted with different varieties of fuels with burden consisting of iron ores (B. Patnaik), and iron ore fines (Orissa Minerals) separately, low temperature carbonized coke (C.F.R.I.), low temperature carbonized coke (Kolsit — R.R.L.), nut coke, and blended fluxes. The calculated basicity $CaO/SiO_2 = P_1$ was maintained at 1·15 and dolomite was added to obtain 8-10 per cent MgO in the slag.

The chemical and screen analyses of the raw materials used in this campaign are given in Tables 46 and 47.

The furnace operated under the following conditions:

Tuyere diameter	75 mm.
Average blast volume	2400-2800 Nm.^{3}/hr.
Average blast pressure	1500-2000 mm.WG.
Hot blast temperature	450-500°C.
Top gas temperature	380-400°C.

The analytical basicity $CaO/SiO_2 = P_1$ fluctuated between 0·8 and 1·1 and on average slag analysed:

CaO %	SiO_2 %	Al_2O_3 %	MgO %	FeO %
32-37	33-36	20·5	5·0-7·0	1-2

while the metal analysed:

C %	Si %	Mn %	S %	P %
2·8-3·2	2·8-3·45	0·5-1·5	0·04-0·10	0·50

The average slag volume was 1·5-1·8 tonnes/tonne of pig iron. CO/CO_2 ratio of top gases was between 6 and 7 and it analysed:

CO %	CO_2 %	CH_4 %	H_2 %	O_2 %	N_2 %
23·0-27·5	3·0-4·5	3·0-6·0	nil-0·8	0·5-1·5	64-66

It was observed that the reducibility characteristics significantly affected the coke rate. Regardless of the nature of the fuel employed, the ore with high reducibility characteristics required lower amount of fuel. The finer particle size of the ore also contributed towards low fuel rate.

Twenty-ninth Campaign

The object of this campaign was to study the effect of oxygen enrichment of the blast and size grading of the ore on the operational and smelting characteristics.

Table 46 — Chemical Analyses of Raw Materials

1. Proximate analysis of low temperature carbonized coke (CFRI)

Moisture %	Ash %	Volatile matter %	F.C. %
8·5	25·9	4·1	61·5

2. Analysis of coke ash (L.T.C. — C.F.R.I.)

SiO_2 %	MgO %	Al_2O_3 %	CaO %	Fe %	P %	S %
53·8	1·88	28·0	5·68	4·6	0·8	0·28

3. Proximate analysis of Kolsit

Moisture %	Ash %	Volatile matter %	F.C. %
2·40	19·3	9·2	69·1

4. Analysis of coke ash (Kolsit)

SiO_2 %	MgO %	Al_2O_3 %	CaO %	Fe %	P %	S %
66·3	2·2	23·3	0·7	4·2	0·028	0·6

5. Analysis of iron ore (B. Patnaik)

Fe %	SiO_2 %	Al_2O_3 %	S %	P %
63·2	2·88	5·6	N.D.	0·05

6. Analysis of iron ore (Orissa Minerals)

Fe %	SiO_2 %	Al_2O_3 %	S %	P %
64·14	3·28	4·57	0·01	0·02

7. Analysis of fluxes

Flux and location	CaO %	MgO %	SiO_2 %	Al_2O_3 %	S %	Fe %
Limestone (Madras)	54·31	1·0	0·88	1·2	0·027	—
Dolomite (Assam)	31·3	20·7	0·4	—	0·6	—

Table 47 — Screen Analyses of Raw Materials Employed

Raw materials	+50 mm %	−50 mm +25 mm %	−25 mm +12·5 mm %	−12·5 mm +6·0 mm %	−6 mm +3 mm %	−3 mm %
Low temperature carbonized coke (C.F.R.I.)	21·3	45·1	31·2	1·6	0·3	0·5
Low temperature carbonized coke (Kolsit — R.R.L.)	17·2	53·8	28·0	0·5	0·15	0·35
Iron ore (B. Patnaik)	—	—	1·5	65·2	31·5	1·8
Iron ore (Orissa Minerals)	—	—	19·9	50·0	26·6	3·8
Limestone (Madras)	—	44·4	34·6	10·5	4·8	5·7
Dolomite (Assam)	—	—	56·0	30·5	8·5	4·1

The burden constituted of two different varieties of iron ores. low temperature blended fluxes. For establishing the benefits or otherwise of oxygen enrichment nut coke was employed as fuel.

Chemical and screen analyses of the raw-materials used in this campaign are given The replacement of lumpy ore with fines necessitated higher blast pressure to obtain similar wind volumes. The operational

Table 48 — Chemical Analyses of Raw Materials

1. Proximate analysis of nut coke

F.C. %	V.M. %	Mn %	Ash %	S %
74·70	2·50	0·20	22·90	0·50

2. Analysis of coke ash

CaO %	SiO_2 %	MgO %	Al_2O_3 %	Fe %	P %
3·8	52·08	2·12	33·0	6·0	0·69

3. Analysis of iron ore (Bolani)

Fe %	SiO_2 %	Al_2O_3 %	CaO %	MgO %	S %	P %
57·0	5·0	6·5	0·10	0·20	0·03	0·05

4. Analysis of manganese ore

Fe %	SiO_2 %	Al_2O_3 %	Mn %	CaO %	P %
2·45	10·8	20·0	47·5	2·5	0·13

5. Analysis of limestone (Madras)

CaO %	MgO %	SiO_2 %	Al_2O_3 %	S %
54·31	1·0	0·88	1·2	0·027

Table 49 — Screen Analysis of Raw Materials

Raw materials	+25·4 mm %	−25·4 mm +12·7 mm %	−12·7 mm +6·35 mm %	−6·35 mm +3·175 mm %	−3·175 mm %
Nut coke	38·30	61·00	—	—	0·70
Bolani iron ore					
Medium (a)	—	—	36·50	50·90	12·60
Coarse (b)	8·25	31·10	35·75	10·90	14·00
Fine (c)	—	—	—	4·15	95·85
Madras limestone	48·00	36·50	9·20	4·70	1·60
Manganese ore	—	18·25	58·34	15·78	7·63

results are summarized in Table 50. The calculated basicity degree of the slag CaO/$SiO_2 = P_1$ was maintained between 0·9-1·15.

Table 50 — Operational Characteristics with Size-grading of Iron Ore

Mean size grading of ore	−25+6 mm	−12+3 mm	−3 mm
Av. hot-blast pressure mmWG	1600	1650	1700
Av. hot-blast volume Nm^3/h	2400	2350	2300
Av. hot-blast temperature °C.	520	480	525
At the tuyere °C.	465	440	470
Av. top gas temperature °C.	470	460	440
Av. top gas analysis CO%	25·2	25·0	25·6
CO_2%	4·8	3·6	3·92
CH_4%	4·0	4·4	3·88
CO/CO_2	6·7	6·9	6·0
F.C. consumed/tonne of pig iron	2·08	1·78	1·48
Basicity degree CaO/SiO_2	1·2	1·3	1·3
Slag volume/tonne of pig iron %	1·26	1·18	1·00
Dust loss/tonne of pig iron %	1·6	1·6	1·4
Flux rate/tonne of pig iron %	800	800	640
Production/day/tonne	6·28	7·20	9·00

The pig iron produced in this campaign analysed:

C %	Si %	S %	Mn %	P %
2·8-3·0	2·5-4·0	0·04-0·10	0·5-0·6	0·4-0·6

while the slag analysed:

CaO %	SiO_2 %	Al_2O_3 %	MgO %	FeO %
34-38	32-34	20·5	5-8	0·5-2·5

In confirmity with the previous observation (Campaign XIX), replacement of lumpy ore by fine-grained ore indicated more efficient utilization of fuel as indicated by CO/CO_2 ratio and coke rate. The flux rate decreased remarkably despite increase in the basicity degree. The associated decrease in slag volume increased the productivity. It was found that ore fines of −12 + 5 mm. can be smelted in low shaft furnace without any operational difficulties. The enrichment of the blast with 1-3 per cent oxygen, in the absence of any other blast additive, such as moisture and/or supplementary fuel, was of marginal consequence and did not lead to any remarkable improvement in smelting efficiency. However, further trials will be conducted for confirmation.

Thirtieth Campaign

With the object of thoroughly understanding the mechanism and the technological implications of fuel oil injection, preliminary campaign was conducted with a burden consisting of a mixture of iron ores (Orissa Mineral, N. V., B. Patnaik, Bolani) with low temperature carbonized coke (C.F.R.I.) with blended fluxes. Chemical and screen analyses of the raw-materials used in this campaign are given in Tables 51 and 52.

The furnace was blown in under the following conditions:

Tuyere diameter	75 mm.
Average blast volume	2500-3000 $Nm.^3/hr.$
Average blast pressure	1700-2000 mm./WG.
Hot blast temperature	450-500°C.
Hot gas temperature	350-450°C.

The metal produced analysed:

C %	Si %	S %	Mn %	P %
2·5-2·9	2·7-4·5	Trace-0·08	0·1-0·6	0·7

The slag associated with metal analysed:

CaO %	SiO_2 %	Al_2O_3 %	MgO %	FeO %
25-35	32-36	21-22	4-5·5	1·5-5·0

Table 51 — Chemical Analysis of Raw Materials

1. Analysis of iron ore (B. Patnaik)

Fe %	SiO_2 %	Al_2O_3 %	S %	P %
63·2	2·88	5·6	—	0·05

2. Analysis of iron ore (Orissa Mineral)

Fe %	SiO_2 %	Al_2O_3 %	S %	P %
64·14	3·28	4·57	0·01	0·02

3. Analysis of iron ore (Bolani)

Fe %	SiO_2 %	Al_2O_3 %	CaO %	MgO %	S %	P %
57·0	5·0	6·5	0·10	0·20	0·03	0·05

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

S %	F.C. %	V.M. %	Moisture %	Ash %
0·23	61·5	4·1	8·5	25·9

5. Analysis of ash from low temperature carbonized coke

CaO %	SiO_2 %	MgO %	Al_2O_3 %	Fe %	P %
5·68	53·8	1·88	28·0	4·6	0·80

6. Analyses of fluxes

Flux and location	CaO %	MgO %	SiO_2 %	Al_2O_3 %	S %	Fe %
Limestone (Madras)	54·1	1·0	0·88	1·2	0·027	—
Dolomite (Assam)	31·3	20·7	0·4	0·6	—	—

Table 52 — Screen Analyses of the Raw Materials Employed

Raw materials	−50·8 mm +24·4 mm %	−25·4 mm +12·7 mm %	−12·7 mm +6·35 mm %	−6·35 mm +3·17 mm %	−3·17 mm %
Low temperature carbonized coke (C.F.R.I.)	59·50	37·50	Nil	Nil	3·00
Iron ore (B. Patnaik)	Nil	2·00	49·85	42·25	5·90
Limestone (Madras)	93·00	4·75	1·60	0·50	0·15
Dolomite (Assam)	15·95	79·80	4·00	Nil	0·25

The average slag volume was at 1·3-1·5 tonnes/tonne of pig iron and the basicity of the slag P_1 value of between 0·8 and 1·0. The gas analysed:

CO %	CO_2 %	CH_4 %	H_2 %	O_2 %	N_2 %
24·5-27·5	3·0-4·0	3·2-4·5	nil-0·5	0·8-1·5	63-66

During this investigation, the burden was calculated to yield a lime basicity degree of the slag $CaO/SiO_2 = P_1 = 1{\cdot}20$-1·25, and the dolomite was added in the burden so as to have 6-8 per cent MgO in the slag. During the entire trial period, the blast pressure was maintained at

1700-2000 mm.WG. while the maximum available blast temperature varied between 450 and 500°C.

In view of the limitations in raising the temperature of the hot-blast by the recuperator system of heating, it was considered that oxygen enrichment of the blast was essential for compensating the heat requirements for the endothermal dissociation of the auxiliary fuels. The investigation was, therefore, broadly divided into three stages, in which the oxygen enrichment of the blast was varied from 1 through 2 to 3 per cent respectively, keeping the raw materials and the operational variables constant as far as practicable. The smelting operation in each individual stage was of sufficiently long period for attaining equilibrium and to ensure correct data.

With oil injection at the rate of 30 kg./tonne of pig iron, the enrichment of the air-blast with oxygen in the three different stages was observed to exercise marked effect on smelting characteristics and the productivity. It was observed that with the continuity of the third stage of operation, at which fuel oil was injected at the rate of 30 kg./tonne of pig iron produced (calculated amount) and the blast was enriched by about 3 per cent O_2, the coke rate dropped by 50 per cent in respect of F.C.

In the third stage of operation, the daily production increased from 6·6 tonnes/day without oil injection to about 10·5 tonnes/day with oil injection and 3 per cent O_2 enrichment of the blast. It is considered that at the oil injection rate of 30 kg./tonne of pig iron, the required degree of blast enrichment should amount to about 3 per cent. Under these conditions the metal analysed: C, 2·75; Si, 3·70; S, 0·06 per cent, while the corresponding slag analysed: CaO, 33·0; SiO_2, 36·00; FeO_2, 3 per cent and top gas CO, 27·6; CO_2, 5·00; CH_4, 3·60; oxygen, 1·0 per cent; hydrogen, nil per cent. With increase in the oxygen content of the blast from 1 to 3 per cent, no conspicuous change in the oxygen content of top gas was noticed; neither CO/CO_2 ratio was altered. It was, however, very interesting to note that despite the high injection rate of 30 kg. of furnace oil/tonne of iron (calculated production) the top gas did not contain any hydrogen which showed optimum utilization of hydrogen dissociated from the fuel oil in the furnace.

On the basis of this preliminary trial, it can be inferred that with the injection of auxiliary fuels through tuyeres coupled with optimum enrichment of air-blast with oxygen, the fuel rate can be effectively lowered and iron productivity significantly increased.

After the termination of fuel oil injection trials with simultaneous enrichment of the blast with oxygen, naphtha was injected through two lances placed in two diametrically opposite tuyeres initially without oxygen enrichment of the air-blast but subsequently with oxygen enrichment of the air-blast. During continuous operation, no operational difficulties were experienced such as formation of naphtha vapour in the lances with development of back pressure thereby disallowing any further naphtha injection in the furnace, coking of naphtha in the injection lances or any other operational hazards. As a matter of fact, the visual examination of injector lances after their withdrawal showed them to be absolutely clean without any evidence of carbonization occurring therein. However, duration of this test trial was not long enough to assess the effect of naphtha injection on the smelting characteristics such as the fuel rate, slag volume and productivity. Further trials with naphtha injection through all the four furnace tuyeres are underway.

74.1 Investigations on Briquetting

Briquetting of Iron Ore Fines

Iron ore fines require agglomeration like sintering, pelletizing for obtaining iron by the conventional blast furnace process. receiving considerable attention as briquetting process is not limited to close particle size classification of the feed. Besides, the uniform size of briquettes and relative freedom from fines affords optimum permeability to the reducing gases in furnace stack and, therefore, economizes the fuel requirement. Investigations were taken up on the hot briquettability of some Indian iron ore fines. The screen analysis of the iron ore fines employed are given in Table 53. Iron ore fine was heated to a temperature of 850°-900°C. The hot ore fines were fed to a briquetting machine which is originally meant for making briquettes from a mix containing iron ore fines, non-coking coal and limestone (0-5 mm.) with the addition of binders.

Although the fine ore fines were pressed in the form of pillow shaped briquettes, 63 mm. long × 45 mm. wide × 36 mm. high, the physical strength was unsatisfactory due to the limitations of low preheating temperature which was not high enough to make it semiplastic and the totally inadequate pressure developed in the briquetting press. The variation in the grain size of the ore did not improve the physical properties. Further investigations are being conducted with minor additions of coking coal to provide plasticity rately high temperatures functioning as a binder.

74.2 Malleabilization Characteristics of Low Shaft Furnace Pig Iron

Smelting of pig iron in the low shaft furnace with low temperature carbonized coke made from non-coking coals yields pig iron containing low phosphorus contents which is ideally suitable for making malleable castings.

It was previously noticed that the presence of small percentage of boron of 0·03 per cent maximum and residual aluminium in the melt significantly lowered the malleabilization cycle. The effects of different amounts of these elements were thoroughly investigated. In order to study the fundamentals of malleabilization, the effect of prior microstructure on the morphology of graphite nodules was studied. Experimental melts containing boron and aluminium were normalized, oil-quenched and water-quenched before subjecting them to malleabilization. It was noticed that such pretreatment had considerably lowered the heat treatment period during malleabilization.

Table 53 — Screen Analyses of Iron Ore Fines

Ore samples	+2·41 mm %	−2·41 +1·00 mm %	−1·00 +0·50 mm %	−0·50 +0·252 mm %	−0·252 +0·152 mm %	−0·152 +0·076 mm %	−0·076 mm %
Orissa Mineral	9·7	22·8	25·8	9·5	10·3	7·6	14·3
Ratnagiri	7·2	6·4	8·3	10·3	25·3	23·3	19·2
Rajhara iron	12·9	6·5	7·9	11·0	21·2	24·0	16·5
Blue dust iron (Noamundi)	32·7	10·7	9·4	6·3	7·8	8·9	24·2

75.0 Pneumatic Method of Steel Making in Basic-lined Side-blown Converter

The object of this Project is to produce acceptable grades of steel from Indian pig iron containing about Si, 1·0-1·2 and P, 0·30-0·40 per cent in half-a-tonne basic-lined side-blown converter, designed and fabricated in the Laboratory. After proper standardization of the operational procedures and techniques for the conversion of hot metal C, 3·20-4·0; Si, 0·7-1·0 and P, 0·25-0·35 per cent to low carbon steel, further investigations were directed towards the following:

The addition of burnt-lime or limestone in the form of lumps or pellets made from the beneficiated limestone and briquettes made from the beneficiated limestone with bentonite and sulphite lye as binders to evaluate their efficiency during steel making and its economics. The results are tabulated in Tables 54 and 55. The ingots were forged and the mechanical properties are recorded in Table 56. In order to find out the soundness of the ingots an ingot was sectioned vertically. The cross-section did not show any unsoundness (Fig. 88). Macro-etching and sulphur print did not reveal any segregation or heterogeneity. It is well known that the extent of oxidation reactions are dependant on bath temperature. Attempts were made to measure the temperature continuously during the period of blow by appropriate insertion of Pt/Pt. Rh thermocouple particularly to enable to reduce the bath temperature by suitable additions for facilitating dephosphorization. Optimum utilization of steel scrap was investigated. Further studies on the process control, yield and techniques of flux addition are underway.

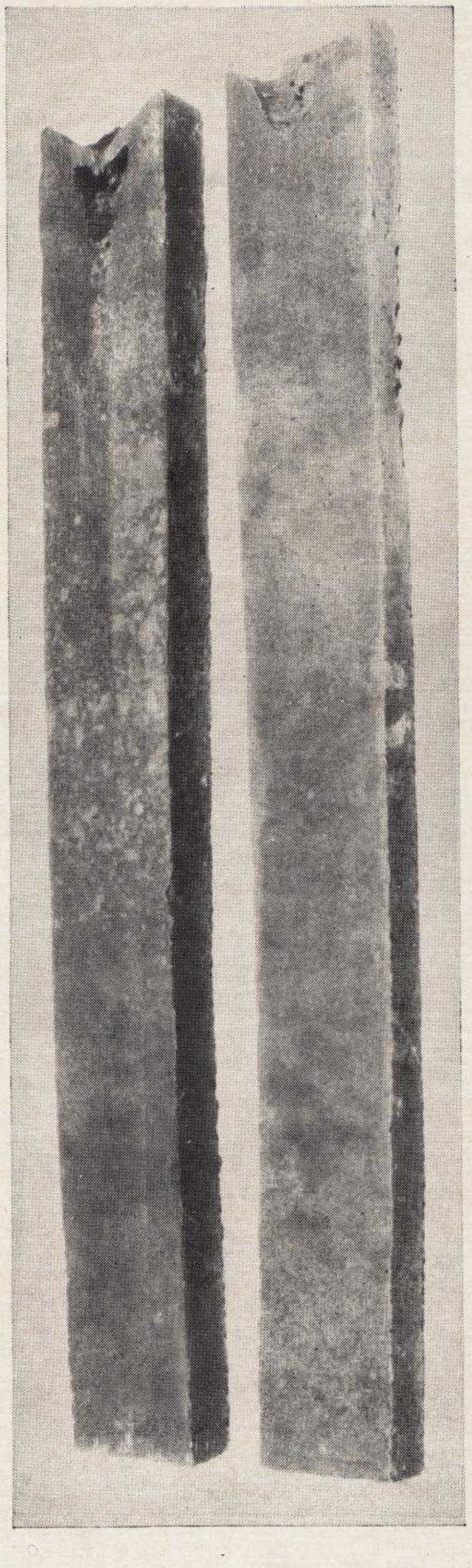


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FIG. 88 — VERTICAL CROSS-SECTION OF AN EXPERIMENTAL STEEL INGOT MADE BY THE BASIC-LINED SIDE-BLOWN CONVERTER PROCESS SHOWING ABSENCE OF BLOW HOLES

Table 54 — Experimental Results of Some of the Blows

Sl. No.	Approx. wt. of hot metal blown kg.	Temp. of molten iron metal °C.	Time of blow min.	Temp. of blown metal °C.	Addition				Sample	Chemical analysis, %						Slag analysis, %					Basicity CaO/SiO$_2$ %
					Lime kg.	Fe-Si kg.	Fe-Mn kg.	Mill scale kg.		C	Si	P	S	Mn	N	CaO	SiO$_2$	MgO	P$_2$O$_5$	Fe$_2$O$_3$ + FeO	
1	520	1180	35	1720	20 kg. after 5 min.	4	4	—	Hot metal	4·05	0·887	0·23	0·06	0·64	—	25·9	23·2	13·5	2·5	32·30	1·11
					15 kg. after 15 min.				Blown metal	0·04	0·149	0·11	0·047	0·82	—	—	—	—	—	—	—
2	540	1180	35	1700	20 kg. after 5 min.	4	—	—	Hot metal	4·0	0·73	0·28	0·06	0·33	—	—	—	—	—	—	—
					20 kg. after 15 min.				Blown metal	0·04	0·02	0·07	0·05	0·08	0·004	9·02	15·7	18·8	1·71	48·35	0·68 1st slag
																10·8	9·0	21·5	4·70	49·60	1·20 2nd slag
3	500	1190	35	1600	20 kg. after 5 min.	4	—	2	Hot metal	3·85	0·66	0·23	0·06	0·58	—	—	—	—	—	—	—
					30 kg. after 15 min.				Blown metal	0·27	0·16	0·03	0·024	0·17	0·006	9·82	17·44	9·48	0·75	59·44	0·56 1st slag
																18·76	9·96	9·41	4·39	55·46	1·60 2nd slag
4	500	1200	30	1600	40 kg. after 5 min.	7	6	—	Hot metal	3·05	1·29	0·42	0·05	0·58	—	—	—	—	—	—	—
					20 kg. after 15 min.				Blown metal	0·59	0·5	0·05	—	0·29	—	10·6	19·64	10·65	2·2	43·77	0·55 1st slag
																16·2	15·32	20·00	1·8	43·44	1·06 2nd slag
5	500	1190	30	1690	30 kg. lumpy after 5 min.	4	—	—	Hot metal	3·6	1·4	0·25	0·05	0·58	—	—	—	—	—	—	—
					20 kg. lumpy & powder after 10 min.				Blown metal	0·72	0·23	0·18	0·025	trace	—	22·54	28·08	13·4	1·2	33·3	0·88

6	350	1200	25	1620	20 kg. lumpy & powder after 5 min.	4	4	—	Hot metal	3·62	1·50	0·41	0·05	—	—	—	—	—	—	—	—
					20 kg. lumpy & powder after 10 min.				Blown metal	0·60	0·09	0·09	—	—	—	5·74	5·24	28·8	2·3	54·28	1·0
7	500	1180	30	1650	20 kg. briquettes after 5 min.	7	6	—	Hot metal	3·8	1·3	0·39	0·04	0·67	—	—	—	—	—	—	—
					30 kg. after 10 min.				Blown metal	0·03	1·1	0·08	—	—	—	8·86	7·48	31·85	1·08	48·05	1·2
8	400	1180	25	1580	20 kg. briquettes after 5 min.	7	6	—	Hot metal	3·375	1·25	0·439	0·058	0·5	—	—	—	—	—	—	—
					22 kg. briquettes after 10 min.				Blown metal	0·09	0·045	0·02	0·1	0·08	—	19·81	13·29	14·35	1·18	41·99	1·49
9	500	1180	25	1675	20 kg. lumpy after 5 min.	8	4	2	Hot metal	3·85	1·25	0·23	0·056	0·58	—	—	—	—	—	—	—
					25 kg. lumpy after 10 min.				Blown metal	0·57	0·41	0·05	0·026	0·9	—	13·0 33·3	30·48 23·60	15·26 12·46	3·2 2·9	35·36 25·50	0·42 1st slag 1·40 2nd slag

Table 55 — Experimental Results with the Addition of Limestone Pellets

Sl. No.	Approx. wt. of the hot metal blown kg.	Temp. of the molten iron °C.	Time of blow min.	Temp. of blown metal °C.	Sample	Chemical analysis, %						Slag analysis, %					Basicity CaO/SiO$_2$
						C	Si	P	S	Mn	N	CaO	SiO$_2$	MgO	P$_2$O$_5$	Fe$_2$O$_3$ + FeO	
1	400	1160	30	1680	Hot metal	3·8	1·6	0·39	0·03	0·67	—	18·74	21·92	11·94	0·72	38·68	0·85 1st slag
					Blown metal	1·74	0·57	0·19	0·067	0·39	—	17·54	20·82	11·00	0·64	41·24	0·84 2nd slag (40% limestone pellets)
2	450	1180	30	1650	Hot metal	3·4	1·2	0·42	0·035	0·61	—	—	—	—	—	—	45% limestone pellets
					Blown metal	0·19	0·32	0·2	0·06	—	—	22·84	26·44	21·84	0·95	26·57	0·82
3	400	1180	30	1680	Hot metal	3·6	1·4	0·29	0·02	0·67	—	—	—	—	—	—	50% limestone pellets
					Blown metal	0·18	1·1	0·16	0·067	0·47	—	21·56	22·23	10·28	0·32	44·90	0·97 1st slag
												21·32	20·68	9·64	0·30	45·45	1·0 2nd slag
4	300	1180	30	1600	Hot metal	3·51	1·654	0·349	0·047	0·587	—	—	—	—	—	—	20% of limestone pellets
					Blown metal	0·288	0·9	0·042	0·057	0·55	—	24·96	24·64	14·64	1·15	29·34	1·0 20% of limestone briquettes

Table 56 — Physical Properties of Low Carbon Steel

Yield stress T.S.I.	Maximum stress T.S.I.	Elong. on 2″ gauge length %	Charpy impact toughness kg/cm.^{2}
18·0	24·00	40·6	14·12

76.0 Experimental Baby Blast Furnace for Studying Injection of Auxiliary Fuels

The experimental miniature blast furace has been designed and fabricated primarily for assessing the fuel requirement under various operational conditions, such as oxygen-enrichment of the blast, injection of various auxiliary fuels, etc. The coke rate for the base period with normal air blast was determined as the initial parameter for comparison with other conditions.

During the second stage, oxygenated air-blast was used in place of normal air-blast keeping all other operating conditions and raw material unchanged. With 3-4 per cent oxygen enrichment of the air-blast, the fuel rate was reduced from 1·43 tonnes of fixed carbon/tonne of pig iron in the base period to 0·9 tonne of fixed carbon per tonne of pig iron and the productivity increased remarkably by 30 per cent. Chemical analyses of metal and slag (Tables 57, 58) became consistent with better slag-metal separation. The carbon saturation was exceedingly low. Investigations with higher percentage of oxygen in blast and injection of auxiliary fuels through the tuyeres are under progress.

Table 57 — Analysis of Pig Iron

C %	Si %	S %	P %	Mn %
2·2	2·1	0·10	0·33	0·40

Table 58 — Analysis of Slag

CaO %	SiO_2 %	Al_2O_3 %	MgO %	Fe %
36·50	33·00	20·50	6·30	3·70

77.0 Investigation on Carbon Saturation in Normal Cupola Practice

Due to the acute shortage of pig iron for the grey iron foundries, a study on carbon saturation of steel scrap in normal cupola operation is of industrial importance.

Investigations were conducted with progressively increasing steel scrap constituting the metallic charge under different basicity degrees of the slag during normal cupola practice using cold air-blast. For attaining neutral, acid and basic slags; charges were melted in both acid and basic lined cupolas.

The chemical analyses of the raw materials used in the investigation are recorded in Tables 59 and 60.

The size classification of the raw materials used is recorded in Table 61.

The charges with 50, 75 and 100 per cent steel scrap and the compositions of the iron before and after melting has been given in Table 62.

Effect of increasing steel scrap on the carbon saturation is shown in Fig. 89. The dependence of slag basicity CaO/SiO_2 on carbon solubility under basic conditions is shown in Fig. 90. On the basis of the investigations it has been concluded that:

(i) 100 per cent steel scrap could be successfully melted in a cupola with a coke-to-metal ratio of 1:3 under normal practice using cold air-blast,

(ii) the carbon saturation of the melt improved with the basicity degree (CaO/SiO_2) of the slag,

(iii) in a basic-lined cupola operated with normal air-blast using 100 per cent

Table 59 — Chemical Analyses of Coke, Limestone and Dolomite

1. Proximate analysis of coke on air-dried basis

M. %	Ash %	Volatile matter %	F.C. (by diff.) %	Total S %
1·15	23·50	2·0	73·75	0·47

2. Analysis of coke ash, limestone and dolomite

	SiO_2 %	Fe %	Al_2O_3 %	CaO %	MgO %	P_2O_5 %
Coke ash	54·3	5·0	28·7	3·04	1·95	1·70
Limestone	0·9	—	1·2	54·3	1·0	—
Dolomite	0·4	—	0·6	31·3	20·7	—

Table 60 — Chemical Analysis of Pig Iron and Steel Scrap

	T.C. %	Si %	S %	Mn %	P %
Pig iron	2·9	2·5	0·07	0·15	0·20
Steel scrap	0·23	0·065	0·07	0·49	0·10

Table 61 — Size Ranges of the Different Raw Materials

Materials	Size in mm
Bed coke	90 to 110
Split coke	50 to 70
Limestone	30 to 45
Dolomite	25 to 35
Pig iron	150 × 70 × 50
Steel scrap	70 × 50 × 12

steel scrap charge, cast iron having a carbon content of about 2·6 per cent could be successfully produced at a slag basicity (CaO/SiO_2) of 1·3,

(iv) for obtaining the desired amount of silicon and manganese, ladle additions of ferro-alloys were necessary and the recovery of these elements were satisfactory.

78.0 Scrap Oxygen Steel Making in L-D Converter

Pig iron and steel scrap are available in Indian foundries and their conversion to steel by electric furnace depends on the availability of cheap power. Work on scrap-oxygen-steel making process was initiated with the objective of refining cold

Table 62 — Charges with 50, 75, 100 Per Cent Steel Scrap and Composition of Iron Before and After Melting

Charge	Pig iron 50% steel 50%		Pig iron 25% steel 75%		Pig iron nil steel 100%	
	B.M.	A.M.	B.M.	A.M.	B.M.	A.M.
T.C.%	1·56	2·34	0·93	2·43	0·23	2·57
Si%	0·28	2·5	0·67	2·52	0·06	2·54
Mn%	0·32	0·4	0·41	0·4	0·49	0·51
P%	0·15	0·14	0·12	0·12	0·10	0·12
S%	0·07	0·06	0·07	0·065	0·07	0·064

B.M. — Before melting
A.M. — After melting

Coke 32 kg.
Limestone 6·5 kg.

in split charges in all cases

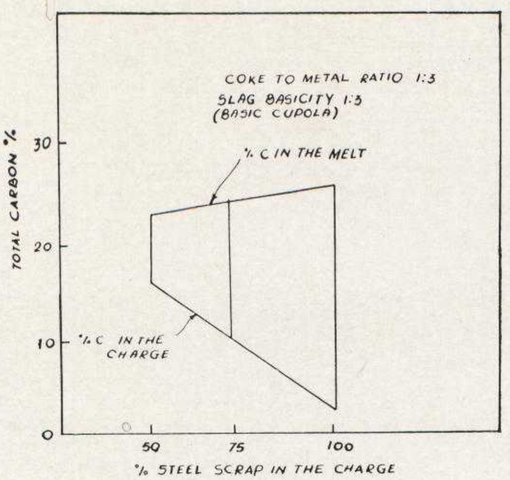

FIG. 89 — EFFECT OF INCREASING AMOUNTS OF STEEL SCRAP ON CARBON SATURATION

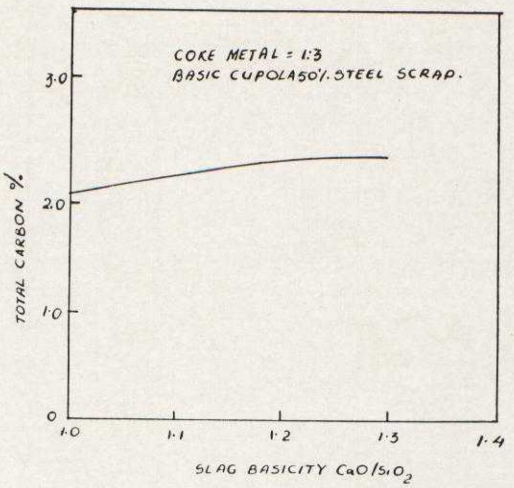

FIG. 90 — DEPENDENCE OF CARBON SATURATION ON SLAG BASICITY UNDER BASIC CONDITION

pig iron and steel scrap to steel by melting in the presence of a cheap solid fuel such as coke along with oxygen followed by refining the melt with oxygen as in basic L.D. Process.

For investigations in pilot plant scale, the L-D vessel was heated, pig iron scrap and solid fuel were charged in alternate layers. The oxygen through 6 mm. dia. consumable lance tube was injected from the top as in normal L.D. Process. On completion of melting of pig iron scrap, lime was added for the removal of oxides of metalloids in the form of a basic slag. Double slagging was necessary for promoting dephosphorization. After completion of blowing the hot metal was poured in ingot mould with the proper deoxidation. The amount of solid fuel and the flux were adjusted to obtain proper molten bath and dephosphorization. The chemical analyses of pig iron scrap used and blown metal are given in Table 63.

The tap to tap time, oxygen consumption, yield, size of pig iron scrap, steel scrap additions and economics of the process were investigated. Besides burnt lime, beneficiated lime stone briquettes or pellets were employed as flux which will reduce the cost of production.

Table 63 — Chemical Analysis of Pig Iron Scrap and Blown Metal

Elements	Pig iron scrap	Blown metal
C%	2·60	0·045
Si%	3·70	0·90
S%	0·05	0·04
P%	0·64	0·065
Mn%	0·70	Trace

79.0 Appraisal of Raw Materials for Iron Making

(*A*) *Iron Ores*

Physical and chemical characteristics of raw materials employed for iron making are of great significance in attaining optimum efficiency and for increasing the productivity. As the metallurgical value of iron ore depends on its chemical composition and its behaviour during handling and smelting, the mineralogical constituents, degradation characteristics, porosity, reducibility, decrepitation behaviour, crushing strength at room and elevated temperatures of a large variety of iron ores collected from different occurrences in India were determined and compared with particular reference to their smelting characteristics in the low shaft furnace.

shatter strength increased with cold crushing strength of iron ores; the effect was more pronounced on abrasion index than on shatter index.

(b) Improvement on the abrasion index of different ores from 60 to 95 per cent on + 6·25 mm. fraction reflected on the reducibility ores.

(c) The undersize (−6·25 mm.) particles of ores adversely influenced the abrasion indices rather sharply, the inference being that ores of poor abrasion index resulted in correspondingly larger amounts of undersize particles during its smelting.

(d) With increase in decrepitation temperature of ores, the reducibility of the ores decreased due to the effect of the particle size on reducibility.

(e) Ores with higher reducibility required lower amounts of fuel for smelting.

(f) For high smelting efficiency, the ores should have satisfactory reducibility, high porosity, high abrasion and shatter indices and reasonable hot compression strength.

(*B*) *Limestones*

It is well known that on heating to 950°C. limestone dissociates to calcium oxide and carbon dioxide. Calcium oxide combines with acidic constituents of the percentage of CO_2 will favour the

$$C + CO_2 = 2\ CO$$

It is necessary to assess the dissociation characteristics of limestone to ascertain the stack height at which CO_2 is liberated. An apparatus was set up to determine the beginning and end of dissociation. The results are given in Table 64 and Fig. 91 which show difference in dissociation characteristics.

80.0 Distribution of the Burden in the Stack of Low Shaft Furnace

In order to improve the smelting efficiency in the low shaft furnace, the distribution patterns of various raw materials were further determined in a previously designed and fabricated half-scale model of the actual 15 tonnes/day furnace. The distribution behaviours were mapped separately with individual constituent composing the burden and also with a few typical composite burdens employed during smelting trials, with or without admission of cold air-blast. The direction of rotation of the charging drum was alternatively reversed to assess its effect on the distribution. The angles of repose of the individual raw material composing the burden and the composite burden were measured. In the

Table 64 — Calcination Characteristics of Limestones

Name of limestone from	Initial temperature of calcination °C.	At constant rate of heating up to 590°C.		
		Time in min.	CO_2 evolved × 1/12 cuft or × 2·36 lit.	Size of limestone
Madras	800	240	8·09	−25·4 +19·0 mm
Bombay I	650	340	5·06	do
Andhra	740	—	—	do
Nepal	740	150	7·05	do
Bombay II	780	110	6·77	do
K.K.T.	800	160	6·64	do
Chowdhury & Dutta	775	150	8·94	do
Bisra	720	150	8·08	do

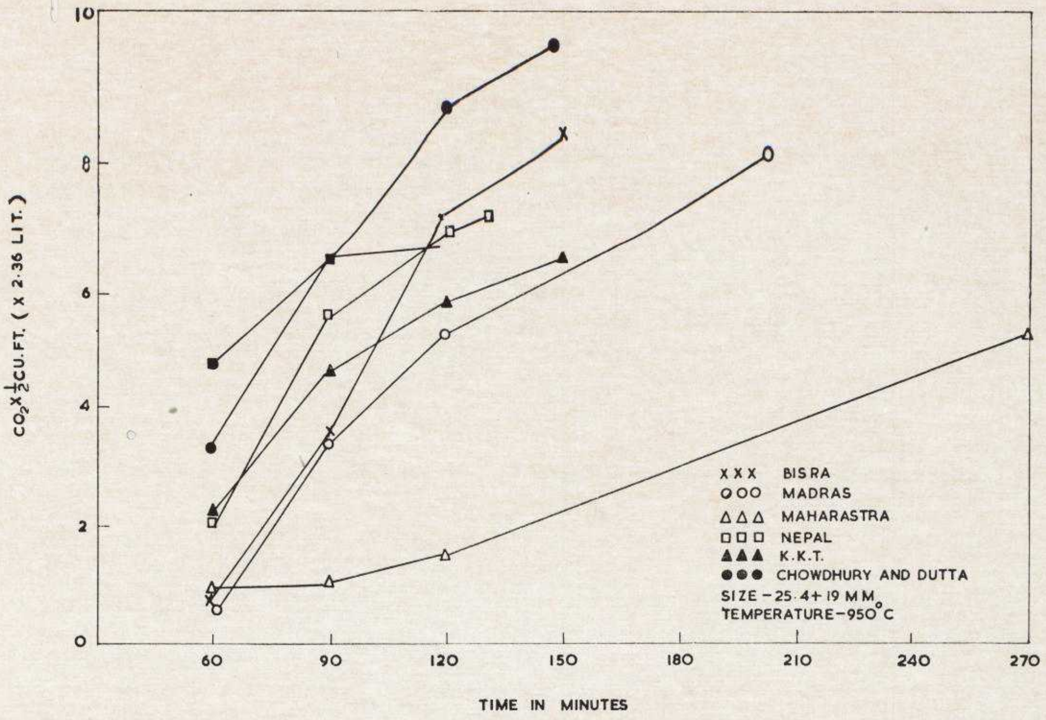

FIG. 91 — DISSOCIATION CHARACTERISTICS OF SEVERAL LIMESTONE SAMPLES

static condition, the blast pressure decreased from 100-130 mm.Hg at the tuyere level to 2-15 mm.WG at the top, depending on particle size of raw materials. The distribution patterns, with or without blast, are depicted in Fig. 92. The blast pressure slightly altered the angle of repose and improved the size distribution but the alternate reversal of the direction of rotation of the charging mechanism assured 'M' type contour, which improves the smelting efficiency.

81.0 Production of Pig Iron from Run-of-Mine Iron Ore, Limestone and Coke Breeze in a Rotary Furnace

Although several alternative methods of iron making have been developed, the classical technique of smelting iron is a blast-furnace smelting which contributes almost the entire world's output. The disadvantages of the blast-furnace process, such as dependance on metallurgical coke, lumpy or agglomerated or burden and partial utilization of the energy from the costly fuel, are well known. The imbalance existing between the known reserves of iron ore and coking coal and the location of coking coal deposits in India needs no emphasis. It was, therefore, considered that smelting of iron ore fines, with any solid fuel producing liquid pig iron will be of commercial importance and with this objective, iron ore fines, run-of-mine limestone and coke breeze were smelted in a rotary furnace. The chemical and screen analyses of raw materials are given in Tables 65 and 66.

After heating the 0·5 tonne acid-lined rotary furnace, a pool of molten pig iron was obtained. The charge was fed intermittantly and the CO liberated inside the furnace was burnt by a consumable oxygen

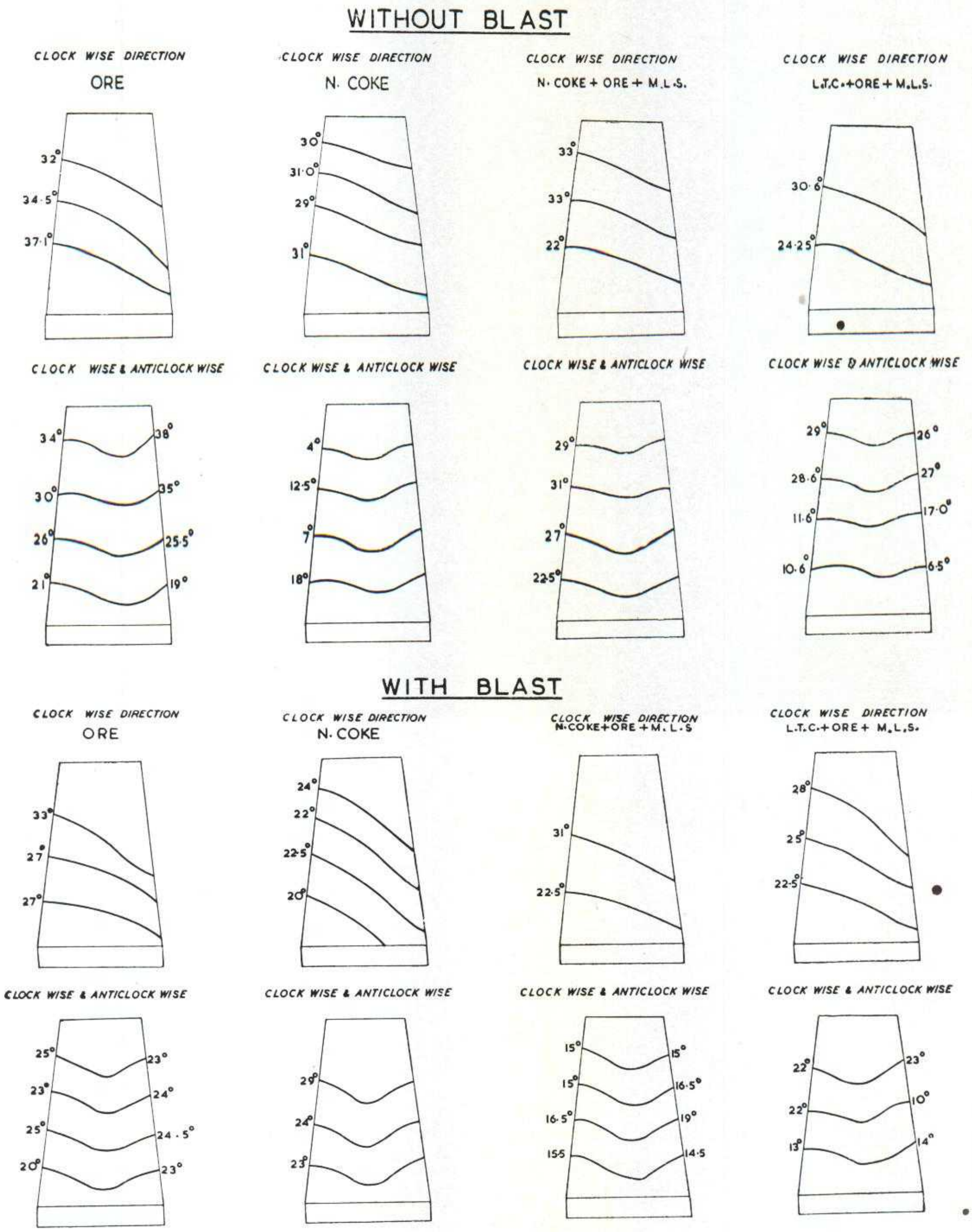

Fig. 92 — Distribution Patterns of Burdening Constituents in a Half-scale Model of the Low Shaft Furnace

Table 65 — Chemical Analysis of Raw Materials Employed for Smelting

1. Raw material

	Fe %	Mn %	SiO_2 %	Al_2O_3 %	CaO %	MgO %
Iron ore	64·14	—	3·28	4·57	Trace	Trace
Manganese ore	5·6	50·7	11·2	10·30	—	—
Limestone (A)	0·47	—	0·3	0·56	32·20	25·07
Limestone (B)	—	—	6·96	1·60	44·8	3·27

2. Analysis of coke breeze

	F.C.	V.M.	Moisture	Ash	S
Coke breeze	70·8	2·5	0·2	26·0	0·5

3. Analysis of ash

CaO %	SiO_2 %	MgO %	Al_2O_3 %	Fe %	S %	P %
3·8	52·08	2·12	33·0	6·0	—	0·69

Table 66 — Sieve Analysis of all the Raw Materials Employed for Smelting

1. Raw materials

	+50·8 mm %	−50·8 mm +25·4 mm %	−25·4 mm +12·7 mm %	−12·7 mm +6·35 mm %	−6·35 mm +3·175 mm %	−3·17 mm % below %
Iron ore (Orissa Minerals)	—	—	0·65	20·98	25·35	53·02
Limestone (B)	—	2·14	7·96	12·47	33·43	44·0
Limestone (A)	—	2·79	59·81	29·38	4·84	3·18

2. Mechanical grading of coke breeze

+7 mesh sieve	−7 mesh +16 mesh sieve	−16 mesh +30 mesh sieve	−30 mesh +60 mesh sieve	−60 mesh +100 mesh sieve	−100 mesh +200 mesh sieve	−200 mesh and below
7·85	23·76	32·13	14·95	9·30	5·60	6·41

lance. The exhaust gas had no chemical heat value. The metal analysed: C, 2·4; Si, 0·10; P, 0·31 while the slag analysed CaO, 18·2; SiO_2, 39·9; FeO, 1·4; MgO, 1·5 and Al_2O_3, 38·5 per cent. The high sulphur content is attributed to the acid slag. Identical work on a basic-lined vessel has been planned.

82.0 Thermal Conductivity of Blast-furnace Raw Materials

Determination of the coefficient of thermal conductivity of the iron ores is of interest in assessing the efficiency of the heat transfer between the descending burden and the ascending gases in the blast furnace.

This study was based on the steady state heat conditions created in the iron ore samples in the form of 1 in. cube. The iron ore sample was placed between two standard 1 in. cube samples made from high purity silver of known thermal conductivity. Good surface contact was established between the iron ore and standard samples, and a piece of 0·020 in. thick by 1×1 in. silver sheet was inserted in each interface to reduce the interfacial resistance. Six thermocouples (BS 28 SWG chromel/alumel) were placed in appropriate

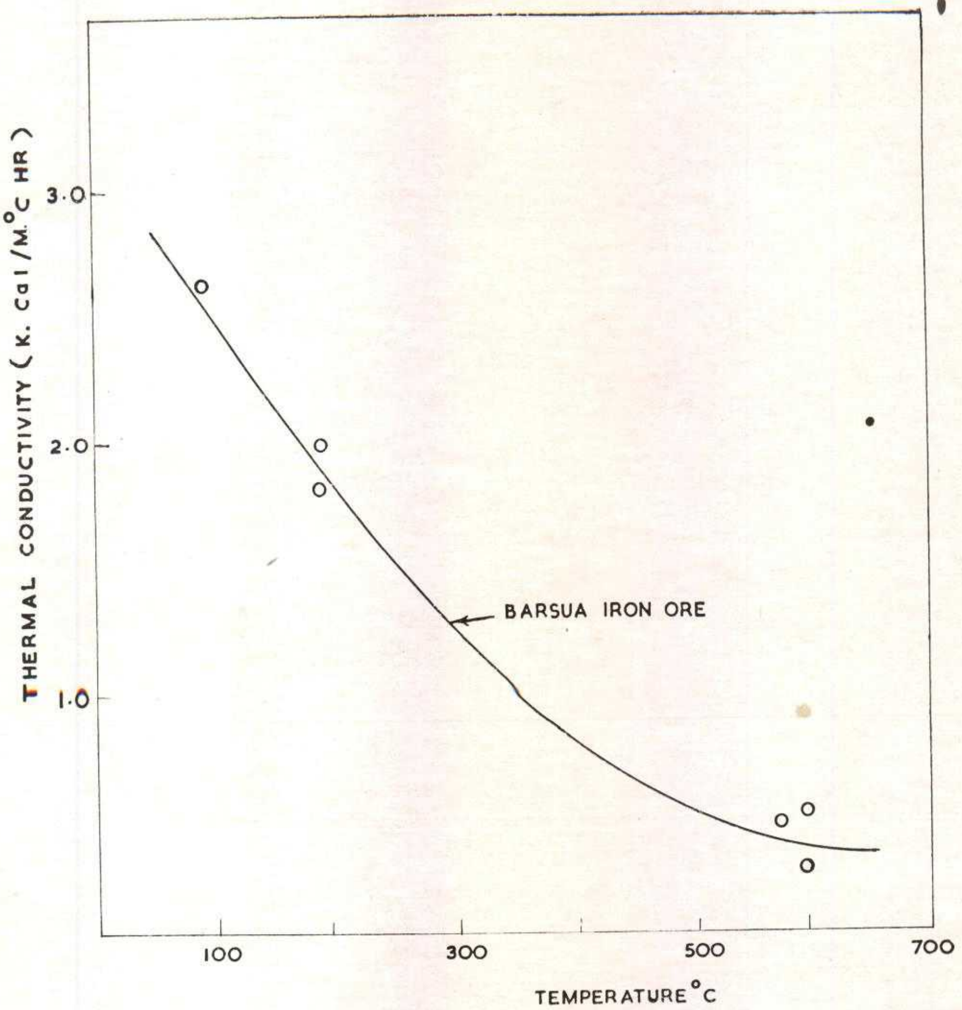

FIG. 93 — DEPENDENCE OF COEFFICIENT OF THERMAL CONDUCTIVITY OF IRON ORE ON TEMPERATURE

grooves in the samples. The tubular furnace was lowered to surround the specimens and the thermocouples in the tubular furnace were positioned directly opposite the interfaces between the samples. Refractory insulating material filled the space between the tube and the samples so as to reduce the heat transfer by convection.

Experiments were conducted at various elevated temperatures and the results on Barsua iron ore are shown in Fig. 93. It was observed that the thermal conductivity of the iron ore decreased as the temperature was raised.

83.0 Electrolytic Manganese Metal Pilot Plant

Regular production of manganese metal was continued in the 100 lb. electrolytic pilot plant for various research projects in the National Metallurgical Laboratory such as the development of magnetic alloys, nickel-free austenitic stainless steels, thermostatic bimetals, etc.

Manganese ore of the following composition was used for production of the metal.

	%
Manganese	34·53
Iron	19·85
Al_2O_3	8·28
SiO_2	5·13
P	0·18

Studies on the appropriate mesh size of the ground ore, with regard to reduction and leaching efficiencies and settling rate, indicated that mesh size −36 to + 72 was most suitable for work on electrolytic manganese. The leaching efficiencies with mesh size −36 + 72 were of the order of 85 per cent generally. Settling rate with regard to the end *p*H point of the leach had also been studied. The electrolytic cell design was further improved by providing spacer blocks between anode diaphragms and cathode chambers, which resulted in better performance of the cell as a whole. The current efficiencies obtained from the cell were of the order of 65 per cent.

83.1 Electrolytic Manganese Dioxide Pilot Plant

Lead antimony anodes were used for production of manganese dioxide. The cell each time was worked at a stretch for 20-24 days. Current efficiencies of the order of 90-95 per cent were obtained. The manganese dioxide obtained on lead antimony anodes contained 0·2-0·4 per cent lead.

84.0 Pilot Plant for Hot-dip Aluminizing

Development Work

Trials were made to get design data on aluminized sheet similar to the 'spangles' on galvanized sheets. Whereas none of the conventional alloying additions for spangles, such as antimony or tin, were helpful, good results were obtained by cold-rolling aluminized sheet sandwitched between thin paper stencils bearing designs.

Wire Aluminizing

Besides routine demonstrations to interested firms on aluminizing of m.s. wire, efforts were made to work out optimum conditions for the aluminizing of high carbon, high tensile steel wires suitable for rope making for M/s. Usha Martin Black (Wire Ropes) Ltd., Ranchi. High-carbon, high-tensile steel wires get softened during aluminizing owing to a high coating temperature of 700-750°C. These have to be redrawn to restore the tensile strength. Cold-drawing results in decrease of coating thickness and loss in torsions. Thick coatings have, therefore, to be given so that the end product may have optimum thickness of coating. 14 and 16 swg wires were aluminized and sent to Usha Martin for rope-making.

A bulk supply of black-iron wire was aluminized for field trials by P & T Dept. on telephone and telegraph lines. Trials at Vishakhapatnam since 1961 have already indicated that aluminized wire has three times the life of galvanized wire under the worst saline atmosphere on a sea beach. Conductivity of aluminized wire is also superior to that of galvanized wire both initially and in service. Additional trials are likely to be taken up at four different places, viz. Jabalpur, Simla, Jaipur and Trivandrum districts.

Aluminizing of Railway Electrification Hardware

890 Kg. of assorted steel hardware samples were aluminized for the Railway Electrification Project for field trials in the newly constructed rail lines with a view to eventually replace galvanized hardware.

Bucket Aluminizing

A substantial amount of imported zinc is consumed for galvanizing of iron or mild steel buckets used mostly for domestic water storage service. As a result of foreign exchange shortage zinc quota of bucket galvanizers has been drastically reduced and many firms face closure that will lead to unemployment in cottage industry. Although plastic buckets will be ideal to substitute galvanized buckets for domestic use, their present cost is too high and production too low. As an interim aid to the bucket trade to survive, aluminizing is being suggested although aluminium coatings may not offer as good superficial protection as zinc in contact with water. Sample buckets were aluminized for Messrs Shree Krishna Pvt. Ltd., Calcutta and Tayabi Bucket Factory, Bombay.

85.0 Pilot Plant for Refractories

Forsterite Project

Studies were conducted on pilot plant scale with a view to (i) assessing the industrial worthiness of the two-stage process developed on the basis of the laboratory scale experiments, (ii) examining the possibility of adopting a more economical one-stage process and (iii) working out optimum conditions for getting uniform and good serviceable end products. The raw materials were crushed and graded and various bodies were compounded. $9 \times 4\frac{1}{2} \times 3$ in. compacts were fabricated at various pressures and processed into the forsterite bricks. Physical properties were taken by standard method. Serpentine rock was crushed into 4 lots of various grain sizes passing through 5, 10, 20 and 48 mesh B.S.S. and the following trials were undertaken:

(i) Refractory bricks were pressed at various pressures and tamped under hammer with different grain sizes of the raw materials by incorporating dead burnt, raw and light calcined magnesite. The bricks were fired to 1500° and 1600°C. with 5 hours' soaking.

(ii) In the second series, attempts were made to increase the bulk density of the product by using a blend of serpentine rock powders, passing through 10 mesh, 20 mesh and 48 mesh B.S.S. along with the additions of 25 per cent dead burnt magnesite. Ten batches were made to determine the packing density and the maximum packing density blends (from number 5 to 9) were pressed at 14,000 p.s.i. and fired at 1550°C.

(iii) In the third series, attempts were made to incorporate a blend of dead burnt and light calcined magnesite keeping the proportions of serpentine rock −10 mesh and −48 mesh B.S.S. as constant. Seven batches were pressed at 14,000 p.s.i. and fired at 1600°C.

(iv) In the fourth series, investigations were undertaken to improve the bulk density and other characteristics of the finished products by incorporating specific percentages of forsterite grog and increasing MgO content in the batch compositions.

Crushing and Grinding Characteristics of Serpentine Rock

Using jaw crusher for primary crushing and the roll crusher for secondary crushing and grinding, data indicated the preponderence of very coarse grains in the products from the 1st and 2nd passes through the roll crusher. From the third pass onwards, the cumulative curve becomes more and more 'S' shaped and the size distributions, showed maxima in the coarse and fine ranges with minima in the medium size ranges. Packing density of all the crushed products are more or less the same as expected. Also the results of chemical analysis indicated that no preferential crushing took place as the analyses of all the fractions were similar within limits of experimental error.

Bodies of First Series

These bodies were compounded to study the effects of grain size of serpentine, the nature of magnesite addition and fabrication pressures on the final 9×4½×3 in. compacts.

The bodies were all workable; those with dead burnt magnesite were easy to press. With raw magnesite additions, bodies with coarser serpentine powders were trouble-free but those with finer powders developed laminations on pressing. Light calcined magnesia developed heat during mixing and caused the bodies to loose workability on keeping. In these bodies lamination cracks became apparent only after drying and increase with increasing forming pressures. If light calcined magnesia was to be used, it was advantageous to soak it in water and allow it to age overnight before incorporating in the mix. Proportionately, light calcined magnesia bodies needed more water for forming than bodies made of dead burnt or raw magnesite.

Weight-loss amongst these compacts varied from 14 per cent in the case of bodies with dead burnt magnesite to 27 per cent in the case of those containing raw magnesite. Bodies with light magnesia lay midway with approximately 23 per cent of weight-loss. The figure was rather high and it was probable that the light calcined magnesia used, contained some undecomposed magnesite.

While the effect of variation in forming pressure was not very marked, in general, a forming pressure of 14,000 lb. per sq. inch in bodies containing coarser serpentine powders and of 10,000 lb. per sq. inch in bodies containing finer serpentine powders seemed to yield comparatively low porosity compacts. Similarly, a higher firing temperature appeared to reduce the porosity.

Compression strength of these compacts appeared to be affected considerably by the factors under study. These varied with the grain size of the serpentine powder, −5 mesh powder and −48 mesh powder yielding compacts with lowest and highest strength respectively.

In all the compacts forsterite formation was complete irrespective of the grain size of the serpentine powder used. Bodies containing dead burnt magnesites and coarse serpentine powders yielded sound compacts after firing. Compacts made of −48 mesh powders or with raw magnesite addition were defective. Bodies with light calcined magnesia formed good compacts except in cases where −48 mesh serpentine powder was used.

Bodies of Second Series

The effect of blending various grades of serpentine powders on packing density of serpentine-cum-magnesite powder mixes and on the porosity of the resultant compacts was studied in this series.

The data brought out the effects of blending various grades of serpentine powder not only on the packing density of serpentine/magnesite powder mixes but also on the porosity and bulk density of the resultant compacts. As expected, the blend having the highest packing density yielded compacts of lowest porosity.

Bodies of Third Series

In this series, the effects of blending light calcined magnesia and dead burnt magnesite were examined.

Bodies containing light calcined magnesia and dead burnt magnesite in the ratio of up to 2 : 3 were easy to mixand press into compacts. With increasing proportions of light calcined magnesia, it needed overnight soaking and the water content of the resultant mix rose more than the optimum necessary for pressing. The mix was then to be partially dried and remixed before pressing.

In these series the effect of addition of forsterite grog was investigated and an all forsterite grog composition was also studied. The data indicated that with an all forsterite grog composition, it was possible to obtain a dense brick (bulk density 2·5 gm./cc.). Incorporation of grog in bodies containing rock powders can bring down porosity appreciably provided the grain sizes were suitably matched.

The results also confirmed that on a large scale, the two-stage process originally designed for this work (i.e. preparation of forsterite grog in the first instance and remaking the grog into final compacts) yielded good compacts of satisfactory physical properties.

General Conclusions

The results given above indicated that it was possible to process serpentine into forsterite bricks by a one-stage process. Sound compacts of suitable properties can be made out of the rock provided the serpentine powders were properly graded and blends of high packing densities were used. The magnesite addition in such bodies should preferably contain certain proportions of light calcined magnesia to obtain satisfactory results. Fabrication pressures of the order of 10,000-14,000 p.s.i. appeared to be optimum for these mixes. While in these trials fired compacts did not attain bulk density above 2·41 gm./cc.,

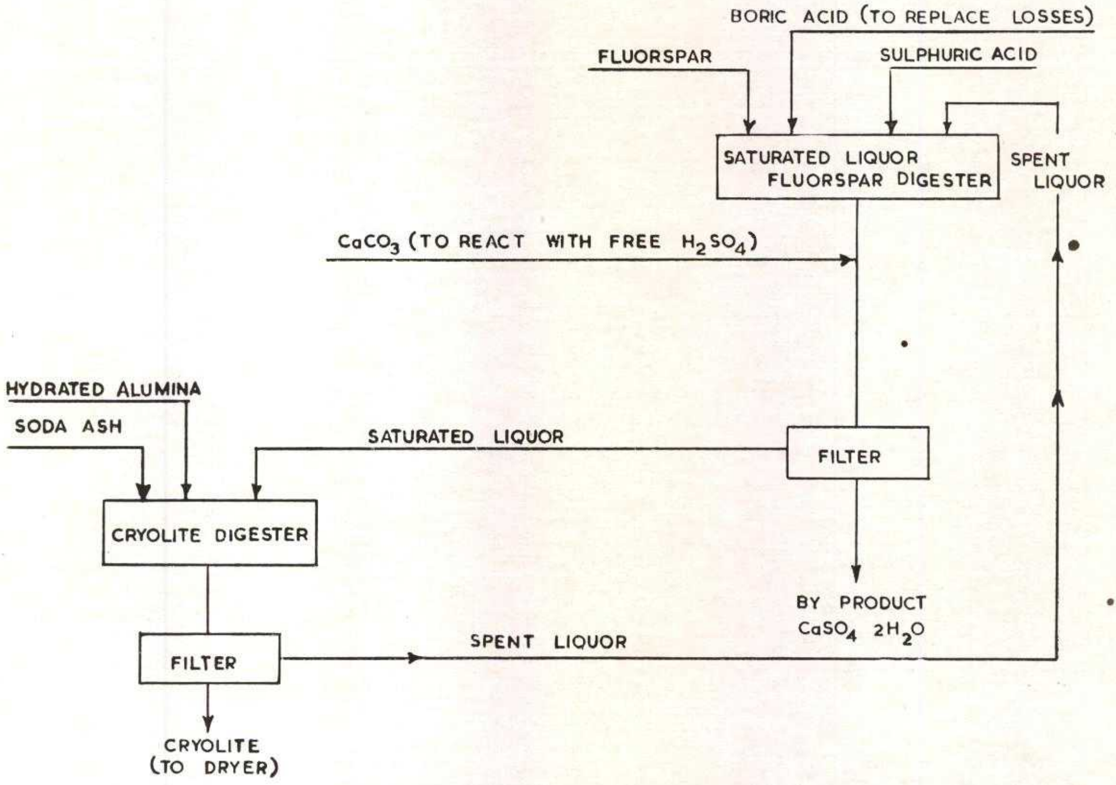

Fig. 94 — Flowsheet of Fluoboric Acid Cryolite Process

it can be stated that with better blending and judicious control of fabrication pressures better bulk density values could be obtained.

86.0 Pilot Plant for Production of Synthetic Cryolite

A Pilot Plant for the production of cryolite by fluoboric acid process is being set up in the National Metallurgical Laboratory. The capacity of the Pilot Plant is 50 kg. of cryolite per day. The building for the plant is ready with a control room, concrete platform, drawings, etc. The specifications and drawings of the equipment and machinery required for the plant were prepared in collaboration with Design & Engineering Division of the National Metallurgical Laboratory. Quotations were called for the equipment and machinery on the basis of the specifications. The structural part of the plant had been fabricated and installed so that the equipment can be installed with minimum delay.

Corrosion studies of different materials in contact with the process liquors were carried out with a view to assess their suitability for the fabrication and lining of the contact parts with the process liquors. The corrosion data obtained by the investigation were taken into consideration while selecting the equipment and machinery. The flowsheet of the process to be followed on pilot plant is given in Fig. 94.

SUMMARY OF RESEARCH THESES SUBMITTED FOR THE AWARD OF DOCTORATE DEGREE BASED ON THE WORK UNDERTAKEN AT THE NATIONAL METALLURGICAL LABORATORY

(i) *High Temperature Phase Equilibria in Some Refractory Mineral System Involving* CaO-MgO-Al_2O_3-SiO_2

Phase equilibria in the quarternary system monocalcium monoaluminate (CA) calcium dialuminate (CA_2), gehlenite and spinel were investigated both in the solid state and above the solidus. The crystalline phases CA-CA_2-gehlenite and spinel form a condensed quarternary sub-system within limiting quarternary system CaO-MgO-Al_2O_3-SiO_2. X-ray analysis of the heated mixtures of these crystalline phases did not indicate any solid solution effects. No new quarternary or ternary phases were encountered in this system either below the solidus or in equilibrium with liquids. Of the six limiting binary systems of this quarternary system, five binary systems such as CA-CA_2, CA_2-gehlenite, CA_2-spinel, gehlenite-spinel and gehlenite-CA showed true binary relationships. The system CA-spinel is not truly binary as CA_2 exists in equilibrium at the liquidus temperature in a portion of the system. The systems CA-CA_2-gehlenite and CA_2-gehlenite-spinel showed true ternary relationships with an eutectic located within the respective composition triangles. In the system CA-CA_2-spinel, the invariant point lies outside the composition triangle. It is a reaction point. The system-CA-gehlenite-spinel does not show true ternary relationships as CA_2 crystallizes as a primary phase in certain portions of the join. Two piercing points are located in this join. Two univariant curves traverse the composition tetrahedron. Along one such curve CA_2, gehlenite and spinel are in equilibrium with the liquids. Along the other univariant curve, CA, CA_2 and gehlenite are in equilibrium with liquids. The quarternary invariant point lies outside the composition tetrahedron. Its composition is not known, but the temperature of the invariant point is 1475°C.± 3°C. as indicated by the crystallization of melts in the system CA-gehlenite-spinel.

(ii) *Transformation Characteristics of Alloy Steel*

The Thesis embodies results of a systematic investigation on the effects of addition of nickel, chromium and manganese, either singly or in combination at 0·34 to 0·36 per cent C steel on the isothermal as well as continuous cooling transformation characteristics of alloy steels. A correlation between the isothermally transformed microstructures and the mechanical properties of a number of alloy steels was established. A study of the cause of the irregularities observed in the intermediate transformation range and a comparison of the mechanical properties of isothermally transformed, and quenched and tempered products were also carried out. In addition, continuous cooling transformation diagrams of a number of alloy steels as well as their detailed microstructural characteristics at different cooling rates were studied. Moreover, continuous cooling transformation diagrams in respect of some alloy steels were derived from their isothermal transformation diagrams.

The experimental techniques employed covered both the dilatometric and metallographic methods, hardness and microhardness measurements and determination of tensile strength, yield strength, reduction in area and percentage elongation. Detailed microstructural analyses were also carried out with electron microscope, using formvar replicas.

The study had established that in most of the alloy steels investigated, pearlite and bainite reactions overlap in respect of time and temperature in the intermediate transformation range between 450-550°C. In this range an acicular type of ferrite, designated as 'X' constituent by Devenport, was precipitated. The quantity of this constituent was found to be maximum in chromium steel and minimum in nickel steel. The work had further established that hardness and strength values of isothermally transformed products increase with the decrease of transformation temperature both in the bainite and pearlite range. In the intermediate range, however, each steel behaved differently. In the case of nickel steel, the hardness increased with decrease of transformation temperature; in the nickel-chromium and manganese steels, the hardness failed to increase; whereas in chromium and manganese-chromium steels the hardness decreased considerably showing irregularity in the hardness curve — the extent of irregularity being maximum in chromium steels. The cause and extent of the irregularities were investigated at higher carbon level, keeping the amount of chromium constant.

The mechanical properties of isothermally transformed structures were compared with those of tempered martensite of comparable strength and hardness. It was observed that tempered martensite exhibited consistently superior properties than the isothermally transformed structures except lower bainite. Continuous cooling transformation characteristics of a number of alloy steel were investigated. Oil cooling and air cooling curves of appropriate size bars were determined to test the reliability of equivalent relationship proposed by Nayer & Steven. It was observed that there was an overall satisfactory agreement between the two sets of curves.

Effect of sub-zero treatment of the continuously cooled specimens was also investigated and it was observed that on treatment at −120°C. and −180°C. there was an appreciable increase in hardness in chromium, manganese-chromium, and nickel-chromium-molybdenum steels at cooling rates, at which considerable amounts of acicular and proeutectoid ferrite or upper bainite had been precipitated. It was also observed that when the specimen was cooled at a rate faster than the critical cooling rate and quenched in water below the Ms temperature, the martensite formed was simultaneously tempered. The effect of alloying elements and cooling rates on such self-tempering was also investigated. C.C.T. diagrams were derived from T.T.T. diagrams on the basis of Schel's theory. It was observed that the calculated " beginning of transformation " temperatures were in general higher than those determined experimentally. Progress of transformation was also calculated at 50 and 99 per cent transformation and these were found to be generally lower than that experimentally determined.

RESEARCH GROUPS

DURING the period under review, the various Research and Development Groups maintained satisfactory progress in their specialized fields of metallurgical discipline. The scope of these Research and Development Groups is fairly wide and flexible to suit the pattern of specific objectives and requirements of the industry. These groups also prove useful in the training of graduate and trade apprentices that are recruited for a two to three years' training programme at the National Metallurgical Laboratory.

High Temperature Creep Research Group

High Temperature Creep Research Group has recently been formed at the National Metallurgical Laboratory to provide the industry with the basic data and information on the creep and rupture properties of the conventional high temperature alloy for design purposes and to promote research and development work on high temperature creep resistant alloys based on maximum effective use of indigenous alloying elements.

Considering that the resources for nickel are scarce in the country, whereas most of the super-alloys for high temperature applications are either based on nickel or contain high nickel contents as the main alloying element, it was decided to develop high temperature super-alloy compositions based on materials available from indigenous resources. With this objective in view, studies of the high temperature creep behaviour of Cr-Mn-N type austenitic steel was undertaken. Two types of alloys in this group were taken up — one with low C (below 0·1 per cent) and the other with high C (about 0·4 per cent). Solution treatment at different temperatures in the range 1050°-1200°C. was given to the creep test specimens made from material hot-forged from 4 in. square ingot size down to about $1\frac{1}{4}$ in. square size bars. Creep tests were conducted at 650°C. under 15 kg. per sq. cm. stress. The data so far obtained revealed that the solution temperature had a strong bearing on the creep resistance of these alloys. The higher temperature treatment led to severe loss of rupture ductility, which was studied on the basis of structural changes in the material. Creep test specimens were prepared from $1\frac{1}{4}$ in. sq. size bars hot-forged from 4 in. square ingots. Hot forging was done at 1180°C. Proper care was given to control the finishing temperature for forging, otherwise cracking of bars was encountered. The hot-forged bars were given solution treatment at different temperatures in the range 1050°-1200°C., by soaking at these temperatures followed by water quenching. Test specimens were machined from these heat treated bars. In a few cases where machining was difficult, hot-forged bars were first given a prolonged aging treatment at 750°C. for about 2-3 hours for ease of machining. The specimens were finally solution treated by heating in nitrogen atmosphere.

Hardness of the material in the as-solution treated condition is given below. These results show that there is progressive drop in hardness with higher temperature treatments.

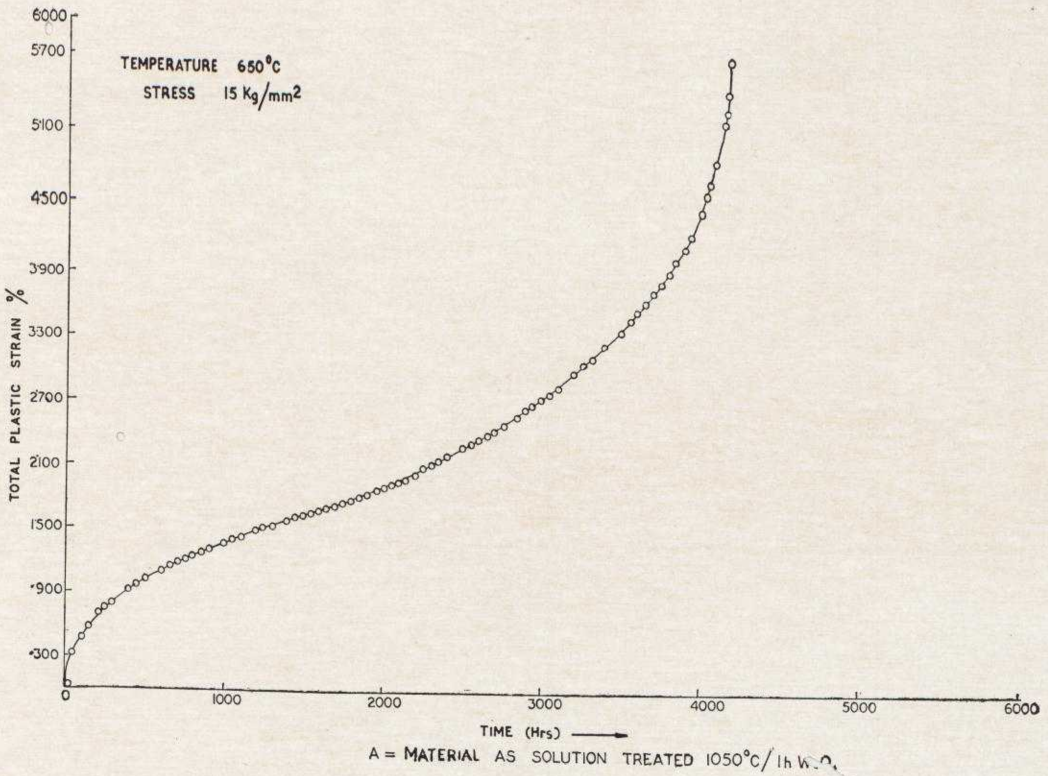

FIG. 95 — CREEP STRAIN-TIME CURVE OF Cr-Mn-N AUSTENITIC STEEL

Solution treatment temperature °C.	Vickers hardness number (HV30)
As hot forged	411
1050	320
1100	315
1150	303
1200	284

High sensitivity creep tests were carried out at 650°C. and 15 kg. per sq. mm. stress according to the corresponding British Standard Specification. In fact the control of temperature was within much narrower limits, viz. ± 15°C. Results of creep tests for the material in the 'as hot-forged' and 'as solution-treated' conditions are shown in Figs. 95 and 96. The results show that there is all round improvement on creep properties on solution treatment: creep rate is reduced and rupture life and ductility are increased. Further tests in this series are in progress.

The materials treated at higher temperature showed large grain growth and there was considerable loss of ductility in the subsequent creep tests. This question of loss of ductility is being more fully examined from structural changes in the material. The work on specimens given different aging and mechanical treatment before creep tests is under progress. This work is expected to lead to the optimum condition of heat treatment required to obtain good creep resistance and fair rupture ductility. Taking the Cr-Mn-N austenitic steel as the base composition the work was directed to evolve suitable composition which would provide in the alloy strong matrix hardened by stable precipitates of alloy carbides or nitrides and suitable intermediate phases.

As some of the creep tests last several tens of thousands of hours, every care has been taken in setting up the creep labora-

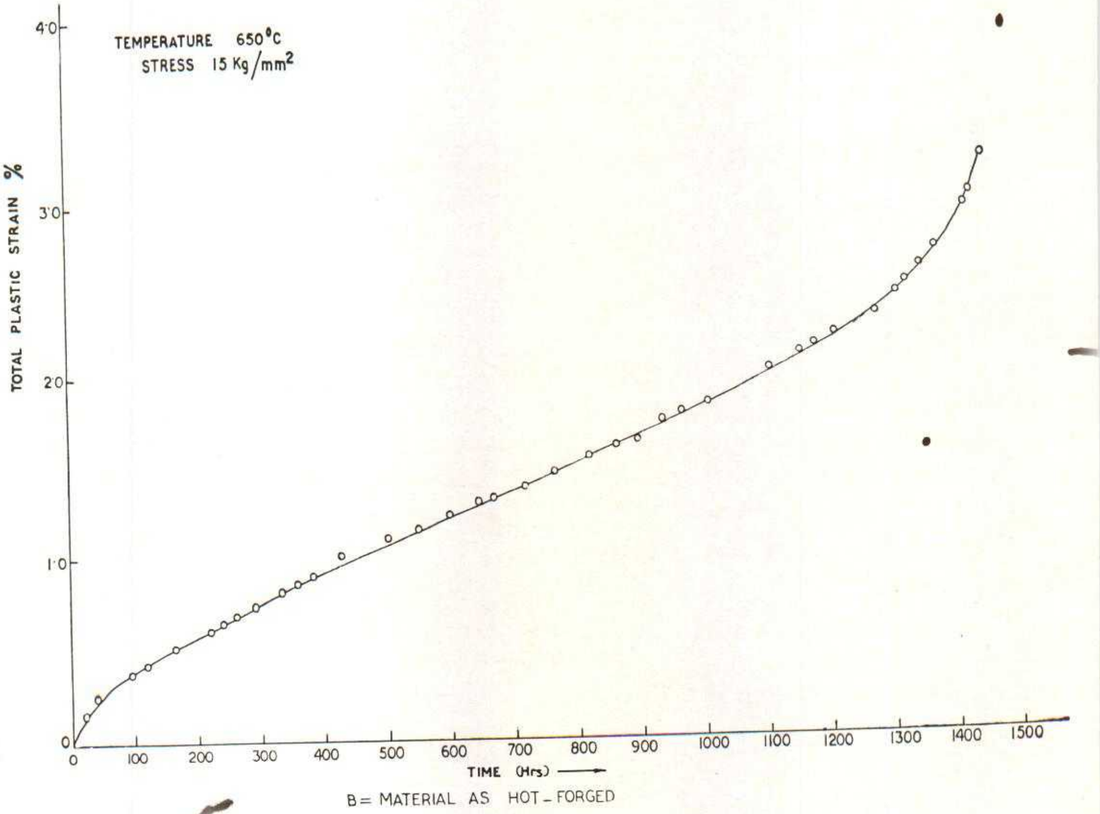

Fig. 96 — Creep Strain-Time Curve of Cr-Mn-N Austenitic Steel

tory to ensure smooth running of the high temperature creep tests according to well-established practices abroad. Loading units had been calibrated, axiality reduced to minimum and the temperature was measured and controlled thrice a day in the three zones of the specimens and kept regulated throughout the test period closer than ± 1·5°C. of the test temperature. The longest test so far conducted was over 6,000 hours. Provision of an alternative power supply, in case of main power failure is being made through an automatic diesel generating set.

Non-Metallic Inclusions Research Group

Non-metallic inclusions are known to materially affect the properties of steel. It is well established that the quality of steel is largely dependent on the amount and distribution of non-metallic inclusions. In general, inclusions are detrimental from the point of view of ductility and tendency to fatigue failure although there are obvious cases in which inclusions produce beneficial results. The non-metallic inclusions in steel are of two main-types — (i) exogenous inclusions formed by mechanical entrapment or hetrogeneous reactions with ladle refractories as their main source, and (ii) endogenous inclusions formed during cooling of the liquid steel due to homogeneous reactions — these are mainly oxides and sulphides. The quantity, size and shape of these inclusions in steel is mainly affected by the deoxidation agents and technique adopted, which in turn affects the mechanical properties of the steel. With a view to study the influence of the number, size, shape and composition of the type of inclusions present in steels, investigations were undertaken with plain carbon steel melts made in a high frequency (10 kg. capacity) furnace using various deoxidizers including: Fe-Mn, Fe-Si, Si, Fe-Ti, Al, misch metal, etc. The inclusions were extracted by normal alcoholic iodine and electrolytic extraction techniques and will be followed up by chlorination method when high carbon and alloy steels will be studied and examined metallographically and by X-ray diffraction studies. Metallographic specimens and extraction samples from each ingot were selected from three positions — top, middle and bottom — after forging the cast ingot into a 1 in. sq. rod from identical positions in each ingot. These studies were followed up by examination of tonnage heats and bigger ingots.

Electric Furnace Development Group

The object of the Group is to evolve suitable refractory cement compositions for use in electric furnaces and also for making suitable designs of furnaces for specific research projects.

The three cement compositions, namely clay-kyanite, phosphoric-acid-kyanite and an aluminous composition with calcium-aluminate developed earlier, were freely used in fabrication of all laboratory furnaces. None of the compositions reacted with kanthal or nickel-chromium resistance wires. The technique of making internally wound heating sheaths developed earlier was utilized for making tubular furnaces required for laboratory work. Platinum wound furnaces were fabricated by the Electrical Section using the technique developed earlier.

Radio Isotope Research Group

A Radio Isotope Research Laboratory has been started in the National Metallurgical Laboratory with a view to use radio-active isotopes as tools in metallurgical research problems such as grain boundary and surface segregation of metals in alloy systems, friction, lubrication and wear studies, zone melting of metals, corrosion of metals, electro-deposition, mineral ore dressing, radio-activation analysis for determination of the minute quantity of elements (10^{-8} to 10^{-12} gm.), etc. Radio-active isotopes will also be used for industrial studies like study of processes in blast furnaces and ancillary metallurgical equipment, studies on the refining and casting of steel, measurement of wear of blast furnace linings, production control using thickness gauges, etc. For radio-tracer studies in the Isotope Laboratory, necessary specimens would be irradiated in the atomic reactor piles available with the Atomic Energy Establishment, Trombay. With these objectives in view, the setting up of the Isotope Laboratory made considerable progress. Following electronic instruments, accessories and materials were set up for the Radio Isotope Laboratory.

(i) *Electronic Instruments*

1. Gamma Ray Spectrometer
   (Type GRS 20 B)
2. Geiger Counting Set-up
   (Type GCS 10A)
3. Utility Scaler
   (Type SS 361A)

(ii) *Accessories*

1. Remote Pipetting Device
2. Aluminium Absorbers set
   (Type A. 1750).
3. Stainless Steel Planchets
   (Type P 1850)
4. Set of Gamma Reference Sources
   (Type R2300B)
5. Set of Beta Reference Sources
   (Type R2100)

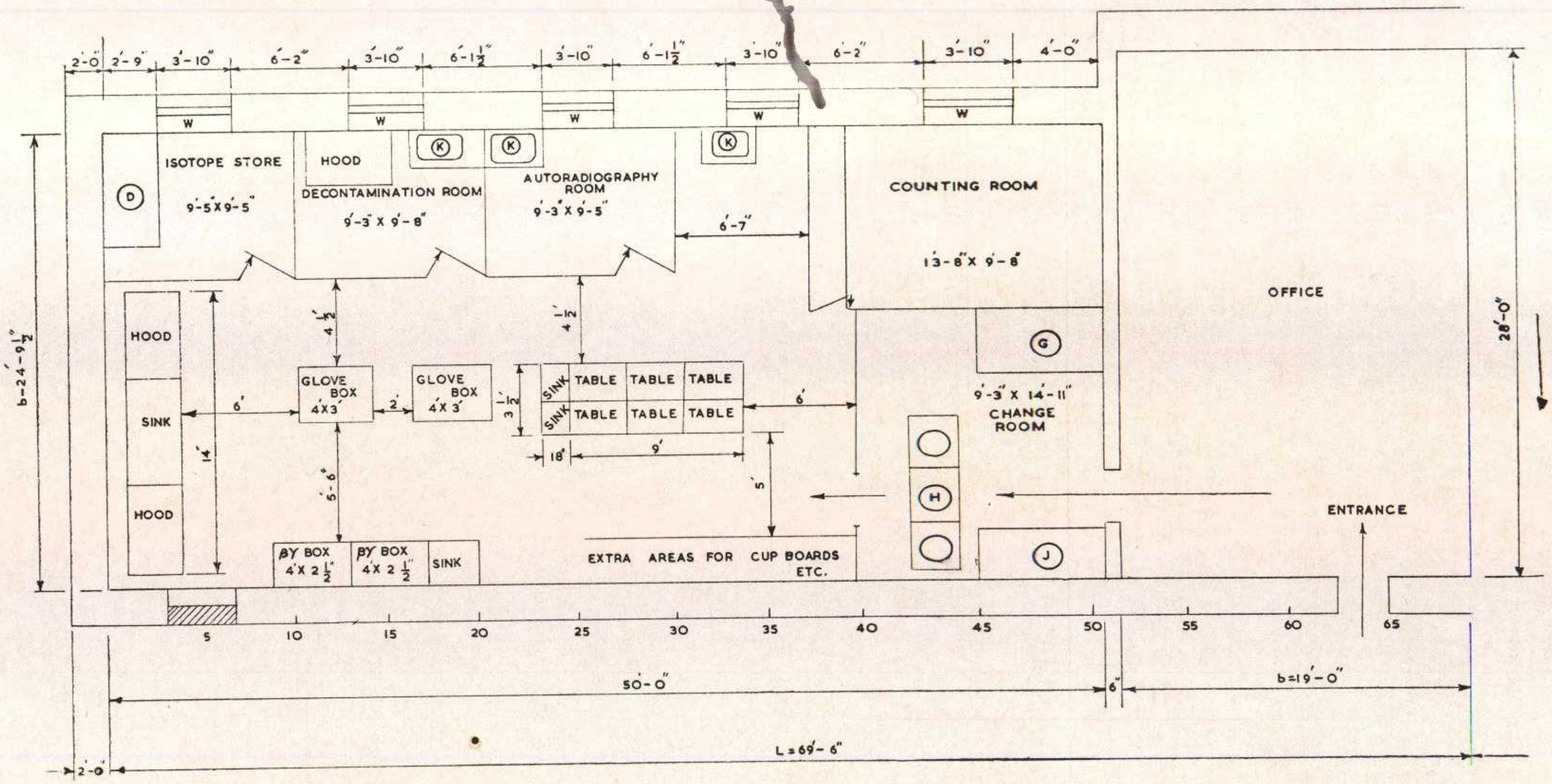

Fig. 97 — Layout of Isotope Laboratory

(iii) *Materials*

Lead bricks
- Type A
- Type B
- Type C
- Type D

In connection with the setting up of the Isotope Laboratory, the following programme of work is in progress

1. The installation of the proper ventilation plant in the Isotope Laboratory.
2. The installation of air conditioning unit for the conditioned air required for the non-active area and the counting room.
3. The fabrication of the necessary glove boxes, beta-gamma boxes and fume hoods required for the tracer studies in metallurgical research problems, in the NML workshop.
4. The painting of the walls of the Isotope Laboratory with high finish plastic emulsion paint.
5. The laying out of linoleum sheet on the floor of the active area of the Isotope Laboratory.
6. Making necessary arrangements for the disposal of the high level active liquid and solid wastes, low level active liquid and solid wastes and low level active wastes which would result from the tracer studies, with the Atomic Energy Establishment, Trombay.

The layout of the NML Isotope Laboratory with the actual location of the fume hoods, beta-gamma boxes and glove boxes is given in Fig. 97.

Standard Metals Development Group

Standard samples are used in industrial and research organizations for checking of methods of determinations of constituents and control of analysis. The requirement of standard samples has been so far met by import. With the stepping up of metallurgical industries the demand for standard samples of indigenous raw materials and products is increasingly being felt. In order to meet this demand effectively, a project has been undertaken, for the preparation and supply of the standard samples from indigenous raw materials.

For the preparation of standard samples avoiding the possibilities of any contamination a separate unit has been set up, the layout of which is given in Fig. 100. The samples will be machined, reduced to suitable size in a lathe and screened in the milling room. After crushing the bulk sample to proper size and screening, the sample is mixed in a mixer to ensure homogeneity, sampled and bottled in the sampling room.

During the period under review, the following quantity of standard samples were produced. In the first instance production of standard cast iron and plain carbon steel was taken up.

For the production of cast iron standard sample, Bhilai pig iron was melted in electric furnace and cast. The millings suitable for cast iron were obtained in a lathe machine after removing one-fourth of an inch from the surface of the bar. The sieve fraction of the millings obtained were as follows:

	%
−10 + 18	54·8
−18 + 25	21·1
−25 + 44	9·4
−44 + 72	6·5
−72 + 100	2·3
−100	5·9

The sieve fraction (−10 + 18) was sampled and analysed in N.M.L., National Test House, Calcutta, and Directorate of Metallurgical Inspectorate, Tatanagar. The analysis report given in following page shows the result obtained after careful verification and checking.

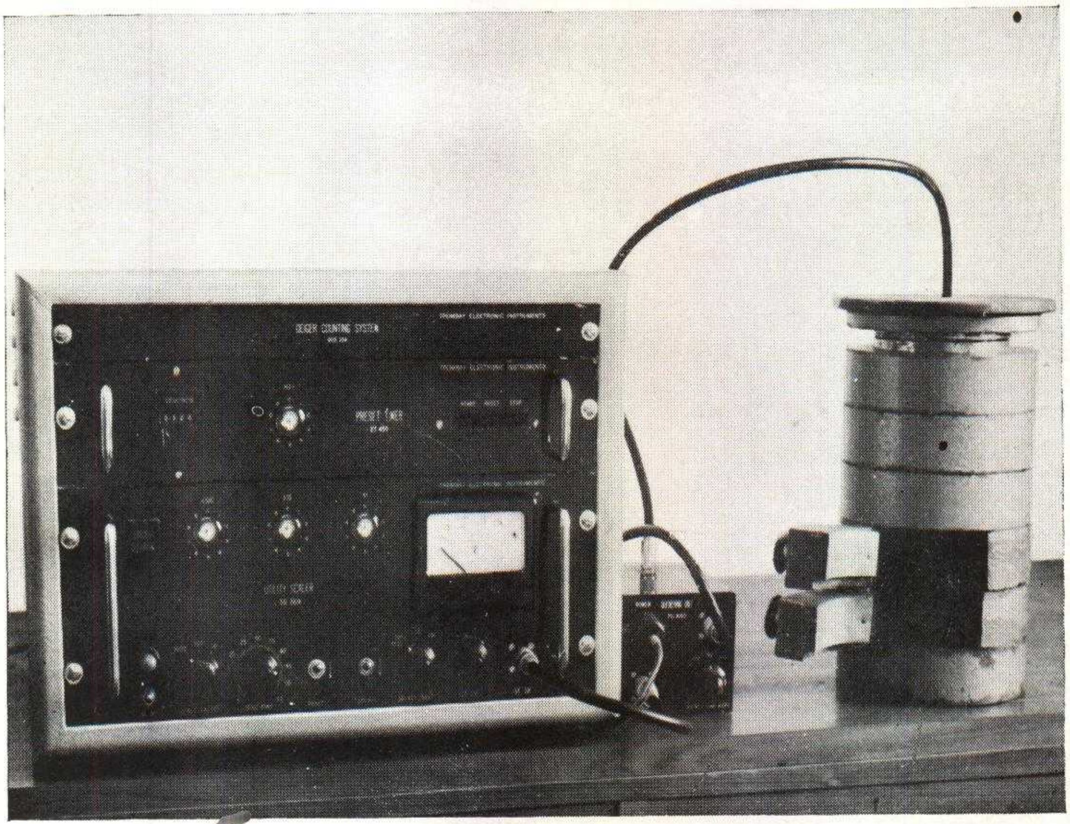

Fig. 98 — Geiger Counting Set-up

%	National Test House	Govt. Metallurgical Inspectorate	N.M.L.
Total carbon	3·66	3·71	3·73
Graphite carbon	2·99	2·95 (by diff.)	2·98 (by diff.)
Combined carbon	0·67 (by diff.)	0·76	0·75
Silicon	0·612	0·60	0·61
Manganese	0·501	0·50	0·50
Sulphur	0·043	0·045	0·042
Phosphorus	0·204	0·265	0·23

Standard brass sample was prepared from a brass piece. It was found that during the milling operation considerable grease was contaminated with the millings. Whole sample was treated with ether in a soxhlet to remove the grease. The result of analysis of the sample obtained in the laboratory is as follows:

	%
Cu	57·04
Zn	39·98
Pb	2·33
Fe	0·10

The sample is being sent to other laboratories for analysis. Mild steel (0·2 per cent C) standard sample was prepared. The suitable millings were obtained from the bar in a lathe after removing one-fourth of an inch all round the surface. The sieve fraction (—32 and + 10) was rejected. The fraction (—10 + 18) was crushed to suitable size, sampled and analysed in the laboratory, and also sent to the other laboratories for analysis which is given below:

%	Govt. Metallurgical Inspectorate	National Test House	R.V. Briggs.	N.M.L.
Carbon	0·20	0·216	0·15	0·21
Manganese	0·57	0·593	0·61	0·59
Sulphur	0·039	0·035	0·03	0·035
Phosphorus	0·024	0·025	0·022	0·025
Silicon	0·083	0·101	0·06	0·085

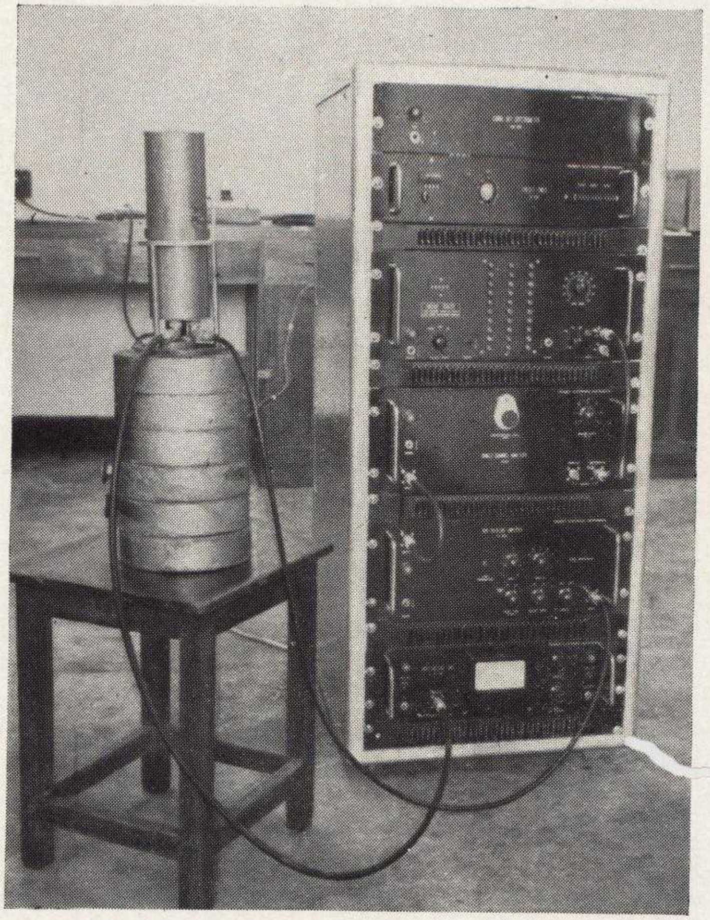

FIG. 99 — GAMMA RAY SPECTROMETER

Metallic Surface Coating Development Group

Co-diffusion of Chromium in Steel for Heat Resistant Coatings

Resistance to high temperature oxidation can be conferred on steel both by chromium and aluminium coatings. Aluminized steel is resistant up to about 800°C. Chromized steels are also useful up to about 900°C., but oxidation is extensive at 1000°C. and above. The possibility of developing coatings which can withstand high temperatures represents an interesting field of research.

Co-diffusion of chromium and aluminium, therefore, suggested itself as one line of approach. The complex oxide films that would result from Cr_2O_3 and Al_2O_3 can be assessed for their protective value to arrive at a suitable coating for use above 1000°C. Only the aluminium-rich end of Al-Cr phase diagram is known. At 800 to 900°C. (temperatures at which coating from liquid-bath could be done by hot-dipping) only 4-6 per cent Cr is in solution. This precipitates on cooling as $Cr\,Al_7$ (Beta phase) along the liquidus. The maximum solid solubility of aluminium in chromium is given as about 29 per cent in the range 975-1350°C.

A master-alloy (Cr 76·4, Al 19·9, Fe 1·2 per cent) was initially prepared by aluminothermic reduction of chromite. An attempt was made to prepare a saturated solution

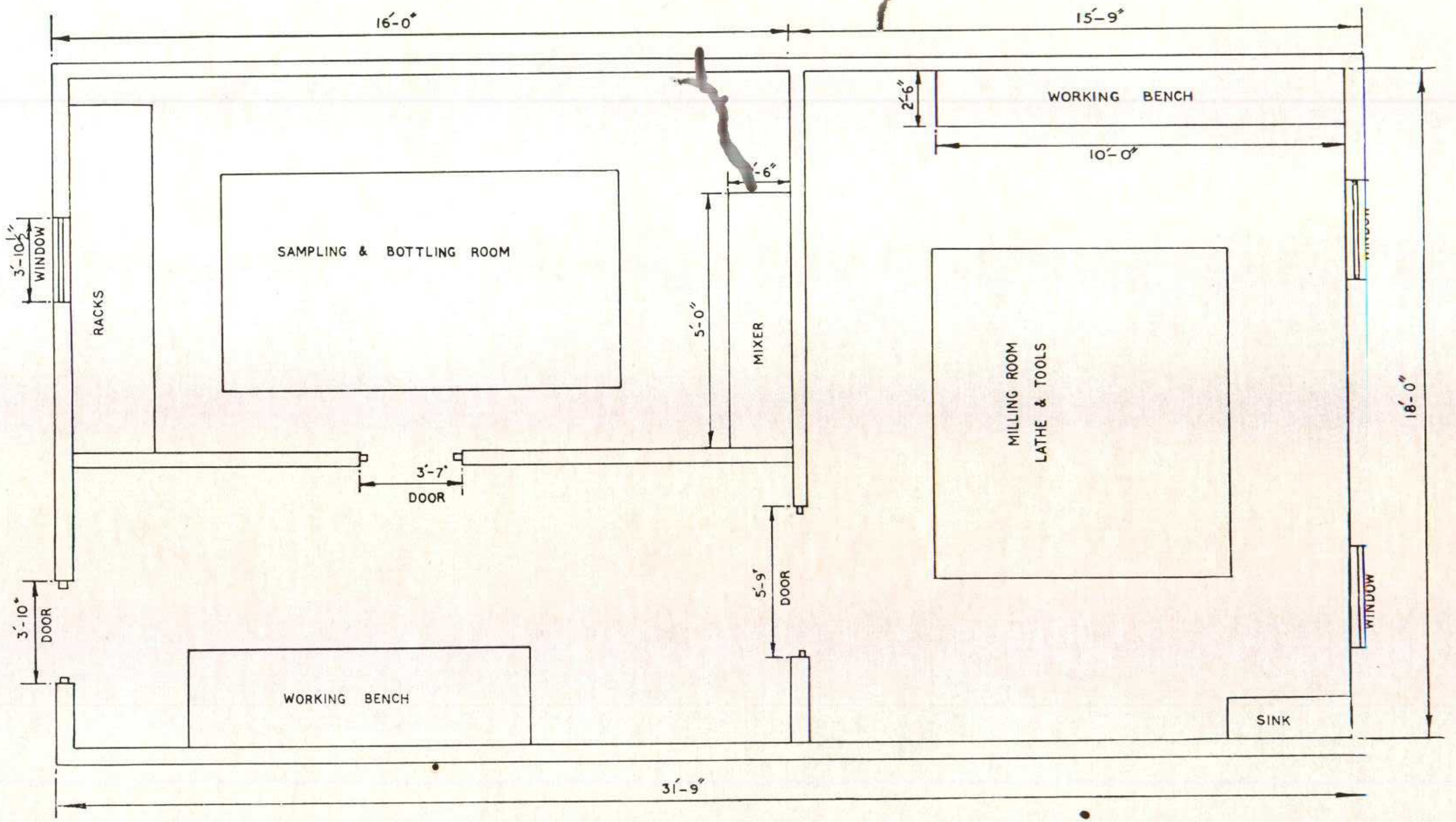

FIG. 100 — LAYOUT OF THE STANDARD SAMPLE LABORATORY

of chromium in aluminium at 800°C. for hot-dip coating. Lumps of the above alloy were kept in molten aluminium and soaked for six hours in an electric resistance furnace at the end of which the bath and the residue analysed as follows:

%	At 800°C.		At 1400°C.	
	Bath	Residue	Bath	Residue
Cr.	0·51	74·8	6·09	43·23
Al	98·15	21·8	88·41	30·2
Fe	1·34	0·8	5·46	21·56

To improve the chromium pick-up by aluminium, the system was soaked at 1400°C. for two hours in a high frequency furnace. The chromium pick-up in the above two experiments not being enough, the hot-dip technique was ruled out as impractical.

Plastic Coatings on Metals

This project envisages several end objectives and end products. Work is being carried out in the first instance on metal-PVC laminates for corrosion protection of steel sheets. The steel sheets are bonded with PVC films of different thickness and the laminates are tested for chemical resistance, heat resistance and corrosion resistance. The bond strength of different adhesives for making metal-PVC laminates is tested by stripping the plastic film from the metal under standard conditions.

Work so far carried out showed that the laminates are quite resistant towards various chemical reagents, viz. N/100 HCl, H_2SO_4, HNO_3, 30 per cent citric acid, acetic acid, sodium hydroxide, sodium carbonate, ammonia, etc. The laminates could be deformed without loss of adhesion in cupping tests. Atmospheric and accelerated corrosion tests are in progress. Work is underway on making plastisols with PVC resins available in the country. An experimental fluidized bed was set up to study the fluidization characteristics of the PVC powders and to evaluate the various factors such as bed density, rate of flow of air, particle size, etc., with a view to design a bigger fluidization tank for dip-coating experiments.

Pack Chromizing

Resistance to high temperature oxidation can be conferred on steel-bath by chromium and aluminium coatings. Aluminized steel is resistant up to about 800°C. Chromized steels are also useful up to about 900°C., but oxidation is extensive at 1000°C. and above. The possibility of developing coatings which can withstand higher temperatures is, therefore, an interesting field of research.

Co-diffusion of chromium and aluminium, therefore, suggests itself as one line of enquiry. The complex oxide film that would result in service from Cr_2O_3 and Al_2O_3 and the lower oxides can be assessed for their protective value to arrive at a suitable coating for use above 1000°C. A master alloy of chromium and aluminium was prepared by alumino-thermic reduction of chromite. It can be easily kept packed in this alloy powder at 800°C. added to the pack to facilitate chromium diffusion through Cr Br_3. Grey-matt oxidation resistant coatings were obtained.

Slag Research Group

Utilization of Basic Slag as Soil Conditioner

Work on utilization of basic slag as soil conditioner was taken up with the objective of determining its suitability as a cheap liming material for agricultural purposes. Basic slag was found to give encouraging results being a source of micro-nutrients such as boron, manganese, molybdenum, copper, zinc, cobalt, tungsten, titanium, etc.

besides P_2O_5. Preliminary field experiments were conducted in collaboration with Directorate of Agriculture, Bihar; with blast furnace and steel plant slags having the following chemical analysis:

%	Rourkela L.D.	Rourkela O.H.	Bhilai O.H.	Bhilai B.F.	NML L.S.F.
CaO	37·17	37·2	29·65	36·96	33·26
SiO_2	18·82	18·2	19·46	31·31	39·82
Al_2O_3	3·64	14·42	13·7	10·45	19·9
P	0·85	0·90	1·63	0·014	0·027
S	0·018	Trace	Trace	0·39	0·575
Mn	9·80	4·48	1·73	0·39	0·75

The slags were applied during last *Rabi* season to 35 microplots, having each an area of 1/400 acre, to study the comparative liming effect with standard liming material, such as lime and dolomite. Each variety of slag and standard liming material crushed to −100 mesh were applied to 5 microplots selected at different directions of the field. The different varieties of slags including lime and dolomite which were calculated on equal lime basis after measuring the *p*H value of the soil, were added to the respective microplots. Similarly, other treatments of the soil such as nitrogen treatment, etc., were done equally to all the microplots for comparative study. Microplots treated with the slags and standard liming material were sown with wheat crops and the following results were obtained.

Treatment	Yield of grain kg/hect.
Control (lime)	1880
Dolomite	1774
Bhilai, blast furnace	2209
Bhilai, open hearth	1938
Rourkela, L.D.	2278
Rourkela, open hearth	2400
NML, L.S.F.P.	1734

The data obtained in the first field trial showed that all the slags were better liming material than standard liming material. Rourkela open hearth and L-D slag were found better than other slags. Since the results of successive three years' field trials on the same crop would be co-related for confirming liming effect of the slag, further field trials are under progress. Similar field trial was initiated with sowing of groundnut and maize in the last *Kharif* season and the results of the field trial is awaited. Work is also underway to determine mechanical and chemical processes to crush the slag at minimum cost. In carrying out this work, it is essential to know the optimum size of the slag to be crushed for efficient use of slag as liming material. As such, Rourkela open hearth slag which had already given better results in the first field trial, was crushed to various mesh sizes and is being applied to the soils in different plots under identical conditions to determine the suitability of the optimum size of slag by actual field trials.

Utilization of Blast-furnace Slag as Rail-road Ballast

This investigation is also a part of broad-based research and development programme on utilization of blast furnace slag as rail-road ballast which was initiated at the instance of Ministry of Railways. In order to conduct preliminary study on the possibility of utilization of blast-furnace slag as rail-road ballast, representative samples of air-cooled slag from the Tisco blast furnace were collected. The slag was crushed to $-2+1\frac{1}{2}$ in., $-1\frac{1}{2}+1$ in., $-1+\frac{3}{4}$ in. and $-\frac{3}{4}+\frac{1}{2}$ in. sizes in jaw crusher and roll crushers for undertaking the abrasion test, crushing test, unit weight, specific gravity, soundness test, etc., as per the ASTM Specification for rail-road ballast. As the specification called for abrasion test to be carried out in Los Angeles Machine was not yet available at the National Metallurgical Laboratory, arrangements were made to conduct this particular test at the Research, Designs and Standards Organization, Lucknow. The results obtained on tests so far conducted, i.e. crushing test, unit weight, specific gravity, soundness test, etc., are very encouraging. It is expected that the air-cooled blast-furnace slag would give equally good results for the wear and abrasion test which are going to be conducted very shortly.

ENGINEERING SECTIONS

Design and Mechanical Engineering

Some of the major design and fabrication projects completed and in progress for different pilot plants, research and development projects are given below:

(i) *Synthetic Cryolite Pilot Plant*

A fully integrated continuous processing unit with a rated capacity of 50 kg. per day was designed for the production of synthetic cryolite from Indian fluorspar. Specifications were drawn up for individual items of equipment and procurement is underway.

(ii) *Ausforming Unit*

Detailed design work was taken up for a Wire Ausforming line involving wire uncoilers, preheating lead-bath, immersed wire drawing and motorized winders. The unit will be engineered for installation during the coming year. Fig. 101 gives a layout of the Wire Ausforming Unit.

(iii) *Plant for the Recovery of Zinc from Dross*

An engineering Project Report was prepared inclusive of layouts, equipment drawings, and cost estimates. The rated capacity of the plant is 120 tonnes per month which is expected to process 100 tonnes per month of zinc blowings and small quantities of ash. Capacity can be further increased to handle upto 80 tonnes per month of zinc ash and 60 tonnes per month of zinc skimmings. The recovery of zinc will be up to 95 per cent. The total capital costs have been estimated at about Rs. $2\frac{1}{2}$ lakhs. Fig. 102 gives the plan of the schematic layout of the plant.

(iv) *Erection of Hot-blast Cupola and Recuperator*

Work was completed on the fabrication of piping connections, stack, damper valves, expansion joint and other components. Installation of the entire unit was completed. A novel 90° vertical motorized skip hoist was designed for charging the raw materials in the hot-blast cupola.

(v) *A Tube Aluminizing Pilot Plant*

Work on design for a pilot plant incorporating magnetized transfer rolls for the semi-continuous aluminizing of steel tubes was started.

(vi) *Strip Aluminizing Plant*

The plant design which was completed earlier was finalized, bills of materials were drawn up and fabrication of equipment was initiated. The plant will be commissioned during next year.

(vii) *High Temperature Creep Apparatus*

Refractory materials used as structural components in high temperature industrial furnaces are subjected to severe external and internal stresses during service. As such their high temperature mechanical properties, particularly their ability to withstand loads at high temperatures, are

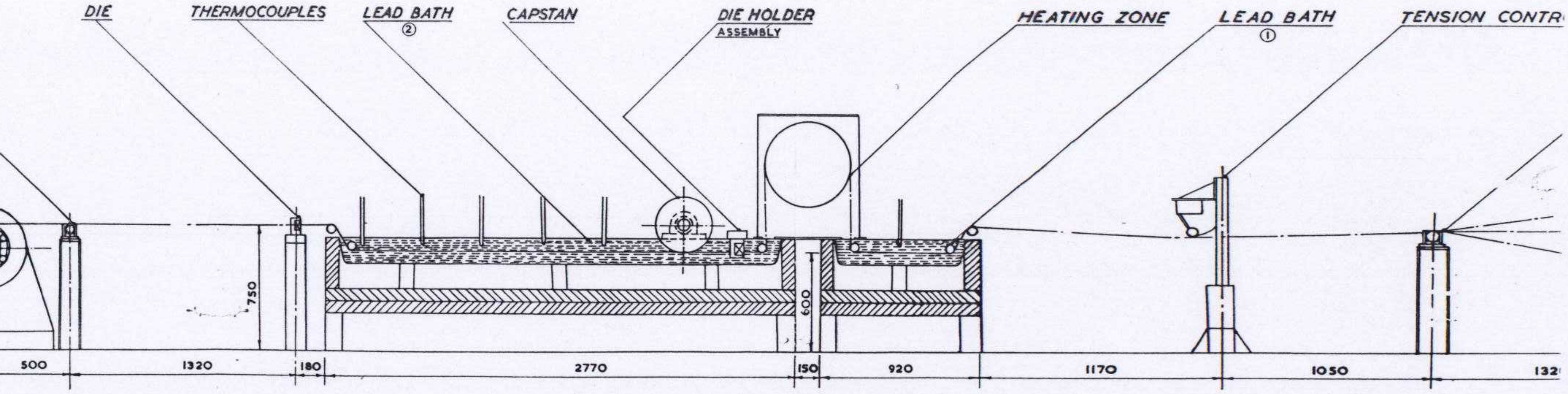

Fig. 101 — Layout of the Wire Ausforming Unit

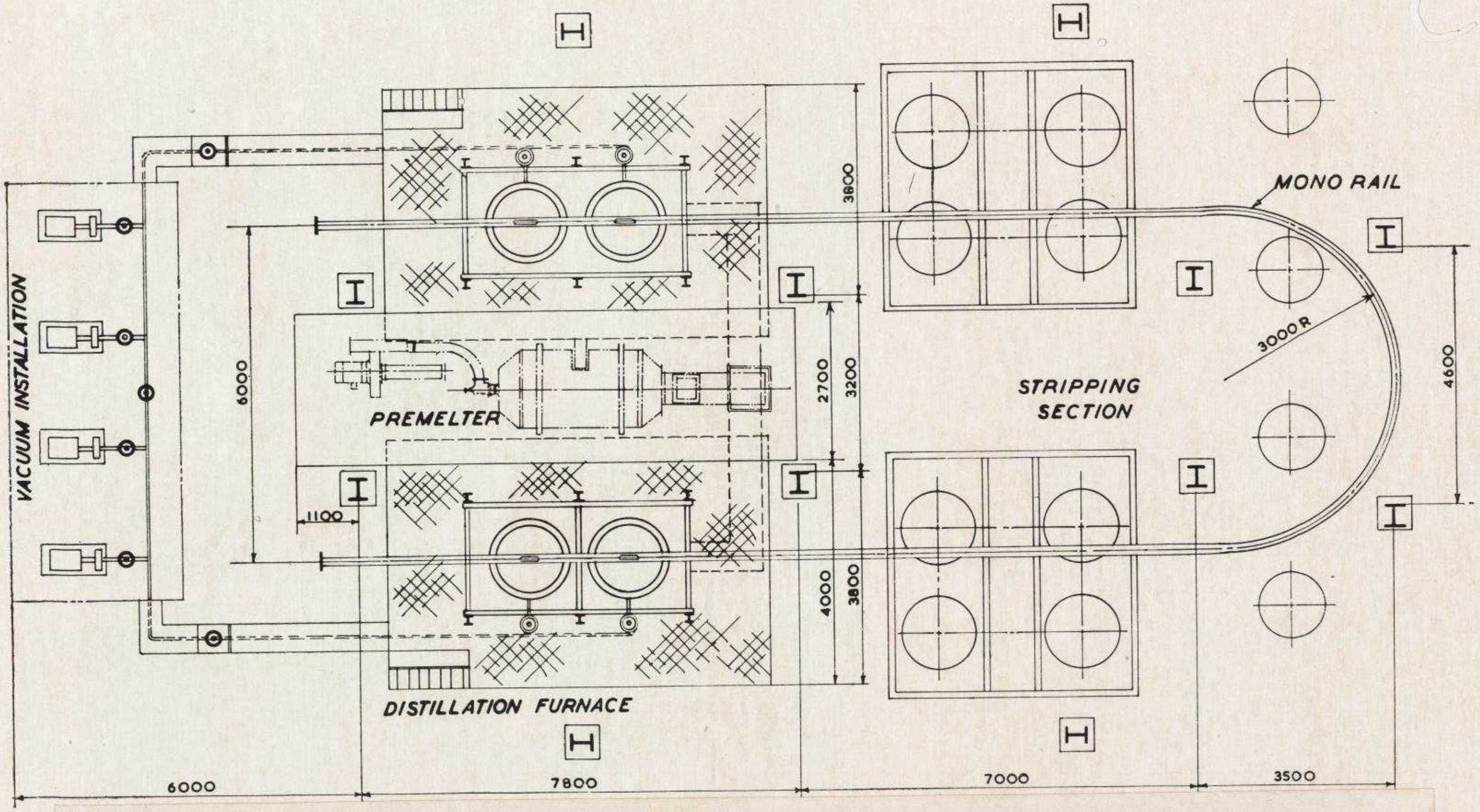

Fig. 102 — Schematic Layout of Vacuum Distillation Unit for Recovery of Zinc from Galvanizing Wastes

important factors in determining their choice and applications. An understanding of the mechanical properties and behaviour of refractories at high temperatures is highly important and work thereon has led to the development of improved refractories, such as direct bonded basic brick successfully used in open hearth furnace roofs. There is comparatively little work done in India on this subject. Keeping in view the importance of the subject, work was taken up at the National Metallurgical Laboratory on correlation of high temperature mechanical properties of some basic refractories with their constitution and structure. An apparatus for study of creep in refractories under torsion was designed and fabricated at the National Metallurgical Laboratory (Fig. 103). Studies on the subject employing this apparatus, possibly the first of its kind in this country, will throw useful light on the nature and behaviour of Indian refractory materials at high service temperatures.

(viii) *Cost Data Correlations*

A project was initiated for the collection of comprehensive cost data for indigenous items of process equipment, ancillaries and plant hardware. Data procured was correlated in terms of capacity versus cost for over forty items for purposes of compiling quick project report cost estimates based upon actual prices.

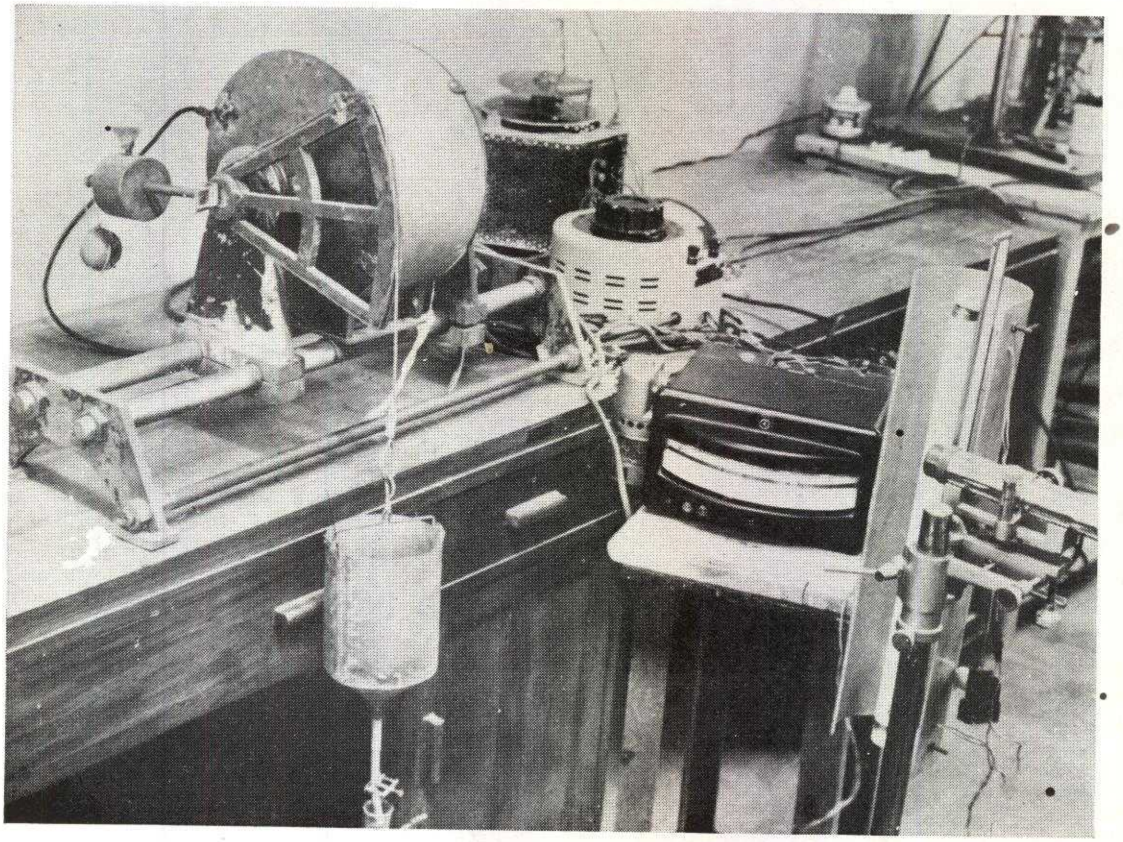

Fig. 103 — Apparatus for Study of Creep on Refractories Under Torsion, Designed and Fabricated at the National Metallurgical Laboratory

Electronics Engineering

(i) *Electronic Potentiostat*

An Electronic Potentiostat for corrosion studies under controlled potential/current conditions with special reference to electrode reactions was developed, designed and fabricated. A thermionic d.c. amplifier gets input from the potential difference between standard electrode and test electrode in an electrolytic cell. The output current of the amplifier flows between the test electrode and a third electrode of platinum. Any change in potential of the test electrode alters the output current to reduce the charge, the system forming a closed loop proportional controller. An output of 0·6 mA. can be supplied with less than 100 mV. variation of electrode potential. In studying the relationship between electrode potential and corrosion rate, either current or potential can be kept constant. For this purpose this potentiostat is very useful. A transistorized version has also been planned.

(ii) *R-C Oscillator*

A simple R-C Oscillator was designed and fabricated for use with an a.c. bridge circuit for corrosion study. Its frequency stability has been found satisfactory. A transistorized version of the oscillator was under development.

(iii) *Voltage Regulator*

A stabilized d.c. negative voltage regulator was designed and is under fabrication. It is a simple stabilizer giving high voltage supply for high-gain differential input. It uses a special type of regulator tube StR 280/80 to provide an initially stabilized input to two other regulator tubes $85A_1$ and $85A_2$. It permits a voltage of approximately 140 volts supply to each of $85A_1$ and $85A_2$ tubes so that 55 volts is available across the series regulator. The output current is almost 8·5 mA. and well stabilized.

Compensatory circuits using thermistors and N.T.C. resistors were designed and incorporated in Photo-transistor Temperature Controllers developed and fabricated at the Laboratory. These compensatory circuits will improve the performance of the photo-transistor temperature controllers for higher ambient temperatures (above 30°C.).

(iv) *Proportional Temperature Controller*

Proportional temperature controller using an adjustable electronic timer was developed, designed and fabricated. Its performance test was undertaken. A synchronous motor-driven microswitch actuates an electronic time delay vacuum switch DLS 10.

Several major maintenance and installation work was carried out during the year under review. Preventive maintenance schedules of all instruments were prepared and necessary spares obtained for some instruments. Complete instrumentation of Mineral Beneficiation Pilot Plant was undertaken which included six flow recorders, two temperature recorders, industrial *p*H recorder, oxygen analyser and gas chromatographs.

Electrical Engineering

A number of development work and installation of melting units was undertaken, the salient features of which are furnished below.

(i) *Design and Fabrication of High Temperature Furnaces*

A graphitization furnace to attain the temperature up to 3000°C. was designed and fabricated (Fig. 104). The furnace was provided with graphite resister and water-

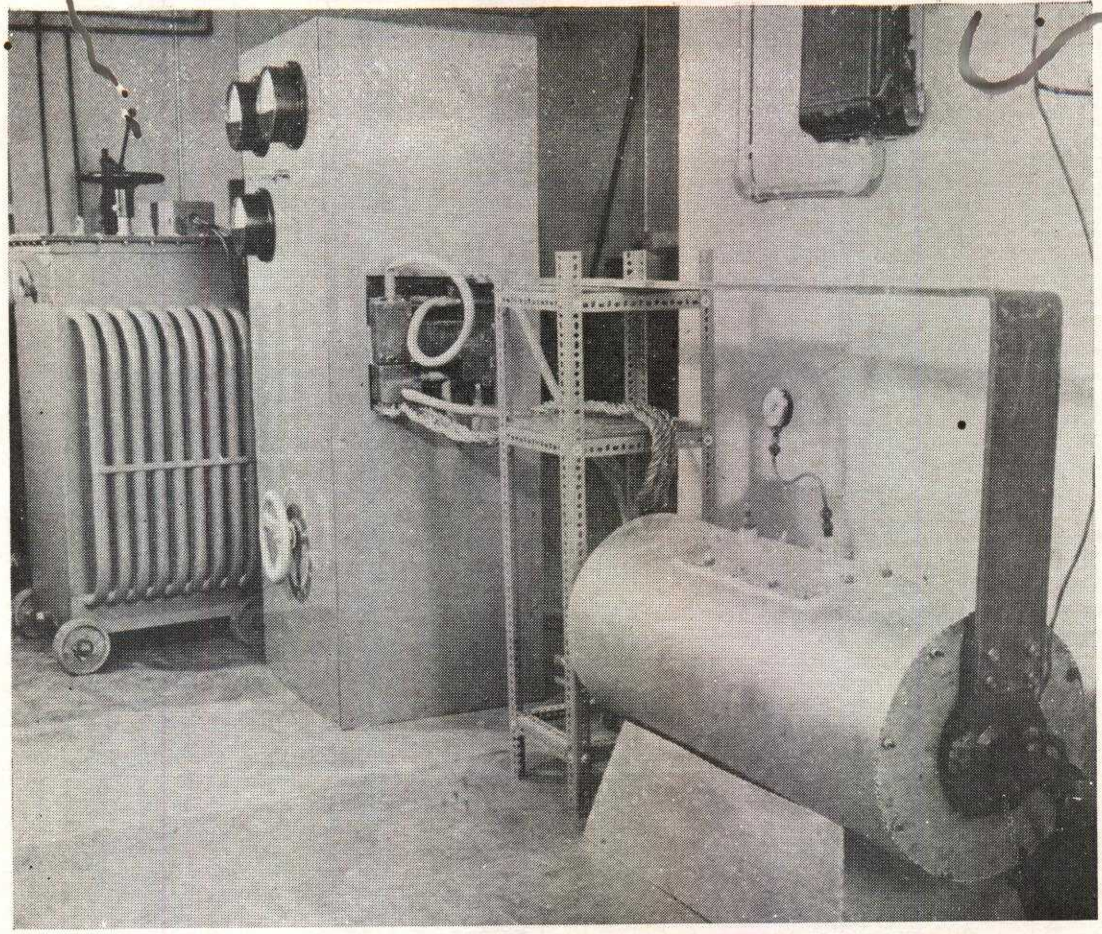

FIG. 104 — GRAPHITE RESISTANCE FURNACE, DESIGNED AND FABRICATED AT THE NATIONAL METALLURGICAL LABORATORY

cooled copper electrodes which would supply high current at low voltage to the furnace. The construction is such that the accessibility of the element is easy for replacement. The temperature of the furnace could be rapidly raised up to 3000°C. and a uniform hot zone of about 60 per cent could be obtained.

Another Platinum — 13 per cent Rhodium wire wound tube furnace having 88 mm. internal diameter was designed with auxiliary winding of kanthal wire. The auxiliary-winding had reduced the cost of the furnace considerably and it had also helped to give a wider constant temperature zone at 1400°C.

(ii) *Installation of High Frequency Vacuum Melting Equipment*

One motor generator type high frequency vacuum melting furnace was installed and commissioned. The generator having the capacity of 50 kW. at 3600 cycles/sec. was vertically coupled with a squirrel cage motor of 66 kW. capacity. The unit was provided with adequate interlocks and overload devices. A valve-generator type high frequency furnace having the capacity of 300 kVA. output at 200 V. and 5 KC/sec. nominal frequency was also designed and fabricated.

(iii) *Extension of Power Supply*

Since the maximum demand of power in NML had increased due to the installation of several high power consuming units, the existing current transformers of the main circuit breaker (6600 V. main) needed replacement with higher capacity current transformers. New current transformers to suit the present requirements as well as to provide for some future increase in maximum demand were installed and commissioned.

New Foundry Bay was equipped with appropriate power distribution panels and luminairs. One 50 kVA., 400 V. direct arc furnace, one 17 kVA. motor and their controlling devices were installed. The illumination at the level of 20 lumens per square foot was designed and fabricated in the bay.

(iv) *Power Scheme for Magnesium Pilot Plant*

Power Supply Schemes for Magnesium Pilot Plant was studied and specification for one 1000 kVA. transformer 6600/415 V., for this plant was prepared.

Besides the above, some major breakdown repairs and planned preventive measures were carried out.

Civil Section

The following major civil works were completed during the period under review:

(i) Installation of foundry equipment in Foundry Bay.
(ii) Construction of metal-cum-refractory recuperator for Refractory Pilot Plant.
(iii) Steel structural erection work for Cryolite Plant.
(iv) Modifications for the installation of the Isotope Laboratory.
(v) Installation of gas producing unit and construction for its building.
(vi) Installation of klin and discharge conveyor.
vii) Installation jobs for the NML Foundry Station at Howrah.
(viii) Construction of gas producing unit and fuel store at Marine Corrosion Research Station at Digha.
(ix) Installation of vacuum and electrical furnaces.
(x) Construction work for Strip Aluminizing Plant is in progress.
(xi) Construction of staff quarters (multistoreyed at Agrico Area — 30 E type quarters and 10 D-II type quarters are under construction).
(xii) Extension of first aid room behind the guest house in NML.
(xiii) Extension of workshop for sheet metal job.
(xiv) Construction of crushing shed at L.S.F.P. area.
(xv) Construction of first floor over transit stores of main building.
(xvi) Construction of first floor store over the back-side store.
(xvii) Construction of motor garage at eastern wing of NML main building.
(xviii) Construction of room for instrumentation of MBPP.
(xix) Fabrication and erection of gantry beam and rails for overhead cranes.

Important Scientific Equipment Designed and Fabricated at the National Metallurgical Laboratory During the Period Under Review

1. Sigma mixer — a jacketed sigma blade mixer for hot refractory mixes.
2. Skip hoist — a motorized skip type of hoist for lifting raw materials to the charging door of the NML Hot-blast Cupola.
3. Centrifuge — a centrifuge for the processing of hot-dipped aluminized components.

4. Vacuum system — for a full scale plant unit.
5. Down Draft Kiln Recuperator — The entire piping system for the hot air lines was fabricated and installed and the recuperator was readied for commissioning.
6. Strip Aluminizing Plant — fabrication of uncoiler cradles was well underway and the manufacture of dross shields has been taken up.
7. Zinc Distillation Retort — A pilot plant scale retort with condenser was fabricated.
8. Beta Gamma Boxes (Tracer Laboratory) — Two Beta Gamma Boxes to the design of the Atomic Energy Commission were fabricated.
9. Chromizing Retort — A stainless steel retort for chromizing, was fabricated.
10. Punch — A spring loaded punch for
11. Control Panels — two control panels for the Mineral Beneficiation Pilot Plant for installation of control instruments were completed.

Design Projects Continued During the Period were:

1. Synthetic Cryolite Pilot Plant — details of piping, steam line and filters.
2. Strip Aluminizing Prototype Plant — finalization of aluminizing furnace and dross shields.
3. Wire Ausforming Unit — detailed design of lead-baths, hot drawing equipment, coilers and uncoilers.
4. Zinc Recovery Prototype Unit — finalization of detailed retort design.

INDUSTRIAL LIAISON, OPERATIONAL RESEARCH, INFORMATION AND EXTENSION SERVICES

DURING the period under review, liaison, information and extension services of the laboratory considerably increased covering a wide spectrum including consultancy work furnishing detailed technical advice, dissemination of research results, preparation and circulation of technical notes of NML-developed methods for their fruitful utilization, arranging practical demonstration of licensed processes, etc.

Consultancy Work

All round consultancy work of importance on different metallurgical aspects with special importance to the preparation of indigenous raw materials for the iron and steel industry had been rendered to the integrated iron and steel and other metallurgical complexes besides small units of the country. Attention had also been given to the problems being faced by non-ferrous industry of vital importance, since the country needs to be more precise in using and avoiding wastage of non-ferrous materials like Cu, Zn, Sn, etc., which are in very short supply. Considerable assistance has also been rendered to Indian aluminium industry which ranks next to iron and steel. During the period under review, NML has done beneficiation and agglomeration of iron ore from Dalli mines for Bhilai Steel Plant, Bailadila iron ore from N.M.D.C. Ltd. and Kiriburu iron ore for Bokaro Steel Plant. A systematic study had been done for the distribution of of hematite in the Kanjamalai magnetite deposit of Salem for Neyveli Salem Steel Plant and reports prepared on the findings of the investigations conducted on beneficiation, pelletization and reducibility of iron ore from Goa had been sent to Messrs Salgaocar E Irmao Ltda for exporting the ore after pelletization. Extensive studies had also been made on the upgrading and reducibility of specular hematite iron ore of Rajasthan and the data obtained on the subject had been furnished to Messrs Kamani Industrial Development Corporation. Studies on the production of high basicity sinters from beneficiated Bolani iron ore had been completed for Durgapur Steel Plant of Hindustan Steel Ltd. Besides, a detailed investigation report incorporating the precise data obtained in the pilot plant scale studies undertaken on Nandini limestone had been forwarded to Bhilai Steel Plant of Hindustan Steel Ltd. Work had also been completed on beneficiation of limestone sample from Rajpipla, Narmada Project and manganese ore from Barajamda. In the non-ferrous group reference may be made to project report submitted on the recovery of zinc from zinc ash for Indian Tube Co. Likewise mention may also be made of the investigation report sent to NMDC., giving full data for beneficiation work conducted on upgrading of copper ore from Khetri. Similarly, studies had also been made on the extraction characteristics of alumina from Mysore bauxite for Director of Geology and Mining, Bangalore.

Priority was given to the research and development work being referred to by defence establishments and necessary technical know-how and technical informations had been provided for all such requests.

Besides the above major consultancy work, National Metallurgical Laboratory had maintained its usual unstinted help to the metallurgical industries by way of conducting numerous metallurgical investigations on failure of parts, breakdown of plants, preparation of raw materials, use of wastages, etc., and on many occasions even on-the-spot studies had been carried out to study the case in proper perspective and suggest remedial measures thereof.

In the field of granting industrial licenses to private and public enterprises for establishment of new undertaking or considerable expansion of existing units, before any final decision is taken by Govt. of India, National Metallurgical Laboratory is effectively consulted. During the period under review 26 such cases were referred to NML through CSIR. Wherever considered necessary representatives of the applicants were invited to discuss the project in full details. Applications envisaging foreign collaboration were critically reviewed in view of foreign exchange expenditures and alternative indigenous ' know-how ' pleaded for adoption, if available in the country.

Active liaison work with first-hand information had been rendered to the industry for using the materials developed at the National Metallurgical Laboratory. Six phosphor-bronze contact springs prepared as per specification of Indian Railways from phosphor bronze developed at the National Metallurgical Laboratory were sent to Indian Railways for field trials. Fosterite refractories of specification supplied by Bhilai Steel Plant had been made from the product developed at the National Metallurgical Laboratory and the same had been sent to Bhilai Steel Plant for trials in the stove checker works. All the above material had been made for field trials in order to replace the imported one. The process developed at the National Metallurgical Laboratory, at the request of All India Handicrafts Board for imparting black colours to Bidri wares (Zn-Cu alloys) had been demonstrated to the persons engaged in the Bidri ware industry.

Collaborative Research and Development Work

The National Metallurgical Laboratory is collaborating with the Commonwealth Committee on Mineral Processing in the field of mineral beneficiation work conducted in India as well as with the International Working Group on Bonding Clays and had undertaken investigations on samples of bentonite obtained from different countries of the world. Besides the above, the National Metallurgical Laboratory is collaborating with the Indian Standards Institution in formulating Standards and undertaking for the Ministry of Defence and ordnance establishments the development and production of special alloys and finished products, etc. Likewise, the National Metallurgical Laboratory is maintaining close liaison with the Department of Atomic Energy, Geological Survey of India, Indian Bureau of Mines, National Mineral Development Corporation and sister laboratories of the C.S.I.R. in its drive towards effective and scientific utilization of indigenous raw materials, development of products and processes, etc.

On behalf of Indian Standards Institution, the National Metallurgical Laboratory had completed the following investigations and reports were submitted:

(i) Investigation to determine the accuracy of potassium permanganate method for the determination of antimony as compared to the accuracy obtained by potassium bromate method. (ii) Determination of lowest ambient temperature below which welding should not be permitted in structural steel for fabrication purposes. (iii) Investigations on standard Rajendra silica sand sample, Rajmahal silica sand, Shertallai silica sand and Neyveli sand. (iv) High silica sand samples collected from different parts of India by Geological Survey of India are being tested for foundry purposes simultaneously in six laboratories including the N.M.L. which is acting as the co-ordinating laboratory. (v) Investigation on Ennore sand regarding its suitability for use as standard sand.

Defence problems referred to the National Metallurgical Laboratory were tackled with top priority basis and all possible assistance required by them were rendered by the National Metallurgical Laboratory. National Metallurgical Laboratory had already reconditioned 6·6 tons of magnesium powder sent by Defence establishments. During the period under review further request for reconditioning 3·5 tons of various grades of magnesium powder had been received from Ordnance Factory, Khamaria, which is being reconditioned. At the request of Director-General, Ordnance Factories, the Laboratory had evolved a process for reconditioning the Mn-Mg powder to defence specification. National Metallurgical Laboratory had also supplied 317 kg. of low-carbon soft iron to Defence Electronics Research Laboratory, Hyderabad; to meet some of their requirements. Besides, about 1200 kg. of ferro-aluminium had been prepared and supplied to Metal and Steel Factory, Ichapur.

Industrial Surveys and Collection of Statistical, Economic and Market Data

Latest statistics relating to production, consumption, demand and production cost of various materials and metal products, ores and minerals, etc., in India and different countries of the world were collected and maintained on a Card Index System. Close scanning of technical news of the industry in the home and overseas press and research developments is undertaken at all times.

Technical Aid to Industry

Technical advice rendered to industry in public and private sector as well as to individuals covering divergent fields of interest maintained a steady flow from NML. Nearly four hundred enquiries on different subjects pertaining to process details, quality production, raw materials, etc, were attended to, during the period under review. Over 82 per cent of the total enquiries attended, during the period, originated from private sector organizations/individuals. Detailed technical notes were prepared to attend to the enquiries effectively wherever necessary. Over hundred short-term investigations and specification tests were conducted on behalf of various industrial organizations and Govt. bodies.

Practical Demonstration

Practical demonstrations of the processes were arranged for the benefit of license-holders of the processes developed at NML. During such demonstrations requisite technical know-how was imparted besides demonstration of actual plant operation and continuous running thereof. Demonstrations were arranged for the following processes during the period under review for the benefit of licencees.

(i) Production of Ni-free electrical resistance alloys for heating elements.
(ii) Production of thermostatic bimetals.
(iii) Production of alnico type permanent alloys.
(iv) Production of flux for submerged arc-welding
(v) Production of C-free ferro-alloys by aluminothermic reactions.

Training

Training had been imparted to technical officers from State Govts., students from technical institutes and universities and trainees from sister laboratories of CSIR in heat treatment, metallography, petrology, spectrographical analysis, ore dressing, refractories, etc., relating to various metallurgical and mineral industries.

Operational Research

The study of the concentration of lead in the liquid Pb-Sn alloy system by statistical analysis was completed. Of the eight samples taken up for study three showed existence of significant correlation to occur in the Pb concentration. The results in respect of other two samples were observed to be non-significant. In the remaining three samples, however, though no significant correlation appeared to occur, there might exist a trend — the trend in case of one sample being positively deviated from the line Y = C, in contrast to the negatively deviated trend in case of the other samples where 'y' represents the Pb concentration and 'c' is a constant. Analysing the above results in the light of experimental conditions under which sample data were collected, it appeared that possibily two variables, e.g. ' sample composition ' and ' temperature of centrifuging ', play significant role. The optimum ' sample composition ' and ' temperature of centrifuging ' appeared to be around 38 Pb to 62 per cent Sn (wt. %) and 320°C. respectively. Deviations from this optimum set of values thus may very likely be responsible for non-existence of significant correlation in five out of eight samples. Furthermore, another significant variable in explaining existence of significant correlation appeared to be ' time of centrifuging ' — the optimum value for which seemed to be around 3 hr. and then again possibly around eight hours.

Report embodying the results of statistical analysis of the data pertaining to foundries in the country and obtained through the sample survey conducted by the Foundry Pig Iron Panel constituted by the Govt. of India had been prepared for their use.

Work was initiated to prepare a comprehensive literature report on Operational Research Techniques as applied to problems of the Iron & Steel Industry so that Operational Research studies can be formulated in a phased programme.

Colloquia

During the period under review, colloquia and related activities of the National Metallurgical Laboratory were effectively maintained. A number of scientists engaged at the National Metallurgical Laboratory in research work on specialized and divergent subjects in the field of metallurgy, delivered extended talks related to their field of specialized activity which were followed by lively discussions. Lectures were also arranged from distinguished scientists and metallurgists from home and abroad.

Exhibition

National Metallurgical Laboratory participated in the 69th Indian National Congress Exhibition held at Durgapur, West Bengal, in January 1965. An impressive stall — Mining and Metallurgy — was put up at the exhibition site in co-operation with C.M.R.S., Dhanbad. Distinguished scientists, engineers and political leaders of repute visited the National Metallurgical Laboratory stall and appreciated the work done at the National Metallurgical Laboratory besides a stream of common daily visitors who were well attended to. A certificate of merit was awarded by Shri P. C. Sen, Chief Minister of West Bengal, to the National Metallurgical Laboratory at the closing ceremony of the Exhibition.

NATIONAL METALLURGICAL LABORATORY FOUNDRY STATIONS AND CORROSION RESEARCH STATION

National Metallurgical Laboratory Foundry Stations

The first NML Foundry Station set up by the National Metallurgical Laboratory at the Industrial Estate, Batala, Punjab, was declared open early in 1964. The NML Foundry Station at Batala has since been engaged in providing testing facilities and technical assistance to the large number of foundries in and around Batala. The field staff have been visiting a number of foundries and giving valuable advise regarding quality and metal control specially in respect of moulding and melting techniques with a view to achieve best results with the existing conditions. The progress achieved by the foundry industry in Punjab with the establishment of National Metallurgical Laboratory Foundry Station at Batala has been acknowledged by the Department of Industries, Govt. of Punjab. Extensive investigations on a project basis has been undertaken in collaboration with the Geological Department of the Govt. of Punjab, on moulding sands available in the northern region with a view to find out their suitability for foundry

FIG. 105 — SHRI R. VENKATARAMAN, MINISTER FOR INDUSTRIES, GOVT. OF MADRAS, OPENING THE NML FOUNDRY STATION, MADRAS. SIR JEHANGIR GHANDY, CHAIRMAN, EXECUTIVE COUNCIL, NATIONAL METALLURGICAL LABORATORY, IS ON THE RIGHT SIDE OF THE MINISTER

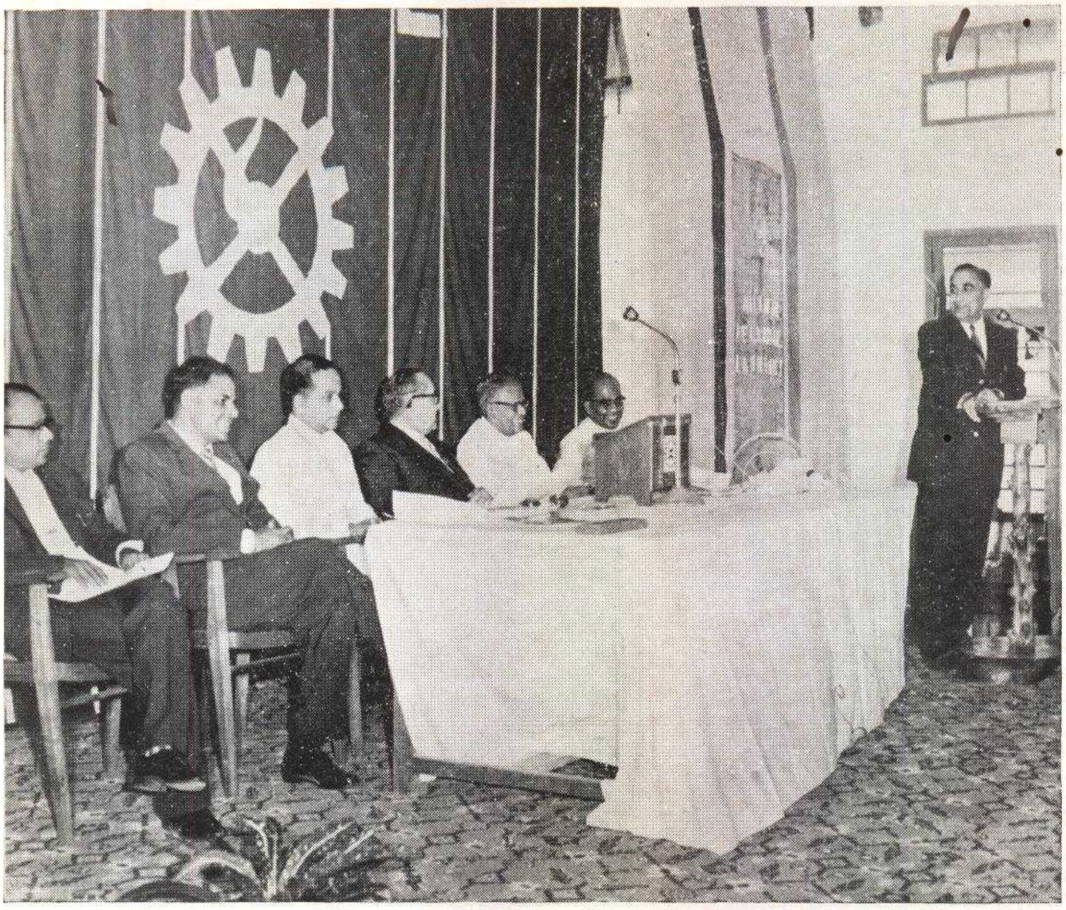

FIG. 106 — DR. B. R. NIJHAWAN, DIRECTOR, NATIONAL METALLURGICAL LABORATORY, DELIVERING THE WELCOME ADDRESS AT THE INAUGURAL FUNCTION OF NML FOUNDRY STATION, MADRAS, WHICH WAS PRESIDED OVER BY SIR JEHANGIR GHANDY, CHAIRMAN, EXECUTIVE COUNCIL, NATIONAL METALLURGICAL LABORATORY

use. All the major sand deposits in the northern region is to be covered by the above investigation.

Considerable progress has been made for the establishment of other National Metallurgical Laboratory Foundry Stations, especially those at Madras and Howrah. The building for the Ahmedabad National Metallurgical Laboratory Foundry Station is at the finishing stage and orders have already been placed for equipment and apparatus. The National Metallurgical Laboratory Foundry Station at Madras and Howrah are ready with equipment and other facilities and work at these centres is expected to commence very soon.

All the National Metallurgical Laboratory Foundry Stations will be equipped with adequate facilities for testing of foundry sands and bonding clays for scientific control of sand mixtures, as well as to find out their suitability for foundry moulding purposes. Stations will also be equipped to undertake chemical analysis of both ferrous and non-ferrous metals, moulding sands and other foundry raw materials. In the second phase, the National Metallurgical Laboratory Foundry Stations will expand the service facilities to include metallographic examination and mechanical testing of metals and alloys. It is also proposed to equip the National Metallurgical Laboratory Foundry Stations with melting units for undertaking experimental work on problems relating to metal melting. Each National Metallurgical Laboratory Foundry Station will have a small library which will gradually be expanded to provide facilities for technical reference on diverse fields of foundry technology.

Programme of Work of National Metallurgical Laboratory Foundry Stations

The programme of work at the National Metallurgical Laboratory Foundry Stations will cover the following scope:

- (i) Periodical testing of foundry sands, bonding clays used in foundries, and raw materials like pig iron, limestone, coke, etc., used for melting, as well as finished products.
- (ii) To tender technical advice regarding selection and application of indigenous foundry sands and other moulding materials, including selection of metal charges for the production of various types of castings.
- (iii) To undertake ad-hoc investigations on the problems faced by individual foundries and to suggest suitable remedial measures thereof.
- (iv) To conduct a regional survey of the moulding materials available in those regions and to conduct a complete study of the chemical and physical properties of the moulding materials.
- (v) To conduct periodical refresher courses to the staff sponsored by the foundries.
- (vi) Standardization of melting and moulding techniques in under-developed foundries.
- (vii) To advise the operational staff on modern foundry technology and to issue technical bulletins periodically.
- (viii) To carry out research and development work, standardization and simplification of moulding, melting and other technological procedures to achieve quality as well as productivity.

Marine Corrosion Research Station, Digha

Since the inception of the Marine Corrosion Research Station in 1963, the laboratory is being equipped with necessary facilities for carrying out studies in various fields of metallic corrosion and its protection. One small analytical laboratory and one machine shop for the preparation of specimens were set up. Work, at present, is mainly confined to study atmospheric and sea-water corrosion. Samples of ferrous and non-ferrous alloys were exposed for long term atmospheric corrosion studies, the results of which are being compared with that of similar samples exposed under industrial atmosphere at Jamshedpur. Studies on effect of proximity to the sea coast were also initiated by exposing samples at a distance of 50 ft. and 520 ft. from the sea coast. Effect of day and night exposures on corrosion rate of mild steel is also being studied, the day samples were exposed daily between 6 a.m. to 6 p.m. and night samples between 6 p.m. to 6 a.m. The tests gave interesting results and comparison between day and night exposure with continuous exposure indicated that the corrosion due to latter is much less than combined weight loss of day and night exposure. A more detailed study on this is under progress.

Both laboratory and field tests on sea-water corrosion were started. Corrosion rate of different metals and alloys are being evaluated in sea water under constant immersion condition in the laboratory. For field test, samples were exposed under alternate immersion condition so that on each day the samples are immersed twice for a period 6-8 hours. The tests are under progress.

LIBRARY, DOCUMENTATION, TRANSLATION AND REPROGRAPHIC SERVICES

Library and Documentation Services

The Library and Documentation services of the National Metallurgical Laboratory with their project-oriented documentation and pin-pointed system of information retrieval play a key role in facilitating research in metallurgy and service to metallurgical industries. These services are built around a vast collection of bibliographic and graphic material acquired from all over the world which has taken over a decade to build. From the very beginning, they were systematically classified and indexed according to the enlargements of the Universal Decimal Classification — UDC brought out by the British Standards Institution. With time and the growth and refinement of classificatory system pertaining to metallurgy and allied subjects, it became imperative that the holdings of the Library be re-classified in tune with modern thinking and discipline. This was particularly urgent in view of necessity to dovetail the entries in the Central Documentation System with macro data in the Library in order to afford a firmer foundation to the system which has now been operating for three years. The reclassification work has been taken up.

The project-oriented documentation system has been extended to cover newer divisions and frontiers like the application of isotopes in metallurgy, adaptation of metallurgy to newer fields like plasma physics, space metallurgy, etc., to name only a few. The micro-information in the patent literatures of most industrially advanced countries like U.K., U.S.A., U.S.S.R., Germany, France and Japan are now being fed into the Central Documentation Grid making data coverage as comprehensive as the means permit. The extraction of data from patent literature has also enhanced the utility and range of service offered by the Library's Patent Literature Section, which is also Govenment of India's Patent Literature Inspection Centre for this region.

Although on a modest scale, chain indexing project of the entire micro data in the Library has been taken in hand. It is hoped that as the work gathers momentum, it would considerably extend the area of information retrieval and reduce the time span of each search.

Translation Service

Articles in French and German of direct interest to research work projects underway at the National Metallurgical Laboratory were translated into English. Oral translations were also rendered to give the research worker a quick appraisal of the results contained in the technical publications with a view to enable him to precisely define the portion of the text of specific interest to him. Additionally other routine translation work was also attended to efficiently. About 300 technical papers published in French and German were translated during the period under review, each paper being of an average of 4,000 words.

Reprographic Service

Photostat, reflex prints and microfilms were made of the scientific and technical papers of interest to the research workers and were widely circulated. Documentary films were taken of important technical functions of the Laboratory as well as outstanding processes and techniques developed. Coloured transparencies and micro-photographs, X-ray photographs, etc., were taken of the various research and investigation projects.

INTERNATIONAL SYMPOSIA

WITH a view to focus attention on a multitude of aspects of micro-metallurgy, a Symposium on 'Micro-Metallurgy — the Role of Minute Additions to Ferrous and Non-ferrous Metals and Alloys' was organized by the National Metallurgical Laboratory from 29th March to 1st April, 1965, so as to provide an international forum to discuss the role of minute additions to ferrous and non-ferrous metals and alloys and to exchange technical 'know-how', examine inter-related problems and facilitate further research studies on the subject.

The Symposium drew a large gathering and more than 200 delegates from all over the world including top ranking scientists and technologists participated in its deliberations.

The following twenty-nine technical papers were presented and discussed in six technical sessions:

1. Pearlite-free structural steels.
2. Rare-earth metals in stainless steel making.
3. Techniques for the study of micro-phases and their influence on material properties.
4. The purification of iron by selective prepurification and oxidation zone melting; property measurements and minor impurity contents.
5. The role of residual trace elements in alloy steels.
6. On the transformation of austenite-martensite and the tempering processes of martensite in iron-nitrogen system.
7. Effect of molybdenum on the grain boundary relaxation of iron.
8. The effect of small amounts of vanadium and niobium on the micro-structures and the strength of pro-eutectoid ferrite of iron-carbon alloys.
9. Effect of minor elements on normal grain growth in singly oriented silicon iron.
10. Scope of research in micro-metallurgy at the National Metallurgical Laboratory.
11. Micro-metallurgy of abnormality in steels.
12. Micro-metallurgy of austenitic grain size control of steels.
13. Effect of aluminium additions on the primary grain size of steel castings.
14. Effect of micro-impurities and alloying elements on zirconium and zircaloy-2.
15. Distribution and function of micro-phases and micro-additions in metals and alloys.
16. Morphology and polygonization of copper whiskers in the form of ribbons.
17. Importance of micro-metallurgy in non-ferrous metals and alloys used in navy.
18. Influence of small amounts of boron and zirconium on hot working characteristics and creep rupture properties of some vacuum-melted nickel-base alloys.
19. Effect of minor additions of alloying elements on irradiation behaviour and mechanical properties of natural metallic uranium.
20. Effect of bismuth additions on the graphitization of spheroidal graphite cast iron.
21. Surface tension of cast iron with additions of different elements.

FIG. 107 — DR. B. R. NIJHAWAN, DIRECTOR, NATIONAL METALLURGICAL LABORATORY, WELCOMING THE DELEGATES TO THE SYMPOSIUM

22. Determination of the distribution of impurities in commercial beryllium by electron microscopy.
23. Studies on micro-constituents in metals and alloys by the electron microprobe and by some specialized chemical technique aided with X-ray diffraction.
24. Effect of trace elements on ageing of some aluminium-base alloys.
25. Study of micro-additions to Al-Mg wrought alloys.
26. Some observations on recent methods of analysis of gases in metals and alloys.
27. A spectroscopic method of determination of micro-amounts of elements in steels.
28. Spectrographic analysis of micro-elements in metals and alloys.
29. Analytical methods in micro-metallurgy.

Dr. B. R. Nijhawan, Director, National Metallurgical Laboratory, in welcoming the distinguished delegates from India and abroad, said that such international Symposia offer stimulation of thought and provide an ideal forum for exchange of technical ' know-how ' and ' know-why ' of diverse aspects of the subject.

Continuing, Dr. Nijhawan said that the subject of Symposium is somewhat unusual, e.g. ' Micro-Metallurgy — The Role of Minute Additions to Ferrous and Non-ferrous Metals and Alloys ' which bridges across the work of fundamental research studies and that of applied research development themes. During the successive Five Year Plans, the National Metallurgical Laboratory is called upon to handle various research and development projects essential to the growth of mineral and metallurgical industries, challenging in their objectives and the challenge is being effectively met. A main theme of activity at the National Metallurgical Laboratory is educational, such as the organization of Symposia on metallurgical subjects of topical interest like the present one. Research and development studies undertaken so far at the National Metallurgical Laboratory have been effectively rewarding whilst the search and research on how so much is contributed by the so small, will be ceaseless and practically unending, in the planning and execution of which, the National Metallurgical Laboratory is actively engaged.

Sir Jehangir Ghandy, Chairman, Executive Council of NML, said that the National Metallurgical Laboratory has undertaken very valuable research work on various aspects of micro-metallurgy. These related to grain-size control in steel, abnormality and strain ageing effects in steel. Research is also being conducted on micro-additions of rare-earth elements to stainless steels and aluminium-magnesium alloys, and on the ageing of aluminium alloys. In addition, studies on phase changes in relation to micro-constituents, micro-metallurgy of aluminium-silicon alloys and heat-resistant alloys and analytical techniques for measuring are being made. The subject of micro-

FIG. 108 — SIR JEHANGIR GHANDY, CHAIRMAN, EXECUTIVE COUNCIL, NATIONAL METALLURGICAL LABORATORY, DELIVERING THE PRESIDENTIAL ADDRESS AT THE SYMPOSIUM

Fig. 109 — Shri Asoka Mehta, Dy. Chairman, Planning Commission, Delivering the Inaugural Address at the Symposium

metallurgy is vast and requires cataloguing of hypotheses and defining those needing attention to research facilities and equipment available for specialized research techniques. Sir Jehangir extended on behalf of the Executive Council of the National Metallurgical Laboratory, their sincere thanks and deep appreciation to Dr. B. R. Nijhawan, Director, National Metallurgical Laboratory, and his colleagues for the valuable research and development work being done by them and also for the fully adequate arrangements made for the Symposium.

Shri Asoka Mehta, Deputy Chairman, Planning Commission, while inaugurating the Symposium said that the subject of the Symposium is of profound interest particularly to developing countries, because the subject tries to find out how the minute additions have far reaching consequences on the properties of metals and alloys. Thus it becomes possible for a country lacking certain crucial resources to make up by this kind of micro-marginal efforts and devices. Then again because a minute particle may have far reaching consequences, it becomes possible to cut down the cost structure. Continuing Shri Mehta said, what is liked about this particular laboratory is that it has succeeded in assembling together a group of men and women, young scientists, young engineers, who have confidence, ability, ingenuity and improvization. These are essential qualities that are needed today and in the next few years. Nothing must be allowed to intimidate. There will be difficulties and if difficulties are going to intimidate then this one-seventh of world's humanity will remain always a load, a burden for the rest of the world to carry. But if there is confidence, ingenuity, willingness to put in whatever intellectual and physical efforts that are to be put in and above all improvize, there is no reason that in the next ten years the country should not be able to move forward in the manner where it can play its part in the concert of the world whatever be the symphony that the concert may be played at the particular time. Shri Mehta stated that it is the responsibility of the Planning Commission as the central highest thinking organization set up by the Government to go out and find out and make it possible for all these creative elements in the country to come together and make their critical and decisive contributions. Concluding, Shri Asoka Mehta congratulated Dr. Nijhawan and his colleagues in organizing the International Symposia and on their splendid work in diverse fields.

Dr. Husain Zaheer, Director-General, Scientific and Industrial Research, addressing the delegates to the Symposium on 'Micro-Metallurgy' complimented the National Metallurgical Laboratory for the valuable work done on diverse fields and the number of project-oriented industrial problems undertaken on behalf of the industries. In the connection, he referred to the tribute paid to the National Metallurgical Laboratory by the Third Reviewing Committee headed by Shri A. Ramaswamy Mudaliar that the National Metallurgical Laboratory research work, has forged close links and won the confidence of the Indian mineral and metallurgical industries. The valuable suggestions given by the Reviewing Committee that the National Metallurgi-

Fig. 110 — A View of the Delegates at the Symposium Session

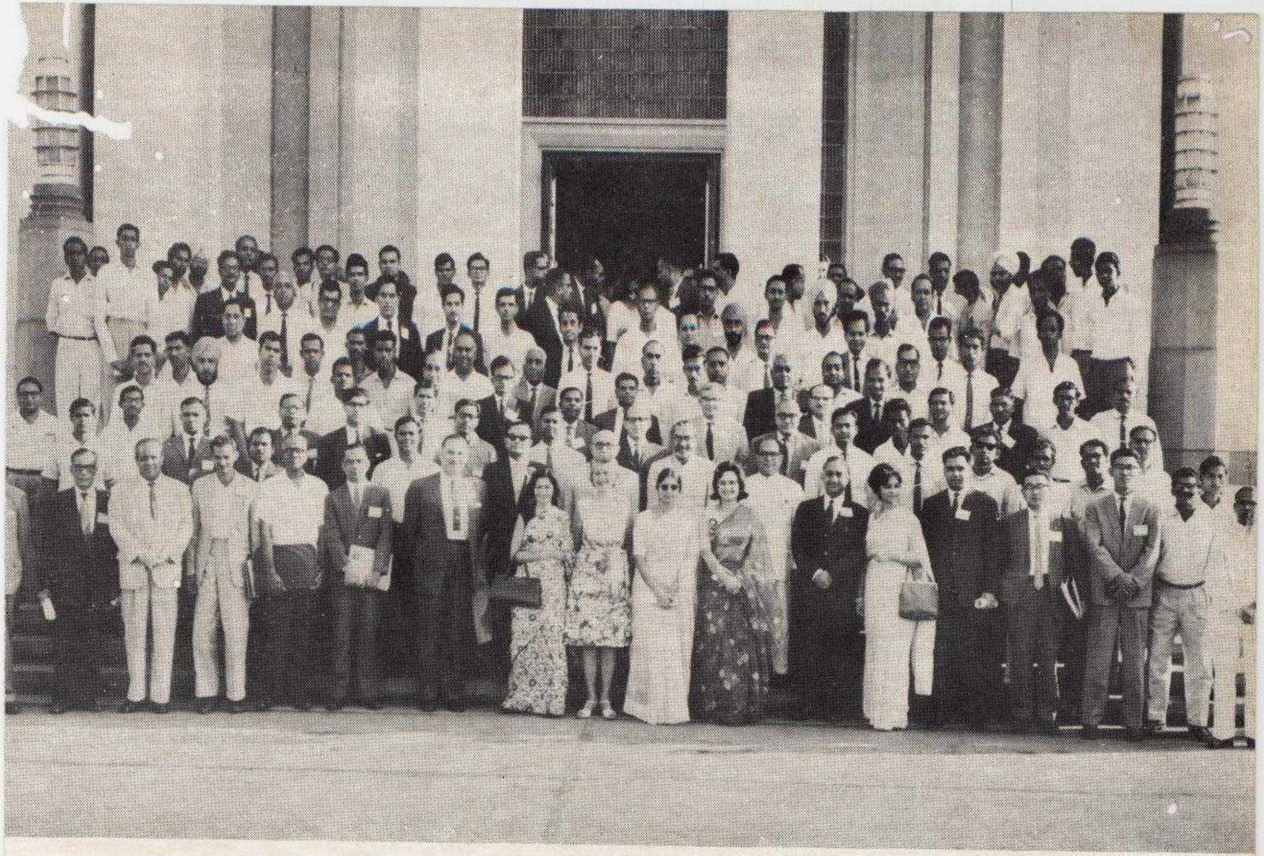

FIG. 111 — GROUP PHOTOGRAPH OF SOME OF THE DELEGATES WHO PARTICIPATED IN THE SYMPOSIUM

cal Laboratory should also focus attention on fundamental aspects of the work has also been implemented immediately by the holding of this Symposium on Micro-Metallurgy in which field this Laboratory has done pioneering work on the fundamental aspects of minute additions to metals and alloys. As far as the National Metallurgical Laboratory is concerned, Dr. Zaheer said, " It has been fortunate to have such a young and dynamic Director in the person of Dr. B. R. Nijhawan, who has done much in bringing this Laboratory to the international forefront and status ". He expressed his sincere hope that this Laboratory will provide the most needed solutions to the multifarious problems faced by the industry in the mineral and metallurgical fields with its vast net-work of pilot plants and the well-talented research staff working under the able leadership of Dr. B. R. Nijhawan. He also paid a tribute to Mr. Asoka Mehta, Deputy Chairman, Planning Commission, for his depth of vision and deep insight in the planned economy of the country and his faith in the indigenous talent in promoting the cause of scientific and industrial research.

Shri M. S. Rao, Chairman, Hindustan Steel Ltd., who was the Chief Guest, referred to the great service that the National Metallurgical Laboratory rendered to Hindustan Steel Ltd. not merely on the basis of general research studies carried out but because the Laboratory had carried out pilot plant studies specifically for Hindustan Steel Ltd. on beneficiation of the iron ores which go to supply the three Steel Plants of Hindustan Steel Ltd. and also studies on sintering of the Barsua iron ore for Rourkela and the Rajhara iron ore for Bhilai Steel Plant. The National Metallurgical Laboratory is regarded as the main research centre for original studies for Hindustan Steel Ltd. Continuing, Shri Rao said, the subject matter of the Symposium is perhaps as entrancing in the vistas which it opens, as the study of the tiny atom, has been in nuclear physics; and placed in man's hand enormous energy, power and speed, so also purposive research of the micro-constituents in metals and alloys with reference to the raw materials available in our country; but, in course of time, it should be possible, in the making of alloy steels in our country to replace by some indigenous elements certain rare and costly elements not readily available in our own country. The prospect of developing new and better alloys is also great.

Giving a resume of the Symposium at the end of the technical sessions, Dr. B. R. Nijhawan, Director of the National Metallurgical Laboratory, said that the papers submitted covered numerous aspects of the subject of micro-metallurgy which represent a vast and challenging field. Dr. Nijhawan thanked all the participating delegates from various parts of the country and abroad for taking part in the deliberation of the Symposium, and expressed his appreciation of the valuable contributions on various technical points of importance which arose during the discussions. Research studies in micro-metallurgy undertaken at the NML so far have been fully rewarding, he added, and this Symposium has played its part in providing an inter- scientists to discuss their views and exchange ideas.

The delegates were conducted round the various research divisions of the National Metallurgical Laboratory, the Low Shaft Furnace Pilot Plant, the Mineral Beneficiation Pilot Plant and other pilot plants of the Laboratory during the Symposium.

The Symposium Supplement

On the inaugural day of the Symposium, special supplements were brought out in masterly fashion by Economic Times and Financial Express. The Supplements contained leading papers from eminent scientists and technologists. Economic Times took a special interest in the Symposium and excellent coverages of day-to day proceedings were made. Excellent coverage was also done by other leading news papers like *Hindustan Standard*, *Amrita Bazar Patrika*, *Statesman*, *Hindu*, *Financial Express*, etc., of the inaugural function and technical sessions of the Symposium.

PUBLICATIONS

NML Technical Journal

The *NML Technical Journal*, a quarterly publication, has stepped into the seventh year of its publication. The journal has established a wide exchange programme with many Indian and foreign technical journals and nearly 200 journals and other publications are obtained on ' free exchange basis '. The number of Indian and foreign subscribers are increasing gradually. The papers published in *NML Technical Journal* are regularly abstracted by leading international abstracting services such as Chemical Abstracts, Journal of Iron & Steel Institute, U.K., American Society of Metals, Batelle Memorial Review, etc.; the contents of the Annual Report of the National Metallurgical Laboratory are likewise abstracted by leading abstracting establishments.

Papers Published

During the period under review 93 scientific papers were published and presented as per Appendix I.

Symposium Proceedings

The Proceedings of the Symposium on ' Utilization of Metallurgical Wastes ' was brought out in a masterly fashion. The proceedings contain thirty-seven authoritative papers from leading scientists and technologists of the world engaged in this field of study. There is a great demand of this publication both from home and abroad.

Research and Investigation Reports

During the period under review 69 research and investigation reports were prepared. The details are furnished in Appendix II.

PATENTS

Patents Filed During the Period

1. *Indian Patent No.* 96661 — A novel method of separating iron from ilmenite and its application in the upgrading of ferruginous ores (24.11.1964).
2. *Indian Patent No.* 96017 — Production of high purity iron (12.10.1964).
3. *Indian Patent No.* 94769 — An improved device for the continuous hot-dip coating of metallic strip and wire (20.7.1964).
4. *Indian Patent No.* 94768 — An improved device for the isolation of dross in molten metallic baths during continuous hot-dip processing of strips or wire (20.7.1964).
5. *Indian Patent No.* 94767 — An improved cast iron pot for the melting and holding of non-ferrous molten metal in general and aluminium and zinc in particular (20.7.1964).

Patents Accepted During the Period

U.K. Patent No. 989802 — Improvements in or relating to the modification of aluminium alloys coating silicon (19.2.1965).

Patents Sealed During the Period

1. *Indian Patent No.* 83968 — A method for reconditioning the coated magnesium powders (29.5.1964).
2. *Indian Patent No.* 83652 — Improvements in or relating to magnesite refractories (29.5.1964).
3. *Indian Patent No.* 65610 — Improvements in or relating to the production of chemically bonded metal-clad or unclad basic refractories (4.9.1964).
4. *Indian Patent No.* 84670 — Improvements in or relating to electrolytic cells (11.9.64).

Processes Released

The following processes were released so far to industries 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-bonded graphite crucibles).
9. Refractory compositions comprising graphite and alumino-silicate materials and glazes to render such compositions resistant to oxidation. (clay-bonded graphite crucibles).
10. Production of carbon-free ferro-alloys by aluminothermic reactions.
11. Production of iron powder for autogenous cutting.
12. Preparation of liquid gold.
13. Production of flux for submerged arc welding.
14. Electroplating of metals on aluminium or its alloys.
15. Chemical polishing of aluminium.
16. Metallization of non-conductors.
17. Brass plating from non-cyanide bath.
18. Production of dense carbon aggregate suitable for being used as base material for carbon products in general and Soderberg paste in particular.
19. Production of electrolytic copper powder.
20. Production of ceramic magnets.

GENERAL

MR. M. C. Chagla, Union Minister for Education, Govt. of India and Vice President of Council of Scientific & Industrial Research, declared open the new extension of National Metallurgical Laboratory on 6th February 1965. Mr. Chagla in his inaugural speech greatly commended the highly valuable work done by the National Metallurgical Laboratory in diverse fields and complimented Dr. B. R. Nijhawan, Director, National Metallurgical Laboratory, and his team of workers on their dedicated work. Sir Jehangir Ghandy, Chairman, Executive Council of National Metallurgical Laboratory, who presided over the function, paid his sincere tributes to Dr. Nijhawan, Director, National Metallurgical Laboratory, and his colleagues for their single-minded devotion and the excellent work they were doing in the field of minerals and metals industry of the country under the tempo of successive Five Year Plans of the country.

The extension work to the main building comprising the eastern, western and central wings was completed. The new extensions

FIG. 112 — HON'BLE MR. M. C. CHAGLA, UNION MINISTER FOR EDUCATION, GOVT. OF INDIA, OPENING THE NEW EXTENSION TO THE MAIN BUILDING OF THE NATIONAL METALLURGICAL LABORATORY

Fig. 113 — Hon'ble Mr. M. C. Chagla, Union Minister for Education, Govt. of India, Delivering the Inaugural Address on the Opening of the New Extension to the Main Building of the National Metallurgical Laboratory

provide an extra length of 160 ft. to the 480 ft. long main building of the laboratory thus providing an additional floor area of 30,000 sq. ft. which accommodates the main new items of equipment that have been installed.

In the Main Side and Central Wings of the National Metallurgical Laboratory the following research facilities had been set up:

1. Vacuum metallurgy equipment containing vacuum-melting and air-melting equipment of pilot plant scale.
2. Extraction Metallurgy including silico-thermal reduction of dolomite to magnesium; hydro-metallurgy including leaching of ores, secondary refining of metals such as zinc dross, skimming residue, etc.
3. X-ray diffraction equipment and a small precision workshop.
4. Radio-active isotope laboratory.
5. Phase transformation studies in refractory materials in slag system.
6. Utilization of metallurgical wastes including blast furnace and steel melting slags and their optimum utilization for a multitude of uses.
7. Study of non-metallic inclusions.
8. Plastic coatings of steel.
9. Electronic instrumentation, repair and fabrication.
10. X-ray fluorescence spectrometer; mineralogical and petrological studies on ores and minerals.
11. Mechanical testing of metallurgical equipment including the vibro-phore pulsator.

12. Differential thermal analytical equipment for fundamental studies into slags and minerals.
13. Precision workshop.
14. Alloy Steel Division.
15. Extractive Metallurgy Research Division including non-ferrous metallurgy.
16. Iron and Steel Research Division.

The Central wing contains the library block, Auditorium and Liaison, Information and Operational Research facilities. Additionally a full-fledged Design Engineering Division is operating on the top floor of the Central wing engaged in original design engineering and provision of working drawings for the fabrication of the Pilot Plants in the National Metallurgical Laboratory workshop and industrial projects.

The value of the equipment housed in these sides and central wings is of the order of over Rs. 1·5 million. The wings have been built up at a cost of rupees 1·2 million and constitute a double-storied structure. Another notable installation work completed was the oil/light petroleum naphtha injection system for trials at the Low Shaft Furnace Pilot Plant.

The New Foundry Block was declared open by Shri Asoka Mehta, Dy. Chairman, Planning Commission, Govt. of India. Equipped with modern machineries for moulding, melting and casting, the foundry block will provide most up-to-date research facilities in the field of foundry. A hot blast cupola fitted with a special device to utilize the heat of the exhaust gases to pre-heat the air-blast, has been installed in this block at a cost of rupees one lakh. Designed and fabricated at the Laboratory, it will raise the temperature of the air-blast to 500°C. The cupola has been so designed that it can be easily fitted on even to the existing cold blast cupolas in foundries.

Direct and indirect arc furnaces and gas-fired furnaces, installed in the new wing to melt special steels and non-ferrous metals and alloys will provide ample scope to the research workers to solve many problems of the growing metallurgical industries of the country.

Attached to the foundry block is a well-equipped sand testing laboratory with facilities to undertake investigations both at room and high temperatures of the properties of the moulding sand mixtures. An automatic shell moulding unit for making shell moulds and cores is another important addition to this wing.

The National Metallurgical Laboratory Foundry Stations at Howrah and Madras are now ready for operation. Construction work for NML Foundry Station at Ahmedabad is almost completed and is being equipped. The construction of the Marine Corrosion Research Station at Digha is completed and research work is in progress in full swing in this station.

The construction relating to 30 E type and 10 D II type multistoried staff quarters is progressing satisfactorily. Construction of staff quarters at Digha is also in progress.

Purchase and Stores Sections of the National Metallurgical Laboratory kept up their unceasing activity for procurement of capital equipment, raw materials and consumable stores and equipment, etc. The administrative work of the Laboratory was efficiently maintained and the heavy budgetary accounts of the Laboratory were also handled with efficiency and speed. Details of National Metallurgical Laboratory budget expenditure are furnished in p. 231 of this report. Co-operative Banking Society of the National Metallurgical Laboratory continued its good work, indeed, throughout the year. The Co-operative Stores set up recently is doing an exceedingly good job in the supply of rationed and other food stuffs, stationery articles, etc., to the staff members.

Award

The Hony. Membership of the Iron & Steel Institute, Japan, was conferred on

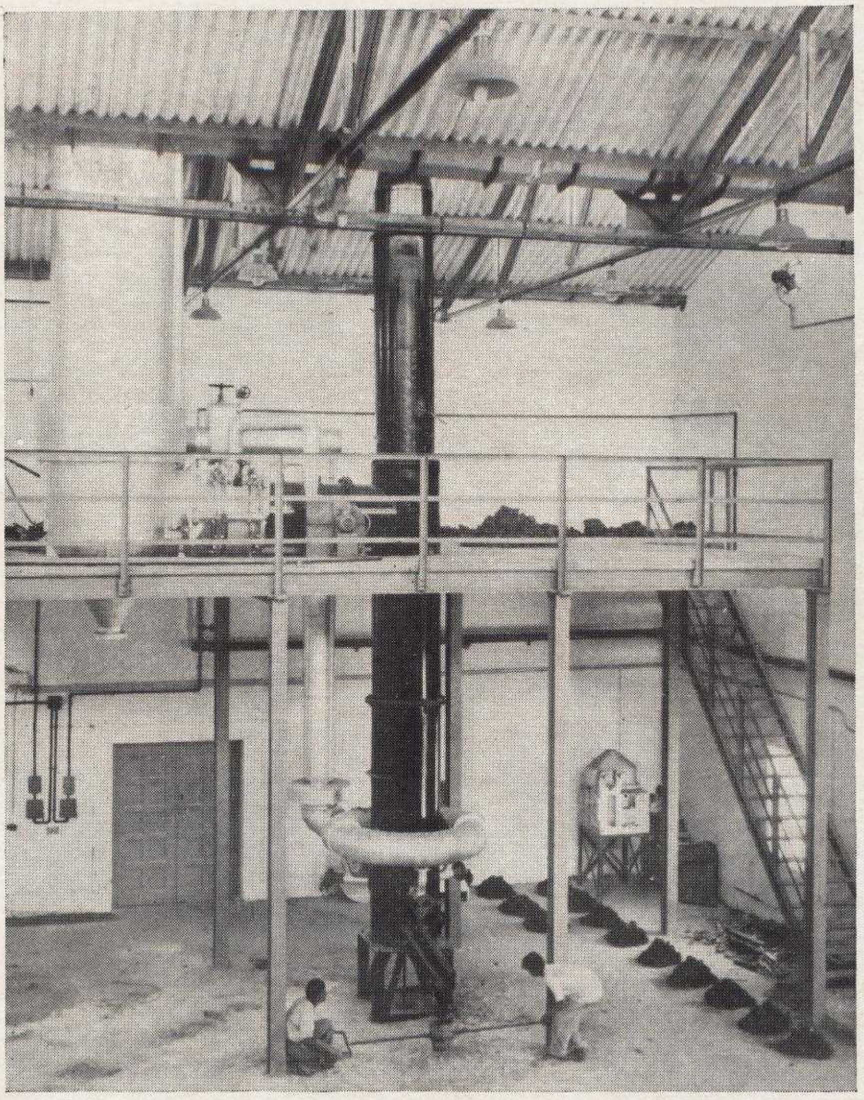

FIG. 114 — A VIEW OF THE HOT-BLAST CUPOLA, DESIGNED AND FABRICATED AT THE NATIONAL METALLURGICAL LABORATORY

Dr. B. R. Nijhawan, Director. He was also awarded a Gold Medal for his outstanding contributions made in the field of metallurgy. Dr. B. R. Nijhawan has also received the coveted Shanti Swarup Bhatnagar Memorial Award for his outstanding contributions in the field of engineering sciences.

Mr. P. P. Bhatnagar, Scientist, has been given "The National Metallurgists' Day Award" for his outstanding contribution to research in non-ferrous metallurgy.

Messrs A. K. Lahiri and R. K. Dubey, have been awarded Ph.D. degrees from Banaras Hindu University.

Safety First and First Aid Section

A safety Committee was formed which met regularly and discussed the ways and means of safety measures and their implementation. A 'No Accident' week was observed and demonstrations were held on first aid, fire fighting, artificial respiration, etc. Safety posters and slogans were displayed and a film show was arranged on the importance of safety measures.

With the installation of Naphtha Injection System and Gas Producer at the Low Shaft Furnace and Mineral Beneficiation Pilot Plant, rigorous safety measures were introduced against fire and gas poisoning hazards. Regular inspection was made of the safety appliances in various plants, machineries and vehicles to minimize the accidents and loss of laboratory property. Three persons attended the Safety Training Course conducted by the Safety First Association, Jamshedpur Branch. First aid was rendered to a number of cases of injury.

BUDGET FIGURES FOR THE NATIONAL METALLURGICAL LABORATORY
FOR THE YEAR 1964-65

		(Figures in lakh of Rupees) *(One lakh =* 10^5 *rupees)*
RECURRING		
P-1	Pay of Officers	8·135
P-2	Pay of Establishment	9·557
P-3	Allowances	4·577
P-4	Contingencies	5·535
P-6	Maintenence	0·298
P-7	Chemicals, Apparatus and Equipment for Research	7·462
F.S.S.—	Fellowships and Scholarships	0·514
	Scientist Pool	0·306
		36·384
CAPITAL		
P-5	1. Works	1·180
P-5	2. Services	1·038
	3. Apparatus and Equipment	1·043
	4. Miscellaneous	0·600
		3·861
PILOT PLANT		
PP-1	(i) Equipment and Installation	3·425
	(ii) Building and Services	3·010
PP-2	Provision for staff	5·222
PP-3	Purchase of Raw Materials	0·528
PP-4	Miscellaneous and Contingencies	3·707
		15·892
LOW SHAFT FURNACE PROJECT		
PP-1	(i) Equipment and Installation	0·994
	(ii) Building and Services	—
PP-2	Provision for Staff	3·281
PP-3	Purchase of Raw Materials	1·291
PP-4	Miscellaneous and Contingencies	6·812
		12·378
CONSTRUCTION OF STAFF QUARTERS		7·047
DEFENCE PROJECTS		0·641
		7·688
	GRAND TOTAL	76·203

APPENDIX I

SCIENTIFIC PAPERS PUBLISHED AND PRESENTED

1. Symposium on Utilization of Metallurgical Wastes — A Review.— J. E. Mannar and V. Muthukrishnan, *NML Technical Journal*, VI (2), (7-12), 1964.
2. Growth Pattern of Iron & Steel Industry in India's Economic Development.— B. R. Nijhawan, *NML Technical Journal*, VI (2), (13-24), 1964.
3. Scope and Function of the Regional Foundry Station of the National Metallurgical Laboratory at Batala.— R. M. Krishnan & B. R. Nijhawan, *NML Technical Journal*, VI (2), (25-31), 1964.
4. The Suitability of Jainti Dolomite for Sintering in Shaft Kilns.— P. C. Sen, M. R. K. Rao & H. V. Bhaskar Rao, *NML Technical Journal*, VI (2), (32-34), 1964.
5. Spectrochemical Determination of Antimony in Lead-Tin Solders.— M. K. Ghose, P. C. Debnath & B. C. Kar, *NML Technical Journal*, VI (3), (6-9), 1964.
6. Chromate Passivation of Zinc.— S. Rao Addanki & A. K. Lahiri, *NML Technical Journal*, VI (3), (10-14), 1964.
7. Rapid Method for the Determination of Manganese in the Manganese-bearing Stainless Steel.— A. C. Biswas & H. P. Bhattacharya, *NML Technical Journal*, VI (3), (15-17), 1964.
8. A Prototype Vertical Kiln for Continuous Thermal Beneficiation.— M. J. Shahani, *NML Technical Journal*, VI (3), (18-21) 1964.
9. Production of Low Residual and Soft Iron.— R. D. Gupta, P. K. Gupte & B. R. Nijhawan, *NML Technical Journal*, VI (4), (4-11), 1964.
10. Thermal Beneficiation of Low Grade Chrome Ore from Enuconda Deposits, West Godavari District, Andhra. — Sipra Samanta, R. N. Misra & P. P. Bhatnagar, *NML Technical Journal*, VI (4), (12-16), 1964.
11. A Comparative Study of Insulation Refractories with Special Reference to Thermal Conductivity.— B. M. Dutta & H. V. Bhaskar Rao, *NML Technical Journal*, VI (4), (17-22), 1964.
12. Development of Aqueous Non-Sludge Forming Zinc Phosphate Bath. — K. P. Mukherjee & A. K. Lahiri, *NML Technical Journal*, VI (4), (23-27), 1964.
13. Scope of Research in Micro-Metallurgy at the National Metallurgical Laboratory.— B. R. Nijhawan, *NML Technical Journal*, VII (1), (45-64), 1965.

The following papers were published in the Proceedings of the Symposium on ' Utilization of Metallurgical Wastes '.

14. Utilization of Important Mining and Metallurgical Wastes Employing Mineral Beneficiation Techniques.— M. S. Chopra, G. P. Mathur & P. I. A. Narayanan.

15. Utilization of Fine Grained Raw Materials by Briquetting for Smelting of Ferromanganese.— A. B. Chatterjea, S. R. Ghosh & J. Goswami.

16. Research and Development Work on the Utilization of Metallurgical Wastes at the National Metallurgical Laboratory.— B. R. Nijhawan.

17. Utilization of Coke Breeze for Production of Pig Iron on Small Scale.— A. B. Chatterjea, J. Goswami, S. K. Biswas, R. Santok Singh & J. S. Padan.

18. Investigations on Granulated Low Shaft Furnace Slag for the Production of Slag Cement. — B. N. Singh, P. C. Debnath, B. C. Kar & B. R. Nijhawan.

19. Scope for Utilization of Slags and Related Wastes from Indian Iron and Steel Plants. — Narinder Singh, K. N. Srivastava, R. M. Krishnan & B. R. Nijhawan.

20. Utilization of Low Shaft Furnace Slag as Light Weight Aggregate for Insulation Concrete. — Narinder Singh, K. N. Srivastava, R. M. Krishnan & B. R. Nijhawan.

21. Utilization of Red Mud.— C. Sankaran & P. P. Bhatnagar.

22. Production of Secondary Aluminium — A Review.— M. C. Sen & T. Banerjee.

23. Recovery of Zinc from Scraps and Wastes.— B. C. Mukherjee, S. C. Aush, M. C. Sen & T. Banerjee.

The following papers (Sl. No. 24 to 31) were published in the Foundry Number (1964) of Indian Construction News, 13 (7, 8 and 9), 1964. The papers were presented at the Annual Convention — 1964 of Indian Institute of Foudrymen.

24. Production of Foundry Grade Pig Iron in Punjab.— A. B. Chatterjea & B. R. Nijhawan.

25. Cupola Operation with Jhama Coal as a Substitute for Foundry Coke.— Jatinder Mohan, J. Goswami, A. B. Chatterjea & B. R. Nijhawan.

26. Design of Risers.— G. N. Rao.

27. Importance of Adsorbed Water on Clay Surfaces in Liquid Limit Determination.— A. T. Chirayath, T. A. Beck, V. S. Bhandary & P. K. Gupte.

28. Differential Thermograms of Certain Indian Foundry Bentonite Clays.— A. T. Chirayath & Jatinder Mohan.

29. Studies on Activation of Indigenous Bentonites.— T. A. Beck, V. S. Bhandary & P. K. Gupte.

30. Effect of Sulphur on the Properties and Microstructure of Cast Iron.— B. V. Somayajulu, P. K. Gupte & B. R. Nijhawan.

31. Study of Eutectic Cells with Addition of Different Elements.— Jatinder Mohan.

32. Some Studies Investigating the Procedure of Manufacturing Porous Bronze Bearing. — S. Ranganathan & P. K. Gupte.— Trans. of Indian Institute of Metals, Vol. 17, June, 1964.

33. Studies on the Properties of Binary Copper-Magnesium Alloys.— A. K. Lahiri, K. P. Mukherjee & T. Banerjee.— Trans. of Indian Institute of Metals, Vol. 17, June 1964.

34. Reaction of Titanium with Titanium Tetrachloride.— S. R. Srinivasan & P. P. Bhatnagar.— Trans. of Indian Institute of Metals, Vol. 17, Sept. 1964.

35. Studies on Silico-Thermic Reduction of Dolomite for the Production of Magnesium.— R. N. Misra, V. S. Sampath & P. P. Bhatnagar.— Trans. of Indian Institute of Metals, Vol. 17, Sept. 1964.

36. Studies on the Chlorination of Vanadium Bearing Titaniferous Magnetite.— S. R. Srinivasan, S. S. Bhoray & P. P. Bhatnagar.— Trans. of Indian Institute of Metals, Vol. 17, Dec. 1964.

37. Effect of Surface Condition of Formabilities of Sheet Metal.— K. N. Ghosh & B. N. Das.— Trans. of Indian Institute of Metals, Vol. 17, Dec. 1964.

38. Electrical Conductivity of Al-Si and Al-Si-Mg Alloys.— Manjit Singh & R. Kumar. — Trans. of Indian Institute of Metals, Vol. 18, March 1964.

39. Trials on the Injection of Light Petroleum Naphtha in the Low Shaft Furnace Pilot Plant.— A. B. Chatterjea & B. R. Nijhawan.— Economic Times Supplement, Feb. 6, 1965 & Financial Express, Supplement Feb. 6, 1965.

40. Technological Innovations to Improve Iron Blast Furnace Productivity.— B. R. Nijhawan.— Economic Times Supplement, Feb. 6, 1965.

41. Micro-Metallurgy: Scope of Research at National Metallurgical Laboratory.— B. R. Nijhawan.— Economic Times Supplement 28.3.65 & Financial Express Supplement 28.3.65.

42. Effect of Trace Elements on Age-Hardening of Some Aluminium Base Alloys.— Ved Prakash. — Economic Times Supplement, 28.3.65.

43. Study of Micro-addition to Aluminium-Magnesium Wrought Alloys.— Y. N. Trehan, S. P. Bhadra, P. K. Gupte & B. R. Nijhawan — Economic Times Supplement, 28.3.65.

44. Spectrographic Analysis of Micro-elements in Metals and Alloys.— M. K. Ghosh, P. C. Debnath, Sumitra Das Gupta, B. C. Kar — Economic Times Supplement 28.3.65.

45. Micro-Metallurgy of Austenitic Grain Size Control of Steel — A. B. Chatterjea & B. R. Nijhawan — Economic Times Supplement 28.3.65.

46. Distribution and Function of Micro-phases and Micro-addition in Metals and Alloys. — Rajendra Kumar & L. J. Balasundaram.— Financial Express Supplement, 28.3.65.

47. Growth of Foundry Industry in India — B. R. Nijhawan — Indian and Eastern Engineer, Annual No. 1965.

48. Blast Furnace Operation with Oxygen Enriched Blast.— A. B. Chatterjea — Indian Industries Annual, 1965.

49. Iron Production in Low Shaft Small Blast Furnace.— B. R. Nijhawan — Science Reporter 1 (2), 1964.

50. Effect of Addition of Sodium Nitrate on Corrosion of Mild Steel in Fused and Concentrated Solutions of Caustic Soda.— S. Rao Addanki, A. K. Lahiri & T. Banerjee.— Iron & Steel (London), 37, April 1964.

51. Role of Research in Standardization — B. R. Nijhawan.— ISI Special Supplement. Nov. 9, 1964.

52. Role of Research in Iron & Steel Industry.— B. R. Nijhawan — Eastern Metals Review, Annual Number, Feb. 1965.

The following papers were presented at the Symposium on 'Micro-Metallurgy — The Role of Minute Additions to Ferrous and Non-Ferrous Metals and Alloys' held in March-April 1965.

53. Scope of Research in Micro-Metallurgy at the National Metallurgical Laboratory.— B. R. Nijhawan.

54. Micro-Metallurgy of Abnormality in Steels.— B. R. Nijhawan, A. B. Chatterjea & S. S. Bhatnagar.

55. Micro-Metallurgy of Austenitic Grain Size Control of Steels.— A. B. Chatterjea & B. R. Nijhawan.

56. Distribution and Function of Micro-phases and Micro-additions in Metals and Alloys.— L. J. Balasundaram & Rajendra Kumar.

57. Surface Tension of Cast Iron with Additions of Different Elements.— Jatinder Mohan.

58. Effect of Trace Elements on Ageing of Some Aluminium-base Alloys.— Ved Prakash.

59. Study of Micro-additions to Al-Mg Wrought Alloys.— Y. N. Trehan, S. P. Bhadra, P. K. Gupte & B. R. Nijhawan.

60. Some Observations on Recent Methods of Analysis of Gases in Metals and Alloys.— N. G. Banerjee.

61. Spectrographic Analysis of Micro-elements in Metals and Alloys.— M. K. Ghosh, P. C. Debnath, Miss S. Das Gupta & B. C. Kar.

The following papers were presented at the 18th Annual Technical Meeting of the Indian Institute of Metals, November 1964.

62. Development of Iron-Aluminium Alloys.— S. M. Arora, S. S. Bhatnagar, P. K. Gupte & B. R. Nijhawan.

63. Structure of Liquid Lead-Tin alloys.— Rajendra Kumar.

64. A Phosphoric-Sulphuric-Nitric Acid Bath for Chemical Polishing of Aluminium.— M. S. Mahanty, S. S. Bhatnagar, P. K. Gupte & B. R. Nijhawan.

65. Thermodynamic Properties of Liquid Metals.— G. Mishra & Rajendra Kumar.

66. Economic Productivity in Iron & Steel Making.— B. R. Nijhawan.

The following papers were presented at the Convention of Indian Ceramic Society, April 1965.

67. Development of Superduty Silica Bricks with Indigenous Raw Materials.— Gurbux Singh Minhas & H. V. Bhaskar Rao.

68. Occurrence and Refractory Properties of Rajasthan Magnesite.— P. C. Sen, Ashimesh Dutt, M. R. K. Rao & H. V. Bhaskar Rao.

69. Investigation of Some Refractory Clays from Jammu and Kashmir.— N. S. Sahota, T. V. Prasad & H. V. Bhaskar Rao.

70. Pilot Plant Studies on the Production of Forsterite Refractories from Indigenous Raw Materials.— H. P. S. Murthy, M. C. Kundra, P. C. Sen & H. V. Bhaskar Rao.

71. Comparative Studies on High Temperature Coke Oven Coal Pitch as a Binder in the Manufacture of Carbon Refractories and Compacts.— H. P. S. Murthy & Bimal Chatterjee.

72. The Role of Monovalent and Divalent Cations in Dehydrorelation of Kaolinite.— P. Prabhakaram.

73. Mineralogy of Some Indian Kaolinitic Clays and Crystallinity of Kaolinite.— P. Prabhakaram.

The following papers (Sl. No. 74 to 81) were presented at the Convention of Indian Institute of Foundrymen, February 1965.

74. Effect of Quenching at the Duration of Eutectic Arrest on the Size of the Eutectic Cell.— Jatinder Mohan.

75. Investigation of Carbon Saturation Normal Cupola Practice.— J. Goswami, A. B. Chatterjea & Jatinder Mohan.

76. Alternative Solid Fuels for Melting Iron in Cupola.— Jatinder Mohan, J. Goswami, A. B. Chatterjea & B. R. Nijhawan.

77. Drying Oils and Its Influence on the Properties of Oil Bonded Sand Mixtures in Foundries.— A. T. Chirayath & Jatinder Mohan.

78. Expansion Characteristics of Indigenous Sands.— V. S. Bhandary, P. K. Gupte & B. R. Nijhawan.

79. Particle Size Determination of Indigenous Clay and Their Properties.— T. A. Beck, V. S. Bhandary & P. K. Gupte.

80. Dephosphorization of Indian Pig Iron in Basic Lined Cupola.— R. K. Dubey, C. A. Naresh Rao, S. S. Bhatnagar & P. K. Gupte.

81. Utilization of T.T.T. Curves for Heat Treatment of Alloy Steel Casting.— R. K. Dubey, P. K. Gupte & B. R. Nijhawan.

82. Iron Smelting with Non-coking Coals in Low Shaft Furnace Pilot Plant.— B. R. Nijhawan & A. B. Chatterjea — Presented at the 50th Annual Technical Meeting of the Iron & Steel Institute of Japan, April 1965.

83. Scope of Research and Development Work on Extraction Metallurgy at the National Metallurgical Laboratory.— B. R. Nijhawan & P. P. Bhatnagar.— Presented at the Eighth Commonwealth Mining & Metallurgical Congress held at Australia, 1964.

84. A New Method of Modification of Aluminium-Silicon Alloys.— B. R. Nijhawan & S. S. Bhatnagar — Presented at the 31st International Foundry Congress held at Amsterdam, September 1964.

85. Factors in the Design and Development of Prototype Plant and Equipment for Metallurgical and Related Processes.— B. R. Nijhawan & M. J. Sahani — Presented at the Symposium on Engineering Design and Development at Indian Institute of Technology, Kharagpur, November 1964.

86. Scope for Port-based Iron and Steel Works in India.— B. R. Nijhawan & C. Sharma — Presented at the Symposium on 'The Desirability of having Port-based Iron & Steel Works in the Country' Organized by Mining, Geological & Metallurgical Institute of India, March 1965.

87. Translation of Technical Literature in the field of Metallurgy.— J. E. Mannar.— Presented at the Seminar on 'Technical Translation' held under the auspices of Insdoc, April 1965.

88. Auxiliary Fuel Injection in Iron Blast Furnace.— A. B. Chatterjea & B. R. Nijhawan — Financial Express Supplement & Economic Times Supplement, 6 February 1965.

89. Scope for Iron Smelting Plant for the Production of Foundry Grade Pig Iron in Punjab.— A. B. Chatterjea & B. R. Nijhawan — Economic Times Supplement, 10 April 1964.

90. Cupola Operation with Jhama Coal.— A. B. Chatterjea, Jatinder Mohan, J. Goswami & B. R. Nijhawan — Financial Express Supplement, 10 April 1964.

91. Recent Trends in Iron Ore Reduction, Pt. I.— A. B. Chatterjea — Blast Furnace & Steel Plant, May 1964.

92. Recent Trends in Iron Ore Reduction, Pt. II.— A. B. Chatterjea — Blast Furnace & Steel Plant, June 1964.

93. Blast Furnace Performance with Prepared Burden.— A. B. Chatterjea — Jr. of Iron & Steel Engineering 5(1), November 1964.

APPENDIX II

SCIENTIFIC INVESTIGATIONS COMPLETED AND REPORTS PREPARED

1. Pilot Plant Studies on Beneficiation and Sintering of a Mixed Iron Ore Sample from (1) Hill No. 1, Bench No. 2 and (2) Hill No. 2, Bench No. 2 of Kiruburu Iron Ore Mines of National Mineral Development Corporation.— A. Peravadhanulu, Y. A. Joglekar, S. B. Das Gupta, G. S. Ramakrishna Rao, G. P. Mathur & P. I. A. Narayanan (I.R. 288/64).
2. Further Pilot Plant Beneficiation Studies on Limestone Sample from Tisco and Proposals for Setting up a 300 tons/day Treatment Plant.— G. S. Ramakrishna Rao, A. Peravadhanulu, B. L. Sen Gupta, M. V. Ranganathan, S. B. Das Gupta, P. V. Raman, G. P. Mathur & P. I. A. Narayanan (I.R. 289/64).
3. Beneficiation of a Gypsum Sample from Messrs Bikaner Gypsums Ltd., Rajasthan. — S. K. Banerjee, G. V. Subramanya & P. I. A. Narayanan (I.R. 290/64).
4. Beneficiation of Coarse Grained Fluorspar Sample from Ambadongar, Baroda.— S. K. Dhar, G. V. Subramanya & P. I. A. Narayanan (I.R. 291/64).
5. Moulding Characteristics of Rajmahal Silica Sand.— A. Das, V. S. Bhandary & P. K. Gupte (I.R. 292/64).
6. Moulding Characteristics of Rajendra Silica Sand No. 1.— S. K. Sinha Babu, V. S. Bhandary & P. K. Gupte (I.R. 293/64).
7. Investigation Rajendra Silica Sand No. 3.— P. R. Sastry, V. S. Bhandary & P. K. Gupte (I.R. 294/64).
8. Moulding Characteristics of Rajendra Silica Sand No. 2.— S. K. Sinha Babu, V. S. Bhandary & P. K. Gupte (I.R. 295/64).
9. Moulding Characteristics of Gundiposi Sand Received for G.S.I., Bihar Circle.— Amitava Das, V. S. Bhandary & P. K. Gupte (I.R. 296/64).
10. Pilot Plant Studies on Beneficiation and Agglomeration of a Low Grade Limestone Sample from Purnapani Quarries, Orissa, for Hindustan Steel Ltd.— Y. A. Joglekar M. V. Ranganathan, B. L. Sengupta, R. K. Kunwar, S. B. Das Gupta, G. S. Ramakrishna Rao, G. P. Mathur & P. I. A. Narayanan (I.R. 297/64).
11. Washing of Selenite from Gypsum, Rajasthan.— P. V. Raman, G. V. Subramanya & P. I. A. Narayanan (I.R. 298/64).
12. Pilot Plant Beneficiation Studies on a Gypsum Sample from Messrs Bikaner Gypsum Ltd., Rajasthan.— M. V. Ranganathan, Y. A. Joglekar, S. B. Das Gupta, G. S. Ramakrishna Rao, G. P. Mathur & P. I. A. Narayanan (I.R. 299/64).

13. Washing of a Selenite sample from Barmer District, Rajasthan.— P. V. Raman, G. V. Subramanya & P. I. A. Narayanan (I.R. 300/64).

14. Pilot Plant Studies on Concentration of Economic Minerals from Manavalakurichi Beach Sand.— N. Chakravorty & P. I. A. Narayanan (I.R. 301/64).

15. Development of Superduty Silica Bricks with Indigenous Raw Materials.— Gurbux Singh Minhas & H. V. Bhaskar Rao (I.R. 302/64).

16. A Comparative Study of Insulation Refractories with Special References to Thermal Conductivity.— B. M. Dutt & H. V. Bhaskar Rao (I.R. 303/64).

17. Study of the Distribution of Hematite in the Band I, Kanjamalai Magnetite Deposit, Salem Dist., Madras.— A. Peravadhanulu, P. D. Prasada Rao, M. C. Chopra & P. I. A. Narayanan (I.R. 304/64).

18. Beneficiation of a Low Grade Manganese Ore from Barajamda Area, Singhbhum Dist., Bihar.— S. K. Banerjee, G. V. Subramanya & P. I. A. Narayanan (I.R. 305/64).

19. Reduction of Silica Content in a Limestone Sample from Rajpipla, Narmada Project. — S. K. Dhar, G. V. Subramanya & P. I. A. Narayanan (I.R. 306/64).

20. Pilot Plant Studies on Beneficiation of Limestone Samples from Nandini Mines of Bhilai Steel Plant, Hindustan Steel Ltd.— M. V. Ranganathan, Y. A. Joglekar, R. Kunwar, A. Peravandhanulu, G. S. Ramakrishna Rao, G. P. Mathur & P. I. A. Narayanan (I.R. 307/64).

21. Damodar River Sand from near Durgapur Barrage Site.— General Metallurgy Division (I.R. 308/64).

22. Studies on the Production of High Basicity Sinters from the Beneficiated Bolani Iron Ore Fines for the Durgapur Steel Plant.— P. K. Sinha, G. V. Subramanya & P. I. A. Narayanan (I.R. 309/64).

23. Beneficiation and Sintering Studies with an Iron Ore Sample from Bailadila Mines of National Mineral Development Corporation.— M. V. Ranganathan, A. Peravadunulu, Y. A. Jogelkar, G. S. Ramakrishna Rao, G. P. Mathur & P. I. A. Narayanan (I.R. 310/64).

24. Studies on Leaching and Ion Exchange Process for the Improvement of Colour of Washed China Clay Sample from Karanjia Quarries, Chaibasa.— R. Ganesh, G. V. Subramanya & P. I. A. Narayanan (I.R. 311/65).

25. Recovery of Metallics from Zinc Ash from Messrs Indian Tube Co. (1953) Ltd., Jamshedpur.— B. V. S. Yedavalli & P. I. A. Narayanan (I.R. 312/65).

26. Moulding Characteristics of Shertallai Silica Sand.— P. R. Sastry, V. S. Bhandary & P. K. Gupte (I.R. 313/65).

27. Moulding Characteristics of Godavari River Sand No. 2.— S. K. Sinhababu, V. S. Bhandary & P. K. Gupte (I.R. 314/65).

28. Investigation on the U.B.I. (Kashmir) Bentonite Received from Director of Mines & Geology, Jammu & Kashmir Govt.— Autar Singh, V. S. Bhandary & P. K. Gupte (I.R. 315/65).

29. Investigation on Rajendra Silica Sand No. 4 Received from M/s. Rajendra Silica Works, Sankargarh, Allahabad.— P. R. Sastry, V. S. Bhandary & P. K. Gupte (I.R. 316/65).

30. Beneficiation & Pelletization of Iron Ore Fines from M/s. Salgaocar E Irmao Ltd., Goa.— B. V. S. Yadavalli, S. K. Dhar & P. I. A. Narayanan (I.R. 317/65).

31. Investigation on the Generation of Fines from Kiruburu Iron Ore during Transport and Handling.— S. K. Dhar, G. P. Mathur & P. I. A. Narayanan (I.R. 318/65).

32. Beneficiation of Specular Hematite Samples from Rajasthan — Part II — Sample from Daralmata, Udaipur Dist., Rajasthan. — S. K. Banerjee, K. N. Rakshit & P. I. A. Narayanan (I.R. 319/65).

33. Beneficiation of Specular Hematite Samples from Rajasthan — Part I — Samples from Amsiwali, Udaipur Dist., Rajasthan.— P. V. Raman & P. I. A. Narayanan (I.R. 320/65).

34. Reducibility of Lumpy Iron Ores from M/s. Salgaocar E Irmao Ltd., Goa.— M. Subramanian, S. B. Mathur & P. P. Bhatnagar (I.R. 321/65).

35. Some Observations on Stress Corrosion Cracking of Hardenable Stainless Steel.— A. K. Lahiri, T. Banerjee & B. R. Nijhawan (R.R. 173/64).

36. Some Observations on Tests to Determine Corrosivity of Solder Flux Residues.— K. P. Mukherjee & A. K. Lahiri (R.R. 174/64).

37. Study of Eutectic Cells with Addition of Different Elements.— Jatinder Mohan (R.R. 175/64).

38. Cupola Operation with Jhama Coal as a Substitute for Foundry Coke.— Jatinder Mohan, J. Goswami, A. B. Chatterjea & B. R. Nijhawan (R.R. 176/64).

39. Defferential Thermograms of Certain Indian Bentonite Clays.— A. T. Chirayath & J. Mohan (R.R. 177/64).

40. Importance of Adsorbed Water on Clay Surface in Liquid Limit Determination.— T. A. Beck, A. T. Chirayath, V. S. Bhandary & P. K. Gupte (R.R. 178/64).

41. Activation of Bakudi Bentonite.— T. A. Beck, V. S. Bhandary & P. K. Gupte (R.R. 179/64).

42. Effect of Sulphur on the Properties and Microstructure of Cast Iron.— B. V. Somayajulu (Late), P. K. Gupte & B. R. Nijhawan (R.R. 180/64).

43. Thermal Dehydroxylation and Phase Transformation of Bayer's Alumina.— P. C. Sen, Ashimesh Dutt & H. V. Bhaskar Rao (R.R. 181/64).

44. Structure of Liquid Lead-Tin Alloys.— Rajendra Kumar (R.R. 182/64).

45. Development of Iron-Aluminium Alloys.— S. M. Arora, S. S. Bhatnagar, P. K. Gupte & B. R. Nijhawan (R.R. 183/64).

46. A Phosphoric-Sulphuric-Nitric Acid Bath for Chemical Polishing of Aluminium.— M. S. Mahanty, S. S. Bhatnagar, P. K. Gupte & B. R. Nijhawan (R.R. 184/64).

47. Production of Low Residual and Soft Iron.— R. D. Gupta, P. K. Gupte & B. R. Nijhawan (R.R. 185/64).

48. Development of Aqueous Non-sludge Forming Zinc Phosphate Bath.— K. P. Mukherjee, & A. K. Lahiri (R.R. 186/64).

49. Curie Temperature of Iron Alloys.— A. N. Sinha & L. J. Balasundaram (R.R. 187/65).

50. Studies on Properties of Rust Formed during Atmospheric Corrosion.— A. K. Lahiri, K. P. Mukherjee, S. Rao Addanki & T. Banerjee (R.R. 188/65).

51. Mineralogy of Some Indian Kaolinitic Clays and Crystallinity of Kaolinite.— P. Prabhakaram (R.R. 189/65).

52. The Role of Monovalent and Divalent Cations in the Dehydroxylation of Kaolinite.— P. Prabhakaram (R.R. 190/65).

53. Phase-transformation in Electro-deposited Copper Cadmium Alloys.— P. L. Ahuja & T. Banerjee (R.R. 191/65).

54. Effect of Quenching at the Duration of Eutectic Arrest on the Size of Eutectic Cell.— Jatinder Mohan (R.R. 192/65).

55. Alternative Solid Fuels for Melting Iron in Cupola.— Jatinder Mohan, J. Goswami, A. B. Chatterjea & B. R. Nijhawan (R.R. 193/65).

56. Investigation of Carbon Saturation in Normal Cupola Practice.— J. Goswami, A. B. Chatterjea & Jatinder Mohan (R.R. 194/65).

APPENDIX III

MAJOR SPONSORED PROJECTS AT THE NATIONAL METALLURGICAL LABORATORY

Sl.	*Title*	*Sponsor*
1.	Pilot plant studies on beneficiation and sintering of a mixed iron ore sample from (i) Hill No. 1, Bench No. 2 and (ii) Hill No. 2, Bench No. 2 of Kiriburu iron ore mines of National Mineral Development Corporation.	Bokaro Steel Ltd.
2.	Investigation on the generation of fines from Kiriburu iron ore during transport and handling.	National Mineral Development Corporation Ltd.
3.	Pilot plant studies on the beneficiation, sintering studies on a mixed iron ore sample from Kondakesa block of Dalli Pahar Deposits.	Hindustan Steel Ltd.
4.	Pilot plant studies on beneficiation and agglomeration of a low grade limestone sample from Purnapani quarries, Orissa, of Hindusthan Steel Ltd.	Hindustan Steel Ltd.
5.	Beneficiation of specular hematite samples from Rajasthan: Part I — Samples from Amsiwali, Udaipur Dist., Rajasthan.	Kamani Engineering Corporation.
6.	Beneficiation of specular hematite samples from Rajasthan: Part II — Samples from Daralmata, Udaipur Dist., Rajasthan.	Kamani Engineering Corporation.
7.	Reducibility of specular hematite iron ore samples of Nathara-ki-Pal deposit, Rajasthan.	Kamani Engineering Corporation.
8.	Beneficiation & Pelletization of iron ore fines from M/s. Salgaocar E Irmao Ltda.	Salgaocar E Irmao Ltda.
9.	Reducibility of lumpy iron ores from Goa.	Salgaocar E Irmao Ltda.