ANNUAL REPORT
1963-1964

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

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FOREWORD

I HAVE great pleasure in presenting the Report of the Director, National Metallurgical Laboratory, for the year 1963-64.

During the period under review, the major expansion programme envisaged during the Third Five Year Plan by the National Metallurgical Laboratory relating to the installation of the integrated Mineral Beneficiation Pilot Plant, the Ferro-alloy Pilot Plant, the construction of Side and Main Wings of the Main Building of the Laboratory, has been completed. With its interest in applied and basic metallurgical research work as commented by the Council of Scientific & Industrial Research Reviewing Committee, the National Metallurgical Laboratory has forged close links and won the confidence of the Indian Mineral and Metallurgical Industries. The Laboratory has achieved outstanding success in multiple fields and has been the main Research Laboratory for the iron and steel industry.

The Reviewing Committee of the Council of Scientific & Industrial Research headed by Sir A. Ramaswami Mudaliar spent some time at the National Metallurgical Laboratory and studied the developments in research and pilot plant activities of the Laboratory. The members of the C.S.I.R. Reviewing Committee and its Chairman have given high praise to the National Metallurgical Laboratory and their Report has acknowledged the outstanding and dynamic work of the National Metallurgical Laboratory whilst recommending further stepping up of research and pilot plant activities in relation to non-ferrous metal industry and preparation of Design and Project Report.

The National Metallurgical Laboratory has continued to actively consolidate the scope of its research and development activities including pilot plant scale trials in direct relation to Indian Metallurgical Industries specific requirements.

Research and development work was undertaken at the National Metallurgical Laboratory for developing suitable coinage alloys particularly for lower denominations based on indigenous alloying elements and to the complete exclusion of the use of imported copper and nickel. It was as a result of the collaborative work undertaken by the National Metallurgical Laboratory with the Government of India Mint at Bombay that the Government of India has recently accepted NML's conclusive findings on full scale processing and fabrication trials and have decided that the 3 paisa coins should be minted from aluminium-magnesium alloys.

A Report on the " Substitution of Imported Copper and other Non-Ferrous Metals by Aluminium and other Indigenous Metals " prepared by Dr. B. R. Nijhawan and Mr. K. N. P. Rao was issued last year by the National Metallurgical Laboratory. The Directorate General of Technical Development of Government of India who examined this Report, have commended it as a comprehensive Report containing valuable recommendations on the subject of substitution of non-ferrous metals and which has a direct bearing on the industry. The full Report of the National Productivity Team on Non-Ferrous Metal Industry which visited U.S.A. and the U.K. with Dr. B. R. Nijhawan, Director, National Metallurgical Laboratory, as the Leader, will be released in the near future. The National Metallurgical Laboratory has been progressively increasing the scope of its work and related research activities in relation to non-ferrous metals and alloys.

Important research projects relating to the development of substitute alloys and stainless steels have continued to make significant progress — the projects relating to the production of nickel-free stainless steel, nickel and cobalt-free heating elements, production of bi-metals, development of indigenous refractories including graphite crucibles, production of high purity iron powder, alumino-thermic ferro-alloys, etc. In some of these fields, the work of the National Metallurgical Laboratory is ahead of many other research institutions the world over.

Pari passu with the applied development research projects including sustained operations of its several Pilot Plants, the National Metallurgical Laboratory has, during the period under review, greatly intensified its research activities in relation to fundamental basic objective research schemes. There are at present over 14 Research Fellows working on different problems and thereby become eligible for higher degrees including the Ph.D. degree at some of the Indian Universities. The number of Ph.D. degrees awarded during the last two years based on work of basic objective metallurgical research has increased.

Work on various projects undertaken on behalf of Railway Research, Designs & Standards Organization, Railway Electrification etc. has maintained steady progress. Investigations on the corrosion of rails used in Indian Railway tracks were undertaken to investigate possible causes of excessive corrosion of rails, viz. their composition and environmental conditions etc. A long range collaborative research programme for the development of corrosion resistant rail steel is now under way. Further work for the aluminizing of a number of steel and malleable cast iron hardware items was undertaken in NML Hot-dip Aluminizing Pilot Plant and the aluminized materials were supplied to the Railway Electrification authorities for their field trials.

On behalf of Indian Standards Institution, National Metallurgical Laboratory has conducted extended investigations on standard Rajendra silica sand sample, Rajmahal silica sand, Ennore silica sand, Shertallai sand and Neyveli sands as also investigations on fireclay and magnesite stoppers and nozzles samples collected from different iron and steel plants of India to prepare Indian Standards thereon. The 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 National Metallurgical Laboratory which is acting as the coordinating Laboratory.

Extensive smelting trials were conducted with raw materials from various State Governments. Based on the Investigation Project Report submitted by the National Metallurgical Laboratory, the Government of Punjab in collaboration with the Central Government is taking steps to establish a 100,000 tons/annum capacity pig iron Plant. Similar action is under way for the Andhra Pradesh Government.

The National Metallurgical Laboratory has established the first Regional Foundry Station at Batala which is functioning to the great benefit of the local foundries. Similar Regional Foundry Stations are shortly to be opened in Howrah and Madras and the fourth one at Ahmedabad. In the field of corrosion research studies, the Marine Corrosion Research Station at Digha in West Bengal will be opened in the near future and will be a landmark in corrosion research studies in this country.

It is gratifying to state that out of twenty-five processes ready for commercial exploitation, twenty-one processes have already been released to the industry.

The *NML Technical Journal* has entered into the sixth year of publication and is maintaining a high standard as one of the leading journals in metallurgy.

A Symposium on 'Utilization of Metallurgical Wastes' was arranged in March 1964. Like other International Symposia organized by the National Metallurgical Laboratory earlier, this Symposium too drew delegates from all over the world, besides India, and offered stimulation of technical and research ideas and practical initiations to important technical themes.

The Proceedings of the International Symposium on 'Recent Developments in Iron and Steel Making with Special Reference to Indian Conditions' held in February 1963 have just been released by the British Iron & Steel Institute covering the technical papers and the discussions that followed during the above Symposium organized by the National Metallurgical Laboratory in coordination with the Indian Institute of Metals and the British Iron & Steel Institute. This Symposium publication would be a valuable Reference Text on the subject and has been most commendably reviewed in the world's technical press.

I do not know of any other single laboratory including those in U.S.A. and Europe which possess the multifarious types of research and pilot plant equipment for work on mineral and metal industries under one roof. The progress achieved in the different directions at the National Metallurgical Laboratory reflects the high efficiency of its operation in basic and applied fields.

The National Metallurgical Laboratory is fortunate in being headed by a dynamic Director, Dr. B. R. Nijhawan. On my behalf and that of the Members of the Executive Council, I would like to express our sincere appreciation of the sustained and devoted work that is being done under his able guidance. The Members of the Executive Council of the National Metallurgical Laboratory deeply appreciate the highly creditable and praiseworthy efforts. Much of the Laboratory's ever-widening scope of research and pilot plant activities is undoubtedly due to the hard work, research and organization ability of its Director, Dr. B. R. Nijhawan.

The National Metallurgical Laboratory will undergo further expansion during the Fourth Five Year Plan. I have no doubt, that the commendable success already achieved augurs well for the future of the National Metallurgical Laboratory.

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

October 30, 1964

INTRODUCTION

THE progress of research and development work at the National Metallurgical Laboratory during the year under review has been steady and rewarding. Besides consolidation of research activities in diverse fields, the expansion has covered the establishment of Regional Research Stations on corrosion studies and foundry technology. The integrated Mineral Beneficiation Pilot Plant and the Low-Shaft Furnace Pilot Plant of the National Metallurgical Laboratory were actively engaged during the year on Industrial Research Projects for the Public and Private Sectors; the former worked on schedule for the iron and steel industry's raw-materials' studies including agglomeration, pelletizing etc. for Goa iron ore mines after prior optimum beneficiation as also for the National Mineral Development Corporation, the Hindustan Steel Company, Bokaro Steel Project. Work on Khetri Copper Project claimed its due share in these research investigations. For the Salem Steel Plant, Pilot Plant investigations were undertaken on behalf of the Consultants to the Salem Steel Plant.

Sir Jehangir Ghandy, Director-in-Charge, Tata Iron & Steel Company Limited, recently in delivering his Address at the Defence Staff College, Wellington, aptly summarized the problem in relation to Indian iron ores in general and that of the Tata Iron & Steel Works in particular.

" Take iron ore, for example. We have an abundance of ore deposits which may last for several hundred years even on the most optimistic assumptions of home demand, but some problems of quality are arising since the growing volume of requirements can no longer be met by selective small scale mining. I must frankly admit that all of us, in the private or public sector plants, failed to foresee the problem of high alumina in the ore, largely because we were rushed into an expansion of steel capacity at somewhat short notice at the beginning of the Second Plan. Serious operating difficulties have had to be faced as a result, and even now this problem is not fully solved. But we are well along towards the goal, thanks to the help rendered by the National Metallurgical Laboratory working in concert with men from the steel plants of Jamshedpur and Bhilai. The time taken by the investigations underlines the intractability of many of the mineral problems and provides an added argument for preparatory work and investigations to be begun in earnest well in advance".

Although the National Metallurgical Laboratory is actively engaged in meeting the demands of the heavy metallurgical industry and the integrated iron and steel complexes, the requirements of medium and small scale mineral and metal industries have been equally well attended to. The National Metallurgical Laboratory has extended the scope of research and development work by the setting up of Regional Research Stations such as the Regional Foundry Stations in different parts of the country and Marine Corrosion Research Station at Digha; the latter has now been completed and has initiated research studies on marine corrosion on a comprehensive scientific basis. Likewise, the establishment of Regional Foundry Research Station at Batala in the Punjab has been completed; the station is doing exceedingly valuable work as acknowledged so well by the Punjab Foundry Industry. Likewise, the Regional

Foundry Stations in Madras, Howrah and Ahmedabad are being established. The utility of the Regional Foundry Research Station has been truly appreciated by the Foundry Industry and there are persistent demands for the establishment of similar Foundry Research Stations in other parts of the country.

An Iron production plant has now been projected by the Punjab Government for the production of 100,000 tons per year of foundry grades of pig iron in which the National Metallurgical Laboratory has most actively collaborated. It is heartening to see the results of the researches by the National Metallurgical Laboratory taking concrete shape in the form of full scale Industrial Plants.

The collaborative research and development work of the National Metallurgical Laboratory with the Government of India Mint, Bombay, has yielded concrete results and the 3 Paisa denomination coin in aluminium-magnesium composition developed by the National Metallurgical Laboratory has been accepted by the Government of India.

Top priority was accorded to various research and development Projects undertaken for Defence organizations and Ordnance Plants and to Projects covering the production of specialized alloys for the Defence Ministry.

During the period under review, plans have been worked out for the establishment of a pilot-cum-industrial proto-type plant for the production of 250 tons of magnesium per year by silico-thermal vacuum reduction of dolomite on the basis of technical know-how developed at the National Metallurgical Laboratory over the last couple of years. This project is being handled by the National Metallurgical Laboratory jointly with the Central Design & Engineering Unit of the Council of Scientific & Industrial Research.

Various short-term and long-range projects have been taken up at the National Metallurgical Laboratory on behalf of Indian Railways and successful results obtained. The National Metallurgical Laboratory has developed the industrial scale production technique for high purity low residual iron ideally suitable to replace the imported Armco iron; the Project has been completed.

With a view to utilize surplus light 'naphtha' from the Petroleum refineries as blast additive for economizing fuel rate, the incorporation of fuel injection in the low-shaft furnace is under way. As light naphtha injection trials for iron smelting, it is gathered, have not been conducted elsewhere, the National Metallurgical Laboratory will perhaps be doing pioneering investigations in this field.

Investigations on pneumatic steel-making in basic-lined side-blown air converter employing hot metal of low and high silicon contents, with or without additions of ferro-silicon demonstrated total metallurgical feasibility of producing steels of medium and low carbon contents with phosphorus within the specified limits. The nitrogen contents of the steel made varied from 0·003 to 0·006 per cent; mechanical properties of the steels conformed to requisite specifications.

The National Metallurgical Laboratory has been offering active coordination to the Indian Standards Institution by way of undertaking short-term and long-range projects for standardization of different metallurgical projects. Research and development work on standardization of fireclay stoppers and magnesite nozzles for steel has already made much progress and tests initiated on samples from all over the country.

At the request of the Foundry Sectional Committee of the Indian Standards Institution, investigations on silica sands as available in the country for foundry purposes have been taken up. The sand samples from different parts of India are being sent by Geological Survey of India and besides testing samples, the National Metallurgical

Laboratory will also act as a coordinating Institute for making necessary arrangements for testing of those samples simultaneously in five other institutions.

The work done on the foaming of low-shaft furnace slag to find out its suitability for use as light weight aggregate for insulation concrete has given very encouraging results. Concrete made with foamed slag aggregates is lighter in weight, more insulating and as strong as conventional concrete. The work on the use of low-shaft furnace expanded slag for structural concrete is under progress. Further work on determination of foaming characteristics of Indian blast furnace slag by the help of mechanically operated foaming bed has also been taken up and designing and fabrication of foaming bed are in early stages. Granulated blast furnace slag has also been obtained from some of the steel plants and investigation initiated on the effect of MnO on the hydraulic properties of slag for cement making. Likewise air cooled blast furnace slags from various steel plants in public as well as private sectors have also been produced and study on the slag system is in progress by employing hot stage microscopy in still as well as movie form depicting the crystal formation, etc. As a part of the broad-based research programme on utilization of steel plant slags, LD and Open-hearth slags from different steel plants are being obtained for carrying out detailed investigation on their suitability for fertilizers.

Research studies on fundamental scientific subjects in physical metallurgy, general and mechanical metallurgy in relation to liquid metals, studies of precipitated nitrides in alloy steels, age hardening, isothermal transformation studies, phase equilibria studies, activity measurements and gas solubility studies, etc., have kept pace *pari-passu* with applied development projects. When adequate funds are available, it would be necessary to establish a division for research and development specifically on non-ferrous metals and alloys in the National Metallurgical Laboratory on the same basis as the Iron and Steel and Alloy Steel Divisions. Whilst the expansion and stepping up of work in the above directions will be taken up during the end of the Third Five Year Plan and early part of the Fourth Five Year Plan, the tempo of work nevertheless on the above subjects has been progressively stepped up at the National Metallurgical Laboratory. Likewise, suitable stepping up of the work and additions to the facilities provided in the Design and Project Division of the National Metallurgical Laboratory will be implemented with the availability of suitable funds during the end of the current Plan period and possibly more so in the Fourth Five Year Plan. In view of the paucity of funds and the emergency prevailing, any addition to the facilities in relation to the above subjects is not being pressed for at this stage but suitable action thereon will no doubt be taken at the appropriate time. The Third Reviewing Committee of the Council of Scientific & Industrial Research which studied the work of the National Metallurgical Laboratory has referred to the dynamic work of the National Metallurgical Laboratory on applied projects which has enabled the Laboratory to forge close links and win the confidence of the metallurgical industry. The full Report of the C.S.I.R. Reviewing Committee is awaited with great interest. The National Metallurgical Laboratory has been laying considerable stress during the last few years on basic objective research and has also been asking for increased number of Senior and Junior Fellows who can besides others, well undertake objective basic research on suitable subjects and also thereby become eligible for higher degrees, including the Ph.D. degree at some of the Indian Universities. At the same time, work at the National Metallurgical Laboratory in the above directions has been considerably stepped up in recent years. The number of Ph.D. degrees awarded during the last couple of years has progressively increased at the National Metallurgical Laboratory based on work of basic objective metallurgical research, the Theses in each case having been examined by leading scientists and metallurgists overseas and at home for the degrees of Ph.D. based on Theses on the following subjects:

1. Low nickel and nickel substantive-free stainless steel.
2. Phase-transformation in electrolytic alloys with special reference to copper-cadmium alloys deposited at constant cathode potential.
3. Studies on age hardening.
4. Development of oxide refractories from beach sands of Kerala.

Additionally, Theses on the following subjects have recently been submitted to the different Universities for the award of Ph.D. and higher degrees:

1. Studies on the phase transformation in alloys by x-ray diffraction and other techniques.
2. Studies in ion-exchange of clays.
3. Transformation characteristics of alloy steels.
4. Chemical and metallurgical factors affecting failure of metals and alloys under combined action of stress and corrosion.
5. Studies on the morphology of phosphorus in Indian manganese ores.

At the same time, others have registered or have applied for registration at different Universities and Indian Institutes of Technology for the degrees of Ph.D. based on research work on the following subjects of basic metallurgical research interest:

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-tin-alloys.
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. Chemical and metallurgical factors affecting the failure of metals and alloys under the combined action of stress and corrosion.
14. Corrosion resistance and metallurgical characteristics of substitute stainless steels in relation to chemical composition and heat-treatment.
15. Studies on the structure of electro-deposited manganese.
16. High temperature ceramics coating on metal or high temperature alloys.
17. Viscosities of CaO-MgO-Al_2O_3-SiO_2 slags.
18. Phase transformation in alloy steels.
19. Plastic coating of steel.

As a part of the expansion programme of the National Metallurgical Laboratory, the three side wings of the Main Building Laboratory were fully commissioned during the period under review. Requisite plant, machinery and research equipment have now been installed; the main items of equipment installed in these wings relate to vacuum and air melting equipment, magnesium Pilot Plant, Electronic research and Instrumentation Division, X-ray fluorescence spectroscope, vibrophore pulsator set up; the side wings now house the Extraction Metallurgy Division, the Iron and Steel and Alloy Steel Division, mineralogical and petrological sections, work on slag projects and Refractories Division's fundamental research section whilst the

Central Wing contains the Library, the full fledged Liaison and Information Division and Design Engineering Division etc.

An International Symposium was organized by the National Metallurgical Laboratory from 10th to 13th March, 1964 on 'Utilization of Metallurgical Wastes' to exchange technical 'know-how', examine inter-related problems and discuss effective utilization of metallurgical wastes along scientific and economic lines. Leading scientists and metallurgists from different parts of the world participated in the Symposium in which thirty-seven technical papers covering research and development work on utilization of metallurgical wastes were presented and discussed in six technical sessions. The Symposium was inaugurated by Prof. Humayun Kabir, Union Minister for Petroleum and Chemicals, Government of India; Sir Jehangir Ghandy, Chairman, Executive Council, National Metallurgical Laboratory, presided at the function.

Preliminary Project Report for the production of electrolytic copper is under preparation at the National Metallurgical Laboratory in which the Indian Copper Corporation have shown keen interest.

During the period under review, the following processes were released to different firms in India for commercial exploitation through National Research Development Corporation on lumpsum premium and royalty basis as per details given below:

1. Iron Powder for autogenous cutting (non-patented)

   — Lumpsum premium Rs. 10,000/- Royalty: 2½ per cent on net ex-factory sale.

   Nature of Licence: Non-exclusive

   Period of licence: 14 years.

2. Carbon Free Ferro-alloys by Aluminothermic Reaction (Indian Patent No. 65231)

   — Lumpsum premium: Rs. 7,500/- Royalty: 2½ per cent of net ex-factory sale.

   Nature of licence: Non-exclusive.

   Period of licence: 14 years.

3. Hot-dip Aluminizing of ferrous materials (Indian Patent Nos. 55289, 57938 and 65230)

   — Royalty: 2½ per cent on net ex-factory sale price.

   Nature of licence: Non-exclusive.

   Period of licence: 14 years.

Action is being taken to see that different firms who have taken up NML processes set up their manufacturing units without undue delay. A number of parties has shown interest in the commercial utilization of the NML developed process on 'Flux for submerged arc welding'. On this basis, the National Research Development Corporation is taking active steps to release the process details to a number of firms on suitable terms and conditions.

During the period,the process on 'Hot-dip aluminizing of ferrous materials' was demonstrated to parties to whom it was released for commercial exploitation.

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

Patents filed

1. Indian Patent No. 91134 — Improvement in or relating to a precision temperature controller for use with electrical resistance furnaces up to 1600°C. (4-12-1963).

2. Indian Patent No. 91827 — Improvements in or relating to an apparatus for automatically recording progressive changes in weights with special reference to thermogravimetric determinations (21-1-1964).

Patents accepted

1. Indian Patent No. 79598 — An improved device for the continuous vapour phase degreasing of metallic wire and strip (22-4-1964).

2. Indian Patent No. 79597 — Improvements in a continuous vertical counter current solid-gas reactor (11-7-1963).

3. Indian Patent No. 81402 — Improvements in or relating to electro-deposition of metals particularly manganese by direct current electrolysis of aqueous solutions containing metal ions (31-8-1963).

4. Indian Patent No. 81403 — Improvements in or relating to devices for the conversion of pig irons into high grade steels (1-7-1963).

5. Indian Patent No. 82191 — An improved jacketed electrolytic cell for the electro-deposition of metals and metallic oxides in general and manganese dioxide in particular (26-6-1963).

6. Indian Patent No. 83652 — Improvements in or relating to magnesite refractories (19-9-1963).

7. Indian Patent No. 83968 — A method for reconditioning the coated magnesium powders (19-9-1963).

8. Indian Patent No. 84670 — Improvements in or relating to electrolytic cells (10-1-1964).

Patents sealed

1. Indian Patent No. 79597 — Improvements in a continuous vertical counter-current solid-gas reactor (21-1-1964).

2. Indian Patent No. 79598 — An improved device for the continuous vapour phase degreasing of metallic wire and strip (23-10-1963).

3. Indian Patent No. 81402 — Improvements in or relating to electro-deposition of metals particularly manganese by direct current electrolysis of aqueous solutions containing metal ions (14-2-1964).

4. Indian Patent No. 81403 — Improvements in or relating to devices for the conversion of pig irons into high grade steels (6-1-1964).

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

RESEARCH PROJECTS

1.0 Further Pilot Plant Studies on Beneficiation of Iron Ore from Bolani for Durgapur Steel Plant of Hindustan Steel Ltd.

A 500-tons representative sample of iron ore from Bolani mines was received for Pilot Plant beneficiation tests from Messrs Bolani Ores Ltd. for preparing beneficiation flowsheet whilst investigating the possibility of incorporating cyclones in place of the conventional classifiers for deslimings and spirals for gravity separation of the fine fraction of the deslimed sands, instead of jigs. One 6 in. cyclone of the specified design was fabricated at the National Metallurgical Laboratory for undertaking these investigations.

The sample as received assayed Fe, 56·89; SiO_2, 5·23; Al_2O_3, 7·1; MgO, 0·40; CaO, 0·20; P, 0·05; S, 0·03; Mn, 0·08 and L.O.I., 7·4 per cent. By washing (scrubbing followed by wet screening) four products viz., −2+3/8 in., −3/8+1/8 in., −1/8 in. sand and slime were obtained. A cyclone was used to deslime the −1/8 in. fraction. An appreciable amount of slime contained in the cyclone underflow was separated and mixed with the cyclone overflow. It was found that the washed lumps (−2+3/8 in.) 65·7 per cent by weight assayed Fe, 60·59; SiO_2, 2·36; Al_2O_3, 6·1; MgO, 0·18; CaO, 0·1; P, 0·05; S, 0·03; Mn (trace) and L.O.I. 6·2 per cent, with a recovery of 70 per cent Fe. The combined slime produced was 7·3 per cent by weight with the rejection of about 30 per cent of the total silica and 14·6 per cent of the total alumina originally present in the ore, for an iron loss of 4·6 per cent only.

Washing tests carried out, using an Akins type spiral classifier, in place of the cyclone, produced lumps (−2+3/8 in.) of almost identical grade (60·85 per cent Fe, 2·6 per cent SiO_2) and 5·8 per cent Al_2O_3 for a recovery of 70·8 per cent Fe. The −3/8 in. sand was comparatively of a better grade. The rejection of silica and alumina were 41·5 per cent and 22·5 per cent respectively in the slime (11·1 per cent by weight) for an iron loss of 6·9 per cent only. The Akins type classifier had thus operated more efficiently than the cyclone in desliming the sand fractions. The combined jig concentrate (−3/8+1/4 in., −1/4+1/8 in. and −1/8 in.) obtained by jigging the −3/8 in. sand product, assayed Fe, 59·37; SiO_2, 3·7; Al_2O_3, 4·85; MgO, 0·23; CaO, 0·1; P, 0·05; S, 0·05; Mn, 0·06 and L.O.I. 6·0 per cent. The upgraded sand was suitable for production of sinter.

The −1/8 in. washed fraction was also passed through the Humphrey's spiral, to compare its performance with that obtained by jigging. The results obtained were not satisfactory, the spiral concentrate assaying Fe, 54·8; SiO_2, 9·0 and Al_2O_3 6·1 per cent, which indicated that jigging was to be preferred to spiral treatment for upgrading −1/8 in. fraction.

Heavy media separation tests with the −2+3/8 in. washed lumps at a medium sp. gr. of 3·0, produced a concentrate (sink) assaying Fe, 63·8; SiO_2, 1·3; Al_2O_3 4·1; MgO, 0·20; CaO (trace); S, 0·05; P, 0·05; Mn, 0·03 and L.O.I., 4·7 per cent.

Attempts were also made to concentrate and recover at least some iron from the slime which otherwise would be a waste, employing both cyclone and shaking table. It was found that a concentrate assaying

58·2 per cent Fe and 8·16 per cent insolubles with a recovery of 34·1 per cent Fe could be obtained by concentration of the slime assaying 38·72 per cent Fe and 27·32 per cent insolubles by tabling of the cyclone concentrate. However, such treatment may not be economical for effecting merely an extra overall iron recovery of 1·5-2·0 per cent.

It was, therefore, concluded that the overall results obtained in relation to the new flowsheet, were not comparable with the flowsheet developed earlier at the National Metallurgical Laboratory on Bolani iron ores. Furthermore, desliming of —1/8 per cent washed fraction was more efficient with an Akins type classifier in comparison with a cyclone. Also concentrate obtained after spiral treatment of —1/8 in. sand was of a poorer grade than that obtained by jigging.

The economic evaluation of beneficiation treatments 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 for Indian iron ores:

(i) The cost of mined ore as mined has been taken at Rs. 5/- per ton, excluding transport charges.

(ii) Operational capacity of the beneficiation plant is 14,000 tons of run-of-mine ore per day.

(iii) Capital cost of the plant excludes the capital cost structure of the mining equipment and township, but includes the capital cost of the beneficiation plant machinery and its erection, building, water and power supply installation of the beneficiation plant.

(iv) For every one per cent of reduction in the alumina content of the ore achieved through optimum beneficiation treatment, the coke and flux rates would decrease by 40 Kg. and 60 Kg. respectively per ton of pig iron smelted.

The increase in productivity of the pig iron would be about 2·5 per cent. Due to these factors, an overall saving per ton of pig iron produced would be Rs. 4·00 after allowing for depreciation, overhead, and indirect costs.

1.1 Further Pilot Plant Studies on Beneficiation and Sintering of a Mixed Iron Ore Sample from (i) The Upper Portion of Main Haulage Road and (ii) Bench 3 of Hill No. 1 of Kiriburu Mines of National Mineral Development Corporation

The National Metallurgical Laboratory had earlier completed systematic and exhaustive pilot plant studies on the beneficiation and sintering characteristics of 100 tonnes representative samples of iron ore from Kiriburu Mines of the National Mineral Development Corporation of the Government of India. The current investigation was carried out on a mixed sample from the upper portion of the Main Haulage Road and from Bench No. 3 of Hill No. 1 of Kiriburu Iron Ore Mines.

Kiriburu Iron Ore Mines are being developed by the National Mineral Development Corporation of the Govt. of India, for supplying iron ore for the Bokaro Steel Plant and till the latter's installation, lumpy iron ore from Kiriburu mines will be exported to Japan. Messrs M. N. Dastur & Co., who were entrusted with the task of preparing the detailed Project Report, desired that comprehensive pilot plant studies should be taken up at the National Metallurgical Laboratory to determine the physical, petrological, beneficiation and sintering characteristics of

Table 1 — Economic Evaluation of Beneficiation of Bolani Iron Ore

	Crushing, dry screening and utilization of fines %			Crushing, dry screening and rejection of $-\frac{3}{8}''$ size %			Crushing, washing wet screening and dewatering of $-\frac{3}{8}''$ fines %			Crushing, washing wet screening, concentration of only fines $-\frac{3}{8}''$ %			Crushing, washing wet screening concentration of both lumps and fines $-\frac{3}{8}''$ %		
	Fe	SiO_2	Al_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
Analysis of final beneficiated iron ore	56·89	5·23	7·1	59·26	3·88	6·6	58·6	3·95	6·53	60·34	2·7	5·8	62·1	1·94	4·3
Yield tonnes/day %	14,000 (100%)			8,876 (63·4%)			12,978 (92·7%)			11,690 (83·5%)			9,282 (66·3%)		
Capital cost of the ore treatment plant	Rs. 2·5 crores			Rs. 2·5 crores			Rs. 3·36 crores			Rs. 4·5 crores			Rs. 5·4 crores		
Cost of treatment per ton of beneficiated ore															
(*a*) Capital cost	Rs. 0·894			Rs. 1·410			Rs. 1·283			Rs. 1·921			Rs. 2·910		
(*b*) Operating cost	Rs. 0·180			Rs. 0·284			Rs. 0·254			Rs. 0·432			Rs. 0·754		
(*c*) Cost of raw ore mined	Rs. 5·000			Rs. 7·886			Rs. 5·395			Rs. 5·985			Rs. 7·541		
Total cost per tonne of prepared ore	Rs. 6·074			Rs. 9·580			Rs. 6·932			Rs. 8·338			Rs. 11·205		
Tonnes of ore required per tonne of pig iron	1·694			1·627			1·645			1·597			1·540		
Total cost of ore required per tonne of pig iron	Rs. 10·29			Rs. 15·59			Rs. 11·40			Rs. 13·31			Rs. 17·26		
Difference in alumina %	—			0·50			0·58			1·3			2·8		
Expected saving in production cost per tonne of pig iron in blast furnace iron smelting due to decrease in alumina by beneficiation	—			Rs. 2·0			Rs. 2·32			Rs. 5·2			Rs. 11·2		

the iron ores collected from different mining levels on the two hills in the area, representing the types of ore to be mined for the next ten years or so. With these objectives in view, representatives of National Mineral Development Corporation, Hindustan Steel Ltd., Messrs M. N. Dastur & Co. and the National Metallurgical Laboratory formulated a procedure for drawing the representative samples from various mining benches. Consequently, five different samples nearly 100 tonnes each, were prepared and investigated at the National Metallurgical Laboratory. These samples were collected from (i) Bench No. 2 of Hill No. 1, (ii) Bench No. 3 of Hill No. 1, (iii) Bench No. 2 of Hill No. 2, (iv) Upper portion of Main Haulage Road, and (v) Lower Portion of the Haulage Road.

Based on the petrological studies conducted on each of the five samples, it was decided that samples from the upper portion of the Haulage Road and from Bench No. 2 of Hill No. 1 should be mixed in the ratio of 4: 1 for the purposes of beneficiation and sintering studies based on their identical chemical and mineral characteristics. The mixed sample assayed Fe, 60·43; SiO_2, 2·78; Al_2O_3, 5·23 and 4·9 per cent loss on ignition.

Screenability tests with different moisture contents in the ore indicated that maximum screening difficulty would be encountered when the moisture content of the ore was 7·5 per cent. Scrubbing followed by wet screening of crushed ore indicated that a clean, sized, washed product of +3/8 in. or +1/2 in. size free from adherent ore fines for direct charging into the iron blast furnace or for export purposes and a —3/8 in. or —1/2 in. slime-free classifier sand product for sintering, could be obtained.

Keeping in view the requirements of ore for Bokaro Steel Project, ore washing (scrubbing followed by wet screening), was done at 2 in. size and 3/8 in. size screen was used to separate the fines from ore lumps. The ore was also washed at 4 in. size and screened at 1/2 in. size for export of lumpy ore to Japan. With washing done at 2 in. and 4 in. of ore size, the lumps assayed Fe, 63·40; SiO_2, 1·20; Al_2O_3, 3·60 and Fe, 63·41; SiO_2, 1·67; Al_2O_3, 3·56 per cent respectively whilst the rejections of silica and alumina in the slime were 43·6, 26·5 and 35·1, 21·3 per cent respectively.

When 4 in. ore size was only wet screened, the rejection of insolubles was somewhat lesser and the grades of washed products were slightly poorer than those obtained from a scrubbed and wet screened 4 in. ore size.

The —3/8 in. classifier sand obtained by washing at 2 in. ore size, constituting 25·9 per cent by weight, assayed Fe, 59·79; SiO_2, 3·47 and Al_2O_3, 6·04 per cent and contained 25·6 per cent of the total iron content of the ore; sinters made from this product were not expected to offer any particular metallurgical advantages due to their high insoluble gangue contents and the —3/8 in. classifier sand was therefore upgraded by jigging treatment. The combined jig concentrate weighing 21·5 per cent assayed Fe, 62·10; SiO_2, 2·30 and Al_2O_3, 4·30 per cent with the iron recovery and loss of 22·0 and 3·6 per cent respectively with respect to the original. Sieve analysis of —3/8 in. classifier sand indicated that the coarser fraction (+3 mesh) of the sand representing 18·2 per cent by weight, could be by-passed from the jigging circuit and mixed with —3 mesh jig concentrate yielding a combined product very similar to that obtained by jigging of the entire —3/8 in. sand, thus requiring jigging of only 81·8 per cent by weight of sand. If jigging was done with only 48·3 per cent by weight of the fines (—6 mesh), the grade of combined concentrate will be slightly poorer, namely Fe, 60·77; SiO_2, 2·45 and Al_2O_3, 5·64 per cent. The present sample represented ore from bench levels 3, 4, 5 and 6. As the ore grade is expected gradually to improve with depth due to

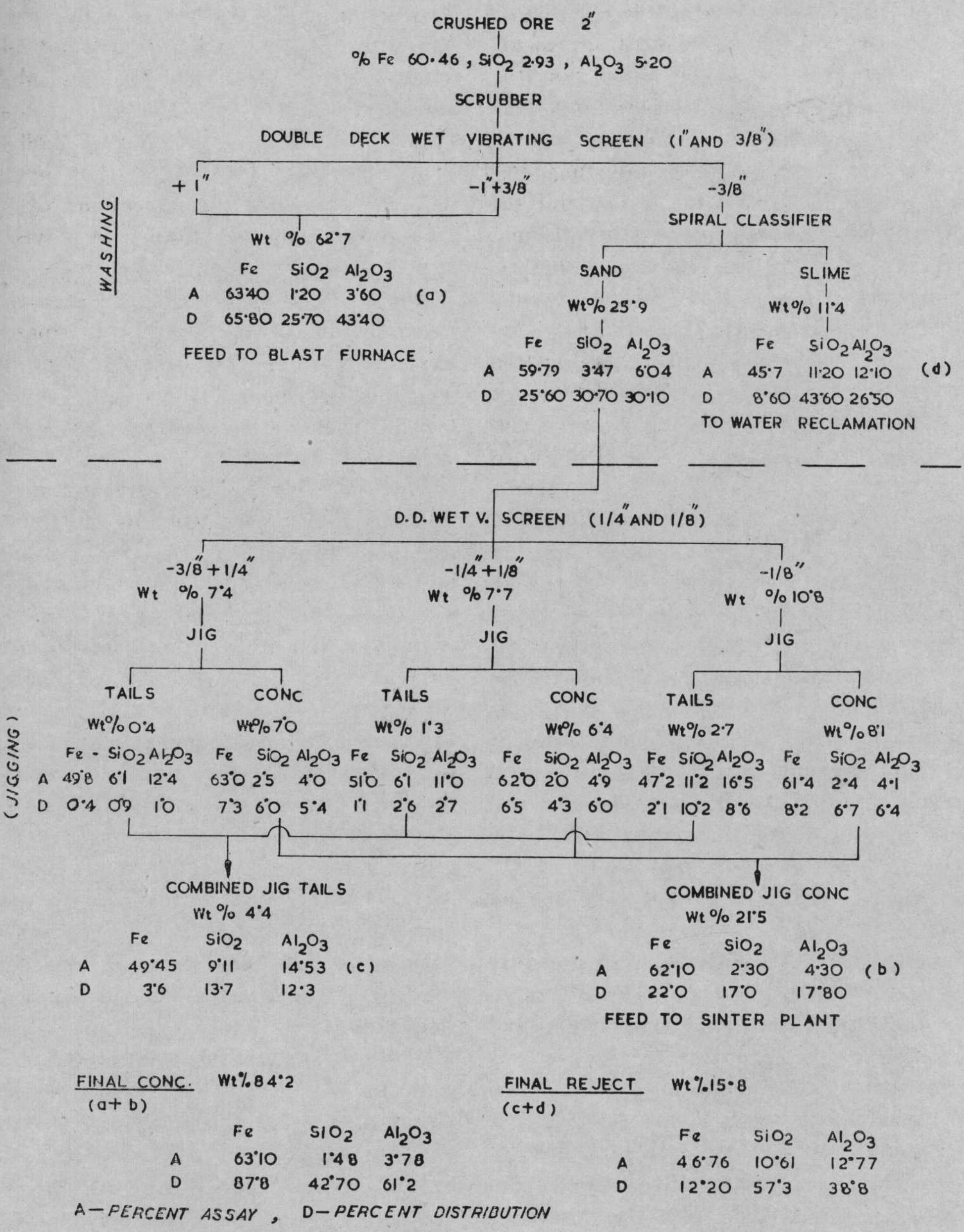


FIG. 1 — SUMMARY OF RESULTS OF BENEFICIATION AT 2 IN. SIZE

less laterite formation, the percentage weight of fines that could be by-passed from the jigging circuit will progressively increase from level 3 to the lower bench levels.

Summary of the results of beneficiation is given in Figs. 1 to 3. Based on these results, the recommended flowsheet is given in Fig. 4. It may thus be observed that grades of the washed lumps as well as the beneficiated fines assumed for supplies from Kiriburu in the Project Report of Messrs M. N. Dastur & Co. for the Bokaro

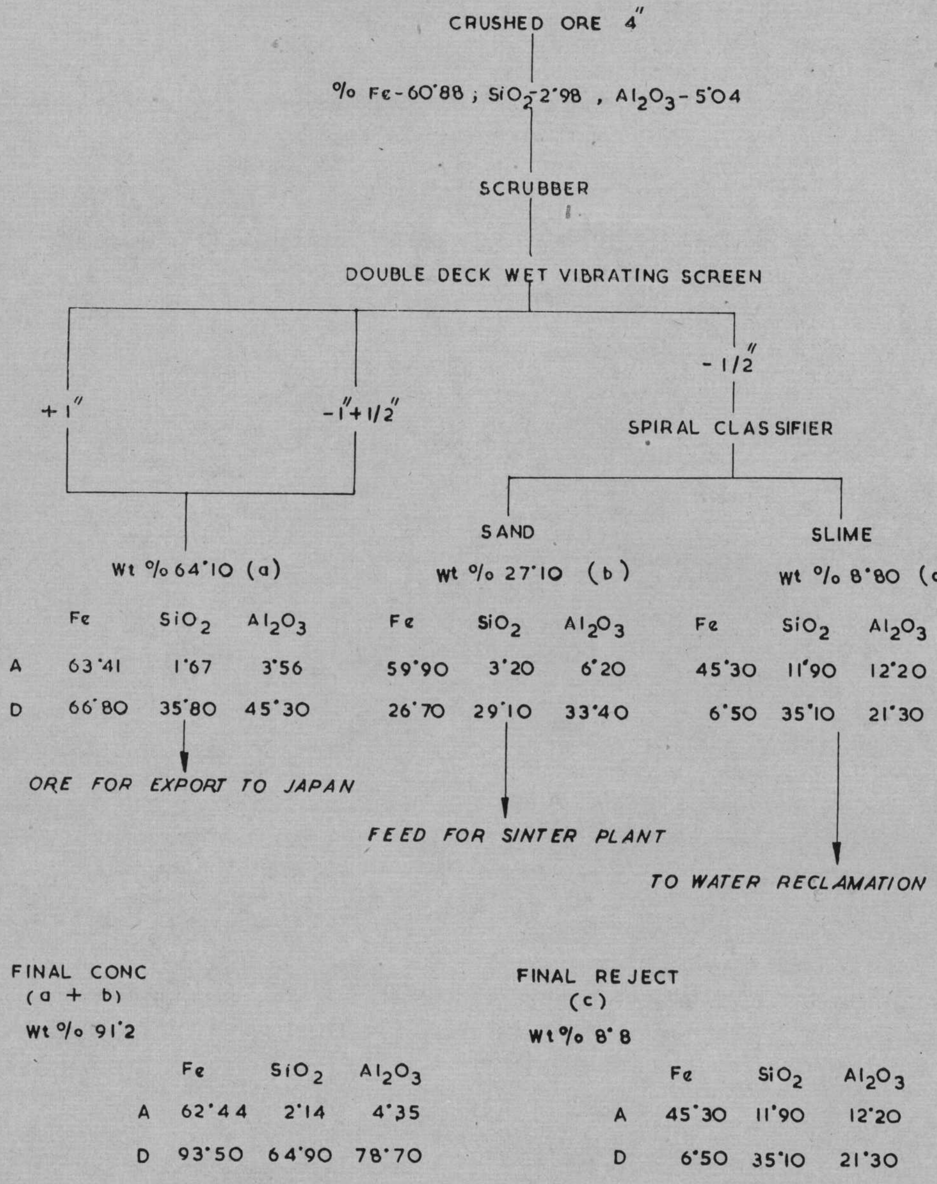


FIG. 2 — SUMMARY OF RESULTS OF SCRUBBING AND WET SCREENING AT 4 IN. SIZE

Steel Project check up fairly close to the pilot plant results obtained at the National Metallurgical Laboratory with 2 in. ore size.

Sintering characteristics of the —3/8 in. washed classifier sand, before and after jigging treatments were studied for making self-fluxing as well as non-fluxed sinters. Unfluxed sinters were made only with washed but unjigged fines; it was found that 7 per cent water content and 4·5 per cent coke represented optimum values for producing a strong sinter. In the case of beneficiated fines, optimum water and coke

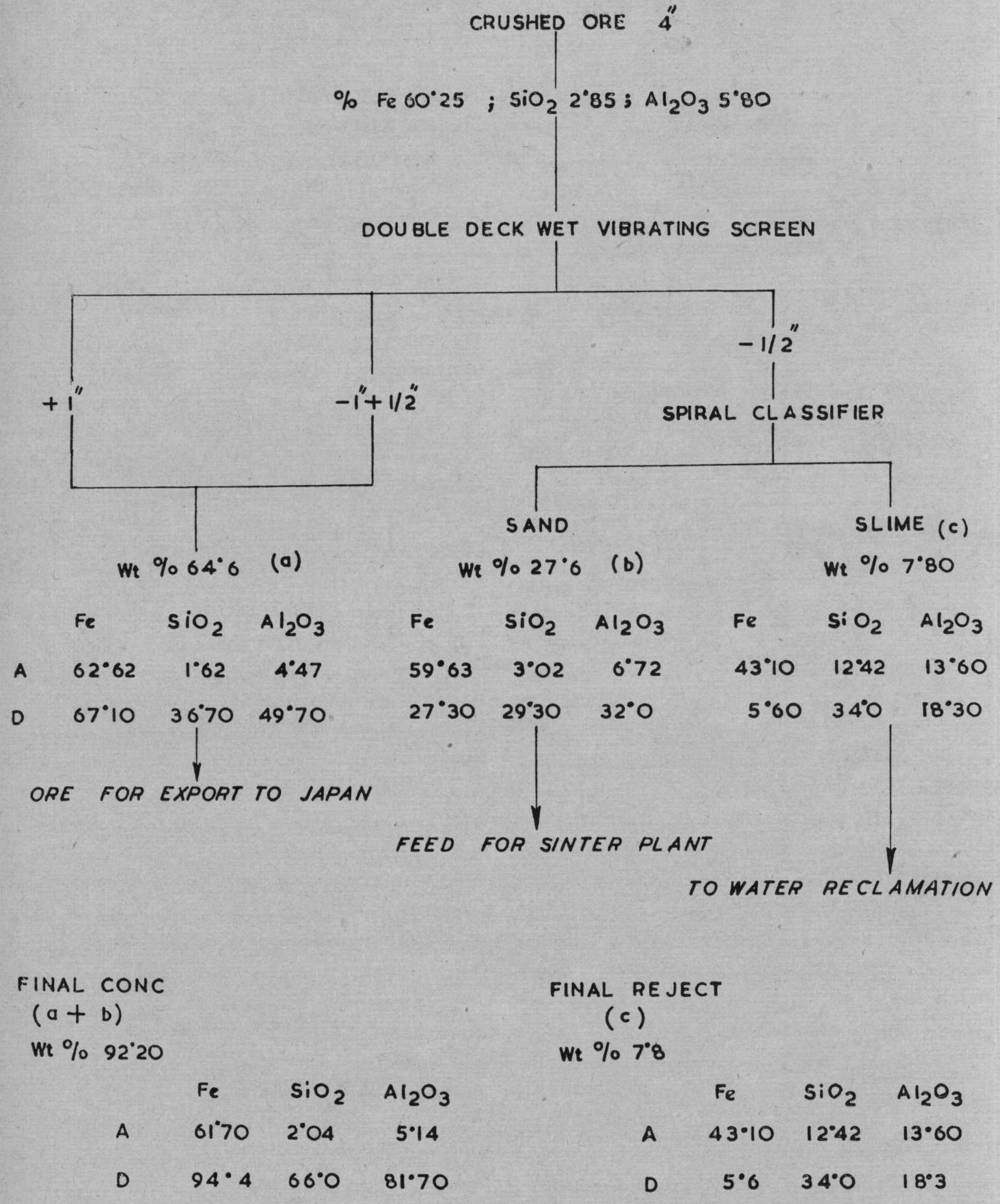


FIG. 3 — SUMMARY OF RESULTS OF WET SCREENING AT 4 IN. SIZE

contents were found to be 7 and 4 per cent respectively. Good quality self-fluxing sinters with a basicity ratio of up to 2·0, could be produced from the beneficiated fines 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 adversely affecting the sinter quality and its metallurgical characteristic.

Varying amounts of blue dust were added to the sinter feed to study the extent to

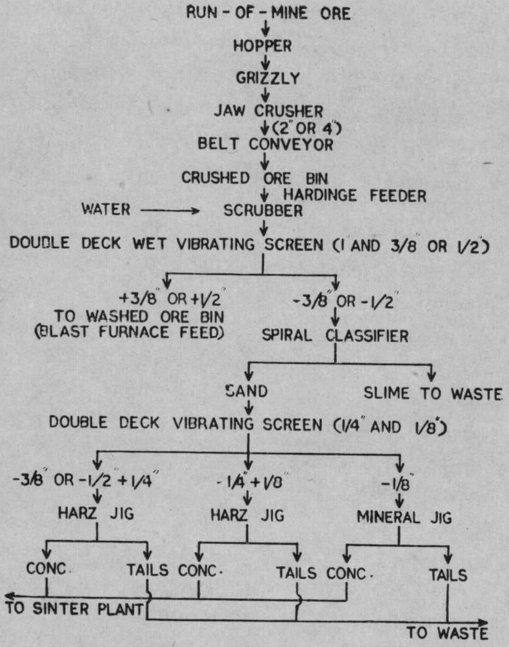


FIG. 4 — GENERAL FLOWSHEET FOR BENEFICIATION OF IRON ORE FROM KIRIBURU

which the former could successfully be incorporated in the latter for making good metallurgical sinters. Though the sintering rate was slightly lowered with increase in the blue dust/ore fines ratio, it was found that blue dust up to 30 per cent with respect to iron ore fines, (blue dust: ore fines=3: 7) yielded a good metallurgical grade sinter of 1·6 basicity ratio possessing highly improved sinter chemistry.

The results further showed that solid fuel coke breeze could be substituted by gaseous fuel (coke oven gas) up to 30 per cent of the total amount of coke breeze needed for proper sintering. The sinters thus produced were of good quality and high strength and additionally possessed an exceedingly high degree of desirable oxidation characteristic in comparison with sinters made by conventional sintering process. Overall economic evaluation of beneficiation of Kiriburu iron ore is given in Table 2 based on the parameters referred to earlier.

1.2 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

This investigation was the second in a series of investigations undertaken on the beneficiation and sintering characteristic of the iron ores from different mining levels of the Kiriburu Iron Ore lease of the National Mineral Development Corporation that are being developed for supplying iron ore on the long run to the projected Bokaro Steel Plant when completed and for export to Japan as a short-term measure.

The samples of iron ore from Kiriburu mines one from Hill No. 1, Bench No. 2 and another from Hill No. 2, Bench No. 2 were mixed in equal proportions for beneficiation and sintering studies. The mixed sample assayed Fe, 57·90; SiO_2, 3·00 Al_2O_3, 7·00; P, 0·047; TiO_2, 0·26 per cent; 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 met with when the moisture content of the ore was 7·5 per cent. Investigations were conducted to produce clean sized products suitable for the iron blast furnaces in the projected Bokaro Steel Plant and also for export to Japan as a short-term measure.

Keeping in view the requirements of ore for Bokaro Steel Plant, the ore after crushing to 2 in. was scrubbed and wet screened at 3/8 in. size. The +3/8 in. 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 rejections of silica and alumina in the slime were 37·5 and 22.1 per cent respectively.

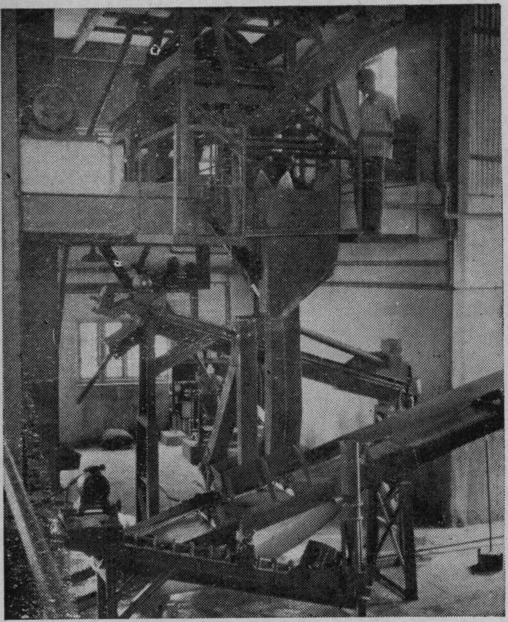


FIG. 5 — IRON ORE WASHING SET-UP SHOWING THE SCRUBBER, VIBRATING SCREEN AND THE CLASSIFIER

The $-3/8$ in. 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 iron 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. These beneficiated fines would produce excellent metallurgical grade sinter than the sinter made from unbeneficiated ore fines.

Scrubbing the ore after crushing to 4 in. and wet screening thereafter to remove the $-1/2$ in. lumps assaying Fe, 61·51; SiO_2, 1·43 and Al_2O_3, 5·35 per cent with an iron recovery of 62·8 per cent. The rejection of silica and alumina through the slime was 25·5 and 13·8 per cent respectively which was less than the corresponding values obtained by scrubbing the ore at 2 in. size.

Mere wet screening of the ore (without prior scrubbing) after crushing it to 4 in. produced $+1/2$ in. lumps assaying Fe, 61·10; SiO_2, 1·52 and Al_2O_3, 5·43 per cent with an iron recovery of 65·8 per cent. The grade of the lumps was almost similar to that obtained by scrubbing and wet screening the ore at the same size, but the lumps retained slightly more silica and alumina contents. The rejections of silica and alumina in the slime were 23·4 and 12·7 per cent respectively, slightly lower than in the earlier case. The $-1/2$ in. classifier sand product when subjected to jigging after sizing into three graded fractions, produced a combined jig concentrate assaying Fe, 59·7; SiO_2, 2·59 and Al_2O_3, 5·65 per cent with an iron recovery of 24·2 per cent of 29·5 per cent present in this fraction. The coarse fraction of the classifier sand was fairly good grade and could be by-passed from the jigging circuit, thus obviating the necessity to jig the entire ore fines.

The results of washing and beneficiation are summarized in Figs. 6-8.

The sintering characteristics of $-1/2$ in. washed classifier sand with or without prior jigging were studied for making self-fluxing as well as unfluxed sinters. Only unfluxed sinters were made from the washed but unjigged ore fines; it was found that 7 per cent water and 5·0 per cent coke were optimum for producing a strong sinter. In the case of jigged beneficiated fines, the values for optimum water, coke, and return sinter fines were determined to be 7, 4 and 25 per cent respectively. Good quality self-fluxing sinters upto a basicity ratio of 2·0, could be produced from beneficiated ore fines incorporating requisite additions of limestone. MgO content of the sinters could be increased by the addition of requisite dolomite to the sinter feed along with the limestone without affecting the sinter quality on the one hand and improving the slag chemistry on the other.

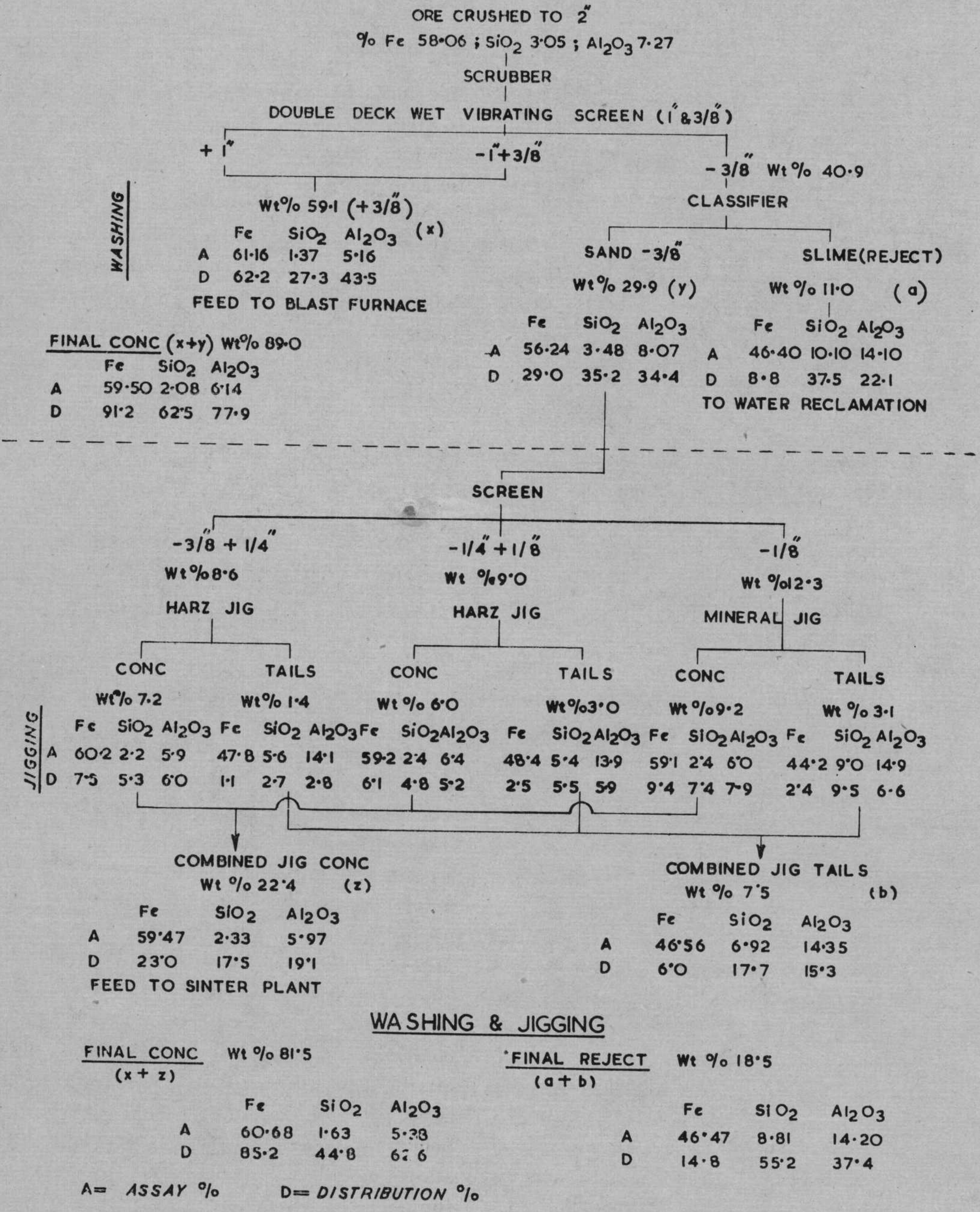


FIG. 6 — SUMMARY AND RESULTS OF WASHING AND JIGGING OF KIRIBURU IRON ORE (H_1B_2, H_2B_2) AT 2 IN. SIZE

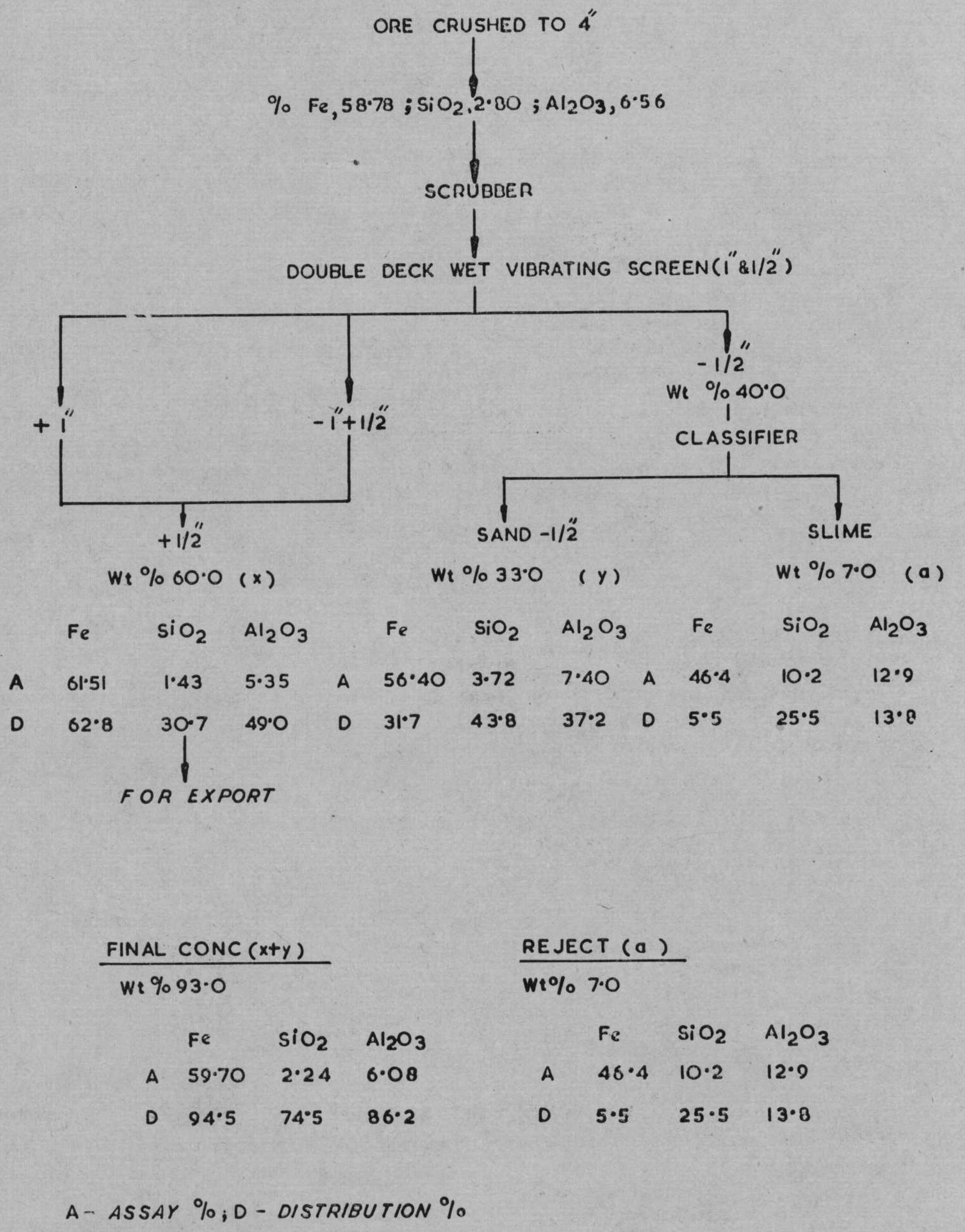


FIG. 7 — SUMMARY OF RESULTS OF WASHING OF KIRIBURU IRON ORE (H_1B_2, H_2B_2) AT 4 IN SIZE

Varying amounts of blue dust were added to the sinter mix, to study the extent to which the former could be incorporated in the sinter feed for making good metallurgical grade sinters. 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

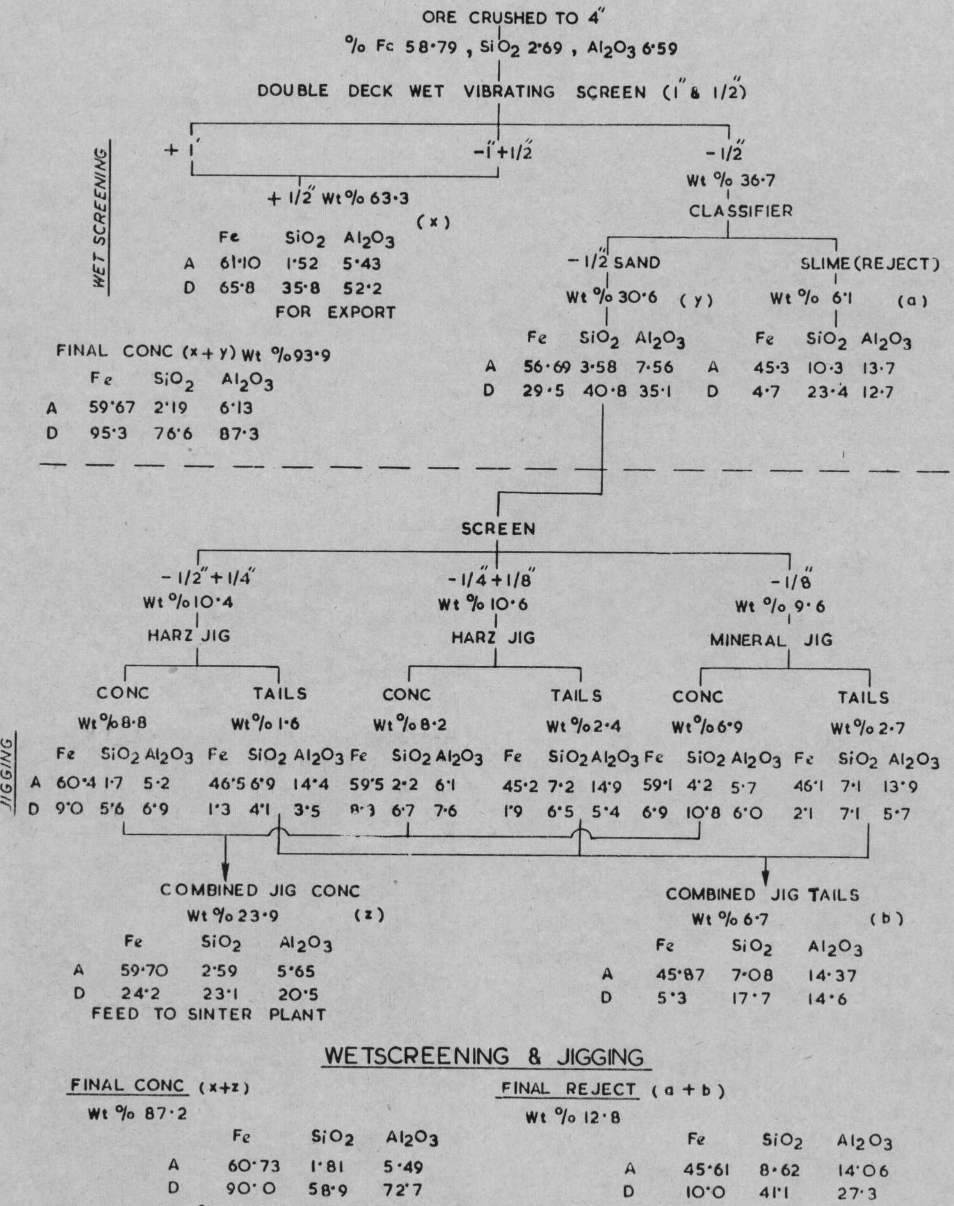


FIG. 8 — SUMMARY OF RESULTS OF WET SCREENING AND JIGGING OF KIRIBURU IRON ORE (H_1B_2, H_2B_2) AT 4 IN. SIZE

a good metallurgical grade sinter of 1·6 basicity ratio considerably improving the sinter and slag chemistry.

The results further showed that solid fuel coke breeze could be substituted by gaseous fuel (coke oven gas) up to 30 per cent of the total coke breeze needed for proper sintering. The sinters thus produced by such mixed firing technique were of excellent quality and possessed good strength with a very high degree of oxidation when compared to sinters made by the conventional sintering process employing the coke breeze alone. The overall economic evaluation of beneficiation of Kiriburu iron ore was evaluated on the basis of parameter referred to earlier in this Report.

1.3 Pilot Plant Studies on Beneficiation of a Float Ore Sample of Low Grade Magnetite from Salem

About a ton of float-ore sample of low grade magnetite from Salem assaying Fe, 37·0; SiO_2, 47·0; Al_2O_3, 0·8; CaO, 0·2; MgO, 1·0; P, 0·067 per cent was received from the Government of Madras for investigation on the advice of Messrs M. N. Dastur & Co., Consultants to the " Neyveli Salem Iron and Steel Project ". The objective in testing the Salem float ore sample was to determine the feasibility of producing a high grade concentrate suitable for iron production based on the beneficiation flowsheet developed in earlier investigations at the National Metallurgical Laboratory with the run-of-mine ore taken from the magnetite ore deposits of Salem, so that the float ore could be mixed with the run-of-mine ore for beneficiation in a single plant. By following wet magnetic separation and tabling, 88 per cent of the total iron in the float ore could be recovered in a concentrate assaying 67·8 per cent Fe and 5·5 per cent insolubles, of which about 6·0 per cent iron was contributed by the table concentrate.

1.4 Studies on Pelletizing of Salem Magnetite Concentrate

The Salem magnetite concentrate assayed Fe, 66·9; Al_2O_3, 0·9, SiO_2, 5·04; S, 0·18 and P, 0·09 per cent, and was as fine as 34 per cent —325 mesh. Pellets made out of the concentrate were found to be of low strength. Pelletization after grinding the concentrate to different degrees of fineness indicated that 60 per cent —325 mesh represented the optimum fineness for further studies. Effects of the additions of different quantities of soda ash and bentonite on the strength of pellets were studied.

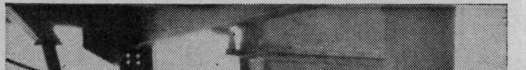


FIG. 9—DRUM TYPE WET MAGNETIC SEPARATOR FOR STRONGLY MAGNETIC MINERALS LIKE MAGNETITE, FERRO-SILICON, ETC.

Table 2 — Economic Evaluation of Beneficiation of Kiriburu Iron Ore

	Crushing to 2″ dry screening and utilization of $-\frac{3}{8}''$ fines %			Crushing to 2″ dry screening and rejection of $-\frac{3}{8}''$ fines %			Crushing to 2″ scrubbing, wet screening and dewatering of $-\frac{3}{8}''$ fines %			Crushing to 2″ scrubbing, wet screening and beneficiation of $-\frac{3}{8}''$ fines only %		
	Fe	SiO_2	Al_2O_3	Fe	SiO_2	Al_2O_3	Fe	SiO_2	Al_2O_3	Fe	SiO_2	Al_2O_3
Analyses of final beneficiated iron ore	60·43	2·78	5·23	63·24	1·80	4·08	62·35	1·866	4·31	63·10	1·48	3·78
Yield, tonnes/day		14,000 (100%)			9,757 (69·7%)			12,420 (88·6%)			11,790 (84·2%)	
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.	1·281		Rs.	1·354		Rs.	1·909	
(b) Operating cost	Rs.	0·180		Rs.	0·259		Rs.	0·266		Rs.	0·429	
(c) Cost of raw ore as mined	Rs.	5·00		Rs.	7·174		Rs.	5·644		Rs.	5·939	
Total cost per tonne of prepared ore	Rs.	6·074		Rs.	8·714		Rs.	7·264		Rs.	8·277	
Tonnes of ore required per tonne of pig iron		1·595			1·524			1·546			1·528	
Total cost of ore required per tonne of pig iron	Rs.	9·690		Rs.	13·27		Rs.	11·23		Rs.	12·65	
Difference in alumina %		—			1·15			0·92			1·45	
Expected saving in production cost per tonne of pig iron in blast furnace iron smelting due to decrease in alumina by beneficiation		—		Rs.	5·75		Rs.	4·60		Rs.	7·25	

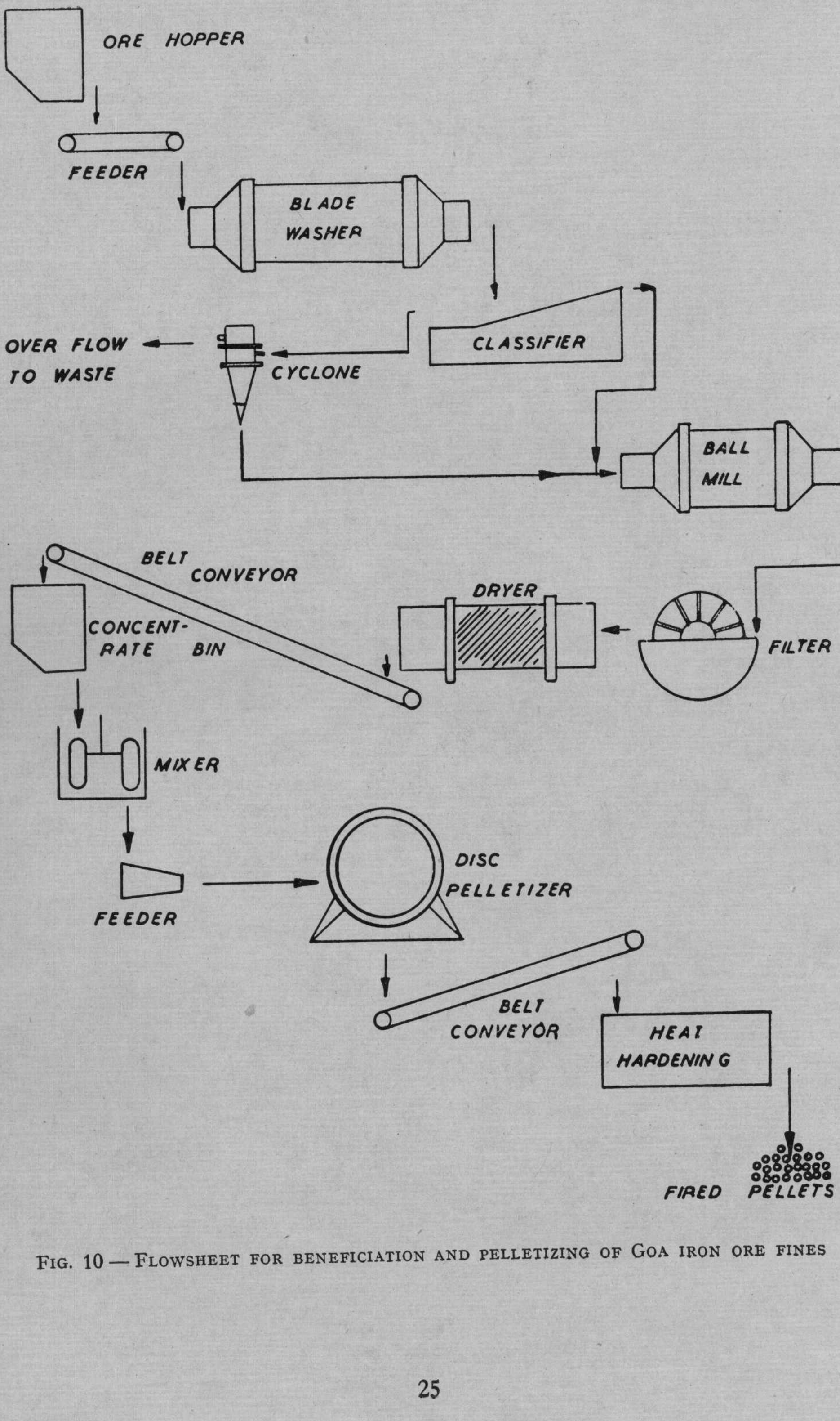


FIG. 10 — FLOWSHEET FOR BENEFICIATION AND PELLETIZING OF GOA IRON ORE FINES

1.5 Beneficiation and Pelletization Studies on Iron Ore Fines from Pale Mines, Goa

A sample of iron ore fines from Pale Mines, Goa, received for beneficiation and agglomeration studies assayed Fe, 60·5; SiO_2, 4·53; Al_2O_3, 3·91; P, 0·07 and L.O.I., 4·47 per cent. The ore consisted mainly of hematite, goethite and magnetite with quartz and ochery coating as gangue. Simple washing and cyclone treatment yielded a concentrate assaying Fe, 64·3; SiO_2, 2·76; Al_2O_3, 2·25; P, 0·05 and 3·56 per cent loss on ignition with a recovery of 93·6 per cent iron. Comprehensive studies on pelletizing of the ore fines concentrate were undertaken. Effect of different grinding treatments on balling characteristics and on strength of the green pellets were determined. It was found that a grind of 75·4 per cent −325 mesh was optimum for the purpose. Firing of the pellets at 1300-1330°C. for heat-hardening purposes yielded pellets of high optimum strength for long distance handling and transport. The heat-hardened pellets analysed Fe, 66·67; FeO, 2·12; SiO_2, 2·96; Al_2O_3, 2·33; CaO and MgO, each 0·13; P, 0·05; and S, 0·12 per cent, possessed good physical strength and characteristics and were ideal for blast furnace iron smelting.

Based on the results thus obtained, a general flowsheet has been developed (Fig. 10) which can form the basis for setting up an economic size industrial plant for the beneficiation and pelletization of iron ore fines from Pale Mines, Goa.

1.6 Studies on the Production of High Basicity Sinters from Noamundi Washed Iron Ore Fines and Incorporating Blue Dust

Samples of −3/8 in. iron ore fines from Noamundi mines, viz. (i) classifier sand and (ii) blue dust, were received from Tata Iron & Steel Works. Comparative studies were undertaken to examine their suitability for making unfluxed, self-fluxing and super-basic self-fluxing sinters.

It was established that sinters could be successfully produced using the classifier sand from the Noamundi Washing Plant of TISCO. Good metallurgical quality super basic self-fluxing sinters with basicity ratios as high as 3·0-3·5 could be successfully obtained. The investigations further showed that with increase in the basicity ratio of the sinter mix, the rate of sintering increased whilst the sinter strength was only slightly reduced.

Varying amounts of blue dust were introduced into the sinter feed in combination with the washed classifier sand. Though the rate of sintering was slightly reduced with the increase in the blue dust/classifier sand ratio, blue dust even up to 50 per cent of the ore mix for the sinter feed could be successfully utilized in producing self-fluxing and non-fluxed sinters.

Introduction of blue dust in the sinter mix improved the sinter chemistry and furthermore minimized the flux requirements whilst yielding good metallurgical grades of sinter of optimum strength and chemistry.

Although considerable handling and screening difficulties under Indian monsoon rainy conditions completely preclude dry screening of iron ores at 3/8 in. size, some tests of academic interest were however performed with dry screened ore fines to produce self-fluxing as well as unfluxed sinters. The sinters made out of dry screened fines, in view of their generally high deleterious gangue contents, are of little metallurgical value or importance and do not confer any of the well established advantages expected from a sinter-burden, such as increased iron productivity, lowered fuel and flux rates etc.

2.0 Beneficiation of Limestone from Tisco

A 700 tonnes sample of low grade limestone assaying CaO, 45·9 and SiO_2,

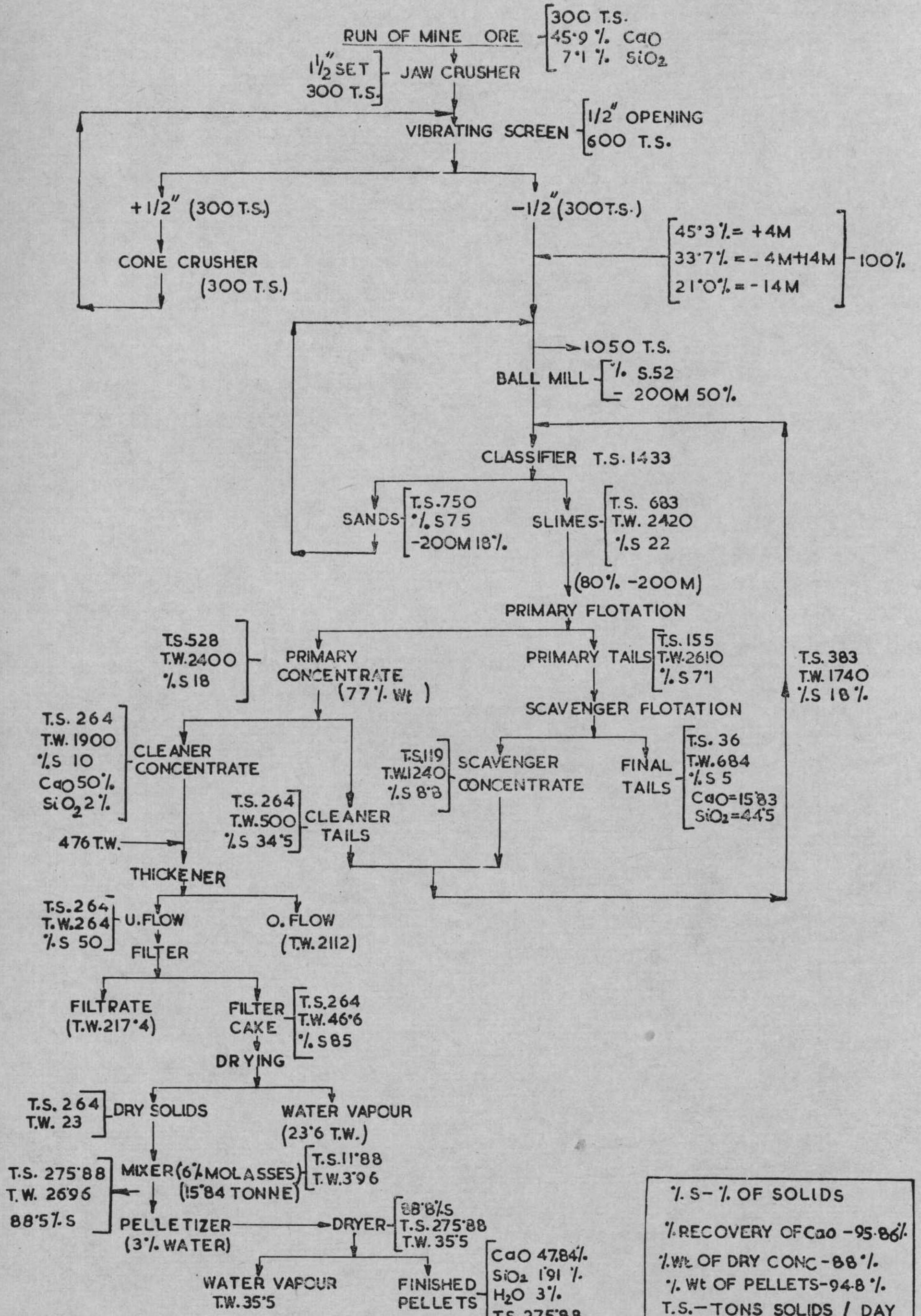


FIG. 11 — QUANTITATIVE FLOWSHEET FOR THE BENEFICIATION AND PELLETIZING OF TISCO LIMESTONE AT 300 TONNES/DAY

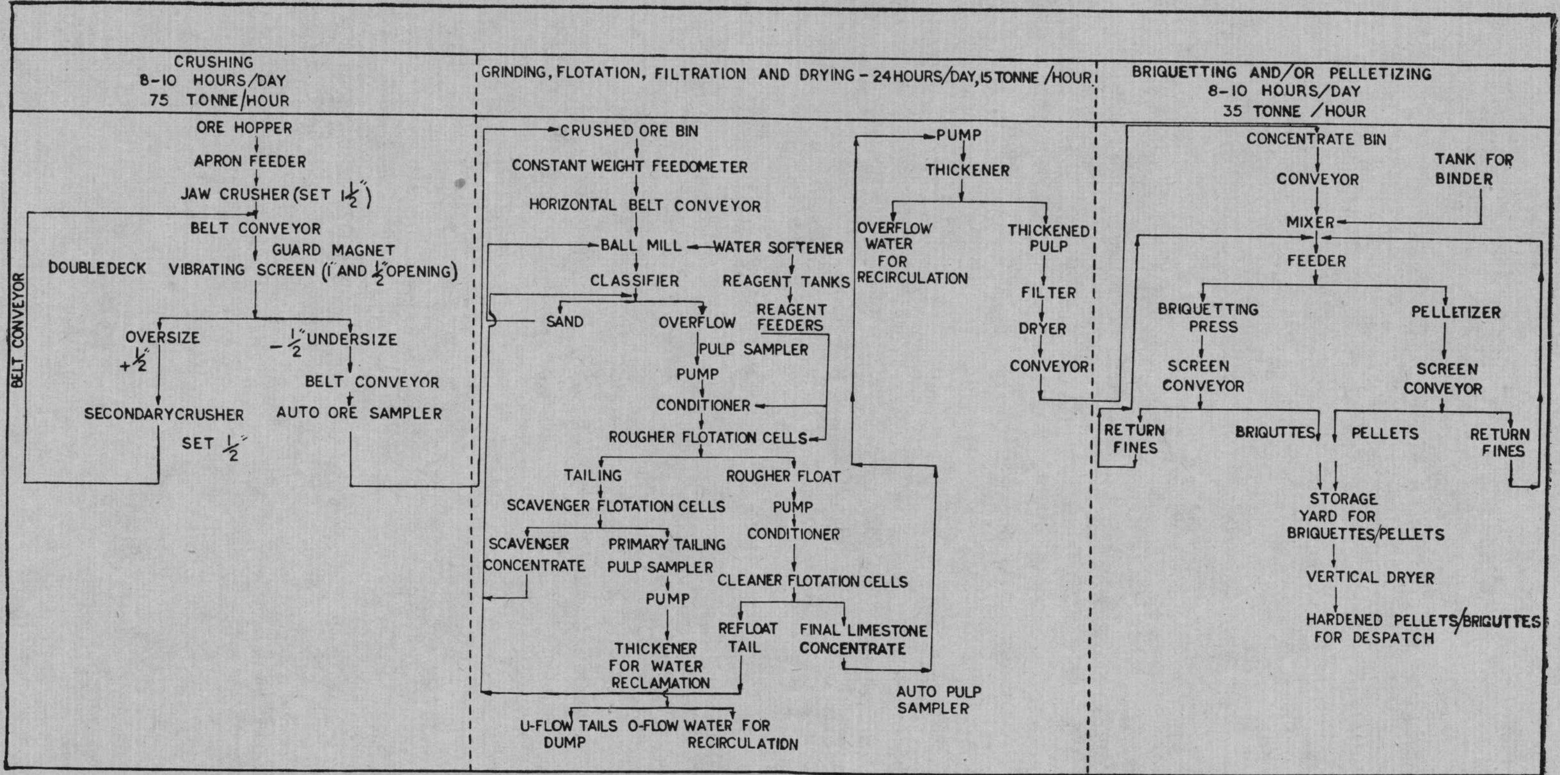


FIG. 12 — SCHEMATIC FLOWSHEET FOR THE BENEFICIATION OF LOW-GRADE LIMESTONE CAPACITY 300 TONNES OF RAW LIMESTONE PER DAY

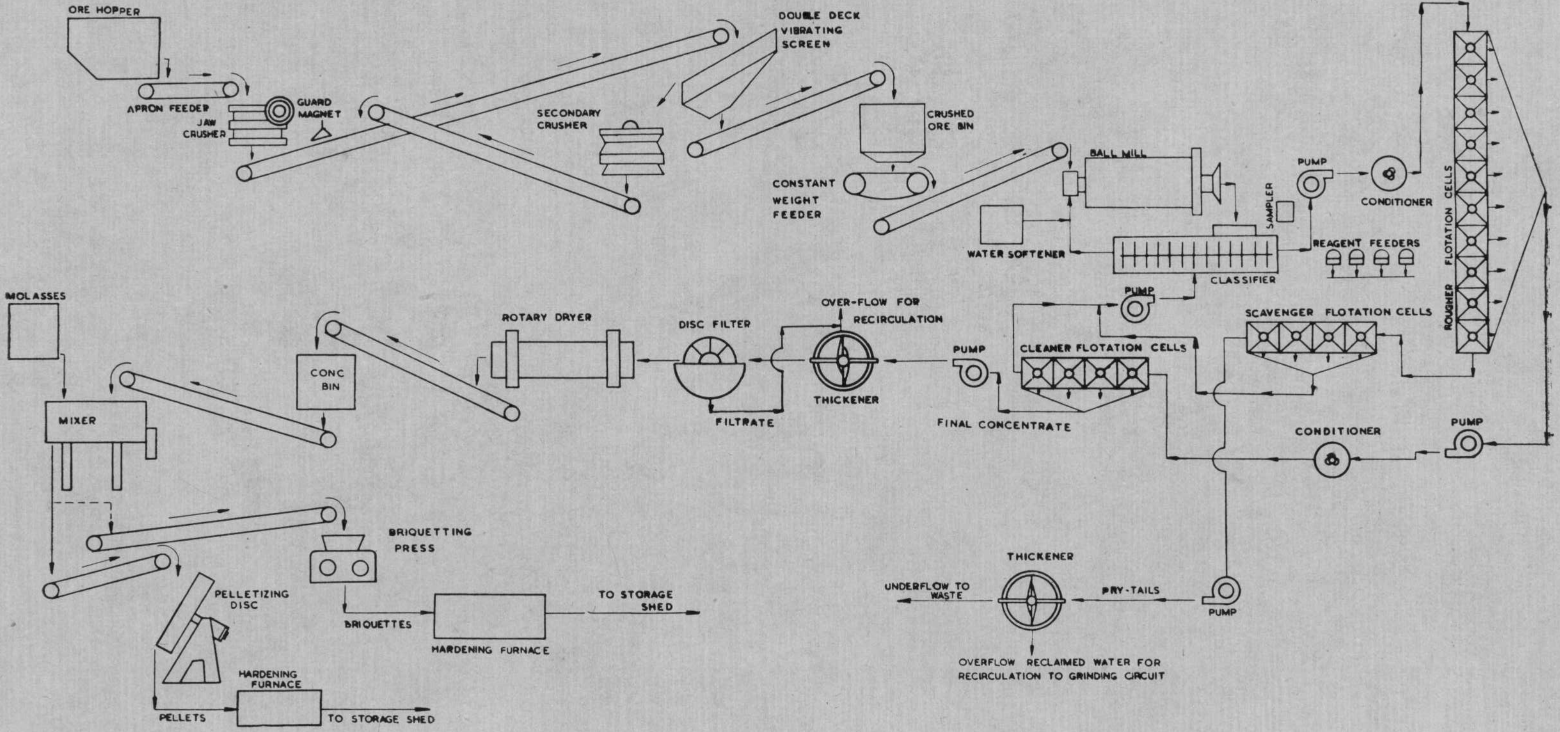


FIG. 13 — DIAGRAMMATIC FLOWSHEET FOR THE BENEFICIATION OF LOW-GRADE LIMESTONE

7·1 per cent was received from Tata Iron & Steel Works, Jamshedpur, for upgrading and agglomeration studies on pilot plant scale and for supplying pellets for trials in their steel-making shops.

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 eliminated producing a concentrate constituting 88 per cent by weight 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 these pilot plant trial investigations indicated a maximum variation of about 0·5 per cent in the assay of final concentrate. The investigation results also indicated that best separation of the insolubles was achieved from a pulp containing not over 22 per cent solids, 80 per cent of which passed through 200 mesh. Small additions of pine oil, (0·227 kg./ton) were found to cut down the collector consumption from 0·73 kg./ton to 0·41 kg./ton for upgrading limestone. The quantitative flowsheet of the entire treatment recommended is given in Fig. 11.

Drop tests conducted on green pellets and crushing load tests on heat-hardened pellets established that strong pellets assaying CaO, 47·84 and SiO_2, 1·91 per cent, could be obtained from the concentrate containing less than 8·5 per cent moisture and employing 6 per cent molasses as the binder.

Based on these pilot plant studies at the National Metallurgical Laboratory, flowsheets for setting up a commercial plant, for upgrading 300 tonnes/day of the limestone are given in Figs. 12 and 13.

3.0 Beneficiation of Dolomite from Tisco

A 100 tonnes sample of low grade dolomite assaying CaO, 28·6; MgO, 20·0; SiO_2, 4·22; Al_2O_3, 1·34; S, 0·12 and CO_2 45·0 per cent was received from Tata Iron & Steel Works for detailed batch as well as pilot plant trial investigations at the National Metallurgical Laboratory, with a view to determine initially by batch tests, optimum conditions under which the silica content could be lowered by froth flotation, so that subsequent pilot plant trials can thereafter be undertaken.

Petrological examination of the sample indicated that quartz, muscovite, chlorite and felspars constituted the gangue and further that grinding the sample to finer than 65 mesh would be necessary to liberate the dolomite from the gangue. Flotation only was attempted for elimination of siliceous gangue from dolomite. Both fatty acid and cationic flotation were tried for the elimination the insolubles. For fatty acid flotation, oleic acid emulsion was employed as the collector for dolomite and sodium silicate as the gangue depressant. Variations in fineness of the feed indicated that grinding the sample to about 80 per cent –200 mesh was necessary to obtain optimum results. Variation in sodium silicate additions indicated that the rejection of insolubles in the tailing was the maximum when 0·68 kg./ton of sodium silicate was employed. A test performed using 0·44 kg./ton of oleic acid emulsion and 0·68 kg./ton of sodium silicate yielded a rougher float assaying 2·7 per cent insolubles and having a weight per cent of 92·0. One cleaning of the rougher floats yielded a refloat concentrate assaying CaO, 30·4; MgO, 21·25; Insolubles, 1·75; SiO_2, 0·84; Al_2O_3, 0·70; Fe_2O_3, 0·70 and CO_2, 45·60 with a distribution of 21·8 per cent insolubles in it. The product is considered suitable for use in refractory industry.

Use of pine oil in conjunction with oleic emulsion considerably assisted in reducing the collector consumption to 0·22 kg./ton, but the rougher float was found to be high in insolubles and cleaning might therefore, be needed to improve the grade. Cationic flotation studies undertaken on the sample indicated that Amine 220, at a pH of 9·7 could suitably be employed, for flotation of siliceous gangue from the sample. The tailing (dolomite conc.) assayed 2·04 per cent insolubles and represented a wt. per cent of 84·9 The recovery of dolomite in the tailing could be improved by cleaning of the rougher floats. Pilot plant tests with the 100 tons sample will determine the technical feasibility of the process and its overall economics.

4.0 Beneficiation of Magnesite Sample from Tisco Employing Flotation

A 100 tons sample of magnesite from Tata Iron & Steel Works was received for pilot plant flotation studies in the Integrated Mineral Beneficiation Pilot Plant, of the National Metallurgical Laboratory. Laboratory scale flotation studies were initially undertaken, ahead of the pilot plant run, to define the optimum conditions for producing a magnesite concentrate low in silica. The sample as received assayed MgO, 45·9; CaO, 0·72; CO_2, 49·5; SiO_2, 3·0; Al_2O_3, 0·94 and Fe_2O_3, 0·36 per cent. Petrological examination of the sample indicated that quartz was the principal gangue followed by serpentine and micas etc. Magnesite was fairly well liberated at about 100 mesh. Flotation only was investigated for the elimination of siliceous gangue from magnesite. Fatty acid as well as cationic flotation studies were undertaken to produce a low silica magnesite concentrate. The flotation tests were conducted in 1000 grams Denver Sub 'A' cell, after grinding the sample to suitable fineness in a batch rod mill. It was found that 1 kg./ton of oleic acid emulsion was needed to recover most of the magnesite in the floats. The effects of variations in the fineness of the feed for flotation were studied which indicated that the sample would have to be ground to 92 per cent −200 mesh to produce good results.

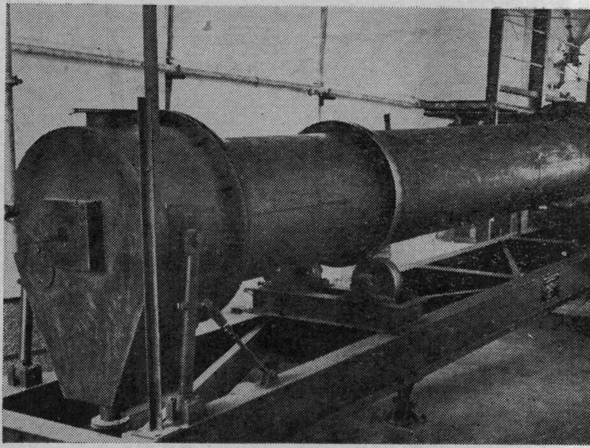
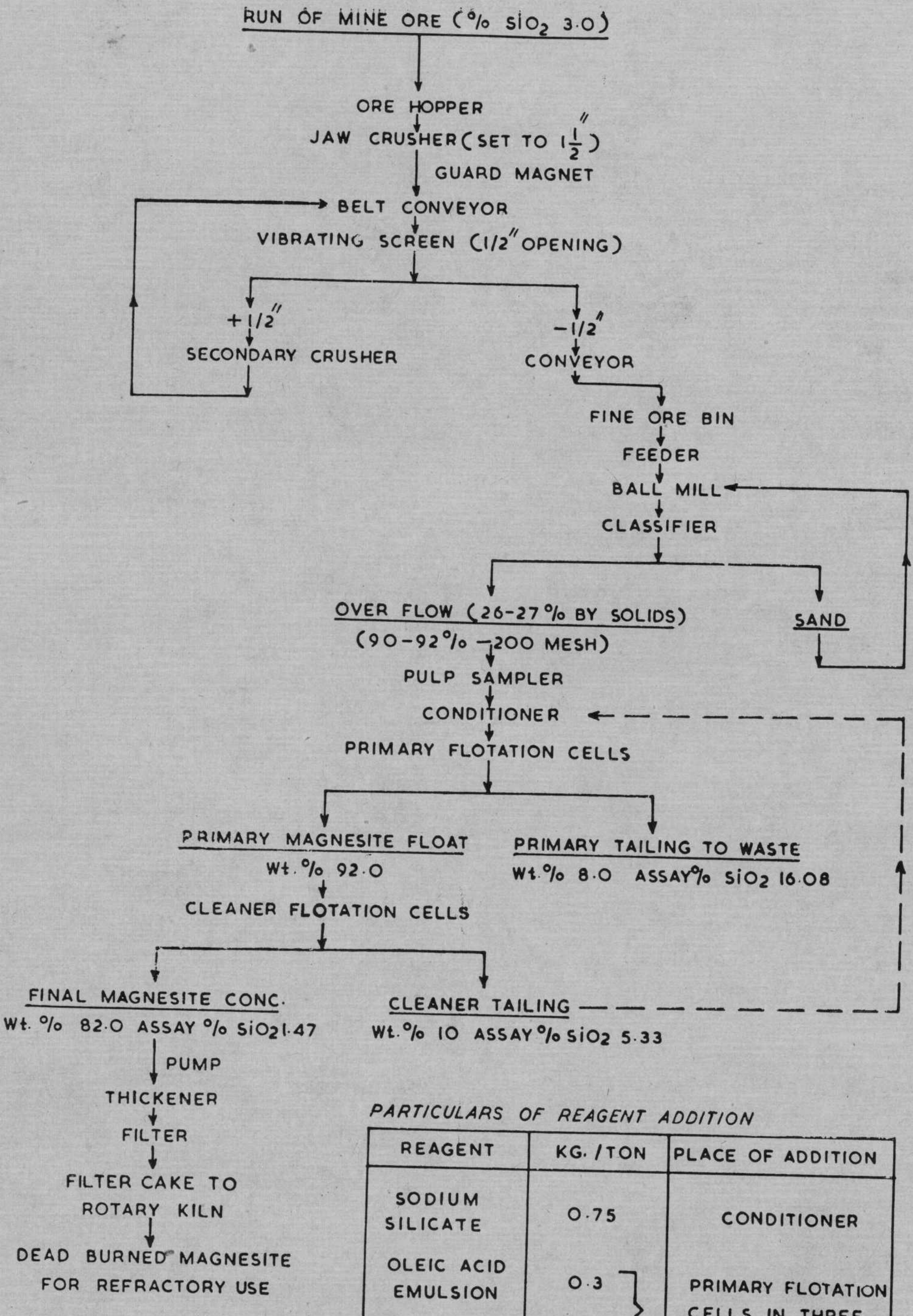


Fig. 14 — Rotary kiln for magnetizing roasting for manganese ores and pellet hardening. The feed end is in front. Size (3′ dia x 35′ long)

Since the collector consumption was as high as 1 kg./ton, attempts were made to reduce the collector consumption employing frother. A test performed using pine oil as frother, indicated that the collector consumption could be brought down from 1 kg. to 0·3 kg. and the results obtained were also equally satisfactory.

Reflotation studies performed on the sample indicated that magnesite concentrate having a SiO_2 range from 0·62 to 1·47 per cent could be produced depending upon the number of cleaning operations to which the rougher float was subjected to. It was established that one cleaning of the rougher magnesite float would produce a magnesite concentrate with a SiO_2 content of 1·4 per cent whilst representing by weight 82 per cent thereof. Recirculation of the cleaner tailings, as done in practice, was expected to increase the yield further. The results obtained from reflotation studies performed on a rougher float obtained from a coarser feed (62 per cent −200 mesh) were not satisfactory.

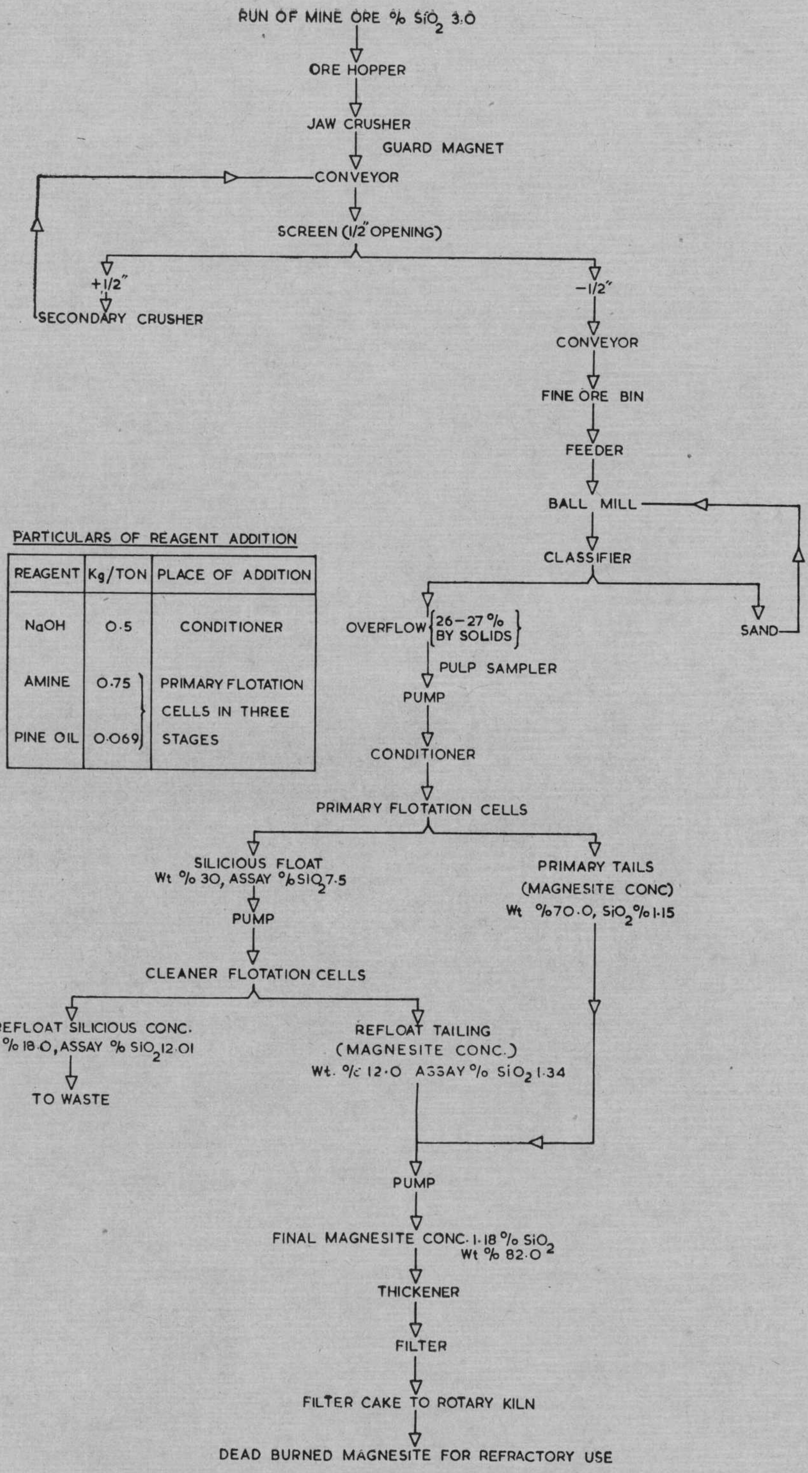
Cationic flotation studies performed on the sample, indicated that Do-Decyalmine could suitably be employed as collector for



PARTICULARS OF REAGENT ADDITION

REAGENT	KG. / TON	PLACE OF ADDITION
SODIUM SILICATE	0.75	CONDITIONER
OLEIC ACID EMULSION	0.3	PRIMARY FLOTATION CELLS IN THREE STAGES
PINE OIL	0.069	

FIG. 15 — FLOWSHEET SUGGESTED FOR THE TREATMENT OF TISCO MAGNESITE BY FATTY ACID FLOTATION



PARTICULARS OF REAGENT ADDITION

REAGENT	Kg/TON	PLACE OF ADDITION
NaOH	0·5	CONDITIONER
AMINE	0·75	PRIMARY FLOTATION CELLS IN THREE STAGES
PINE OIL	0·069	

FIG. 16 — FLOWSHEET FOR THE TREATMENT OF TISCO MAGNESITE BY CATIONIC FLOTATION

siliceous gangue leaving magnesite in the tailing. A magnesite concentrate having a SiO_2 content of 1·15 per cent, representing a recovery of 70 per cent could be thus produced. To improve the yield, the rougher siliceous float was subjected to reflotation and the resultant refloat tailing when mixed with the primary tailing gave a final magnesite concentrate assaying 1·18 per cent SiO_2 for a recovery by weight of 82·0 per cent. The final magnesite concentrate produced either by fatty acid or cationic flotation will have to be thickened, filtered and calcined in a rotary kiln as done in foreign countries for production of refractory magnesia from sea water. Based on the results obtained, flowsheets (Figs. 15 and 16) were suggested for flotation of magnesite from the Tata Iron & Steel Co.

Pilot plant studies, with the 100 ton magnesite sample will establish the technical feasibility of the process and its overall economics.

5.0 Beneficiation of Limestone Sample from Sirka

With the object of reducing the silica content of calcined lime, two samples of siliceous low grade limestone analyzing CaO, 34·95; SiO_2, 24·3 and CaO, 36·3; SiO_2, 21·8 per cent respectively were received from Messrs Bird & Co. The samples after calcination assayed CaO, 50; SiO_2, 34·7 and CaO, 52·0; SiO_2, 31·0 per cent respectively. Froth flotation did not yield satisfactory results. On the other hand, hydro-classification after slaking of the calcined limestone could yield concentrates assaying CaO, 64·46; SiO_2, 3·31 and CaO, 60·5; SiO_2, 6·0 per cent with lime recoveries of 64·7 per cent and 66·1 per cent respectively. These concentrates may be considered suitable for use in the building industry.

5.1 Beneficiation of Limestone Sample from Rajpipla, Narmada Project, Gujarat State

A 500 kg. sample from Rajpipla deposits was received from Assistant Engineer, Narmada Project for beneficiation studies so that the concentrate could be used for the manufacture of cement required for the project. The sample as received assayed CaO, 36·14; CO_2, 31·53; MgO, 3·2; SiO_2, 17·1; Al_2O_3, 7·31; SO_3, 0·73; Fe_2O_3, 2·98; S, 0·29 and P, 0·03 per cent. Petrological studies on this sample were undertaken followed by flotation studies. Full Report on the subject is under issue.

6.0 Beneficiation of Manganese Ore from Barajamda from Messrs Kharswan Minerals

The sample as received assayed Mn, 30·56; MnO_2, 44·46; SiO_2, 28·32; Fe, 10·22 and Al_2O_3, 4·66 per cent. Tabling after hydroclassification of the —35 mesh sample yielded a concentrate assaying Mn, 47·5; Fe, 9·27 and SiO_2, 5·7 per cent with a recovery of only 38 per cent Mn. Straight dry magnetic separation at —35 of the sample, yielded a concentrate assaying 43·0 per cent Mn with a manganese recovery of only 53·2 per cent Mn. The —3 mesh original sample was subjected to batch reduction roast treatment at 500-600°C. followed by low intensity magnetic separation, which yielded a non-magnetic product assaying Mn, 35·8; Fe, 4·04 and SiO_2, 37·3 with a recovery of 72·8 per cent Mn. The non-magnetic fraction was subjected to high intensity magnetic separation for removal of silica content. A full Report on the subject is under issue.

7.0 Beneficiation of Fine Grained Fluorspar from Ambadonagar, Gujarat

Two samples of fluorspar, one veinous variety and the other disseminated variety weighing about a tonne each, were received from the Geological Survey of India for beneficiation studies. The investigation covered only with the disseminated variety of fluorspar. The sample as received, assayed CaF_2, 34·7; SiO, 56; $CaCO_3$, 1·7; Al_2O_3, 1·72; Pb, trace; S, 0·3 and Fe_2O_3, 3·3 per cent.

Petrological studies on the sample showed that most of the fluorspar present in the sample was of violet variety followed by honey yellow light green and little white varieties. The chief gangue mineral was quartz with minor amounts of calcite, felspar, iron oxides and galena. Fair liberation of fluorspar grains occurred at sizes below 150 mesh.

Flotation tests using an optimum feed of 60·7 per cent −200 mesh, and employing 0·7 kg./ton of oleic acid emulsion and 0·8 kg./ton of sodium silicate, eliminated about 50 per cent by weight of gangue with 6-7 per cent loss of fluorspar. Cleaning of the rougher concentrate did not yield even a metallurgical grade of concentrate. The rougher concentrate was ground to about 200 mesh and cleaned several times using a combination of sodium silicate and 'Katha' in one case, and 'Katha' alone in other case. In both the cases, a metallurgical spar assaying CaF_2, 82·4-87; SiO_2, 6-6·2 per cent, and an acid grade spar assaying CaF_2, 96·6-97·9; SiO_2, 0·16-0·8 and $CaCO_3$, 0·4-0·7 per cent could be produced simultaneously, the overall recoveries being 79·5 and 82·1 per cent respectively. In actual practice, the recoveries were expected to considerably improve due to recirculation of the middlings. The concentrates being in a powdery form will need to be suitably agglomerated for effective handling and rail transport for use in metallurgical and steel industries. Based on the results obtained a general flowsheet (Fig. 17) was developed which could form the basis for setting up an upgrading plant for the production of both acid grade and metallurgical grade fluorspar concentrates from this sample.

7.1 Beneficiation of Coarse Grained Fluorspar 'V' from Ambadonagar, Gujarat

A coarse grained fluorspar sample assaying CaF_2, 70·0; SiO_2, 24·0; $CaCO_3$, 1·50; Al_2O_3, 0·40; S, 0·20 and Fe_2O_3, 2·10 per cent was received from the Geological Survey of India, for systematic beneficiation studies. The main gangue mineral was quartz whilst minor amounts of felspar, barite and calcite with traces of biotite and chlorite formed the other gangue.

Visual examination of the original sample showed the presence of an appreciable quantity of fluorspar grains free from gangue mineral, which indicated that gravity separation methods even at coarse size could be applied to obtain metallurgical grade of fluorspar.

Jigging of −6+20 mesh fraction of the original sample yielded a concentrate assaying CaF_2, 88·33 and SiO_2, 8·7 per cent with a recovery of 19·5 per cent CaF_2. Flotation of the jig tailing mixed with −20 mesh fines followed by four cleanings yielded two concentrates assaying (i) CaF_2, 88·92 and SiO_2, 5·13; and (ii) CaF_2, 97·81 and SiO_2, 1·21 per cent with recoveries of 13·4 per cent and 53·5 per cent CaF_2 respectively. Final loss of CaF_2 in the tailings was found to be 13·7 per cent which can be further decreased by recirculating the middlings in actual plant practice.

Heavy media separation of the original −1 in.+6 mesh sample yielded a concentrate assaying 88·1 per cent CaF_2 and 9·0 per cent SiO_2 with a recovery of 57·3 per cent CaF_2. Flotation of the heavy media separation float mixed with −6 mesh fines followed by four cleanings yielded again two concentrates assaying (i) CaF_2, 85·94 and SiO_2, 5·65 per cent; (ii) CaF_2, 96·50 and SiO_2, 1·52 per cent with recoveries of 9·7 per cent and 26·7 per cent CaF_2 respectively. Final loss of CaF_2 in the tailings was only 6·3 per cent; which can further be reduced by recirculating the refloat tailing in actual plant practice.

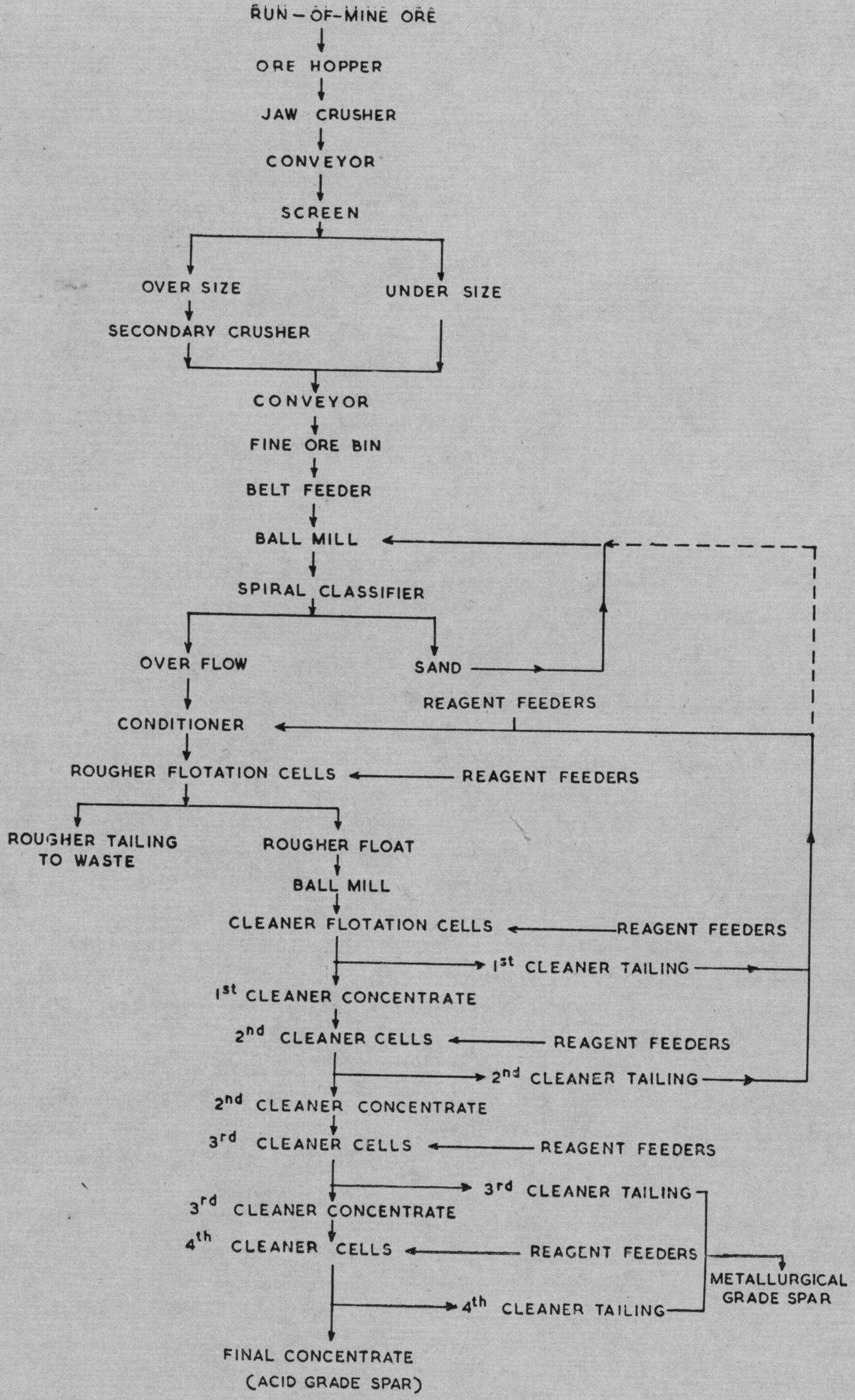


FIG. 17 — GENERAL FLOWSHEET FOR THE BENEFICIATION OF FLUORSPAR FROM AMBADONAGAR MINES, GUJARAT STATE

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Al_2O_3, 2·00; C
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The sample
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Direct flotation followed by four cleanings of the rougher float yielded concentrates (i) CaF_2, 97·0 and SiO_2, 1·44 per cent; and (ii) CaF_2, 89·1 and SiO_2, 5·1 per cent with recoveries of 65·5 per cent and 20·8 per cent CaF_2 respectively which could further be improved in an operating plant by recirculating the middling products.

A general flowsheet which could form the basis for setting up a commercial plant is shown in Fig. 18, employing a combination of heavy media separation and flotation for upgrading this coarse grained fluorspar sample. The results obtained with a fine grained fluorspar sample received simultaneously from the region had shown that flotation was the only method which could be employed for beneficiating that sample. Therefore, before putting up a plant to treat both types of fluorspar samples, several factors need careful consideration, viz. the ore reserves represented by the two varieties of fluorspar, capacity of the plant, market for the finished products, sizes of the finished products acceptable etc. A straight flotation plant can treat both types of ores for the production of acid and metallurgical grades of concentrate, but the choice of whether a gravity section could also be incorporated in the flowsheet can only be decided after a study of the various factors influencing the overall economics of the Project, its capital cost structure and operational expenses being major considerations.

8.0 Beneficiation Studies on a Low-grade Nickeliferous Ore from Assam

Beneficiation studies were conducted on a low grade nickeliferous ore from Moreh area deposits, Assam, received from the Geological Survey of India. The sample as received assayed Ni, 0·48; Fe, 8·94; SiO_2, 37·00; Al_2O_3, 2·00; CaO, 0·04; MgO, 32·83; Co, 0·05 and S, 0·146 per cent.

The sample consisted predominantly of green coloured serpentinous matrix with a small amount of fine opaque mineral grains disseminated throughout the matrix. The presence of any nickel mineral could not be conclusively established by petrological examination of the sample.

Wet magnetic separation of the sample after grinding it to −100 mesh and −200 mesh sizes did not yield satisfactory results. Tabling after hydro-classification of the −65 mesh ground sample followed by high intensity magnetic separation of the table concentrate also did not improve the grade to any desirable extent. The nickel content was found to be almost uniformly distributed in all the products indicating the futility of applying normal ore dressing methods for concentration of the sample.

9.0 Beneficiation of a Gypsum Sample from M/s. Bikaner Gypsum Ltd., Rajasthan

A low-grade gypsum sample assaying SO_3, 36·06; CaO, 28·9; CO_2, 2·95 and SiO_2, 8·22 per cent was received from Messrs. Bikaner Gypsum Ltd., Rajasthan; for the production of a concentrate suitable for making 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 per cent −200 mesh) yielded a concentrate assaying 46·3 per cent SO_3 (99·3 per cent gypsum) with a recovery of only 43·4 per cent SO_3. However, most of the gypsum lost in the cleaner tailings, may be recovered by re-circulation cycles 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.

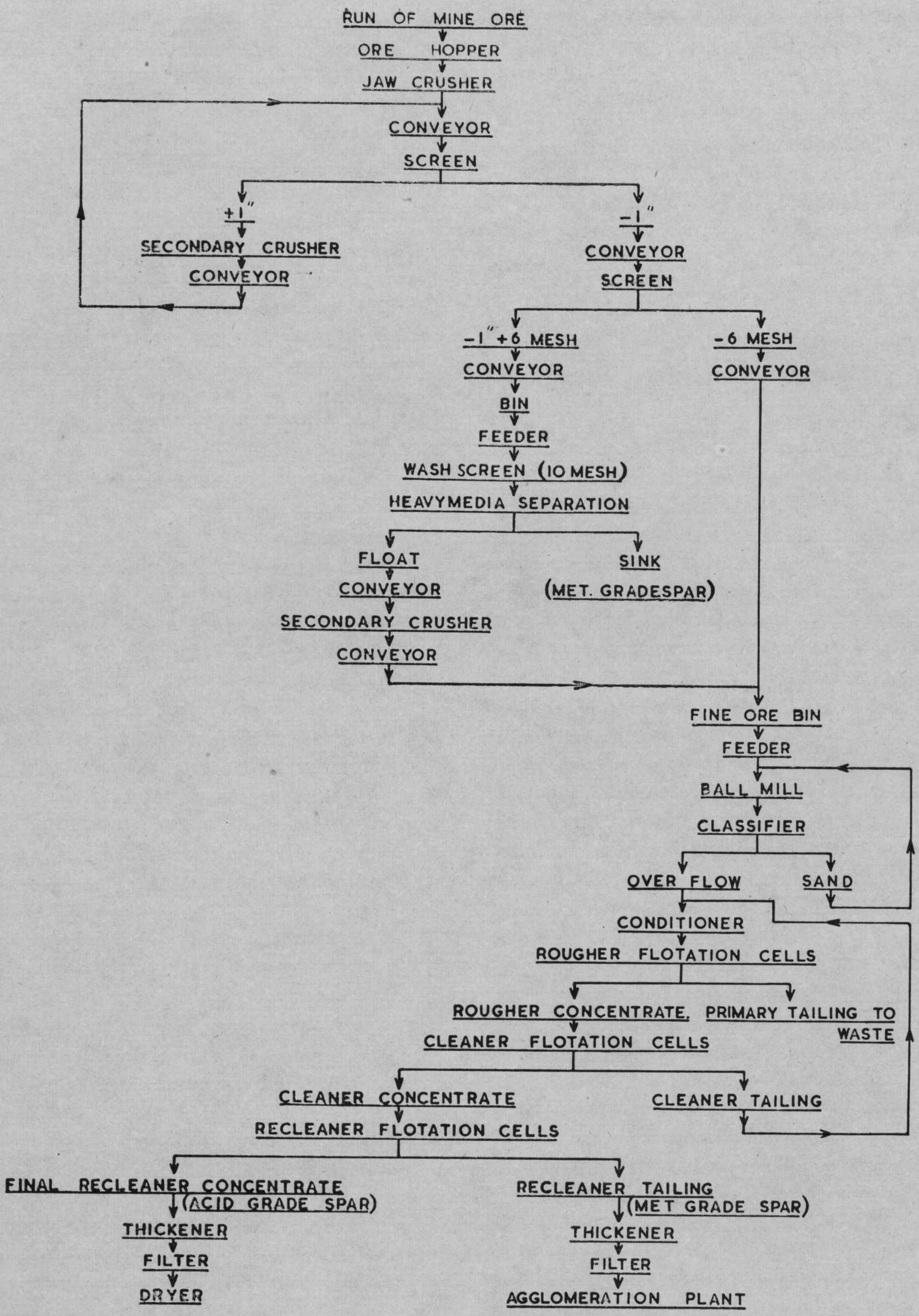


FIG. 18 — A GENERAL FLOWSHEET FOR THE BENEFICIATION OF COARSE GRAINED FLUORSPAR FROM AMBADONAGAR, GUJARAT, BY HEAVY MEDIA SEPARATION AND FLOTATION

10.0 Beneficiation of Selenite from Rajasthan

A 0·5 ton sample of selenite, from Thob area in Jalore District, was received from the Director of Mines and Geology, Rajasthan, to investigate the possibilities of upgrading it to " Plaster of Paris " grade. Petrological studies, followed by washing tests on the sample, are in progress to remove the adherent clay material.

11.0 Studies on the Morphology of Phosphorus in Indian Manganese Ores

Under the Coucil of Scientific and Industrial Research Fellowship Scheme, studies were carried out on the morphology of phosphorus in Indian manganese ores.

Nearly 150 polished ore sections and a few thin sections were examined and several chemical analysis were done to find out the relation between the phosphorus carrying minerals and other mineralogical constituents in the different manganese ores from Maharashtra, Madhya Pradesh and Rajasthan. From the studies carried out, e.g. ore microscopic and petrological, chemical analyses, isodynamic (magnetic) separation, x-ray powder photograph and contact print studies, the following conclusions were arrived at:

(i) Most of the ores studied were high in phosphorus content which varied between 0·2 and 0·58 per cent.

(ii) Chemical analyses of the sieve fractions showed that the phosphorus was uniformly distributed in all the sieve fractions.

(iii) Chemical analysis of the magnetic separation products indicated that in the majority of the cases, it was not possible to obtain a good manganese concentrate low in phosphorus. However, a good manganese with low in phosphorus could be obtained as a magnetic concentrate with rejection of phosphorus in the non-magnetic tailing in a few cases.

(iv) Most of the ores from Maharashtra and Madhya Pradesh were of primary origin with braunite type ore except the Dongribuzurg ore which was found to be secondary in nature with pyrolusite type ore. Rajasthan samples were also secondary in nature.

(v) Apatite was the only mineral which carried phosphorus in its chemical composition that could be identified. Although the manganese and iron minerals were suspected to carry phosphorus in solid solution, no definite identity thereof could be established.

(vi) Contact print studies on the polished ores ections indicated that in the majority of the cases, phosphorus was uniformly distributed. It was also observed that the siliceous bands were comparatively free from phosphorus reaction.

(vii) Garnets were found to act as carriers of phosphorus and their removal by electrostatic separation would be expected to reduce the phosphorus content to some extent.

(viii) Various modes and ways of formation and alteration of the manganese minerals suggested that the original composition of these minerals could be modified in various ways mainly depending upon the physico-chemical conditions. It was quite likely that the phosphorus may enter into the chemical composition of some of the unstable manganese minerals.

12.0 Flotation of Ferruginous Manganese Ore

Under the Council of Scientific & Industrial Research Fellowship Scheme, studies were continued on the separation of manganese minerals by flotation techniques. Flotation experiments were conducted with goethite using tannic acid as a depressant. Goethite

FIG. 19 — A WELMAN GAS PRODUCER (INSIDE DIA 42″) AND THE GAS HOLDER (CAPACITY 15,000 CFT) INSTALLED AT THE NATIONAL METALLURGICAL LABORATORY

did not float, thereby confirming the depressant action of tannic acid. Similar experiments were also conducted with psilomelane. The flotation ranges in this case, were *p*H 5·0-9·7 and *p*H 5·6-10·2. The complete data indicating the flotation ranges for the various minerals at different concentrations of tannic acid are given below: —

The system of geothite and psilomelane also gave identical flotation results in the sense that even though geothite was not expected to float, it floated partly and so did psilomelane. The systems of goethite-pyrolusite and psilomelane-hematite showed similar results indicating the absence of depressing action of tannic acid on goethite

Tannic acid conc. mg./100 ml.	Oleic acid mg./100 ml.	Flotation range (*p*H)			
		Pyrolusite	Psilomelane	Hematite	Goethite
0·5	1	3·7-10·5	—	3·7-6·9	—
1·0	1	3·9-10·1	—	3·9-6·9	—
1·5	1	3·9-10·0	—	4·0-6·9	—
2·5	1	4·7-10·1	5·0-9·7	4·5-5·9	Not floating
5·0	1	6·0-9·6	5·6-10·2	4·6-5·9	Not floating

whilst in association with the manganese minerals. Flotation of the iron minerals with tannic acid, in presence of the manganese minerals, may be either due to (i) adsorption of Mn ions liberated into the pulp by the manganese minerals on to the iron mineral surfaces, or (ii) the removal of tannic acid, adsorbed on the iron mineral surfaces. These features were verified by a series of tests conducted with hematite and goethite separately introducing known amounts of $MnSO_4$ into the tannic acid solutions prepared for the flotation. Tannic acid strength was kept constant at 5 mg./100 cc. and the $MnSO_4$ proportion was varied. Results are tabulated below:

$MnSO_4$ in the solution mg/100 cc.	Flotation range for hematite (*p*H)
0·5	7·9-9·9
1·0	2·9-6·5 & 7·0-10·4
2·5	2·9-5·6 & 7·0-10·4
5·0	3·1-5·9 & 7·5-10·7

The flotation behaviour of goethite was studied in a *p*H range of 6·2-9·9 with the $MnSO_4$ concentrations of 1 mg., 5 mg., and 10 mg./100 cc. of the pulp. It floated sluggishly. It might be concluded from these tests that the flotation of iron minerals was effected by the presence of manganese ions liberated into the pulp and goethite did not float as actively as hematite.

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

The pilot submerged arc furnace was installed 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 for the production of ferro-alloys. 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 will be accepted on contract for researches on their amenability to electro-thermal reduction techniques. A full report giving recommendations on smelting practice, power consumption and size of installation required for a given output will be made based on these smelting trials. During the period under review the furnace was operated for the production of high carbon ferro-chrome and silico-chrome.

Foundry grade high carbon ferro-chrome, conforming to the ISI Specification No. 1178/1950; 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 chrome ore was sieved to remove —1/8 in. fractions. The average sieve analysis was as follows:

Size fractions	Chromite ore %	Coke %	Quartzite %
$-1''$ to $+\frac{3}{4}''$	8·51	50·55	48·43
$-\frac{3}{4}''$ to $+\frac{1}{2}''$	16·00	8·33	36·48
$-\frac{1}{2}''$ to $+\frac{1}{4}''$	59·22	12·50	10·7
$-\frac{1}{4}''$ to $+\frac{1}{8}''$	9·22	9·03	2·51
$-\frac{1}{8}''$	8·05	19·59	1·88

The chemical analyses of chromite ore and coke were as follows:

Analysis of chrome ore	Per cent	Analysis of coke	Per cent
Cr_2O_3	51·45	Fixed carbon	70·9
FeO	10·63	Volatile matter	2·18
Fe_2O_3	4·47	Moisture	1·52
SiO_2	6·5	Ash	25·4
MgO	17·86		
CaO	0·2		
Al_2O_3	7·35		
P	0·016		

After preliminary heating for two days, the submerged arc ferro-alloy furnace was charged. The furnace operations were quite smooth; the tappings were quite easy. During the smelting campaign, the reductant was 170 per cent of the stoichiometric requirements; it was varied between 160 and 180 per cent, but for the grade of product needed, the above value gave the best results. The charge feed rate was 130 kg.

per hour during the test period. The power consumption was 5,300 kWH. per tonne. During the campaign, it was observed that 30 per cent of silicon contained in the charge was reduced to the metallic form. The raw materials requirements per ton of alloy were: chrome ore 2·283 tons, coke 0·8974 ton, quartzite 0·2586 ton, power 5300 kWH/ton. Recovery of metals in alloys were: chromium 73·71 per cent, iron 90·41 per cent, silicon 34·9 per cent, slag: metal ratio was 0·89. Average alloy analysis was Cr, 61·01; Si 8·6 and C, 6·5 per cent.

To study the production characteristics of low silicon high carbon ferro-chrome, two campaigns were undertaken. In course of these campaigns, the coke and quartzite in the charge burden were varied to get a product conforming to the ISI Specification No. 1170/1960 containing Cr, 65; C, 6 and silicon below 3 per cent.

Charge	Charge variations			
	A kg.	*B* kg.	*C* kg.	*D* kg.
Chrome ore	100	100	100	100
Coke	35	34	32	30
Quartzite	5	7	5	5

The alloy analysis progressively decreased in its silicon content and standard grade ferro-chrome was produced.

The sieve analysis of the raw material was as follows:

Size fractions	Chrome ore %	Coke %	Quartzite %
−1″+¾″	6·06	54·55	46·5
−¾″+½″	30·31	18·18	48·3
−½″+¼″	42·42	13·13	3·3
−¼″+⅛″	12·12	6·81	1·6
−⅛″	9·09	6·81	Nil

The alloy analysed Cr, 68; C, 8·01; and Si, 2·47 per cent. Average content of the chromium in the alloy was 67 per cent.

With the experience gained during the smelting campaigns, systematic studies were undertaken to study: (i) the effects of different particle size of raw materials on power consumption and chromium recovery, (ii) effects of varying the size fractions of the reductant on the electrical conductivity of charge and the electrical efficiency of the furnace.

Silico-chrome

Smelting trials were also conducted for the production of silico-chrome employing off-grade high carbon ferro-chrome. The charge composition during three periods of the short campaign were:

	High carbon ferro-chrome kg.	Coke kg.	Quartzite kg.	Magnesite kg.
Period 1	100	107	130	—
Period 2	100	109	160	5
Period 3	100	106	260	3

The high carbon ferro-chrome in the charge was crushed to 1-1½ in. size, rest of the burden material size being the same as given earlier After two days of preliminary heating, the furnace was put on the production of silico-chrome. The metallic nature of the burden did not give any trouble in furnace operation which could be well operated on automatic control system. It was practically a slagless operation; fluidity of the metal was good throughout the campaign, with the average power consumption at 5300 kWH per ton of silico-chrome produced. Composition of the alloys produced during the different periods was as follows:

Period		Si %	Cr %	C %	Fe %	Remarks
1.	A	21·0	52·34	2·5	23·09	
	B	20·61	53·15	4·1		
	C	20·74	51·21	2·03		
2.	A	26·6	48·38	3·40		Increased SiO_2 in charge
	B	28·4	42·79	2·22		
	C	30·7	42·1	—		
3.	A	37·0	39·27	0·27		Increased SiO_2 and MgO in charge
	B	36·59	36·64	0·20		
	C	36·54	33·63	0·40		

It was found that only 30-35 per cent of the charge entered the metal. Effects of varying MgO contents in the charge were investigated.

14.0 Smelting of Vanadium Bearing Titaniferous Magnetites

Vanadium bearing magnetites occur in large quantities in Bihar and Orissa. The vanadium content of the magnetites averaged from 0·6 to 1·8 per cent. The TiO_2 and Fe contents ranged from 8 to 12 per cent and 55 to 65 per cent respectively. It was proposed to reduce the ore with suitable flux in the ferro-alloy submerged electric arc furnace. The iron and vanadium in the ore would be reduced to form a vanadium bearing pig iron. The vanadium bearing pig iron could thereafter be subjected to oxidation by blowing air or oxygen over the molten pig iron in a converter and all the vanadium thereby oxidised to form a vanadium-rich slag with a vanadium content ranging from 10 to 20 per cent. The slag so obtained could further be treated for the production of V_2O_5 by roasting and leaching cycles. The chief advantages in the above process were (a) bulk of material to be handled in roasting and leaching treatment for vanadium pentoxide production was considerably reduced, (b) iron values from the ore could thereby be simultaneously recovered, (c) titanium rich slag was obtained for recovering TiO_2 by any established processes.

Trials were initially carried out in a gas fired furnace. The charge consisting of a mix of ore, coke and limestone crushed to

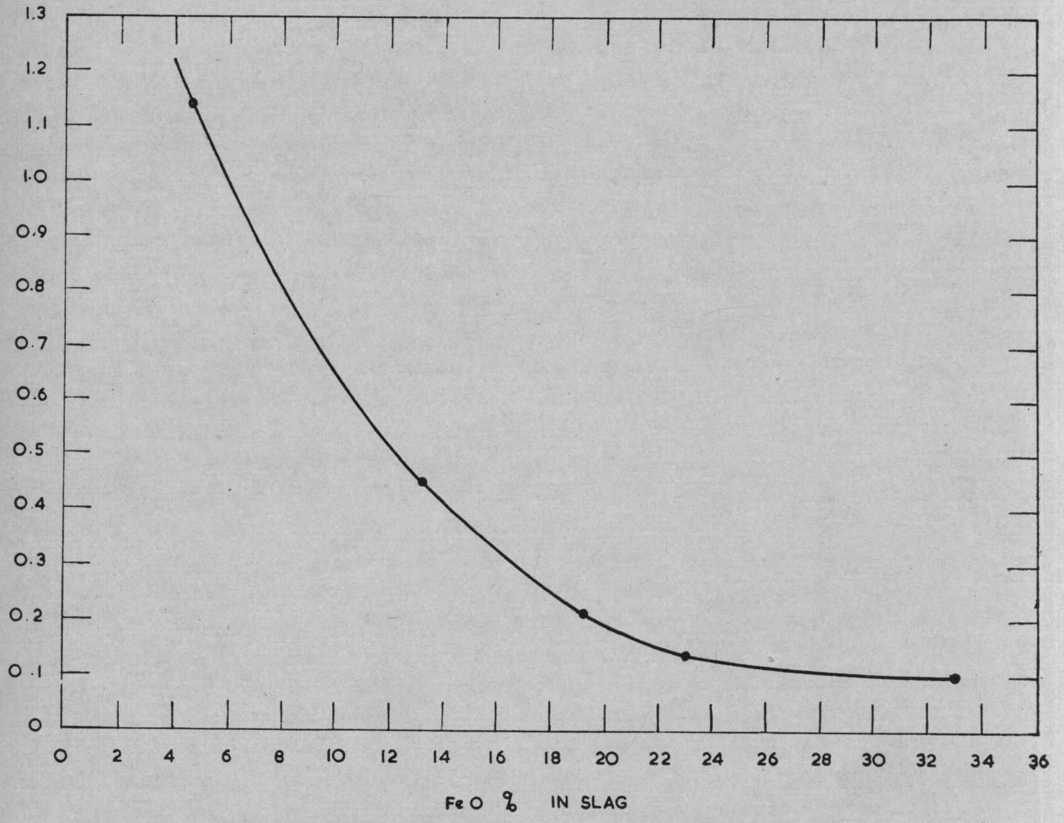


Fig. 20 — The relation between FeO in slag and vanadium in the metal

—80 mesh was heated in a graphite crucible. The maximum temperature obtained in the gas fired furnace was around 1400°C., which was not high enough for the purpose and a maximum recovery of 90 per cent iron and 30 per cent only vanadium was obtained. The slag composition with TiO_2, 45·5; CaO, 22·7; SiO_2, 24·8 and MgO, 6·8 per cent was aimed at to provide the lowest melting point and free flowing slag for the raw materials used. Further experiments were not carried out in the gas fired furnace as the vanadium recoveries were poor.

Trials were carried out in a 50 lb. capacity 50 kVA. direct arc furnace. Ore lumps of size —½ in. with —3/4 in. coke and 1/2 in. limestone were smelted together. A maximum recovery of 56·7 per cent vanadium was thus obtained. Good reduction could not be effected as the slag became viscous due to its attack on the magnesite lining of the furnace shell. Coke was found floating and the reduction took place only at the slag coke interface.

A 50 lb., 50 kVA. rocking type-indirect arc furnace, lined with magnesite was used for subsequent trials. The charge constituents, viz., ore, coke and limestone were crushed to —40 mesh (B.S.S.) mixed together with 2 per cent molasses as a binder. Briquettes were dried at 120°C., and found to be hard and entirely suitable for furnace charging.

Preliminary trials carried out, varying only the carbon contents in the charge mix, indicated that the vanadium content in the pig iron increased with decreasing iron oxide content of the slag. A recovery of 61 per cent vanadium was obtained with the slag having 4 per cent iron. It was also observed that a minimum of about 5 per cent iron would have to be maintained in the slag in order to render the slag fluid. Overreduction of the slag rendered the slag highly viscous as overreduction was accompanied by the formation of titanium carbides and nitrides which contributed to the high viscosity of slags. Studies were undertaken to determine the effect of slag basicity on the vanadium recovery and its fluidity etc. Results of the smelting tests carried out, varying the carbon contents of the charge are shown in Fig. 20.

15.0 Extraction of Magnesium Metal Electrolytically from Magnesium Chloride

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

The diaphragm composition developed after several previous trials effectively withstood the corrosive action of the molten electrolyte. The working of the cell at a current load of 360 amp. was not smooth. Cathodically liberated metal could not be directed to the metal collecting pool due to lifting action of anodic chlorine. Liberated magnesium got burnt with chlorine. The cell pot also failed to withstand the working conditions, cracked and leaked. Hot spots developed on cell body opposite to the gas burner. The oxide content of the charge and that formed during fusion of the charge were substantially heavy which altered the bath composition. The oxides instead of settling down remained as a colloidal matter in the electrolyte. These factors gave rise to series of failures. Metal collection was small with very poor efficiency of metallic recoveries. Attempts to rectify some of the defects, such as dipping the bath to adjust composition, reducing the voltage and current to reduce the anode gas lift agitation, placing a low voltage A.C. to clean the cathode surface, reducing the coke oven gas to minimise the hot spot, etc., did not materially improve operating conditions. On the other hand, electrolyte got considerably thickened with the drop of working temperature. The trial was stopped after 22 hours of continuous operation.

To rectify the difficulties encountered, the cell pot design, the furnace arrangement and the position of the gas burner were changed so that the gas did not strike the cell vessel directly. The dimensions of the cell pot were increased to provide more space below the cathode; the top part above cathode was isolated leaving enough space for chlorine. Separate arrangements for feeding the charge were provided. Conditioning of molten charge prior to charging with a view to discard the major portion of oxides was arranged separately. The cell along with all other accessories was installed for further trials.

16.0 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.

Caustic soda bath was used in the electrolysis with tinplate scrap as anode and either the side walls of the steel tank holding the electrolyte or steel plates at fixed positions, as cathode. The tin was leached out with the help of anodic oxidation and deposited at the cathode in spongy form, which was scrapped out, washed, dried and melted. The detinned steel formed a valuable by-product. The electrolytic tin was 99·8 per cent pure and could be used for solders and other alloys. Following are the operating conditions and results of a typical electrolysis:

Amount of tinplate scrap (used as anode) — 300 gm. (1·61 per cent tin)

Volume of electrolyte — 2 litres containing 70/g/L NaOH and 8 g/L Na-acetate and 10 ml/1 of 1 per cent glue soln. as addition agent.

Cathode — Two stainless steel sheets of 5 in. × 3 in. × 1/16 in. one at each side of the anode. Effective surface area 54 Sq. in.

Temperature — 60-70°C.

Current density at cathode — about 14 amps./sq. ft.

Voltage — 2·1 V.

Time of electrolysis — 1 hour.

Experimental Results

Tin metal recovery as cathode deposit	97%
Detinning of scrap at anode	98%
Purity of deposit	99·8%
Current efficiency at cathode	70·8%
Power consumption per kg. of tin metal deposit	2·25 kWh.

17.0 Production of Ferro-alloys by Alumino-thermic Reactions

The alumino-thermic technique for the production of ferro-alloys is to-day well established for their versatility, ease of operation and adaptability on industrial scale to produce carbon-free and pure ferro-alloys. The process consisted in the reduction of oxides by aluminium which combined with the oxygen content of the metallic oxides until these were consumed and the slag consisting mostly of aluminium oxide and a metal regulus was thereby obtained. The reactions were highly exothermic and high temperatures were thus obtained. The development of alumino-thermic technique is of considerable importance under Indian conditions as the industry can be established without involving high capital cost structure or expenditure of foreign exchange. The following carbon-free ferro-alloys were thus produced:

Ferro-tungsten: The ferro-tungsten analysed as W, 74-77; Fe, 20-25; Si, 0·5-1·3; Al, 0·1-0·3; and Mn, 0·13-0·45 per cent.

Ferro-chrome: The ferro-chrome analysed Cr, 77-80; Fe, 18-20; and Al, 0·5-1·0 per cent.

Ferro-titanium: The ferro-titanium from ilmenite analysed Ti, 25-27; Al, 4·5-8; and Fe, 63-65 per cent.

18.0 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 currently 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

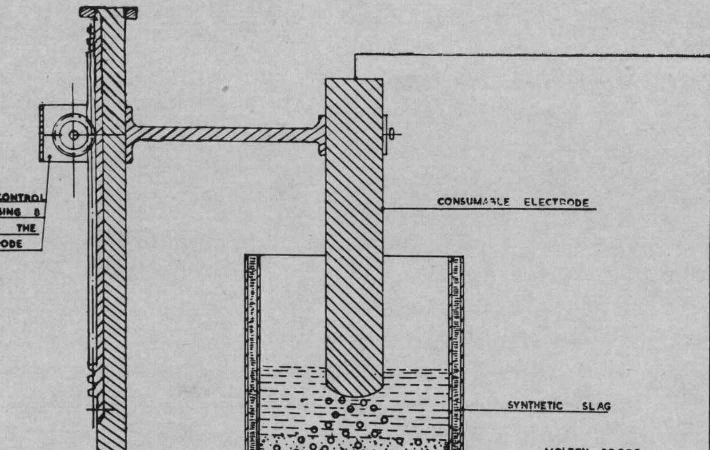


FIG. 21 — SCHEMATIC DIAGRAM FOR ELECTRO-SLAG REMELTING

metallurgical field. Since the process has potential possibilities in the manufacture of special steels with indigenous resources, a project was taken up at the National Metallurgical Laboratory to develop requisite technical know-how of the process especially suited to Indian conditions.

Basically, the process consists of refining an alloy made by conventional melting and processing methods. Ingots made to specification by conventional steel-making are forged or rolled to suitable size billets to be used as consumable electrodes, in electro-slag remelting and refining. Fig. 21 shows a schematic diagram of the set-up. A low voltage high current arc is maintained between the electrode and the water cooled mould with a steel pad at the bottom through a synthetic slag consisting mainly of fluorspar and alumina. A small quantity of self fusing slag containing Al-Mg alloy is also used to initiate the reaction.

During the electro-slag refining, the temperature of the slag is of the order of 1700-1900°C. which results in the melting of the electrode and molten metal drips through the slag in the form of droplets whereby it gets adequately refined. Sulphur content of the electrode material is reduced by over 30-60 per cent (depending on the initial sulphur content of the metal) non-metallic inclusions are few and the refined product is much purer. By electro-slag refining, the physical properties are considerably enhanced. The cast ingot surface is much better in relation to metal-mould reaction in conventional melting.

Trials were carried out in a 2 in. diameter water-cooled steel mould. Fig. 22 shows a remelted electrode. Fig. 23 shows the worked ends of the two electrodes. End (a) indicates the absence of molten slag cover on the remelted material while (b), a clean end, is an indication of a regular molten slag cover all through the experiment.

Typical chemical analyses of the electrode and the remelted material are given in Table 3.

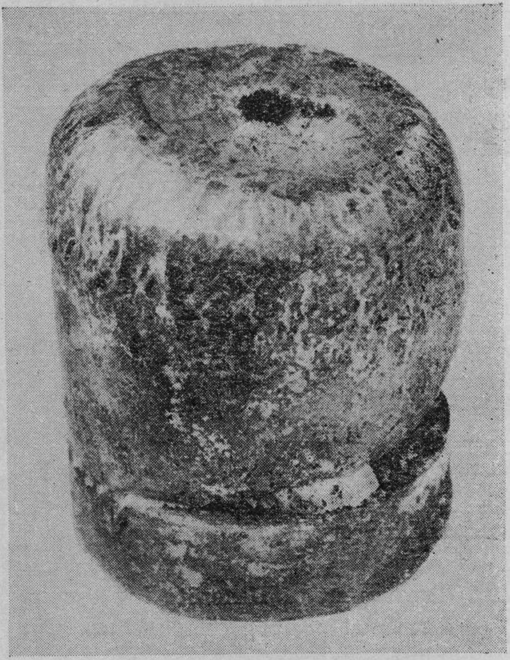
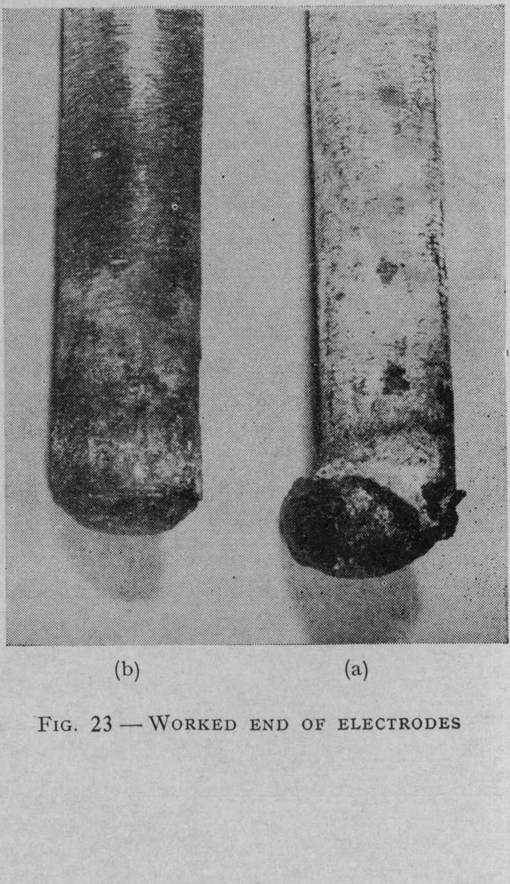


FIG. 22 — REMELTED ELECTRODE



(b) (a)

FIG. 23 — WORKED END OF ELECTRODES

Table 3 — Chemical Analyses of the Electrode and Electro-slag Remelted Electrode

Exp.	Sample	C %	S %	P %	Si %	Mn %	Al %
I	Electrode	0·24	0·03	0·05	0·06	0·64	Trace
	Remelted electrode	0·21	0·008	0·04	0·14	0·61	0·48
II	Electrode	0·21	0·028	0·018	0·11	0·60	Trace
	Remelted electrode	0·17	0·01	0·019	0·12	0·56	0·02

Further work is in progress. Macro and micro-structures and the mechanical properties are under study. It is proposed to study the effect of this process of remelting on the manufacture of high quality alloy steel and sinter-alloys.

19.0 Treatment of Ilmenite for the Removal of Iron, Chromium and Vanadium

The beach sands of Kerala contain large deposits of ilmenite associated with rutile, zircon, monazite etc. The Indian ilmenites though high in titania are associated with small quantities of chromium and vanadium. The treatment of ilmenite with concentrated sulphuric acid to produce pigment grade TiO_2 not only consumes considerable quantity of sulphuric acid, but also the ferrous sulphate thus produced has no indigenous market and generally it is dumped as waste. The present study was undertaken to develop a suitable process for the removal of undesirable constituents.

The ilmenite concentrate from Kerala employed in the present investigation analysed TiO_2, 48·22; FeO, 32·91; Fe_2O_3, 14·32; Cr_2O_3, 0·07 and V_2O_5, 0·21 per cent. Systematic studies on the reduction and removal of iron were carried out. It was observed that about 25 per cent of the total iron could be removed from the reduced calcine without any acid treatment. Percentage of TiO_2 increased from 48 to 65. Some novel techniques for the removal of reduced iron are being systematically examined discarding the known methods for effecting such refining cycles.

20.0 Pilot Plant Study on the Recovery of Vanadium from Vanadium Bearing Titaniferous Magnetite

Vanadium bearing titaniferous magnetite ore occurs in large quantities in Bihar and Orissa; these ores analyse on an average, V, 0·6-1·8; TiO_2, 8-12 and Fe, 55-65 per cent. Based on the laboratory scale studies, a pilot plant to treat one ton of the ore per day was designed and fabricated at the National Metallurgical Laboratory to study the roasting, leaching and precipitation conditions on a fairly large scale.

Vanadiferous magnetite containing 0·9 per cent vanadium collected from Dublabera (Orissa) was crushed and ground to —30 mesh B.S.S. Systematic studies on roasting conditions were studied. Twelve per cent Na_2CO_3 mixtures gave a recovery of 70 per cent vanadium. Experiments were undertaken to obtain data on the rate of dissolution of vanadium and to study the settling characteristics of the leached residue. A Project Investigation Report was prepared for a plant with a production capacity of 100 tons of V_2O_5 per year.

The use of soda ash for roasting entails heavy acid consumption during the precipitation of vanadium pentoxide and as an alternative to soda ash, common salt was tried only for roasting purposes. In the laboratory scale experiments, it was observed that poor recoveries were obtained, unless oxidizing conditions were effectively maintained. The effect of roasting temperature on the vanadium recovery is given in Table 4.

Table 4 — Effect of Roasting Temperature on the Vanadium Recovery

Ore = 200 *gm.*
NaCl = 24 *gm.*
Time = 105 *min.*

Temperature of roasting °C.	Percentage vanadium rendered soluble
700	10·6
750	30·3
800	35·4
850	56·2
875	60·5
900	60·4

Large-scale experiments in a rotary kiln were carried out and similar recoveries as those obtained by soda ash roasting were thereby obtained. Laboratory scale experiments were also undertaken to determine the optimum conditions for the precipitation of vanadium as ammonium vanadate from salt-roast leach liquors. Vanadium pentoxide obtained by ammonium vanadate decomposition analysed 98 per cent V_2O_5. Experiments were also undertaken to produce high purity vanadium pentoxide by solvent extraction.

21.0 Reconditioning of Magnesium Powder

Magnesium powder during storage for long periods deteriorated due to the formation of oxide coating on the metallic surfaces and could not be used in pyrotechnics. The investigation was taken up at the instance of the Defence establishments for improving the metallic content to over 99 per cent form powders containing less than 80 per cent metallics.

Magnesium powder of grades 0, III, IV and V was reconditioned to their required specifications of over 99 per cent metallic magnesium from their original metallic contents ranging from 65 to 80 per cent.

Reconditioning temperature and drying procedures had to be completely modified in relation to earlier methods formulated at the National Metallurgical Laboratory when dealing with very fine magnesium powders which tended to readily reoxidise if the treatment methods regarding the contact period with acid, washing and drying conditions were not carefully followed.

22.0 Reducibility of Kiriburu Iron Ores and Sinters

Reducibility studies on unwashed, washed and beneficiated Kiriburu iron ores is being systematically studied at the instance of Hindustan Steel Ltd, with a view to evaluate optimum crushing size offering maximum reducibility for iron smelting in the projected Bokaro Steel Plant of the Government of India. Likewise, reducibility tests of non-fluxing and fluxing sinters were carried out for evaluating the effect of different variables such as mix composition, degree of oxidation, basicity, amount of return fines, etc.

Reducibility of Ore Samples:

Reducibility tests of lump iron ores of Kiriburu samples from two different lots were carried out. The former being from Hill No. 1 Bench 3 and Main haulage road — Upper position, mixed in the proportion of 1:4 respectively while the latter is a combination of Hill No. 1 Bench No. 2 and Hill No. 2 Bench No. 2 mixed in the proportion of 1:1. The lumps represented different physical characteristics in respect of colour, specific gravity and mineralogical constituents. Reducibility tests were carried out on several ore lumps under each size grading of −50+10 mm., −75+50 mm., −100+75 mm.

The sample was taken in a stainless steel tube kept in a horizontal tubular furnace.

It was heated to 800°C. in nitrogen. When the temperature was steady, the sample was reduced with hydrogen gas flowing at a constant rate. The product of reaction namely water was condensed by passing through a condenser which was externally cooled with ice cold water. The percentage reduction was thereafter calculated. The reducibility index was taken as the time required for 90 per cent reduction of the ore. The results obtained on reducing ores of washed ores from Lot I are given in Table 5.

Table 5 — Results of Reducibility Tests Carried on Washed Ores from Lot I

Temperature — 800°C.
H_2 flow — 3·8 *litres per minute.*

Sample No.	Physical character	Size in mm.	Weight of sample in gm.	Time for 90% reduction in min.
1	Porous laminated ore	−40+30	160	80
2	Massive laminated ore	−50+40	268	145
3	Massive laminated ore	−50+45	341	160
4	Massive and hard	−50+45	360	172
5	Porous with laterite material	−50+45	304	100
6	Porous, laminated with occasional blue dust laminae	−60+50	365	150
7	Porous, laterite ore	−50+45	265	70

Experiments carried out with −50+45 mm., −40+30, −60+50 mm. samples exhibiting various physical characteristics showed that the time required for 90 per cent reduction ranged from 70 min. to 172 min. It was observed that the reducibility was dependent on porosity of the sample. The laminated massive ores on reduction were found to be cracked and this facilitated the reduction. Further experiments are under progress with −100+75 mm., −75+50 mm. size samples of lump ores.

Reducibility of Sinters:

Reducibility tests were done on sinters produced from −10 mm. size fraction of washed and beneficiated Kiriburu iron ores. Non-fluxed and self-fluxing sinters prepared under different conditions were examined. Reducibility tests were done by loss-in-weight method in a thermal balance. 150 gm. of −67+47 mm. size fraction of various sinters were reduced with hydrogen, flowing at a constant rate of 3·8 litres per minute at 800°C. The course of reduction was followed by the loss-in-weight of the sample. The time required for 90 per cent reduction was taken as the reducibility index. The relative reducibility of different sinters are indicated in Table 6.

Table 6 — Relative Reducibility of Different Sinters

Sample weight = 150 *gm.*
Size = −67+47 *mm.*
Temperature = 800°C.
Hydrogen gas flow — 3·8 *litres/min.*

Nature of sinter	Basicity	FeO	Time required for 90% reduction	Remarks
Beneficiated ore	—	—	105	—
Unbeneficiated ore	—	—	110	—
Non-fluxing	—	24·4	115	—
Non-fluxing	—	18·7	95	4% coke addition
Self-fluxing	0·8	—	85	Limestone addition
Self-fluxing	1·0	—	85	do
Self-fluxing	1·4	10·8	80	do
Self-fluxing	1·6	—	80	do
Self-fluxing	1·4	—	125	Limestone and dolomite
Self-fluxing	1·6	9·3	106	Limestone and dolomite
Self-fluxing	1·6	5·8	69	2·1% coke addition only

It was observed that reducibility of the non-fluxed sinters were similar to the beneficiated ore. The self-fluxing sinters had much higher reducibility than non-fluxed sinters. With the increase of basicity from 0·8 to 1·6, time for 90 per cent reduction decreased from

85 to 80 minutes. It was also observed that self-fluxing sinters prepared with limestone and dolomite additions possessed slightly lower reducibility than that prepared with limestone alone. Self-fluxing sinters prepared with 2·1 per cent coke instead of the usual 4 per cent coke additions in other sinters required only 69 minutes for corresponding reduction. It was observed that reducibility of the various sinters improved with the decrease of their FeO contents. Further experiments are under progress with different types of sinters prepared under varying conditions.

23.0 Utilization of Ferro-manganese Slag

The investigation was taken up with a view to recover the manganese value of ferro-manganese slag in the form of silico-manganese.

Ferro-manganese slag produced during the production of high carbon ferro-manganese by electric smelting contains about 16-20 per cent manganese. In order to recover manganese by pyrometallurgical process, various reducing agents were tried. It was found that silicon and carbon were not efficient reducing agents for recovering manganese from such slags. The feasibility of recovering manganese in the form of silico-manganese by aluminium reduction was studied. Experiments were carried out in salamander crucibles in a batch type gas fired furnace. Manganese slag treated with varying amounts of aluminium additions indicated that manganese recovery was very poor with the theoretical quantity of aluminium required for the reduction of manganese in the slag, whereas with 100 per cent excess of aluminium over the theoretical requirement, an alloy containing 44·5 per cent Mn and 30 per cent Si, with 85 per cent manganese recovery could be obtained.

In order to improve the manganese to silicon ratio in the alloy to conform to standard grade silico-manganese, manganese content of the slag was enriched by the addition of pyrolusite. After melting of the slag with MnO_2 and limestone, aluminium was added and the experiment was carried out for 1 hour. Silico-manganese containing 71 per cent manganese, 21 per cent silicon with traces of aluminium was produced. Experiments with varying slag basicity values from 2·0 to 3·5 indicated that whilst manganese recovery was improved at an optimum basicity of 3, the grade of the alloy could not be further improved. With a basicity of 3·5, the slag became viscous and the recovery of manganese dropped. Further experiments were undertaken to decrease the silicon content of the metal and also to improve the overall manganese recovery.

24.0 Studies on the Recovery of Alumina from Indian Bauxite

Studies were taken up to determine the suitability of Bayer's process for the recovery of alumina from Indian bauxites. The properties studied included mineralogical and chemical composition and factors which affected the alumina extraction, viz., particle size, concentration of alkali, molar ratio of caustic to alumina, leaching period and calcination temperature.

Bauxite sample from Lamba Mines, Gujarat

The bauxite sample received analysed Al_2O_3, 59·60; SiO_2, 1·34; Fe_2O_3, 6·54 and TiO_2, 2·24 per cent. Petrographic examination of the sample revealed that cliachite was the predominant alumina mineral, though a very minute quantity of gibbsite and kaolinite were also present. The sample was found to contain 92·49 per cent alumina as cliachite, 5·6 per cent alumina as bohemite and 1·91 per cent alumina as diaspore. It was observed that 92·23 per cent of the Al_2O_3 could be extracted after digestion for 60 minutes. The ore crushed to −10 mesh was leached with caustic soda solution containing 265 gm. per litre

(Na_2CO_3 basis) and $Na_2O:Al_2O_3$ as 2:1. The effect of calcining bauxite at various temperatures on its solubility in caustic solutions showed that the Al_2O_3 recovery decreased from 92·23 per cent at 100°C. to 68·23 per cent at 500°C. which was due to conversion of the readily soluble trihydrate into a difficultly soluble monohydrate.

Bauxite Sample from Lamba Mines, Zanjaria, Gujarat

The bauxite sample received from Lamba Mines, Zanjaria, Gujarat analysed Al_2O_3, 55; SiO_2, 2·05; Fe_2O_3, 5·40 and TiO_2, 3 per cent. Petrographic examination of the sample revealed that the ore consisted chiefly of amorphous cliachite and minor amounts of cryptocrystalline gibbsite, relict rutile and iron oxide as a coating on the alumina minerals. The sample was found to contain 90·10 per cent alumina as cliachite, 6·98 per cent as bohemite, 2·92 per cent as diaspore with trace of kaolinite. It was observed that 93-94 per cent of the Al_2O_3 could be extracted by digesting the ore prior crushed to $-10+100$ mesh for an hour with a caustic soda solution containing 265 gm. per litre (Na_2CO_3 basis) and a ratio of $Na_2O: Al_2O_3$ as 2:1.

Bauxite sample from Jantilal and Chunilal Co. Ltd.

The ore sample analysed as Al_2O_3, 64·03; SiO_2, 1·16; Fe_2O_3, 2·3 and TiO_2, 4·15 per cent. It contained 76·94 per cent alumina as gibbsite, 8·96 per cent as bohemite, 14·02 per cent as diaspore and 0·98 per cent as kaolinite. It was observed that 74·4 per cent alumina could be extracted after digestion for an hour. The ore was crushed to -10 mesh and leached with caustic soda solution containing 265 gm. per litre (sodium carbonate basis) and $Na_2O:Al_2O_3$ ratio as 2:1.

25.0 Studies on the Recovery of Vanadium from its Sodium Complex Obtained from the Bayer's Process Liquor

Vanadium content of the bauxite ores of Bihar averaged 0·05 per cent and nearly 30-35 per cent of the vanadium entered in the Bayer's process liquor for alumina extraction. The vanadium concentration cannot be allowed to build up in the cyclic process liquors as its presence in the aluminium adversely affected the latter's electrical properties. However, if the process liquor is cooled to 25°C., the vanadium separated only as a complex salt containing phosphate, alkali etc. Systematic studies on the production of high purity V_2O_5 from the sodium complex salt produced by the alumina plant were taken up.

The sodium complex salt analysed as follows:

	Per cent
Free alkali (as NaOH) . . .	23·60
Ca . . .	0·1
PO_4 . . .	12·8
V_2O_5 . . .	6·2
SiO_2 . . .	0·14
Al_2O_3 . . .	30·0
Na_2O . . .	19·9

Preliminary experiments were carried out for studying the optimum conditions for the dissolution of vanadium and phosphate in water as well as the separation of the part of the phosphate from the liquor by fractional crystallization. The sodium complex salt was dissolved in water with continuous stirring for 1 hr. and the leach liquor was separated from the insoluble part.

The insoluble part of the complex salt was 42·5 per cent of the original and analysed as follows:

	Per cent
Al_2O_3 . . .	62·5
PO_4 . . .	5·5

Loss on ignition at
750°C. for 4 hrs. = 32·6 per cent

The leach liquor containing 7·3 gm. per litre of vanadium and 20·73 gm. per litre of PO_4 was allowed to recrystallize in order to eliminate the PO_4 and free alkali. Results of recrystallization from 400 c.c. of leach liquor step by step is given below:

Degree of evaporation %	Amount of crystals (in gm.)	PO_4 in crystals (in gm.)	Vanadium in crystals (in gm.)
25	1·42	0·3721	0·02203
25-37	2·9	1·595	0·04163
37-50	6·3	2·65	0·05657

Further work has related to the separation of the vanadium in the liquors by precipitation as ammonium vanadate.

26.0 Treatment of Zinc Waste 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 a ton of zinc consumed in galvanizing, about 0·24 ton of zinc is lost in the form of zinc dross; on an average, for every ton of zinc dross produced, about 0·8 ton blowings, 0·5 ton skimmings and 0·7 ton ashes are obtained. This project aimed at the recovery of the metallic value from various secondary products for further use in galvanizing industry. Distillation under low pressures or at atmospheric pressure could be suitably applied for obtaining zinc in pure state. Finer fractions of metallics may be classified and treated for oxide removal for applications in the chemical industry. Zinc salts may be suitably treated to obtain pigment grade zinc oxide.

Samples of zinc dross, blowings, skimmings and ashes were received from Messrs. Indian Tube Co. Ltd., for recovering the metallic zinc. Zinc ash and blowings were sieved into different fractions and the chemical analysis of the fraction together with the analysis of zinc dross and skimmings are recorded in Table 7.

Zinc Dross

Zinc dross was broken into small pieces and distilled under vacuum in a tubular furnace using a water cooled condenser. The results of distillation experiments are indicated in Table 8.

Table 8 — Results of Distillation Tests

No.	Temperature of distillation (°C.)	Pressure in the system in mm. of Hg.	Weight per cent of residue left undistilled
1	550	1·0	9·35
2	600	1·0	5·0
3	650	1·0	3·6
4	650	1·5	4·0

Further work is under progress to study the rate of distillation at different temperatures and pressures.

Table 7 — Chemical Analysis of Zinc Waste Products

Constituents	Zinc ash sample			Zinc blowings		Zinc dross	Zinc skimmings
	+10 mesh	−10+40 mesh	−40 mesh	+40 mesh	−40 mesh		
Zn	95·3	59·6	13·1	94·3	81·3	93·6	12·3
ZnO	2·0	32·6	60·4	3·2	14·7	—	54·7
Pb	0·72	0·31	0·41	0·62	0·55	0·41	—
Fe	0·28	0·58	0·62	0·28	0·45	3·39	—
NH_4Cl	—	2·77	3·41	—	Trace	—	6·6
$ZnCl_2$	—	5·4	9·2	—	Trace	—	19·4

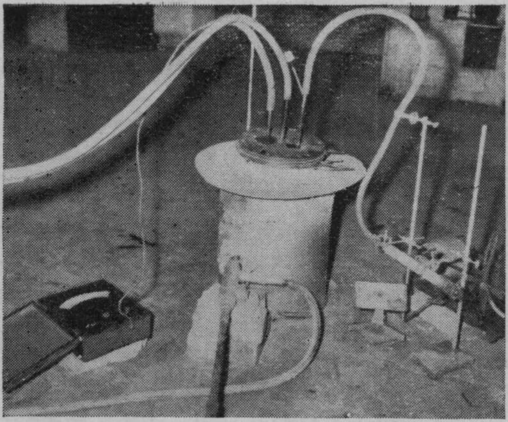


FIG. 24 — A VIEW OF THE VACUUM DISTILLATION UNIT FOR ZINC RECOVERY

Zinc Blowings

Zinc blowings which appeared mostly as metallics were sieved into fractions and the analysis of the products are shown in Table 9.

Table 9 — Sieve Analysis of Different Fractions of Zinc Blowings

No.	Particle size (B.S.S.) mesh	Per cent weight of each fraction	Per cent metallic zinc
1	+60	37·5	97·00
2	−60+100	8·3	86·2
3	−100+170	11·7	82·6
4	−170	42·5	78·06

The various fractions of the sample were treated separately in different concentrations of chromic acid for the preferential removal of the oxides. Considerable improvement in their metallic contents was thus observed. Further work related to the study of the effects of temperature on leaching, washing and drying cycles.

Zinc Ash

Zinc ash was subjected to a preliminary grinding by passing through a roll crusher, to separate the coarse metallics from oxide and flux contaminants. The sieve analysis of the fractions are given in Table 10.

Table 10 — Sieve Analysis of Different Fractions of Zinc Ash

No.	Particle size (B.S.S.) mesh	Per cent weight of each fraction	Fe content of sample %
1	+10	24	0·25
2	−10+40	32	0·44
3	−40	44	0·43

The coarser fractions were amenable to distillation treatment and could be thus treated along with the dross after washing the sample free of flux and other loosely adherent particles of extraneous matter, in a tumbling mill.

26.1 Recovery of Zinc from Galvanizer's Dross

The feasibility of recovering zinc by direct distillation under atmospheric pressure, from galvanizer's dross, on commercial scale was investigated.

Zinc dross obtained during galvanizing of iron and steel articles is a mixture of compounds of iron and zinc with entrained zinc and generally amounts to 25 tons for every 100 tons of zinc consumed. The chemical analysis of the dross investigated showed an average of 3·6 per cent Fe. Experiments were carried out by distilling the dross in a batch type gas fired furnace. Galvanizer's dross was kept in a salamander crucible, to 1100°C., the zinc was distilled out. The distilled zinc vapours were allowed to pass through a hollow cylindrical tube lined with fireclay which was kept slanting towards horizontal. The zinc vapours condensed inside the hollow tube and were collected near the end. Experiments were carried out to define the optimum conditions for efficient condensation. The tube was kept inclined 45° to the horizontal so as to quickly settle the condensing zinc inside a collecting vessel in the liquid form. The factors studied were the rate of distillation, distillation temperature, inclination of condenser tube, thickness of lining of the condenser tube, the inside diameter of the condenser tube, etc. It was observed that with 2·5-4 cm. inside diameter and 2 cm. thick lining and at 40° inclination zinc condensed efficiently. However, in initial condensation, zinc dust was formed to a somewhat greater extent together with oxidation of the zinc vapours. In order to avoid the oxidation and burning of the zinc vapours, the open end of the condenser tube was immersed inside a pool of molten liquid zinc and the zinc vapours condensed without undue oxidation. The effects of temperature of the pool of molten zinc were studied; it was observed that the optimum temperature for efficient condensation was 450-500°C. Further studies undertaken related to the distillation of zinc on a much larger scale. The distilled zinc analysed, Pb, 0·04 and Fe, 0·28 per cent.

27.0 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 India, need for its synthetic preparation is most imperative, particularly in the context of known extensive deposit of fluorspar in Rajasthan and Madhya Pradesh. Fluorspar is one of the chief raw materials used in the production of synthetic cryolite. The indigenous fluorspar are low-grade and need beneficiation in the first instance. Extensive work on laboratory and pilot plant scale on beneficiation of fluorspar was conducted in the Laboratory.

The process investigated at the National Metallurgical Laboratory for the production of cryolite consisted of decomposing the metallurgical grade fluorspar (CaF_2, 85; SiO_2, 3·8; $CaCO_3$, 9·2 per cent, etc.) in aqueous medium with sulphuric acid in the presence of boric acid. The reaction took place almost to completion at 95°C. in two hours

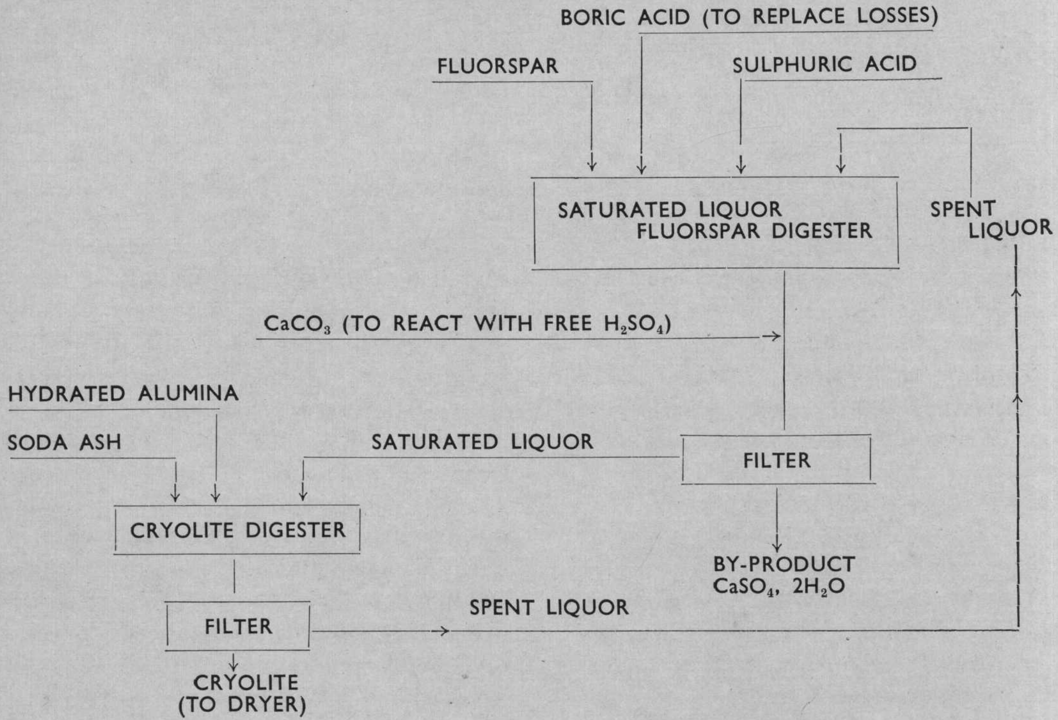


Fig. 25 — Flowsheet of fluoboric acid cryolite process

remelting in the formation of fluoboric acid and calcium sulphate. The slurry was analysed and the residual sulphuric acid removed by the addition of calcium carbonates. The fluoboric acid was neutralized with hydrated alumina and sodium carbonate to form cryolite. Boric acid was regenerated during precipitation of cryolite. Cryolite was filtered and dried. Spent liquor was recirculated for leaching fresh fluorspar after the addition of sulphuric acid.

Cyclic trials on bigger scales were carried out.

The data are given below:

Soln. volume ... *3 litres*
Sod. sulphate ... *75 gm.*
Temp. of leaching ... *95°C.*
Time of leaching ... *2 hours*

No. of cycle	Fluorspar gm.	Conc. H_2SO_4	Boric acid gm.	Residual H_2SO_4 gm.	Sodium carbonate gm.	Hydrated alumina gm.	Cryolite gm.
1	700	Theoretical	360	70·2	240	240	513
2	700	do	360	nil	240	265	472
3	700	do	360	193·9	230	177	448
4	700	do	360	189·0	210	160	420
5	700	do	360	164·8	180	200	400
6	700	do	360	92·1	200	220	477
7	700	do	360	77·6	230	255	425
8	700	do	100	67·8	230	250	450
9	700	do	100	24·2	180	220	320
10	700	do	100	150·0	210	250	320

Laboratory scale experiments were completed and pure cryolite was supplied to an Indian aluminium smelter which reported that silicon and iron contents of the sample were within the accepted limits. Cryolite was also sent to Defence Science Research Organization, Kirkee, for their studies.

A pilot plant for the production of cryolite on 50 kg./day basis is now under installation.

28.0 Preparation of Anhydrous Magnesium Chloride

Hindustan Aircraft Ltd. requires 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.

In the straight drying of the hydrated salt, a product $MgCl_2$, 1·25 H_2O containing approximately 20/22 per cent moisture with 2-3 per cent MgO was obtained efficiently without undue high oxide content. Further drying gave higher oxide and hydrolized oxychloride which are undesirable in the products. Though a dry HCl atmosphere during further drying reduced the oxide and oxychloride formation, it was difficult to get a suitable equipment. Attempts were, therefore, made to use double salt mixture with NH_4Cl and hydrated $MgCl_2$. The NH_4Cl checked the hydrolysis and could be substituted for HCl; this double salt after a certain stage of drying in an oven required special equipment to obtain a product 97-98 per cent $MgCl_2$. In consideration of space and facilities available, the special rotary drier designed on the basis of laboratory results could not be installed for trial. The NH_4Cl, $MgCl_2$ double salt raw material was also tried in a fluidized bed drying unit, designed and fabricated at the National Metallurgical Laboratory where the whole drying range of different temperature region can be attempted. Since the air heater unit designed and fabricated gave a maximum of 128°C. air temp., drying experiments were carried up to this range only. The product obtained which was $MgCl_2$, $NH_4Cl\ 4H_2O$; compared favourably with the drying oven products. One high temperature air heater for final air temperature of 400°C. was designed and fabricated. The continuous feed and product discharge arrangements were incorporated in the unit. Further trials to determine the feasibility of the process were undertaken.

In view of the difficulty of producing oxide free dried magnesium chloride, a design of a furnace for chlorination of magnesium oxide to produce anhydrous magnesium chloride was made and its fabrication from indigenous components was undertaken.

29.0 Nickel-free Austenitic Stainless Steel

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

Comprehensive investigations were carried out on the determination of physical, mechanical 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 cost of production, yield value and range of industrial products obtainable under Indian market conditions and requirements. Tests were conducted to determine the low-temperature and sub-zero tensile and impact properties, high-temperature tensile strength, weldability, oxidation resistance and ageing characteristics of different compositions of the nickel-free austenitic stainless steels.

During the period under review, investigations were conducted to determine the tempering characteristics of 12 per cent chromium stainless steel modified with Mn, Mo, V and N. Chemical compositions of the steels are given in Table 11.

Table 11 — Chemical Compositions of Chromium Stainless Steels Investigated

Heat No.	Composition percentage						
	Cr	Mn	C	N	Mo	V	Si
T-7	12·41	3·75	0·10	0·07	2·74	1·08	0·17
T-8	12·89	5·95	0·13	0·09	3·89	0·89	0·40
T-11	11·16	4·21	0·08	0·04	5·5	0·61	0·20
T-12	12·31	8·21	0·11	0·09	6·15	0·68	0·47

Heat Nos. T-8, T-11 and T-12 showed some promising results. Investigation is being continued with these steels.

Attempts were also made to determine the effects of replacing carbon with nitrogen on the properties of 12 per cent chromium stainless steels. Chemical compositions of the heats investigated are given in Table 12.

Table 12 — Chemical Compositions of Heats Investigated

Heat No.	Chemical composition, %			
	Cr	Mn	N	C
T-2	12·83	0·36	0·04	0·38
T-1	12·23	0·58	0·14	0·05
T-3	12·44	1·03	0·16	0·15

Tempering characteristics of these steels were determined on specimens quenched from 950°C. The results showed that the tempering behaviour of these steels was not materially affected by the nitrogen contents.

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

With a view to determine the suitability of nickel-free chromium-manganese-nitrogen austenitic stainless steels developed at the National Metallurgical Laboratory for high temperature applications, the study of the ageing characteristics at elevated temperatures was undertaken.

Test specimens were taken from some heats which had been hot-rolled into sheets and then solution-treated. The metallographic examination of those steels showed single phase austenitic structure after solution-treatment. The steels investigated contained 17-22 per cent chromium, 8-18 per cent manganese, 0·4-0·9 per cent nitrogen and carbon being less than 0·1 per cent. The test specimens were subjected to different holding temperatures ranging from 300 to 1000°C. for periods varying from 1 hr to 1000 hr in the electric furnaces. After ageing, the specimens were quenched in water at room temperature. Metallographic examination, hardness measurements and corrosion tests in the media of 65 per cent boiling nitric acid, citric acid, vinegar, limejuice and salt-spray were carried out after ageing treatment.

Corrosion tests in boiling nitric acid showed a maximum corrosion of these samples at the optimum temperature of 600-700°C. Above these temperatures, the rate of corrosion decreased though their metallographic study showed an increase of the precipitated phase at higher temperatures. This interesting aspect of less corrosion though having a profuse precipitation may be attributed to the diffusion of chromium to the chromium-depleted areas. Corrosion in other media like vinegar, limejuice, citric acid and salt-spray was negligible.

Hardness measurements of the specimens, in general, did not show any significant change in their hardness values.

Metallographic examination was carried out with the help of optical microscope as well as with the electron microscope during the period under review. Plastic Formvar and carbon replicas were prepared from the different heat-treated specimens for transmission electron microscopic examination. The surfaces of the test specimens were carefully prepared mostly by the electrolytic method for micro-examination. The metallographic examination revealed that ageing at 300° or 400°C. even for a prolonged period did not bring about any marked precipitation. The precipitated phase increased with time and temperature and this precipitation appears to have taken place by nucleation and growth process. The precipitated phase was found mostly along the grain-boundaries in the form of dark-etching nodules having lamellar structure. The inter-lamellar spacing in the nodules was marked to be less at lower temperatures whilst it increased with rise in the ageing temperatures. It was further noted that a higher percentage of nitrogen content promoted more precipitation in the steels.

Further work has related to the study of the ageing characteristics of nickel-free stainless steels of different compositions by the identification of the different phases formed after ageing of the steels under various conditions of treatments by the electron diffraction method with the help of electron microscope.

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

(i) Cold working caused the break-down of austenite into ferrite in Cr-Mn-N-C steels, as it did in the conventional austenitic stainless steels. Ferrite is formed only when the cold work exceeded 50 per cent and its lattice parameter is 2·868 Å.

(ii) During the subcritical ageing treatment, $Cr_2 N$ and $Cr_{23} C_6$ type of precipitates were identified, depending on the temperature of ageing and carbon and nitrogen.

(iii) X-ray spectro-chemical analyses of the $Cr_2 N$ type of precipitate showed, that it contained, in addition to chromium, considerable amounts of iron and manganese.

The amounts of these various elements in the precipitate were found to be functions of time of ageing at any particular temperature level.

(iv) The activation energy of the formation of Cr_2N type of precipitate in Cr-Mn-C steels during ageing was also determined. The values were found to vary between 33·85 and 38·71 Kcal. gm. mol.

30.0 Development of Substitute Alloy Steels

Objectives of this Project are based on the basic themes to develop indigenous 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-à-vis ruling sections and optimum heat-treatment cycles and fabrication techniques.

(i) *Tool Steel*

Several heats of tool steels were made having the basic composition C, 0·6-0·7; Mn, 2·0; Si, 0·2-0·3; W, 6-7; Cr, 4-5; and V, 2 per cent. Additions of Al, Ti, B, Cu, Cb and Zr were made to the above base compositions. Nitrided ferro-chrome was added for introducing nitrogen in the steel instead of nitrided manganese. The hardness of the ingots in as cast and annealed conditions is given in Table 13.

Table 13 — Hardness of Tool Steel in as Cast and Annealed Condition

Heat No.	V.P.N. Hardness	
	As cast condition	Annealed condition
TS_{43}	657	590
TS_{44}	583	225
TS_{45}	594	400
TS_{46}	695	720
TS_{47}	730	740
TS_{48}	736	752
TS_{49}	606	330
TS_{50}	590	518
TS_{51}	539	454

After forging trials, only a few of the ingots could be forged properly in the forging range 1250-900°C. Boron steel ingots could not be fully forged at all as these cracked badly during initial forging itself. Attempts were made to forge the ingots by varying the forging conditions such as soaking time and preheating temperatures. Specimens cut from ½ in. and 1 in. forged bars are being heat treated to determine the correct hardening and tempering procedure.

(ii) *Die Steel*

Die steels of the compositions given in Table 14 were studied for development of indigenous die steels.

The ingots were first forged to ½ in. sq. and 1 in. sq. bars, packed in lime and homogenized for 20 hrs at 450°C. The specimens cut from these bars were then heat-treated for

Table 14 — Compositions of Die Steels Studied

Steel	Composition, %						Forging temp. °C.
	C	Si	Mo	Cr	V	Ti	
DS_1	0·8/0·9	0·30	0·60	1	—	—	1150
DS_2	0·8/0·9	0·30	0·60	2	—	—	1150
DS_3	0·8/0·9	0·30	0·60	—	—	0·5	1150
DS_4	0·8/0·9	0·30	0·60	—	0·25	—	1150

determination of the correct hardening and tempering procedure. Table 15 shows the results obtained with steel DS_1. Further work on these steels is under progress.

Table 15 — Vickers Hardness of Steel DS_1 after Tempering at Different Temperatures for Different Time

Tempering temp. °C.	VPN hardness				
	½ hr.	1 hr.	1½ hr.	2 hr.	2½ hr.
150	840	826	819	805	795
200	823	810	790	772	736
250	765	740	716	700	690
300	700	680	675	666	648
350	648	639	631	626	612
400	615	610	602	494	580
450	575	560	552	548	547
500	495	478	465	460	459
550	451	438	425	405	390
600	385	370	368	365	360

(iii) *Iron-Aluminium Alloys*

Air-melted calcium-deoxidized iron-aluminium alloys were hot workable up to 12 per cent aluminium content and beyond this aluminium percentage only cast alloys will have to be used for heat resistant applications. Cold working and finishing of alloys was restricted to 6 per cent aluminium content. The tensile strength of 5 per cent alloys was 42 TSI which fell to 31 TSI for an 8 per cent alloy. Elongation values also fell from 6·25 to 2. Rapid decrease in ductility with increase in the aluminium content as observed in mechanical working appeared to be inherent with the system and was not due to hydrogen which did not increase in the same way. The corrosion resistance and heat resistance increased with aluminium content of the alloys. The 5 per cent aluminium group of alloys can safely be employed up to 800°C. whilst a 12 per cent Al alloy appeared to possess adequate oxidation resistance at this temperature but follows the same pattern at 1000°C. Air-melted calcium deoxidized ferritic iron-aluminium alloys can be employed in hot worked condition up to 12 per cent aluminium content and as cast condition beyond up to 35 per cent.

Minute additions of Ti, Zr, V, B and Ce resulted in grain refinement, increase in hardness and tensile strength. Additions of Si and C to Fe-8 per cent Al alloys increased the fluidity of melt. The rolled sheets had high tensile strength of the order of 55-60 TSI, the ductility, however, was poor, elongation being around 1·5 per cent only. Addition of vanadium to fix the carbon as vanadium carbide (VC) gave excellent results improving the ductility, elongation rising to 12·5 per cent. Work on this project was completed.

(iv) *Isothermal Transformation Characteristics of Alloy Steels*

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

Isothermal transformation experiments were carried out on a number of low alloy steels, diagram of which are furnished in Figs. 26-29. The chemical composition of steels are given in Table 16.

Table 16 — Chemical Composition of Steels Studied for Determining Isothermal Transformation Characteristics

Steel	C %	Si %	Mn %	P %	S %	N %	Cr %	Mo %
1. Ni-Cr	0·42	0·19	0·75	—	—	1·34	1·25	—
2. Ni-Cr-Mo	0·36	0·25	0·62	0·016	0·01	1·30	0·66	0·12
3. Cr	0·39	0·2	0·60	0·01	0·02	—	1·43	—
4. Cr	0·51	0·29	0·69	0·03	0·028	—	1·15	—

ISOTHERMAL TRANSFORMATION CURVE

TYPE OF STEEL	METHODS OF DETERMINATION
Ni— Cr— Steel	*DILATOMETRIC & METALLOGRAPHIC*

ANALYSIS (WEIGHT%)

C	Mn	Si	S	P	Ni	Cr	Mo	W	V
·42	0·75	0·19	—	—	1·34	1·25	—	—	—

GRAIN SIZE

PREVIOUS TREATMENT

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

AUSTENITIZING CONDITION

TEMPERATURE	TIME
850 °C	30 MINUTES

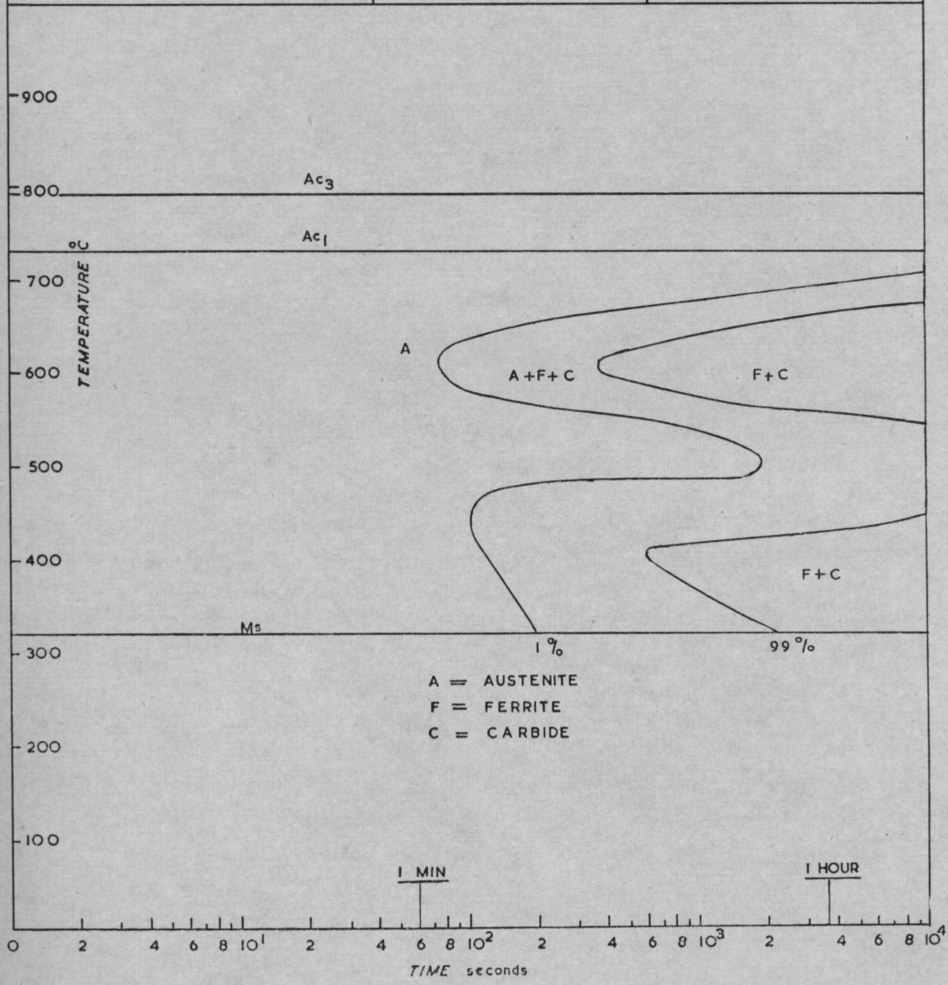


FIG. 26

ISOTHERMAL TRANSFORMATION CURVE

TYPE OF STEEL	METHODS OF DETERMINATION
Ni-Cr-Mo-Steel	*DILATOMETRIC & METALLOGRAPHIC*

ANALYSIS (WEIGHT %)

C	Mn	Si	S	P	Ni	Cr	Mo	W	V
0.36	0.62	0.25	0.01	0.026	1.30	0.66	0.12	—	—

GRAIN SIZE

PREVIOUS TREATMENT

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

AUSTENITIZING CONDITION

TEMPERATURE	TIME
850° C	30 MINUTES

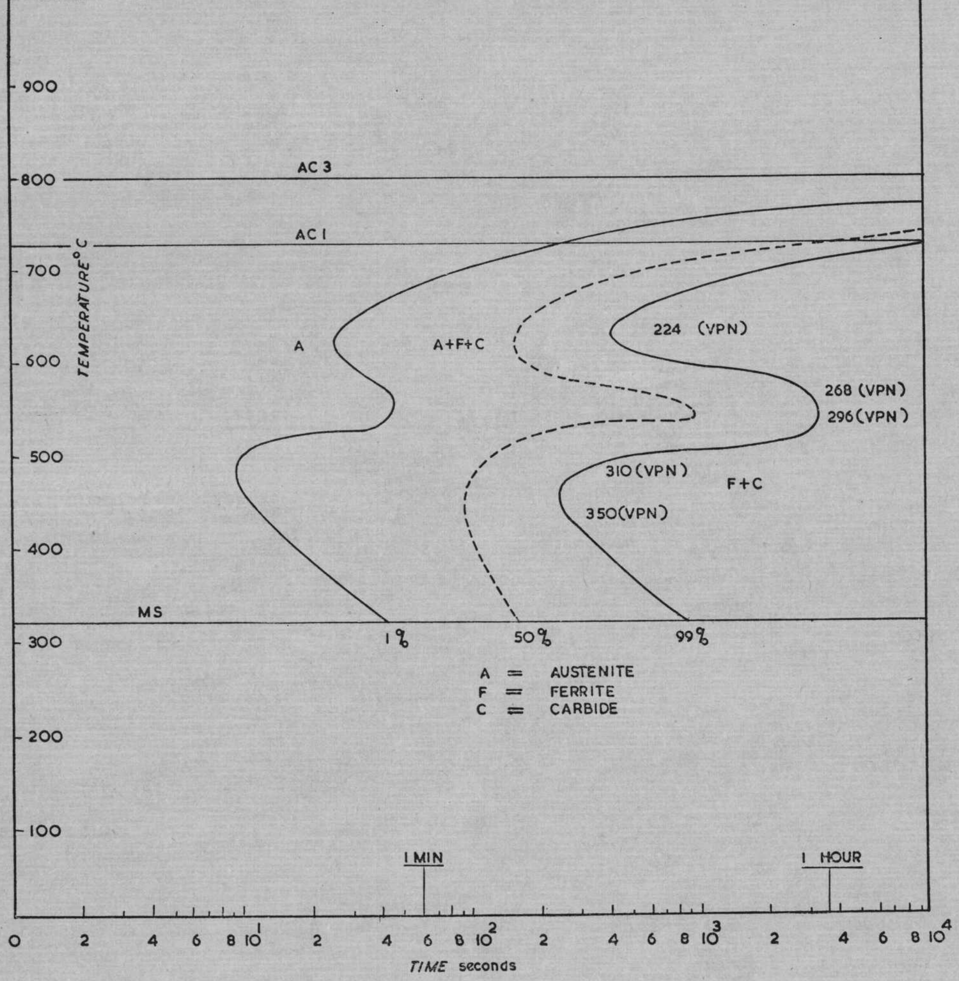


FIG. 27

ISOTHERMAL TRANSFORMATION CURVE

TYPE OF STEEL	METHODS OF DETERMINATION
1½% CHROMIUM STEEL	*DILATOMETRIC & METALLOGRAPHIC*

ANALYSIS (WEIGHT %)

C	Mn	Si	S	P	Ni	Cr	Mo	W	V
0·39	0·60	0·2	0·02	0·01	—	1·43	—	—	—

GRAIN SIZE

PREVIOUS TREATMENT	AUSTENITIZING CONDITION	
	TEMPERATURE	TIME
H.F 70 lbs. INGOT 40 lbs. FORGING 0·5" SQ BAR. ANNEALED	850 °C	30 MINUTES

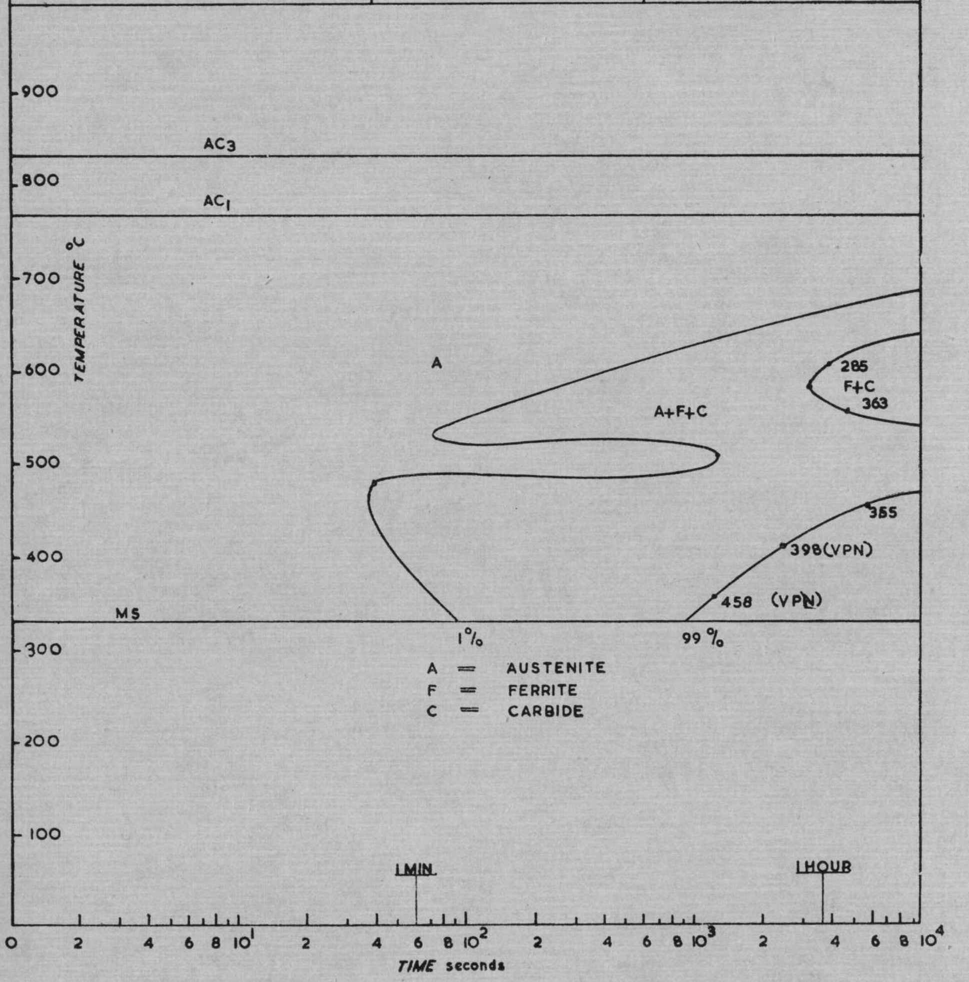


FIG. 28

ISOTHERMAL TRANSFORMATION CURVE

TYPE OF STEEL	METHODS OF DETERMINATION
CHROMIUM STEEL	*DILATOMETRIC & METALLOGRAPHIC*

ANALYSIS (WEIGHT %)										GRAIN SIZE
C	Mn	Si	S	P	Ni	Cr	Mo	W	V	7
51	0·69	0·29	0·028	0·03	–	1·15	–	–	—	

PREVIOUS TREATMENT	AUSTENITIZING CONDITION	
	TEMPERATURE	TIME
H.F. 70 lbs INGOT 40lbs FORGING 0·5" SQ BAR ANNEALED	850 °C	30 MINUTES

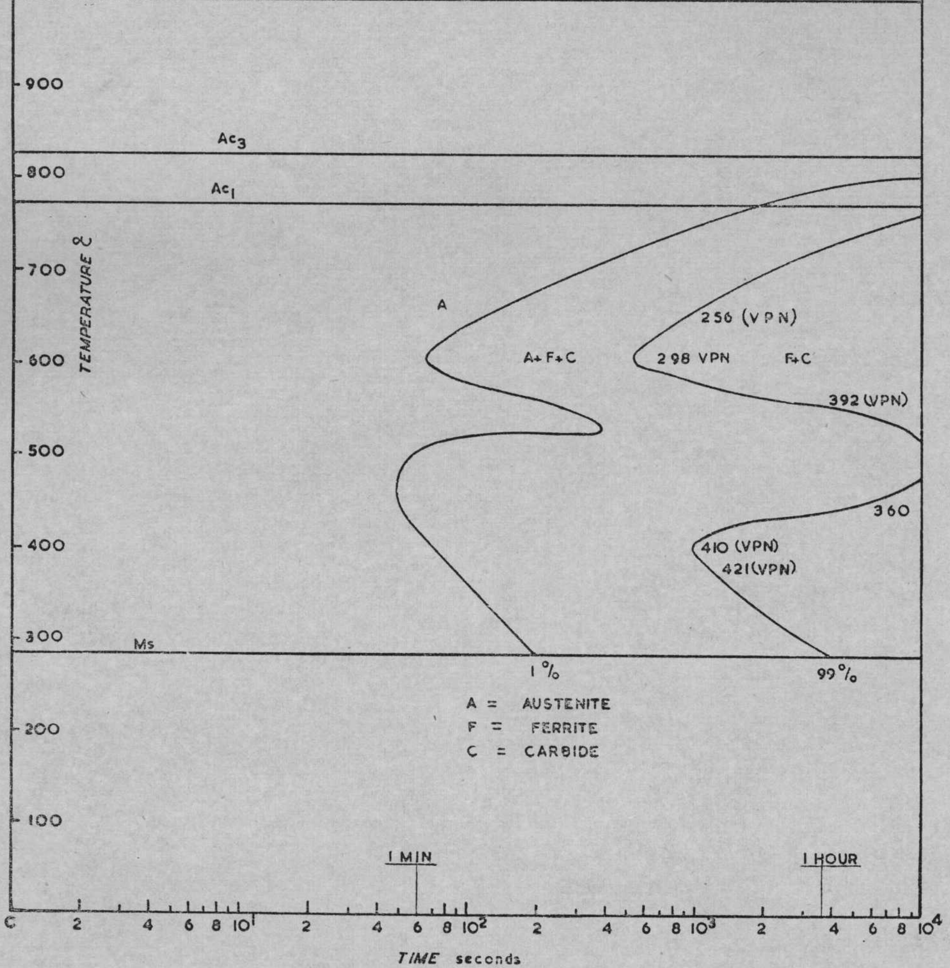


FIG. 29

Table 17 — Chemical Composition of Steels Studied for Determining Continuous Cooling Transformation Characteristics

Steel	C %	Si %	Mn %	P %	S %	Ni %	Cr %	Mo %
1. Ni	0·34	0·15	0·47	0·02	0·029	1·43	0·03	—
2. Ni-Cr	0·35	0·21	0·65	0·03	0·02	1·37	0·72	—
3. Cr	0·36	0·2	0·60	0·01	0·62	—	1·15	—

(v) *Continuous Cooling Transformation of Alloy Steels*

The project was undertaken with a view to study the continuous cooling transformation characteristics of Indian alloy steel and to prepare an atlas of the continuous cooling transformation diagrams for use in industry

Experiments were conducted on steels of the compositions given in Table 17.

The continuous cooling transformation diagram of these steels are furnished in Figs. 30-32.

31.0 Development of Low Alloy High-tensile Structural Steel

This broad-based investigation was taken up with the objective of developing indige-

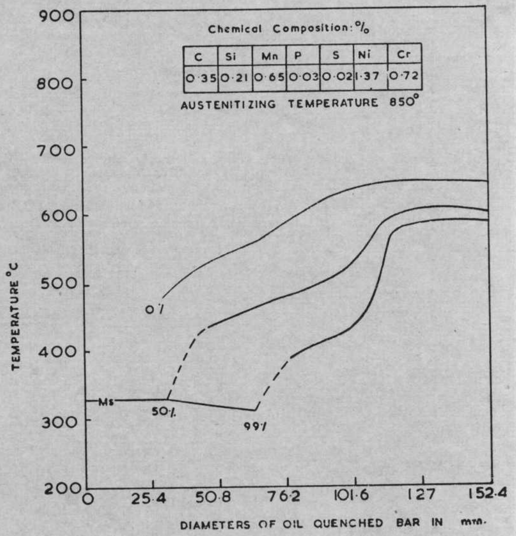


FIG. 31 — CONTINUOUS COOLING TRANSFORMATION DIAGRAM FOR NICKEL-CHROMIUM STEEL

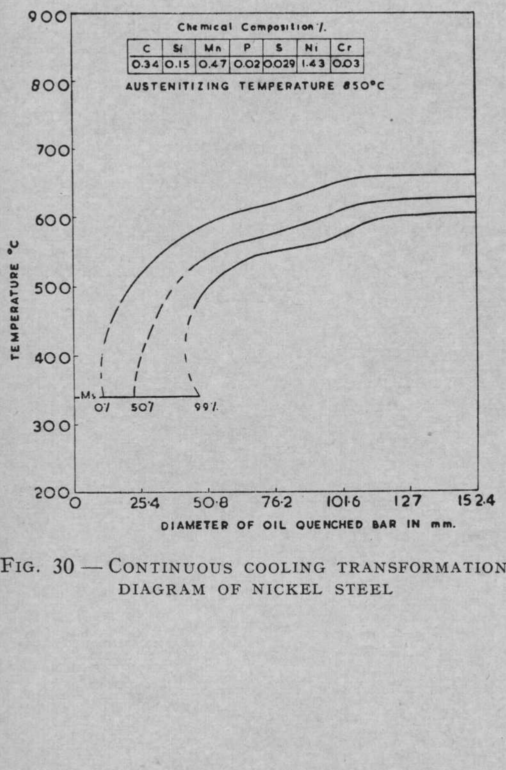


FIG. 30 — CONTINUOUS COOLING TRANSFORMATION DIAGRAM OF NICKEL STEEL

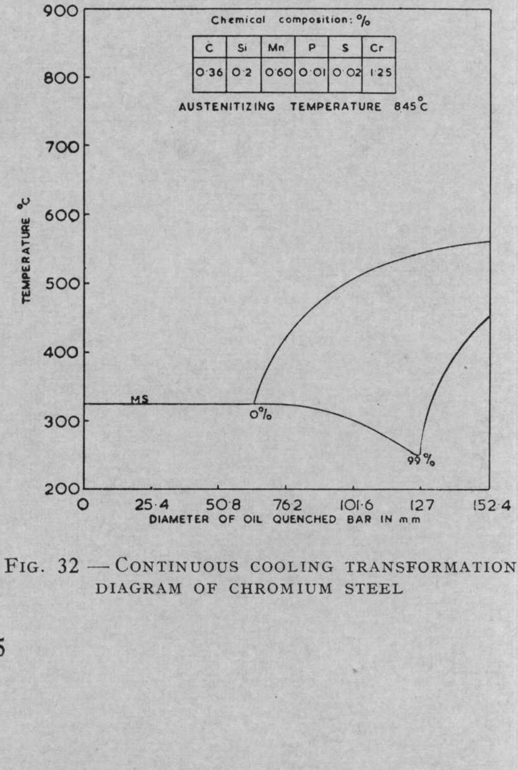


FIG. 32 — CONTINUOUS COOLING TRANSFORMATION DIAGRAM OF CHROMIUM STEEL

nous low-alloy structural steel possessing adequate mechanical properties, hot and cold working characteristics, optimum corrosion resistance and favourable welding characteristics based essentially on indigenous alloying elements.

Earlier, several experimental heats were made with slight modifications in the percentages of alloying elements of the existing low alloy high tensile steels in use. Main alloying elements in these heats were manganese, chromium, silicon and copper. After standardizing the melting procedure on laboratory scale production, some basic compositions were selected for studying the effects of different elements singly or jointly. While studying the effects of silicon, which is known to be beneficial for both gas and arc welding purposes when present in suitable amounts in structural steel, two basic compositions were selected with the following limits:

		Per cent
(i)	C	0·15-0·25
	Mn	1·2-1·8
	Cu	0·3-0·4
	Ti/V	0·1-0·2
	P	0·06
	Si	0·35/0·5
(ii)	C	0·09-0·12
	Mn	0·9-1·4
	Cu	0·2-1·
	V	0·1-0·15
	P	<0·06
	Si	0·35/0·5

During the period under review, several experimental heats were made in which silicon was kept between 0·35 and 0·5 per cent. The test results of the heats made according to the specified compositions are given in Table 18. It may be seen that except the first one, all other heats gave almost identical tensile physical properties. All the three heats were weldable and their tensile strength values ranged between 35 and 40 t.s.i. The high yield point in these heats was remarkable although the phosphorus content was less than 0·04 per cent. Sub-zero charpy impact tests of one of these heats were carried out and quite high impact strength was found even at temperature as low as −60°C. From the chemical analysis, the carbon content of the first heat No. 41/IR was of the order of 0·2 per cent and phosphorus of the order of 0·07 per cent. The tensile strength of this heat was 50 t.s.i. with a yield point of 40 t.s.i. The yield ratio was, therefore, about 0·8. This steel was however, not weldable. It may be used as high tensile corrosion resistance structural steel. The steel also showed fair elongation values. Little variations in impact strength properties were observed between room temperature to −60°C. The corrosion resistance of this steel was investigated.

The next aim was to produce the steel on tonnage scale and also to standardize the production technique for a particular composition. Three heats were made in this

Table 18 — Results of Tests Conducted on Heats of Low Alloy High Tensile Structural Steels

Heat No.	Tensile test results				Results of weld-ability test	Charpy impact values (ft. lb.) at					
	Max. stress T.S.I.	Y.P. T.S.I.	R.A.%	E% GL-1″		R. temp.	0°C.	−20°C.	−40°C.	−60°C.	−70°C.
41/1R	49·68	40·82 38·32	55	28	*N.W.	20	22	20	19	19	—
41/2R	36·46	28·28 34·53	66	32·8	+W	134	122	82	56	38	—
41/3R	36·80	33·59	52	28	+W						
41/4R	38·14	27·12 31·32	69	34·4	+W						

Table 19 — Results of Tests Conducted on Tonnage Scale Heats of Low Alloy High Tensile structural Steels

Heat No.	Tensile test results				Results of weld-ability test	Charpy impact values (ft. lb.) at					
	Max. stress T.S.I.	Y.P. T.S.I.	R.A.%	E% GL-1″		R. temp.	0°C.	−20°C.	−40°C.	−60°C.	−70°C.
Arc 1	32	25·4	71	34	+W	150	178	63	54	56	36
Arc 2	31·16	21·6	73	35	+W						
Arc 3	A35·63	27·22	65·5	35	*N.W.						
	N48·4	—	47·5	25							

*, Not weldable; +, weldable; A, annealed; N, normalized.

connection. The tensile properties of these heats with some other properties are given in the Table 19. Handling of the big ingots during hot working was found to be somewhat difficult with the accessories available. Arrangements were, therefore, made for easy handling with improved holding device and mono-rail pulley system. However, in most cases, ingots had to be gas cut into three pieces and then hot worked. Chemical analyses revealed that none of these big heats strictly conformed to the required specifications. Silicon content in the first two heats, was much lower than the requirement whereas in the third heat, copper content was much higher than that specified. Possibly, lower silicon may be the cause of its lower strength given in the first two heats. The third heat whilst it gave the required strength was slightly difficult to weld. Test results indicated lower strength and hardness of the material in the annealed condition. Arrangements were made to make more heats in order to standardize the conditions for getting the final results. Some heats were also made according to the specification of Tiscor and Tiscrom, required to compare the corrosion rates of the steels under development at the National Metallurgical Laboratory.

Further work will comprise preparation of 800 kg. heats according to the specified compositions to study their properties more elaborately. Work for effecting further improvements in chemical composition from the point of economy of alloying elements and the cost of production would be continued.

32.0 Development of Electrical Resistance Alloys for Heating Elements

Conventional types of 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, intensive research and development work was taken up which led to the development of indigenous based electrical heating elements. The work was centred round Fe-Cr-Al system based on various considerations including availability of raw materials from indigenous resources and the requirements of physical and mechanical properties for suitable heating element. Several heats were made using Mn, Si, Zr, V, misch metal, etc., in high-frequency furnace. The hot and cold working behaviour of these alloys were studied and their physical and mechanical properties were also determined. It was seen that heats which contained relatively higher percentage of aluminium and low percentage of chromium showed better life.

Production Technology

Based on the experience gained on the behaviour of the alloys during melting and mechanical working into finished product, production technique for the electrical resistance alloys was standardized which covered essential details of raw materials viz. low carbon iron, low carbon ferro-chrome, aluminium, misch metal, ferro-titanium and zirconium etc; melting and casting procedure; forging and rolling of the ingots to wire rods, and the wire drawing schedules with intermittent heat treatment cycles. Selection of the charge was found to be critical and elements such as carbon must be limited to low level. Dressing of the ingots, billets, wire rods at various stages was essential to remove surface defects against which special precautions were taken. For hot forging and rolling, it was necessary to pre-heat the charge at a low temperature before slowly heating to the hot working temperature of 1150°C. The finishing temperature during rolling had to be controlled to dull red heat as the higher finishing temperature led to coarse grain structure which was highly detrimental to the subsequent wire drawing. It was seen that the slight scaling during intermittent process annealing at 700°C. was also helpful to retain lubricant during cold drawing operation. The cold reductions between successive annealing stages should not exceed a certain percentage level.

Heats were made using synthetic slag containing some MgO, CaO, Al_2O_3, in order to protect the alloys from gaseous and non-metallic inclusions. Some improvements were noticed in the working behaviour of these ingots prepared, but further work would be carried out to draw full inferences therefrom. The entire process of making the alloy from the selection of raw materials to the finished wire products was demonstrated to the industrial firms to whom this process has been released through the National Research Development Corporation of India.

Weldability Studies

Experiments were carried out to develop proper conditions of welding of the electrical resistance wires to make up for the occasional breakage during wire drawing. Wires of different gauges (from 0·064 to 0·128 in. dia.) were electrical resistance welded. The welding current and mechanical pressure were varied. It was found that except in a few cases, the welds showed brittle behaviour due to coarse grain structure. Use of argon gas and suitable flux has been contemplated to avoid oxygen pick up during welding and further work is being done to ensure fine grain size by proper adjustment of the welding parameters and post welded mechanical treatments.

Accelerated life test:

The results of the life test at 1150°C. on the experimental samples of the heating element are given in Table 20.

Table 20 — Accelerated Life Test of Heating Elements

Heat No.	Testing temp. (°C.)	Life (hr.)
JE_1A	1150	55*
JE_2B	1150	60*
J	1150	82*
ESF	1150	10*
D_2	1150	Passed 80 hours
$2E_{41}B$	1150	80

*These alloys are modified experimental heats to study various conditions.

Most techanical details of the newly developed alloys were successfully worked out and the project is now in its concluding stage. However, such items as the welding techniques are still under investigation. Experience about the accelerated life test as per A.S.T.M. specifications had shown the inadequacy of this test procedure, specially in high temperature tests, the wires show excessive creep even at very small load applied to the free end of the vertically suspended wire. The adoption of a system using a 'U' type of specimen with both ends fixed may be considered desirable. This process has since been released to the industry on lumpsum premia and royalty basis.

33.0 Development of Nickel-free Coinage Alloy

Although nickel is eminently suitable for coinage alloys, its non-availability in India poses a real problem. This project was taken up to develop alloys containing no nickel and having metallurgical and physical properties, like those of the present cupronickel coinage alloy.

FIG. 33 — NEW 3 PAISA COIN MADE OF ALUMINIUM-MAGNESIUM ALLOY DEVELOPED AT NATIONAL METALLURGICAL LABORATORY AND GOVERNMENT OF INDIA MINT, BOMBAY

Studies carried out so far with copper-base manganese bearing alloys indicated that these alloys can be successfully used for coinage purpose. However, to get a good surface finish suitable for stamping, great care was needed during hot rolling. Further, the alloys were found to be tough and required greater power for rolling compared to other commonly used copper alloys.

The possibilities of using aluminium alloys for lower denomination coins were also examined in view of the fact that aluminium is indigenously available. Cold rolling, polishing and stamping properties of commercial alloy, Al-1·1 per cent Mn, Al-1·5 per cent Mg, Al-4·2 per cent Mg were studied in details in collaboration with the Govt. of India Mint, Bombay. On the basis of detailed experimentation it was found possible to successfully produce polished coins from both annealed and cold rolled aluminium alloys.

Erosion-corrosion tests carried out under simulated laboratory conditions indicated that to get an alloy having necessary corrosion and wear resistance properties, the alloy containing 3·5-5·0 per cent Mg was most suitable. On the basis of these studies the use of Al-3·5-5 per cent Mg was advocated at present for minting low denomination coins.

34.0 Development of Manganese Bearing Brasses

Brass is extensively used in India for utensil making. Due to shortage of production of copper in India and import of the entire requirement of zinc, this project was taken up with a view to reducing the copper and zinc contents in brass aiming at their partial substitution by manganese.

During the period under review, the investigation was extended to study the high temperature regions and a detailed survey of literature was made. The design of the apparatus was finalized and fabrication work undertaken.

35.0 Development of Magnetic Materials

The demand for magnetic materials is multiplying in India following the rapid growth of telephone, electronics and other industries. At present, most of the demand in India is met by import. This project was taken up with a view to developing detailed technical know-how for their production and to develop, if possible, permanent magnets and other magnetic materials from indigenous sources. The work on development of magnetic materials was pursued actively under the following heads:

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

(i) *Tempering characteristics of some Alnico type magnets*

The electron microscopic study of precipitation during tempering of alnico specimens containing Co, 12; Al, 11; Cu, 6; Ni, 19 and Fe, 52 per cent was carried out. The critically cooled specimen 2°C./sec. showed a finely distributed phase, which appeared to first go into solution during tempering at 600°C. and then grew in size on prolonged tempering. Changes in the coercive force observed during tempering of Alnico magnets could well be explained in terms of changes in the dimensions of this phase. A complete report of the tempering characteristics of the Alnico type of magnets is under preparation. Similar work on Alcomax type of magnets undertaken to study the effects of different alloying elements on the cooling rates to develop optimum magnetic properties made good progress.

(ii) *Study of Magnetic and Electrical Properties of Ceramic Permanent Magnets of Hexagonal Structures*

During the period under review X-ray structure studies on phases formed during sintering at various temperatures were carried out. It was found that calcined powders of barium ferrite containing PbO showed the presence of only one hexagonal phase of lattice parameter $c = 23{\cdot}054$ Å, $a = 5{\cdot}863$ Å. This phase corresponds to $BaO,6Fe_2O_3$. After sintering above 1200°C., a tetragonal phase $PbO, 2Fe_2O_3$(c=15·8917 Å., a=7·8142 Å.) was also formed. In heavily leaded ferrites the dominant phase was $PbO, 6Fe_2O_3$, isomorphous with $BaO,6Fe_2O_3$. Metallographic examination of sintered compacts showed that the $Ba0,6Fe_2O_3$ phase progressively formed as the sintering time was increased. The $PbO,2Fe_2O_3$ phase could not be microscopically identified.

(iii) *Study of binary and ternary alloys of manganese-aluminium-iron alloys*

During the period under review, work was continued on the above subject, but difficulties were encountered in sintering the compacts without oxidation. However, a substantial quantity of $Mn_{1{\cdot}1}\,Al_{0{\cdot}89}$ powder was made. This ferro-magnetic powder was re-pressed into compacts for final sintering, which however, was particularly difficult in view of the tendency for oxidation and evaporation. Some compacts did however, sinter very well but were very brittle and unsuitable for mechanical working.

(iv) *Soft Ferrites*

Work on soft ferrites was initiated to study the suitability of indigenous raw materials for their manufacture. Preliminary work on nickel-zinc ferrites using purest raw materials was taken up. The oxides (NiO-ZnO-Fe_2O_3) were taken in mol. proportion of 35:15:50, mixed together thoroughly, calcined at 1100°C., pulverised to −300 mesh, and then pressed in the form of anchor rings for testing. Final sintering was carried out at 1250°C. and magnetic properties determined. Suitability of some indigenous raw materials is also being looked into.

37.0 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. Work was carried out on the production of bigger melts of Al-8 per cent Mg-3 per cent misch metal, which was earlier found to be most suitable alloy regarding its workability. Ingots 4 in. sq. × 4 ft. long were obtained and after machining to remove the oxide skin, were hot forged and hot worked. It was observed that during hot forging as well as during hot rolling edge cracks developed in some billets. It was analysed that the failure was due to precipitation of hard phases. The billets were normally rolled down to 3 mm. thickness. Samples of the rolled sheets were given cold reduction of 10-60 per cent

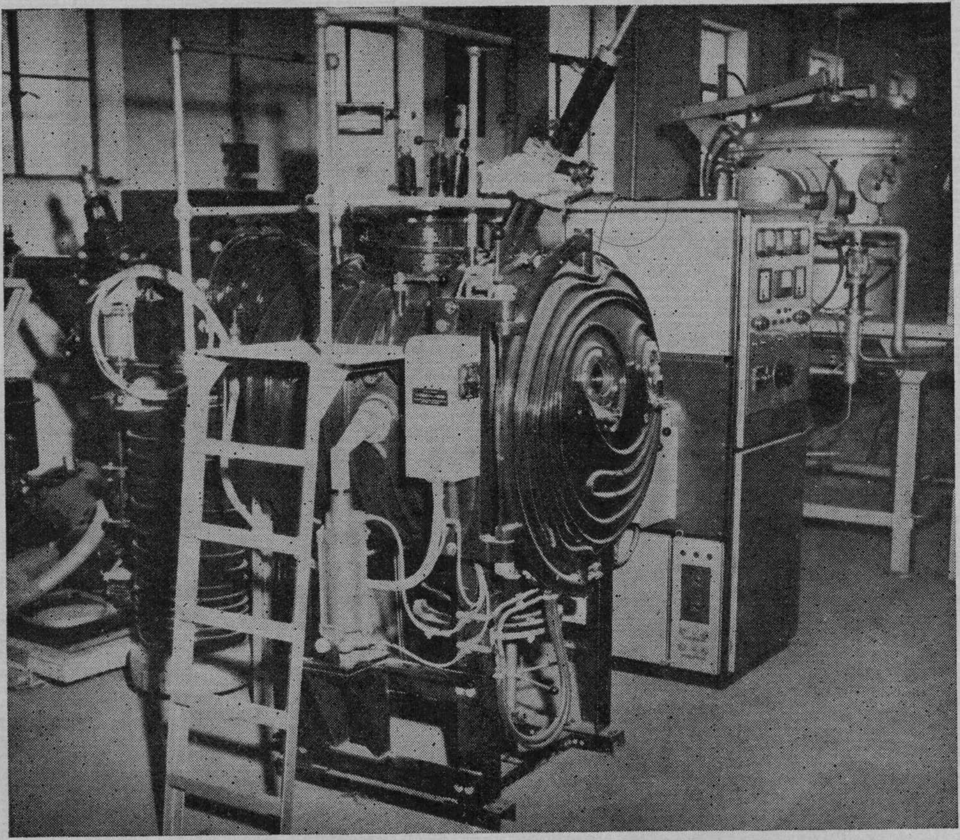


FIG. 34 — A VIEW OF THE 60 LB VACUUM INDUCTION MELTING AND CASTING FURNACE INSTALLED AT NML

to determine whether there was any improvement in their tensile strength and elongation. Following average values were obtained:

Reduction %	Av. tensile strength T.S.I.	Average elongation %
10	29·55	6-8
20	29·77	6-8
30	30·29	6-8
40	32·14	6-8
50	31·97	6-8
60	33·23	6-8

It was observed that while the average tensile strength value increased from about 29 t.s.i. in the original sheet to 33·23 t.s.i. after it had been given 60 per cent cold reduction, there was no change in its ductility.

Samples machined from forged billets of the alloy were used for wire drawing, aiming at drawing a wire about 3 mm. dia. While it was possible to obtain small lengths (about 2 m. long) by cold drawing from a 12·5 mm. dia. rod, it was not possible to obtain any appreciable lengths, possibly because of fractures caused by poor ductility

Stress corrosion of misch metal treated aluminium-magnesium alloys was studied after imparting permanent stress of varying degree to strips 220 mm. long × 12·5 mm. The samples were kept dipped in a 3 per cent NaCl solution for about 26 weeks. From the trend of weight loss of the samples, it was inferred that there was no significant difference in the corrosion rates of the stressed and

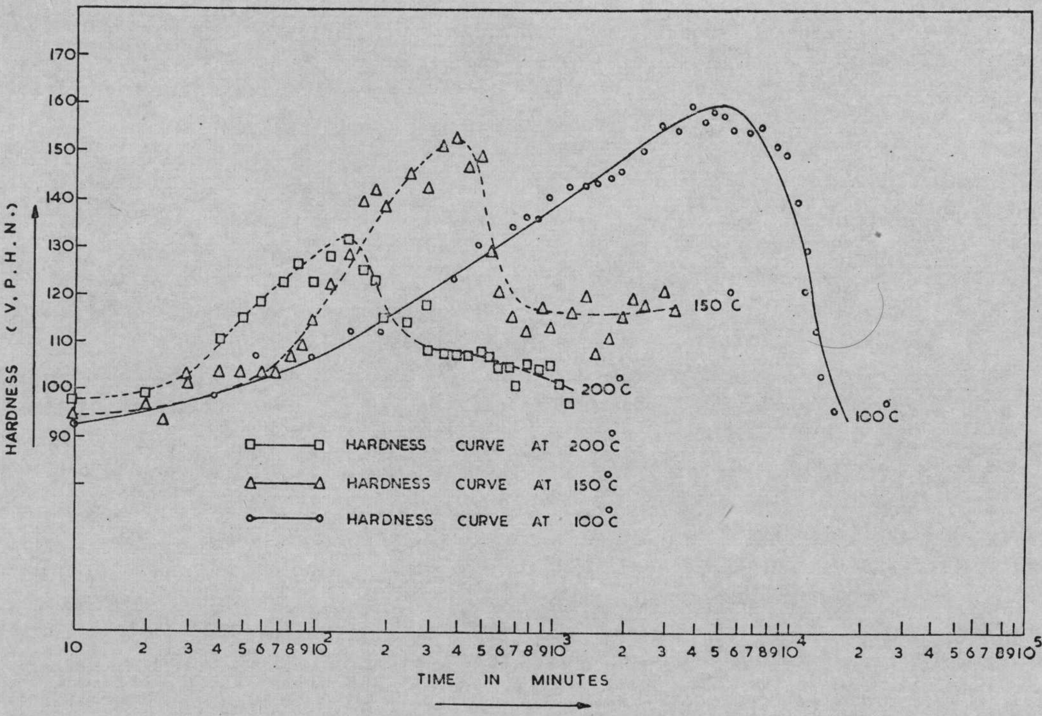


FIG. 35 — AGEING OF AL-6%ZN-3%MG ALLOY AT DIFFERENT TEMPERATURES

unstressed samples under the experimental conditions. Further experiments on corrosion under stress of these alloys under different corrosive media or different corrosive atmospheres were undertaken. Work was continued on age-hardening characteristics of aluminium 7-9 per cent magnesium alloys. It was observed that when solutions treated samples of these alloys were aged at 100°C., there was no increase in hardness over as long a period as 1000 hr. Since the heats from which these samples had been made were melted in air, it was considered desirable to make some heats in vacuum or in an inert atmosphere. Melting in vacuum with tungsten heating elements resulted in extensive sublimation of both aluminium and magnesium. The alloy was melted in an argon atmosphere, but even then sublimation took place when argon pressure was below 10^{-2} mm. Hg. Work was also continued on determination of the age-hardening characteristics of alloys having the nominal composition of Al-6 per cent Zn-3 per cent Mg. Ageing of these alloys was studied at 100°, 150° and 200°C. The results obtained are shown in a graph (Fig. 35).

37.1 Aluminium-copper Alloys

Microporosity observed in the case of Al-4½ per cent Cu alloy is attributed partly to its wide solidification range which renders it into a mushy state thereby making the escape of gases from the solidifying metal difficult. It was, therefore, contemplated to study in the first instance, the effects of micro-additions on the narrowing down of the solidification range of such alloys. The cooling curves for pure Al and Al-4½ per cent Cu in alloys were obtained with the help of a quick temperature recorder. An addition of misch metal was made to the Al-4½ per cent Cu alloys and cooling curves were again obtained. It was observed that the difference between the solidus and liquidus temperature ranges was decreased in the case of the Al-4½ per cent Cu which treated earlier with misch metal.

As one of the consequences of narrowing down the solidification range is the increase in the feeding distance fluidity of the metal, standard fluidity moulds were poured in Al-4½ per cent Cu with and without misch metal addition. It was observed that the fluidity of the metal was appreciably increased and in one instance even a full length spiral was obtained. The porosity of the specimen was examined. The metallographic method did not give any dependable clue to the absence of porosity in the specimens. For a quantitative determination of porosity in these alloys a suitable apparatus is being designed and set up.

38.0 Study of the Properties of Indigenous Foundry Moulding Materials

This project was taken up with a view to assess the moulding characteristics of Indian foundry sands and bonding clays and to determine their suitability for various types of castings. During the period under review, investigations were completed on twenty-five sand samples, four bentonite samples, and one sample each of silica flour and cellulose binder. A summary of the work done is given below:

I. *Moulding Sands*

(i) *A.C.C. Sand* — The sand received from Associated Cement Companies Ltd., Shahabad Dist., Gulbargah, Mysore State, was reddish white in colour and was a coarse grained sand having an A.F.S. Grain Fineness No. of 219 and A.F.S. clay content of 0·46 per cent. Grain shape was sub-angular to angular. The sand contained 28·85 per cent silicon and low alkalies. The mineral constituents of the sand were mostly quartz along with traces of biotite and opaque minerals. The sand had a high sintering range of above 1500°C. For moulding purposes, the sand should be mixed with higher percentages of bentonite as it imparted low strength when bonded with 5 per cent Bihar bentonite. The sand could well be used as a core sand in the cast iron and steel foundries. However, a proper coating of blacking or other fine grained refractory addition to improve surface finish and present metal penetration will be necessary.

(ii) *Gundiposi Sand* — This sand, sent by Geological Survey of India, Bihar Circle, was fine to coarse grained with an A.F.S. Grain Fineness No. of 43·8 and a clay content of 5·8 per cent in the as received condition. Grain shape was rounded to sub-angular. The sand assayed 91·60 per cent silica and 5·65 per cent alumina. The minerals were mostly quartz and feldspar, sericite was also present. The refractoriness of the sand was poor being in the range of 1150-1200°C. Owing to the low refractoriness, non-uniform grading and low permeability, the sand was not suitable for ferrous castings in the as received condition but may be used for non-ferrous jobs after mixing with a suitable grade high silica sand.

(iii) *Jamna Sand*—The sand was received from Messrs Central Indian Machinery Mfg. Co. Ltd., Birlanagar, M.P. It was a fine grained having an AFS G.F. No. 129 and containing 2·8 per cent AFS clay. It contained 82·2 per cent silica. The sand grains were angular to sub-angular. The sintering range of the sand was between 1200-1250°C. This sand could well be used for non-ferrous and cast iron foundry castings after mixing with a coarse silica sand.

(iv) *Bhind Sand* — (Received from Messrs. Central India Machinery Mfg. Co. Ltd., Birlanagar, M.P.) — Bhind sand was fine grained (G.F. No. 127), associated with a high clay content of 15 per cent. It had a silica content of 8·8 per cent. This sand was not suitable for foundry moulding purposes as received in view of the uneven grain distribution and low sintering temperature range of 1125-1150°C. Mixing it with a coarse silica for obtaining better grain distribution may make it usable for foundry usage.

(v) *Banmore Sand* — (Received from Messrs. Central India Machinery Mfg. Co. Ltd., Birlanagar, M.P.) — Banmore sand was a medium to fine grained sand with A.F.S., G.F. No. 55·7 in the as received state, and was associated with a clay content of 16 per cent. The sand sample as received assayed 57·06 per cent silica with a sintering range between 1225-1275°C. The sand can be successfully used for cast iron foundry purposes.

(vi) *Zircon Sand* — The zircon sand received from Manavalakurichi, Kerala State; was dull brown in colour and had a G.F. No. of 96·12 in the as received state and contained 0·15 per cent AFS clay. The sand was fine grained with sub-rounded to rounded shape. The sand consisted mostly of zircon mineral (98 per cent). Refractoriness of the sand was very high showing that the sand can withstand high temperature without fusion, chemical analysis showed 66 per cent ZrO_2 and 32 per cent SiO_2 along with Al_2O_3, Fe_2O_3, etc., in small quantities.

Study of the moulding and core characteristics of the sand showed it to be suitable for both the purposes. But its permeability values were slightly lower; the sand was susceptible to slight changes in moisture. In view of the high cost of the sand, its fineness and high sintering temperature, it would be advantageous to use it as a facing material rather than a moulding tool.

(vii) *Punjab sand samples* — Sixteen sand samples received from Department of Industries, Punjab, were investigated. Investigations were undertaken with respect of the following tests:

1. Mechanical grading and clay washing,
2. Sintering range,
3. Petrological examination, and
4. Chemical analysis.

Of the sixteen sand samples investigated, only six sand samples designated as B-1, B-2, M-2, D-1, Jg-1 and SP-2 may be used in the as received condition for ferrous foundry purposes.

B-1. The sand sample B-1 was a coarse grained sand having an AFS G.F. No. 18 in the as received state and AFS clay content of 2·23 per cent. The sand grains were angular to sub-angular. The as received sand contained 98·52 per cent silica and alkalies in very low quantities. The mineral was mostly quartz accompanied by traces of other minerals. The refractoriness of the sand was high (above 1350°C.). The sand can be used for steel casting purposes.

B-2. The sand sample B-2 was a coarse grained sand (G.F. No. 22), associated with 67.2 per cent AFS clay grade matter. Grain shape was sub-angular to rounded. It contained 93·4 per cent silica. The sand contained quartz predominantly with small amounts of feldspar, mica, and traces of tourmaline and hornblende. Its sintering temperature was between 1300°-1350°C. The sand may be used for light steel castings.

M-2. This sand sample was a medium to fine grained sand with AFS G.F. No. 89 and clay content of 0·8 per cent. The sand grains were sub-angular to sub-rounded. It contained 83·99 per cent silica. Mineralogically it contained predominantly quartz followed by micas, feldspar and opals. Its sintering temperature was between 1200-1250°C. The sand may be used for cast iron jobs but may have to be blended with coarse grained silica sand.

D-1. The sand sample D-1 was medium to fine grained (AFS G.F. No. 57) associated with clay content of 2·30 per cent, 22·06 per cent silica. The minerals present in order of their abundance were quartz, feldspars, mica, garnet, etc. The sintering range of the sand was approximately between 1200-1250°C. The sand could well be used for iron foundaries but a proper blending with coarse grained silica sand may be required to obtain better casting results.

Jg-1. The sand sample Jg-1 was fine grained (AFS G.F. No. 116 in the as received state) associated with 4·6 per cent AFS clay. Grain shape was angular to sub-angular. It contained 89·82 per cent silica. The minerals present in order of their abundance were quartz, feldspar, hornblende, muscovite, tourmaline etc. The sand was found to sinter approximately in the range of 1200-1250°C. The sand can be successfully used for cast iron foundries after proper mixing with coarse silica sand.

SP-2. The sand sample SP-2 was medium to fine grained (AFS G.F. No. 67) having an AFS clay content of 1·2 per cent. Grain shape was angular to sub-angular. The sand contained 78·8 silica and 10·75 per cent alumina. The mineral constituents of the sand were quartz, feldspar, hornblende, muscovite, garnet and zircon. It has sintering range between 1250°-1300°C. The sand was highly suitable for iron foundries after mixing with coarse grained silica sand to obtain better casting results.

Jg-2. The sand sample Jg-2 was a medium fine grained sand having an AFS grain fineness No. of 89 and containing 2·2 per cent AFS clay grade matter. The sand grains were angular to sub-angular. The as received sample contained 80·46 per cent silica, 6·82 per cent alumina and 2·4 per cent alkalies. The minerals present in order of their abundance were quartz, feldspar, hornblende, muscovite, tourmaline, ferruginous matter, etc. The sintering range of the sand was between 1150-1200°C. The sample can advantageously be used for cast iron jobs but a proper mixing with a silica sand will be necessary.

Jg-3. The sand sample Jg-3 was very fine grained with an AFS G.F. No. 148 and clay content of 20 per cent. The sand grains were angular to sub-angular. The sand contained 75·86 per cent silica, 11·70 per cent alumina and 2·1 per cent alkalies. The mineral contents of the sand were quartz, feldspar, hornblende, muscovite, ferruginous matter etc. The sintering range of the sand was low being 1100-1150°C. In view of low refractoriness, excessive fines and high clay content, the sand was not suitable for cast iron castings but can be used for non-ferrous foundry castings.

SP-1. The sand sample SP-1 was a fine grained sand having an AFS G.F. No. 140 and clay content of 7·4 per cent. Grain shape was angular to sub-angular. The sand assayed 76·5 per cent silica, 10·05 per cent alumina, and 3·2 per cent alkalies. The mineralogical constituents of the sand were quartz, feldspar, hornblende, garnet, silliminite etc. The sand was found to sinter in the range of 1200-1250°C. The sand can be usefully used for cast iron castings after proper blending with a coarse silica sand.

F-1. The sand sample F-1 was a fine grained sand having an AFS G.F. No. 145 and clay content of 9·65 per cent. The sand was angular to sub-angular. The sand gave 76·06 per cent silica, 12·07 per cent alumina 3·56 per cent iron oxide and 3·3 per cent alkalies. The minerals present were quartz, feldspar, hornblende and traces of zircon also. The approximate sintering range of the sand was between 1200-1250°C. In view of excessive fines, this sand cannot be used for foundry practice but may be employed only after mixing with a suitable silica sand.

F-2. The sand sample F-2 was also fine grained with an AFS G.F. No. 149 and clay content of 1·93 per cent. Grain shape was angular to sub-angular. The sand contained 82·20 per cent silica, 8·26 per cent alumina and 3·0 per cent alkalies. The mineralogical constituents of the sand were quartz, feldspar, hornblende, tourmaline and zircon in order of their abundance. The sand had sintering temperature of 1250-1300°C. The sand can be used for cast iron practice only after suitable mixing with a coarse grained silica sand as it contained more than 80 per cent fines.

F-3. The sand sample F-3 was a fine grained sand having an AFS G.F. No. 152

and clay content of 13·3 per cent. The sand was angular to sub-angular. The sand assayed 81·92 per cent silica, 4·28 per cent alumina, and 3·2 per cent alkalies. The mineral contents of the sand were quartz, feldspar, hornblende and tourmaline; some clayey matter was also present. The sand was found to sinter approximately in the range of 1200-1250°C. The sand may be used for cast iron castings but its proper blending with a coarse silica sand will be required.

F-4. The sand sample F-4 was a fine grained sand (AFS G.F. No. 143 and clay content 9·40 per cent). The sand contained 79·51 per cent silica, 7·40 per cent alumina and 2·9 per cent alkalies. The minerals present in order of their abundance were quartz, feldspar, hornblende, tourmaline and garnet. The sintering temperature of the sand was between 1200-1250°C. In view of the excessive fines, the sand may be used for foundry purposes only after proper mixing with a suitable grade silica sand.

D-2. The sand sample D-2 was a fine grained sand having an AFS G.F. No. 132 and clay content of 4·6 per cent. The sand grains were sub-rounded to rounded. The sand assayed 82·80 per cent silica, 7·10 per cent alumina and 2·40 per cent alkalies. The mineralogical constituents of the sand were quartz, feldspar, garnets, mica, hornblende, traces of zircon and kyanite were also present. The sand was found to sinter in the range of 1200-1250°C.

D-3. The sand sample D-3 was a fine grained sand having an AFS G.F. No. 125 and clay content of 15·10 per cent. Grain shape was sub-rounded to rounded. The sand assayed 75·95 per cent silica, 12·0 per cent alumina and 2·5 per cent alkalies. The minerals present were quartz, feldspar, hornblende and opaques. Traces of garnet and tourmaline were also observed. The sand had a sintering range of 1175-1225°C.

M-1. This sand sample was a very fine grained sand (AFS G.F. No. 154 and clay content 4·1 per cent). The sand was sub-rounded to rounded in shape. The sand contained 76·08 per cent silica, 11·82 per cent alumina and 2·5 per cent alkalies. The mineral content was quartz predominantly followed by feldspar, mica, hornblende, garnet and traces of zircon. The sand was found to sinter in the range of 1250-1300°C.

(viii) *Kandleru River Sand* — The Kandleru river sand received from Geological Survey of India, Andhra, was coarse to fine grained having an AFS G.F. No. 2·4 in the as received condition and containing 1·2 per cent AFS clay. The sand grains were angular to sub-angular in shape. The sand assayed 77 per cent silica and high percentage of alkalies. Sintering range of the sand was found to be 1300-1350°C. In view of its high permeability, excellent green strength, and good sintering range, this sand can be successfully used for grey iron foundries and may be used for non-ferrous foundries with suitable addition of the fine sand to improve the surface finish and reduce metal penetration.

(ix) *Rajendra Silica Sand* (Received from Messrs Rajendra Silica Works, Allahabad) — *Sample* I was of a high silica sand having AFS G. F. No. 40·6 and clay content 1·84 per cent. It contained coarse to medium grains of rounded to sub-rounded shape. It contained 97·8 per cent silica. The sample essentially consisted of quartz with traces of other impurities. Sintering range of the sand was above 1350°C.

The sample may be used as a core sand for iron and steel castings. It can also be used as a moulding material for light and medium steel casting after mixing it with a proper percentage of silica flour and application of mould washes.

Sample III — It was a silica sand sample having grains rounded to sub-rounded in shape with AFS G. F. No. 72 and contained 5·16 per cent clay. It contained 95 per cent silica. The sand consisted primarily of quartz followed by feldspar, etc. The sintering range of the sand was above

1350°C. Study of the moulding and casting characteristics showed that the sand was suitable for grey iron and steel castings.

II. *Bonding Clays*

(i) *Cutch Bentonite* (Received from Messrs. Mukund Iron & Steel Co. Ltd., Bombay) — This bentonite sample was of cream colour and in powder form. It was associated with moisture content of 11·20 per cent which is in agreement with the specifications for Western bentonites laid down by S.F.S.A. It contained SiO_2, 41·05; Al_2O_3, 18·87 and total alkali oxides, 2·71 per cent. The ratio of sodium to calcium was 2: 15. Its *p*H, gel, swelling index capacity values were found to be 8·75, 28, 9·1 respectively. The total base exchange capacity was 72 m. equts./100 g. The sintering range of the sample was 1025-1100°C. Petrological examination of the sample revealed that the predominant mineral constituent was montmorillonite. Study on the bonding characteristics of the sample with high silica Rajmahal sand showed that the sample develops satisfactory green and dry strengths in the moisture range of the bentonite; it was practically a sodium base bentonite and may be used as a binder in the foundries.

(ii) *Madras Bentonites* (Received from Messrs. Fomics Private Ltd., Rajajinagar, Bangalore) — The bentonite was of a dull light brown colour. It was associated with 6·65 per cent moisture content, a value just in agreement with the specifications of S.F.S.A. for Western bentonites. Its *p*H, gel, swelling index and total base exchange capacity values were 72, 10, 2·5 to 5 and 69 m. equts./100 g. respectively. On wet sieving, about 94 per cent of the sample passed through 325 mesh sieve of U.S. Series. The sample sintered in the temperature range of 1050-1100°C. It contained 51·36 per cent SiO_2, 27·84 per cent Al_2O_3 and 1·05 per cent total alkali oxides (the CaO content was 0·8 per cent). The ratio of Na/Ca was found to be 0·5. Petrological examination indicated the presence of kaolinite in the sample which was confirmed by D.T.A. Though this bentonite was contaminated and of low swelling type, it could develop sufficient strength on bonding with Rajmahal silica sand of grain fineness 55.

(iii) *Kashmir Bentonite* — Two samples of bentonite designated as PB-1 and SB-1 which occur in brown deposits in Kashmir were received from the Directorate of Mining & Geology, Jammu and Kashmir Government, Srinagar.

PB-1. This bentonite had pinkish colour with a dull waxy lustre and a soapy touch. It was associated with high moisture content of 17·3 per cent. Silica and alumina contents of the sample were 52·6 and 16·6 respectively. Na/Ca ratio was 0·2746, *p*H, gel and swelling index of the bentonite were 9, 9 and 2·5 respectively and the total base exchange capacity was found to be 73 m. equts./100 g. Examination of the minerals present indicated them to be predominantly montomorillonite with minor amounts of halloysite and chlorite.

When used as a bonding medium with high silica Rajmahal sand, this was found to develop high green and fairly satisfactory dry strength value. The bentonite was found to be suitable for foundry use.

SB-1. This sample was similar in colour, lustre and touch as the Pb-1 sample. This also contained high moisture content (16·8 per cent). It contained 50·0 per cent SiO_2, 19·34 per cent alumina, 0·56 per cent K_2O and 0·47 per cent Na_2O. Na/Ca ratio was 0·2216. Mineralogically, it was almost similar to the sample PB-1. Standard tests gave values of 8·75, 10, 2·44 and 62 m. equts./100 g. for *p*H, gel, swelling index and total base exchange capacity respectively.

When used as a binder with Rajmahal silica sand, it imparted high green and fairly satisfactory dry strength to the sand specimens. The bentonite may be used as a binder in iron and steel foundries.

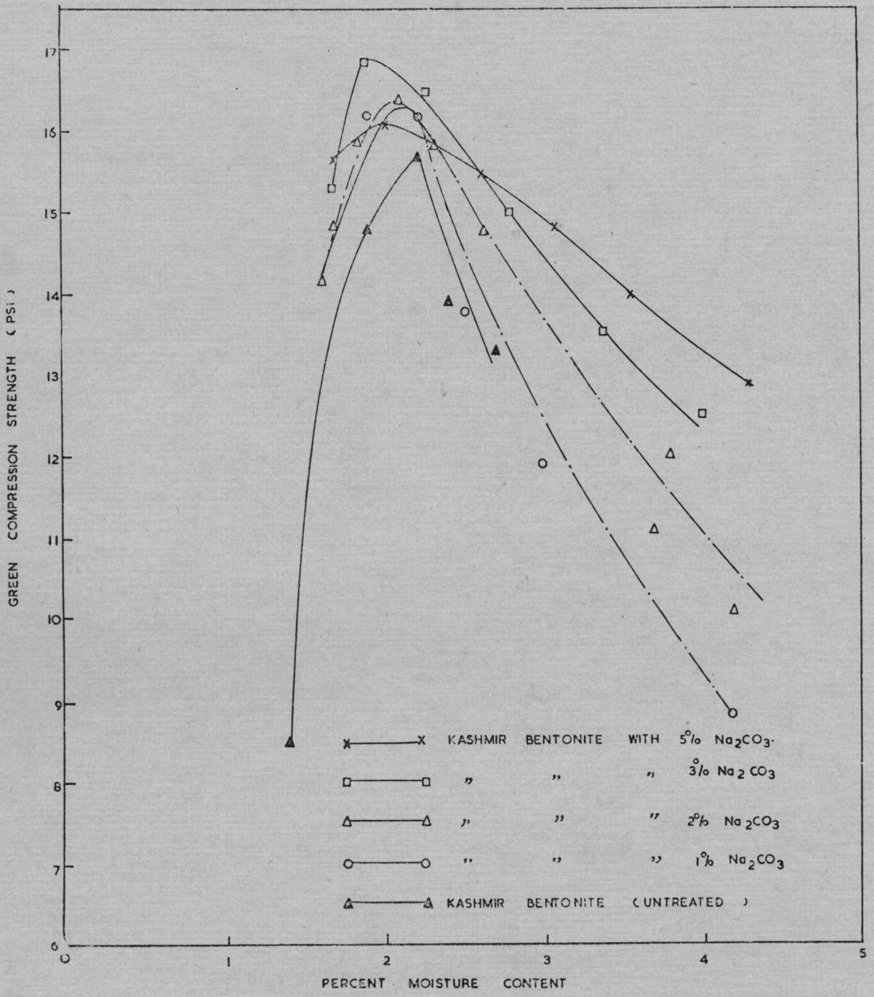


FIG. 36 — EFFECT OF Na_2CO_3 ACTIVATION OF KASHMIR BENTONITE ON THE GREEN COMPRESSION STRENGTH

(iv) *Activation of Bakudi Bentonite* — Clays are agregate minerals and possess a special property of absorbing cations from a solution or watery medium. These cations are not strongly held and are readily replaceable by other ions. This property is known as cation or base exchange capacity of the clay. Many physical properties of the clay minerals are dependent on the types and extent of cations that are exchangeable. A lean clay having poor physical properties can be improved by replacing the Ca^{++} ions by Na^{+} ions, thus transforming a Ca-base clay into a Na-base clay. Such a transformation of the clay is termed as activation.

Experiments were carried out on activation of Bakudi and Kashmir bentonite samples investigated earlier. Activation was carried out by adding Na_2CO_3 (varying from 1 to 5 per cent) in the slurry of bentonite in water both at room temperature and at 75°C. The resulting mass was dried at 100°C., crushed and used for further experi-

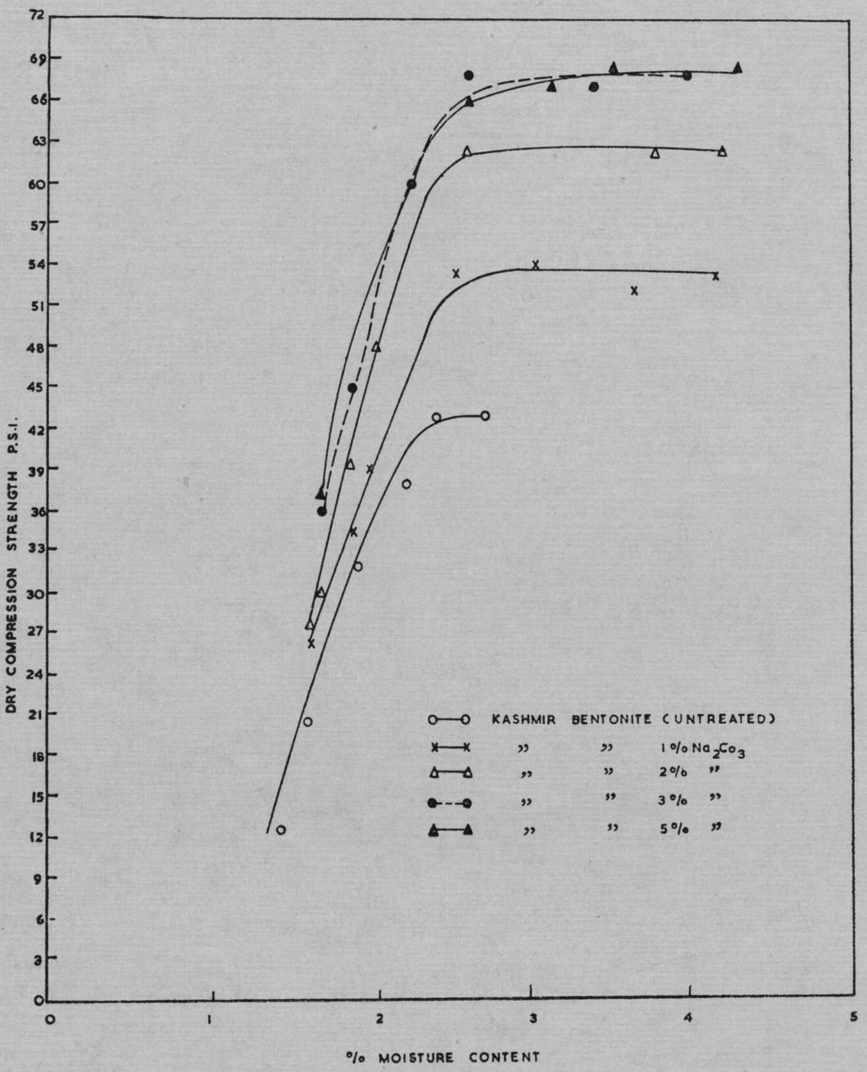
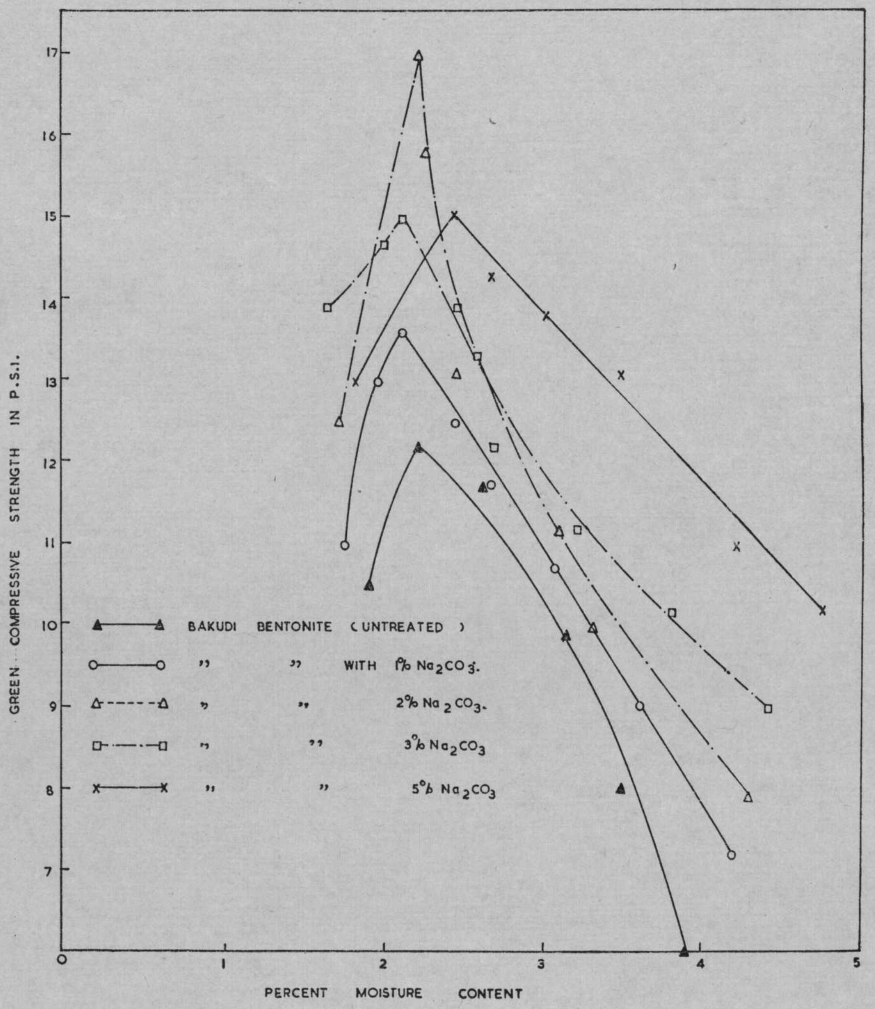


FIG. 37 — EFFECT OF Na_2CO_3 ACTIVATIONS OF KASHMIR BENTONITE ON THE DRY COMPRESSION STRENGTH

ments. The addition of Na_2CO_3 gradually increased the *p*H and gel values of the bentonite samples. The moisture content of the sample, though it increased with the addition of Na_2CO_3, was not so marked. With Bakudi bentonite, best results in green and dry strength values were found with 2 per cent Sorara bentonite with 3 per cent addition of Na_2CO_3. The treatment at 75°C. seemed to further improve the Bakudi bentonite but Sorara bentonite appeared to be unaffected. Figs. 38 and 39 show the development of green and dry strength values with the untreated and treated bentonite samples.

III. *Cellpro Core Binders*

In continuation of earlier work on 'Cellpro' core binders of 'HV' and 'LV'



Fig, 38 — Effect of Na_2CO_3 activation of Bakudi bentonite on the green compression strength

grades received from Messrs. Imperial Chemical Industries, further experiments were done to determine: (i) the optimum amount of cellpro binders to be used alone, (ii) use of lower per cent of cellpro core binder alone or in conjunction with linseed oil.

Moulding characteristics of the Rajmahal high silica sand mixtures bonded with cellpro 'HV'/'LV' ranging from 1 to 10 per cent were studied to determine the correct amount to be used. It was observed that 5 per cent and higher additions did not much improve the green or dry strength properties of the moulding mixtures and at the same time the sand mixtures were difficult to handle due to the sticky characteristics it developed. The working moisture content of the cellpro bonded sand mixture was found to be about 1·5 per cent for 1 per cent addition of binder and increased by approximately 1 per cent for increase in addition of 1 per cent of the binders up-to 5 per cent. The 'HV' grade developed

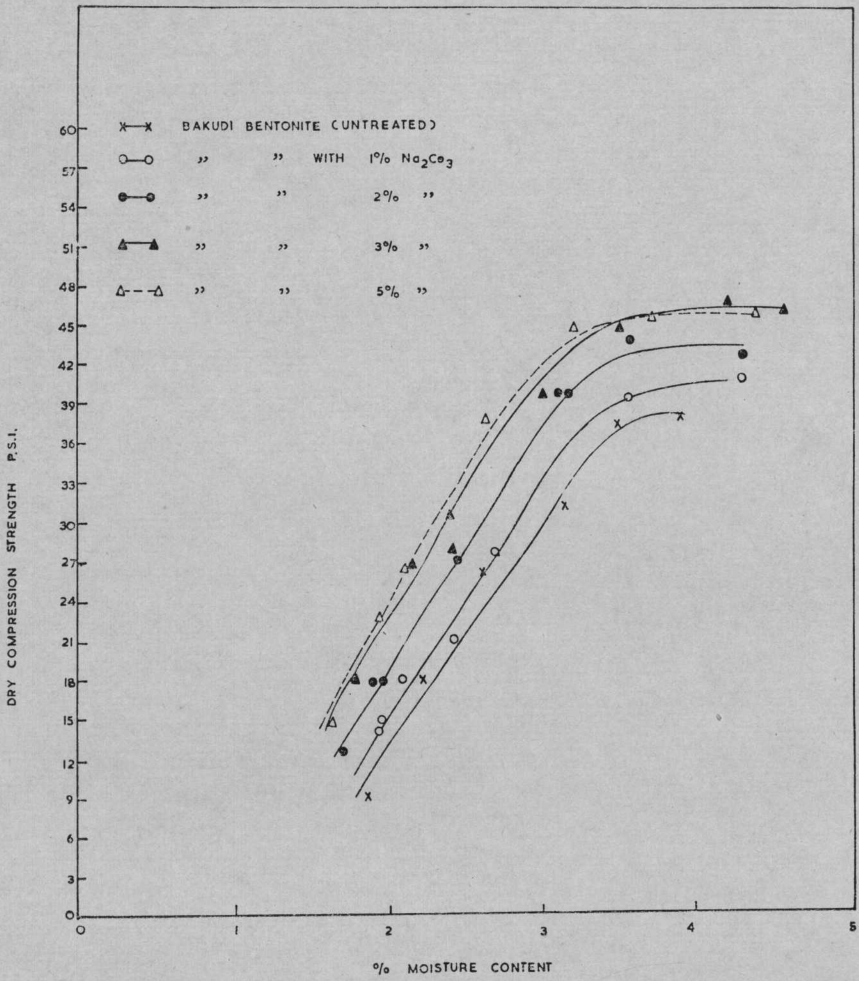


FIG. 39 — EFFECT OF Na_2CO_3 ACTIVATIONS OF BAKUDI BENTONITE ON THE DRY COMPRESSION STRENGTH

better green strength of the two but the reverse happened in case of developing dry strength. Experiments were conducted with additions of 0·5, 1·0 and 1·5 per cent cellpro binder alone and in conjunction of 1 per cent linseed oil. It was observed that additions of the cellpro binders alone in the quantities mentioned were inadequate but in conjunction with linseed oil, gave sufficient green strength for handling the sand specimens. The baking time and temperature which were optimum for oil cellpro bonded mixtures were 1½ hr. and 200°C. respectively. It was concluded that the cellpro binders may be used in conjunction with linseed oil considering the complexity and handling problem of the cores.

VI. *Silica Flour*

Investigation on silica flour sample sent by Messrs. Calcutta Mineral Supply Company Private Limited, was undertaken to study its suitability for foundry applications.

The silica flour was white in colour in the as received condition. It had a silica content of 97 per cent containing quartz of fine grain size; the grain shape was angular to sub-angular. It showed sintering temperature above 1500°C. Synthetic sand moulds with the silica flour facing gave a much superior surface finish compared to unfaced sand moulds. In view of the very fine quality of the silica flour, fairly high quartz content and high sintering temperature; it could be successfully used as a facing material for steel casting, as well as a mixing medium for coarse grained foundry sands for obtaining better distribution.

39.0 Aluminium-silicon Alloys

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

The new method developed for the 'Modification of Aluminium-silicon Alloys' was patented in India and overseas countries. A paper prepared on the subject was accepted as an exchange paper from India to be presented in the International Foundry Symposium to be held in Netherlands.

The effect of this new modifying agent is under study on certain aluminium-base commercial piston alloys. Experiments are being carried out to develop a method of making foam aluminium.

40.0 Powder Metallurgy

Work was taken up with a view to (i) produce copper powder by continuous electrodeposition and (ii) to make porous bronze bearings:

Main operations for making electrolytic copper powder are the initial electro-deposition and subsequent annealing operations. While copper powder of standard sieve analysis can be made, data were obtained as to how the apparent density of the powder could be enhanced to maximum capacity by electro-deposition operation itself. Annealing of the electro-deposited powder results in affecting the apparent density of the powder. How the change in apparent density can be controlled was studied in full detail. Table 21 gives the results of making copper powder by depositing and annealing under different conditions. Studies were also made of the response to sintering of copper powders made under different conditions. It was found that other properties being the same, the higher the temperature used for annealing the less rapid was the rate of sintering of the copper powder in the making of porous bronze materials.

As regards porous bronze bearings, the prevalent practice of their manufacture calls for strict standardization of the manufacturing conditions within close tolerance. While the prevalent practice was thus confined to limitations imposed by somewhat severe restrictions, experiments were continued which established the usefulness of certain chemical reagents for liberalizing the method of manufacture free from critical conditions. Thus, the use of KI and phosphate to a small extent only in the Cu, Sn, graphite and stearate powder mixture made sintering possible without the need for metal powders possessing special properties whilst imposing restrictions on operating conditions. Making these bearings and other porous bronze parts would be possible with the added advantage of

Table 21 — Results of Annealing and Electro-depositing of Copper Powders under Different Conditions

Sl. No. of powder	Sieve analysis of powder −270 mesh fraction %	Annealing treatment		A.D. g./cc. of powder		Remarks
		Heating time hr.	Temp. °C.	Before anneal	After anneal	
1	57	3	600	2·2	2·2	No change in powder property took place
2	51	2½	640	2·2	2·4	—
3	69	3	670	1·7	2·0	Powder fritted up and light grinding for repowdering was necessary of the annealed powder
4	60	2½	720	2·0	2·0	Large quantity of powder was annealed with the result no change in powder property took place
5	60	14	720	2·0	3·0	Large quantity of powder was annealed and furnace cooled after annealing. The powder fritted up and milling of the hard powder cake was necessary
6	49	4	700	2·5	2·5	The powder was annealed coated with glycerine. No change in powder property took place after annealing
7	50	1½	750	2·0	2·3	The powder was annealed coated with glycerine. Repowdering after annealing was necessary
8	70	2	750	1·5	2·4	The powder was annealed coated with linseed oil. No repowdering after annealing was necessary of the powder
9	45	2	800	2·4	2·6	do

greatly simplified manufacturing conditions by the suitable adaptation of these experimental results. Fig. 40 shows a type of porous bronze bearing of well developed porous structure and high permeability.

41.0 Production of Iron Powder by Direct Reduction

The investigation was taken up with a view to produce iron powder directly using direct reduction technique. The direct reduction characteristics of fine flaky mill-scale by coke breeze were studied. It

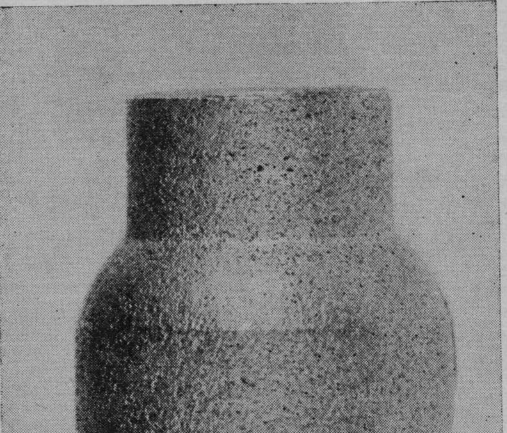


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FIG. 40 — POROUS BRONZE BEARING OF WELL-DEVELOPED POROUS STRUCTURE AND PERMEABILITY

was established that mill-scale can be reduced by solid reductant, such as coke breeze successfully at 900°C. in 8 hr. time. The degree of reduction attainable was over 98 per cent. Experiments were conducted to find the optimum mesh size of the mill-scale to enable its maximum degree of reduction in minimum possible time. The suitability of mill-scale for pelletizing prior to reduction was also studied, using molasses as binder. It was concluded that mill-scale has to be crushed to —200 mesh for successful pelletizing. Experiments were carried out with a view to ascertain the minimum amount of coke breeze required for complete reduction, so that carbon pick-up is low and reduction time is also minimum. 50 kg. iron ore of Goa origin was received from Messrs. Electric Control Gear Mfg. Co. for investigation on its direct reduction characteristics. 5 tons of iron ore had also been received from Government of Maharashtra for direct reduction studies and its suitability. Iron powder ideally suited to autogenous cutting was produced from Noamundi blue dust by direct reduction.

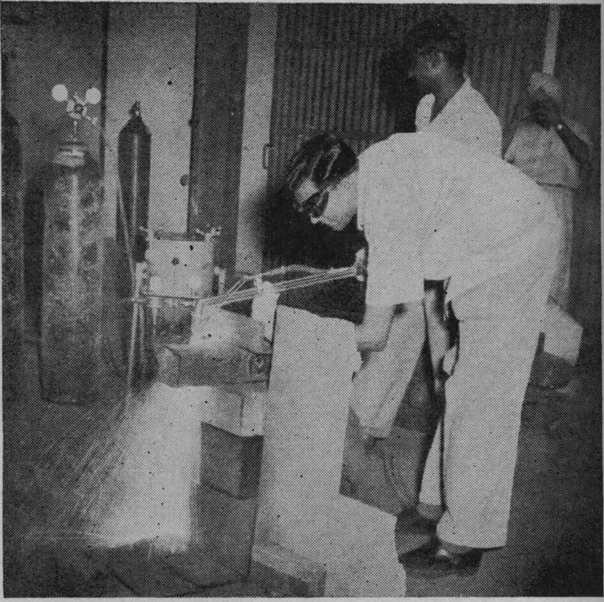


FIG. 41 — IRON POWDER PRODUCED FROM 'BLUE DUST' AT NATIONAL METALLURGICAL LABORATORY BEING USED FOR CUTTING INGOTS

42.0 Siliconization of Basic Pig Iron

In view of the prevailing shortage of foundry grade pig iron, it was felt necessary to investigate, whether Indian foundries could charge basic iron in the cupola and siliconize it to the desired level by the addition of ferro-silicon in order to yield suitable grade of foundry pig iron.

Experiments on melting of the basic pig iron were carried out in an acid lined cold blast cupola with a rated capacity of 18 to 20 cwt./hr. employing a coke to metal ratio of 1: 7. Since better silicon recoveries were obtained when ferro-silicon was added to the bath, the siliconization studies were carried out by addition of powdered ferro-silicon (6 mesh and down) to the basic iron melted in the cupola and received in a ladle. The pig iron employed in these investigation contained 1·03 per cent Si which after melting in the cold blast cupola come down to 0·87 per cent, thereby indicating a silicon loss of ·15 per cent during cupola melting which is considered normal. Two per cent addition of 75 per cent grade ferro-silicon to the molten metal in the ladle increased its silicon content to 1·89 per cent which amounted to a silicon loss of about 20 per cent. However, it was observed that the consistent recovery of silicon from ferro-silicon addition could not be obtained and at times, it was as low as 70 per cent. Temperature of the metal at the spouts was only about 1320°C. Temperature of the metal after 2 per cent ferro-silicon addition was not more than 1200°C. In these trials, 100 per cent pig iron charge was used which may not be so in full fledged foundries where the pig iron was used with proportionate amounts of return iron and steel scrap. In the latter cases, there was a greater fear of temperature fall during transfer of metal to the ladle and during siliconization.

On the basis of 100 per cent basic pig iron charge in the cupola, siliconization is estimated to increase the cost of foundry grade pig iron by about Rs. 30 per ton of pig iron melted. It was considered that siliconization of basic pig iron to foundry grade pig iron which contained 3·25 per cent Si was metallurgically difficult and economically highly expensive to be acceptable on commercial scale under Indian conditions.

42.1 Solidification of Cast Iron

Cast irons containing aluminium are resistant to scaling at high temperatures. These have been little used commercially because of their brittleness, poor hot workability and additionally casting problems. When aluminium was added to cast iron, two graphitizing ranges were observed. The first graphitizing range was from 1-8 per cent Al and the second graphitizing range was from 19 to 26 per cent Al. In between the two ranges, say, from 8 to 19 per cent Al, the cast irons were hard, brittle and unmalleable. However, in this intermediate range, the cast irons were reported to possess maximum resistance to scaling at high temperatures. Another difficulty with these cast irons was the abnormal Al_2O_3 inclusions would also lead to brittleness of the iron. Research investigations were, therefore, taken up to study the effect of optimum additions to cast iron containing 8-19 per cent Al to establish if improvement could thereby be obtained in respect of hardness and machinability and to develop suitable composition which will have resistance to scaling at high temperatures.

43.0 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. The production of low residual soft iron in India would prevent foreign exchange drawing by eliminating its import for Defence, Posts & Telegraphs, Railways and Telephone industry. Optimum refining techniques were investigated for the production of low residual iron in 0·8 ton direct electric arc furnace. Low residual iron in the following range of composition was produced:

		Per cent
Carbon	...	0·03 max.
Sulphur	...	0·01-0·02
Phosphorus	...	0·004-0·016
Silicon	...	0·01-0·12
Manganese	...	Trace-0·11
Aluminium	...	Trace-0·21

Plain carbon steel scrap was used as the starting material for the production of low residual iron. Fe-Si and aluminium were the basic deoxidizers used in the experiments. For soft magnetic material, Fe-Si was used as a final deoxidizer in addition to aluminium. The material is rollable and forgeable and possesses good magnetic property. The tests carried out showed that a coercive force of 1·1 oersted was attained with a saturation inductance of 18857 gausses.

Low residual iron, suitable as a base for the production of special steels and super alloys, was kept as low in its silicon content as possible, which was achieved by blowing the Fe-Si deoxidized bath by oxygen and finally deoxidizing the bath with aluminium. The initial deoxidation with silicon was considered necessary in order to achieve very low sulphur content (of the order of 0·01 per cent). Trials were conducted on the forgeability of the low residual iron for use as a basic raw material in melting. With low silicon and manganese, difficulty was experienced in its forging. Attempts were successfully made to cast the molten iron directly in the form of shots. Success

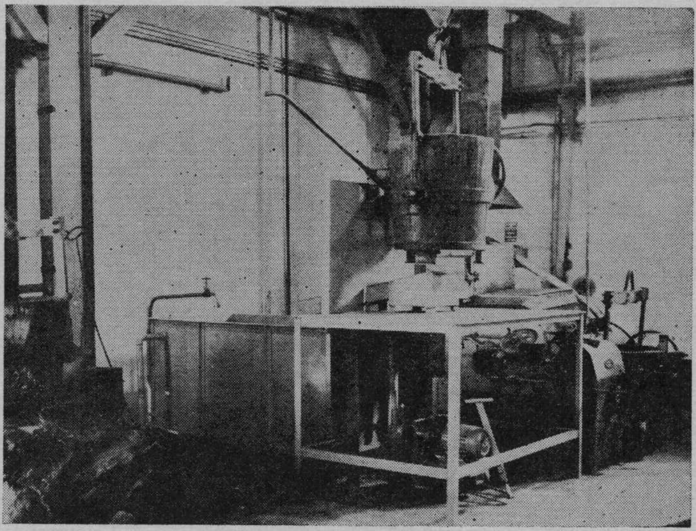


FIG. 42 — SET UP FOR CASTING SHOTS

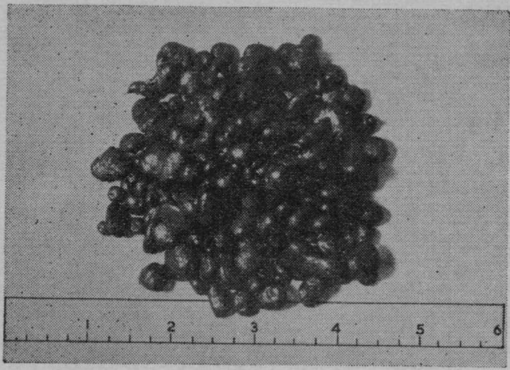


FIG. 43 — LOW RESIDUAL IRON SHOTS

had been achieved on experimental scale. Large scale trials are in progress. Fig. 42 shows the set up used for shot making whilst Fig. 43 indicates the type and size of shots that are attainable.

44.0 Degassing of Liquid Steel During Teeming

The investigation was taken up with a view to develop a method for degassing (mainly hydrogen) molten steel during teeming by the use of inert gases such as argon or nitrogen instead of the use of vacuum degassing techniques which involve the use of elaborate equipment. The gas when used under pressure broke up the pouring stream and thus reduced the gas content of liquid steel. The set up is shown in Fig. 44, which is based on a set up earlier

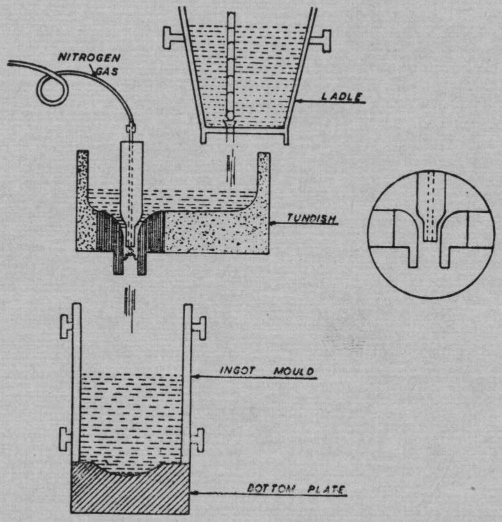


FIG. 44 — SPRAY DEGASSING OF LIQUID STEEL DURING TEEMING

tried out at BISRA's Laboratory at Sheffield.

Small scale experiments were initially carried out to be followed by pilot plant scale trials. The procedure for the small scale experiments was as follows:

A 50 lb. mild steel heat was made in a direct electric arc furnace. After the usual refining, the metal was poured in a ladle. The metal was then teemed through a tundish. Tundish consisted of one stopper fixed in position which allowed the metal to have annular flow round the stopper. The stopper was drilled through the full length and the inert gas (N_2) was passed through this cavity at a pressure of the order of 70-100 lb./sq. in. Due to sudden release of pressure at the end of the stopper the gas expands and breaks the stream of metal into droplets resulting in the reduction of hydrogen content of steel. After the teeming was over, the gas system was closed. Temperature of the metal was recorded by immersion thermo-couple at various intervals as follows:

(i) In the furnace prior to tap,
(ii) In the ladle,
(iii) In the ingot.

A temperature of the order of 1640°±20°C. was required in the ladle prior to the commencement of teeming. It was observed that ingot moulds could be filled in a few cases completely without any gassing trouble. Hydrogen samples were taken before and after teeming in the ladle. Silica tube could be conveniently used for taking hydrogen samples. Hydrogen content as analysed for one of the experiments is given below:

Ladle sample 4·6 cc./100 gm.

Ingot sample 3·6 cc./100 gm.

Hydrogen samples needed immediate quenching in water whilst not permitting the temperature to go below 300°C. It was then cooled in liquid air for arresting the diffusion of any hydrogen. For the time being, scrap graphite electrodes were used as stopper for the tundish which were ultimately to be replaced by the regular type of stopper heads.

45.0 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.

Several medium-size melts using aluminium-bronze scrap were melted in a magnesite lined 17 KW. indirect electric arc furnace. These were melted in 3 kg. charges with and without fluxes. Three typical heats representative of the three processes used, are detailed in Table 22. Carbon-tetrachloride was added in the mould just prior to tap in order to protect

Table 22 — Effect of Melting Procedure on Chemical Composition of the Charge and Mech. Properties of the Cast Alloys

Exp.	Alloy	Chemical composition (%)				Flux used and metallic loss %	Mech. prop. of cast alloy		
		Cu	Al	Fe	Mn		U.T.S. kg./mm².	0·2% proof stress kg./mm.	Elong. %
1	Scrap charge	85·62	10·4	3·7	0·93	—	—	—	—
	Cast alloy	85·38	10·96	2·67	0·71	17	53·68	40·50	10
2	Scrap charge	86·76	10·11	3·9	0·22	NaCl, KCl	—	—	—
	Cast alloy	86·36	10·34	2·67	0·54	5·7	63·58	33·22	17·9
3	Scrap charge	88·04	10·10	1·69	0·66	Albaral	—	—	—
	Cast alloy	86·30	10·10	2·97	0·64	15·0	54·38	22·98	7·9

Table 23 — Chemical Analyses and Mechanical Properties of Some Al-Bronzes Produced from Virgin Metal

Heat No.	Chemical composition (%)					Mech. properties		
	Cu	Al	Fe	Mn	Sn	U.T.S. kg./mm².	0·29 proof stress kg./mm².	Elong. %
1	86·22	10·05	2·13	0·59	0·39	(i) 68·34	24·0	26
						(ii) 62·5	28·6	
2	86·17	9·7	3·53	0·34	0·36	64·07	24·0	19 (broken on gauge length mark)
3	86·00	9·13	2·81	0·45	0·36	(i) 64·07	23·2	32
						(ii) 64·4	23·68	24

the pouring stream from oxidation. A mould wash was given of a mixture of KCl and NaCl, to protect the surface of cast ingot. Table 22 also gives the experimental details regarding the use of fluxes, charge composition, final composition of the melted product and mechanical properties.

Having found that NaCl and KCl flux gave high yield and good mechanical properties in the melting of Al-bronze, experiments were started on the production of aluminium-bronze by melting from virgin metal with the use of different fluxes. Illustrations of some heats prepared are tabulated along with their chemical composition and mechanical properties in Table 23, which were made by melting copper, commercial aluminium, electrolytic manganese and iron. Iron was added as an alloy of iron-aluminium to facilitate easy mixing.

Further work is underway to study (i) attempts to replace Fe-Al by direct iron additions, (ii) studies of structure inclusions and segregation etc., (iii) study and comparison of the mechanical properties of the vacuum melted alloy vis-a-vis air melted alloy.

46.0 Study of Temper Brittleness

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

The work continued on the basis of earlier pattern of study on the subject particularly on nickel-chrome steels. Efforts were made to grow coarse grained specimens of nickel-chrome steels suitable for X-ray diffraction studies by cold pressing tapered specimens and annealing the pressed specimens in argon atmosphere, the temperature of annealing being raised slowly from 800 to 1000°C. From the experiments carried out so far, it was found that a 4 per cent cold work would be highly suitable for the growth of coarse grains whilst aiming to get single crystals of the nickel-chrome steels. Further work on this long range project is actively continuing.

47.0 Curie Temperature of Iron-chromium Alloys

The objective of this project was to determine the electron distribution of iron contributing to ferro-magnetism. There is a difference in the ferro-magnetic behaviour of iron on the one hand and that of cobalt and nickel on the other. It is felt that curie temperature which is taken to be an index of ferro-magnetism may reflect this difference. The experimental procedure contemplated related to the preparation of the alloys of the required composition and thereafter to their homogenization.

Alloys given in Table 24 were prepared in the high frequency furnace of 25 lb. capacity with magnesite lining. After homogenizing and forging, specimens were prepared and welded to ' low-moor ' iron on either side and their curie temperatures were determined. The values are given in Table 24.

Table 24 — Curie Temperature of Fe-Sn and Fe-Cu Alloys

Alloy No.	Sn %	Cu %	Curie temperature °C.
Fe-Sn Series			
1	3·2	—	757
2	5·3	—	750
3	8·3	—	747
Fe-Cu Series			
1	—	5·9	752
2	—	7·2	750
3	—	8·7	747
4	—	13·3	746

48.0 Preferred Orientation in Cold Rolled Aluminium Sheets

The usual method of determining the preferred orientation produced in cold-rolled sheet is based on the study of the nature and intensity of X-ray reflections. This method could advantageously be replaced, if possible, by methods based on the values of the electrical conductivity and the velocity of propagation of stress waves in rolled sheet as a function of the angle to the rolling direction.

The textures developed during cold working of annealed sheets of commercial aluminium under various rolling sequences are summarized in Table 25.

It was found that surface and interior textures differed only in relation to the amount of scatter; reversing the direction of rolling did not cause any appreciable change in the textures developed during uni-directional rolling except a slight difference in their scatter. The textures developed on cross rolling and those developed on cross rolling and reversing combined, were the same differing only slightly in their scatter. The investigation has been completed and a full Report is under preparation.

Table 25 — Texture Developed During Cold Working of Aluminium Sheets

Rolling sequence	No. of passes	% reduction	Textures	
Direction of rolling same in each of the successive passes	19	92·3	(110) (112) (123)	$(1\bar{1}2)$ $(1\bar{1}1)$ $(1\bar{2}1)$
Direction of rolling reversed after each pass	18	92·3	(110) (112) (123)	$(1\bar{1}2)$ $(11\bar{1})$ $(1\bar{2}1)$
Cross rolled after each pass	18	91·6	(110) (123) (100)	$(1\bar{1}2)$ $(1\bar{2}1)$ $(00\bar{1})$
Cross rolled and reversed alternately	18	91·6	(110) (123) (100)	$(1\bar{1}2)$ $(1\bar{2}1)$ $(00\bar{1})$

49.0 Conductivity of Al-Si and Al-Si-Mg Alloys

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

Variations in the conductivity of Al-0·5 per cent Si alloy during ageing at a series of temperatures were determined. During

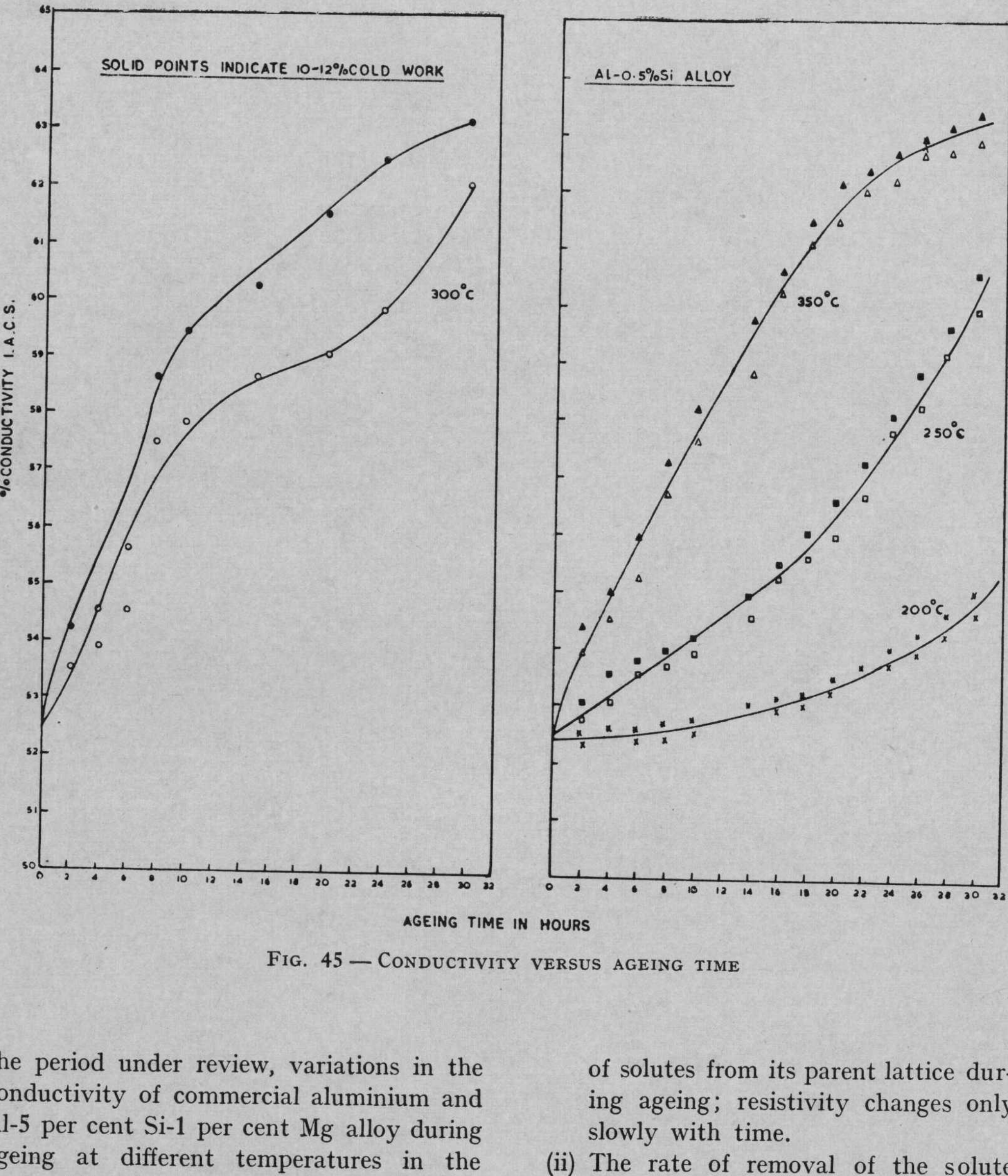


FIG. 45 — CONDUCTIVITY VERSUS AGEING TIME

the period under review, variations in the conductivity of commercial aluminium and Al-5 per cent Si-1 per cent Mg alloy during ageing at different temperatures in the range 250-490°C. were studied. Effects of cold-work to the extent of 10-12 per cent on the acceleration of precipitation processes were also examined. Results are shown in Figs. 45 and 46. Work is now in progress on alloys of commercial aluminium containing 1 and 2 per cent Mg respectively.

Experimental observations so far made are summarized below:

(i) Solution treated and quenched commercial aluminium resists the removal of solutes from its parent lattice during ageing; resistivity changes only slowly with time.

(ii) The rate of removal of the solute atoms is faster in the Al-Si alloy than in commercially pure aluminium and increases as the ageing temperature is raised to 350°C., there is not much difference in the kinetics of ageing at 350°C. and at 400°C.

(iii) Ageing of Al-Si alloy at 350-400°C. for 30 hr. increases the conductivity to about 63 per cent I.A.C.S.

(iv) The addition of magnesium to Al-Si alloy substantially increases the rate

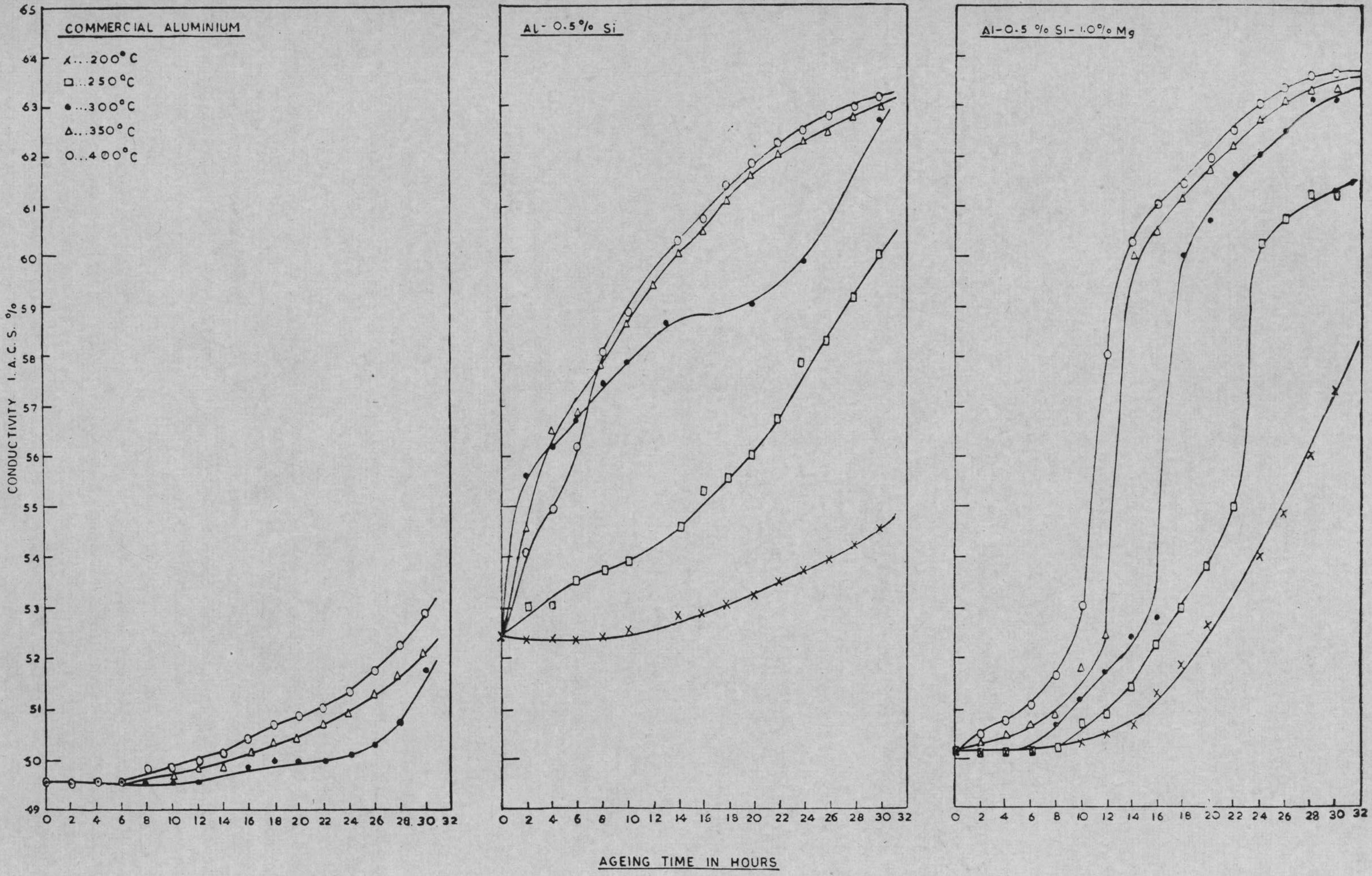


Fig 46 — Conductivity versus ageing time of removal of dissolved silicon. The rate of ageing increases with the rise in ageing temperature, but the kinetics of later stages of ageing at 350° and 400°C. are not much different from one another.

(v) Ageing for 14 hr. at 350-400°C. increases the conductivity to 60 per cent I.A.C.S., but the conductivity may be increased to about 63 per cent I.A.C.S. by ageing for nearly 30 hr. in the same temperature range.

50.0 Structure of Liquid Metals

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

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

(a) *Study of interactions between Al-Cu, Al-Cu-Mg, Pb-Sn alloys* — The Al-4 per cent Cu alloy was centrifuged at 200 r.p.m. at 700°C. for 1, 3, 5, 7 and 9 hr. Samples were chemically analysed and metallographically studied. In order to eliminate the statistical error, several samples were centrifuged under the above conditions. The results obtained are being analysed.

Samples weighing approximately 100 g. of Pb-Sn alloys with nearly eutectic compositions were centrifuged under the conditions given in Table 26.

Table 26 — Centrifuging Conditions of Pb-Sn Alloys

Sl. No.	Temp. °C.	Speed r.p.m.	Radius of gyration from the centre of rotation to the middle of the crucible cm.	Force developed × g.
A	250	265	90	70
B	320	200	90	40
C	320	200	90	40
D	320	265	90	70
E	400	200	90	40

Hypo-eutectic and hyper-eutectic alloys were also centrifuged in order to study the effects of composition on the interactions. Many samples from different positions were taken from the solidified samples for chemical analysis. It was noted that a sedimentation equilibrium was set up after centrifuging for one hour which was not altered even when the centrifuging was extended over 6 or 8 hr.

The results obtained also confirm the existence of 'clusters' in liquid metals. From an analysis of sedimentation equilibrium, size of the clusters was calculated for all the alloys as shown in Table 27. The ease with which sedimentation equilibrium was obtained under small centrifugal forces suggested that forces between the clusters were weak.

Table 27 — Cluster Size of Alloys at Different Temperatures

Series	Temperature °C	Size of the cluster dia., Å.
A	250	30
B	320	—
C	320	35
D	320	30
E	400	35

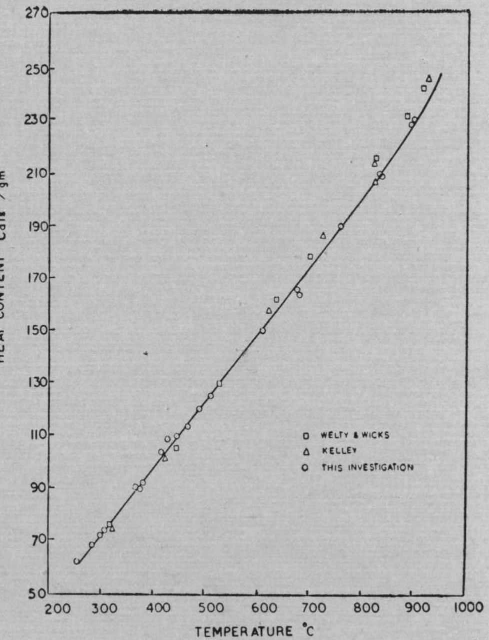
A new centrifuge capable of operation at 1000-1500 r.p.m. and developing a centrifugal force of about 550 g. was also designed and fabricated at the National Metallurgical Laboratory.

(b) *Study of partitioning of magnesium between aluminium and lead* — Several experiments were carried out to determine the distribution coefficient of magnesium between immiscible liquids — lead and aluminium containing 0·5-1 per cent magnesium at 700°, 800° and 900°C. From the results of the chemical analyses, it was qualitatively observed that the addition of copper to the liquid aluminium-magnesium, increased the tendency of magnesium to enter lead. As there was considerable scatter in the values of distribution coefficient no quantitative conclusions could be drawn. The scatter was possibly due to the non-attainment of equilibrium between the two immiscible liquids. The experimental set up has been modified to promote constant agitation and mixing to ensure that Pb and Al-Mg liquids have maximum contact.

(c) *Enthalpy of liquid alloys* — Apparatus for the determination of the heat content of the liquid metals was standardized and calibrated against Merck's Al_2O_3 (chromatographic). The values obtained are shown in Fig. 47, which also gives the data of Kelley and United States Bureau of Standards for comparison purposes. Work is now in progress on aluminium and aluminium-copper alloys.

A photo-transistor temperature controller was filled to the platinum wound furnace to control its temperature. The photo-transistor temperature controller was developed at this Laboratory and maintained the furnace temperature within ±1·5°C. over the operating temperature range of 200-1000°C.

(d) *Field Freezing* — It is well known that solute distribution in metals and alloys can be controlled by careful freezing. One such example is the development of the process of zone refining. An electric field can also produce substantial changes in the concentration in a liquid solution by virtue of the differences in the ionic mobilities, and is a field of topical current technical interest. It is commonly known as ' Field freezing '. Its possible applications are the separation of components and controlling the effective value of the distribution coefficient.



HEAT CONTENT OF ALUMINIUM OXIDE

FIG. 47

Preliminary experiments were carried out to study the movements of components in a binary Pb-Sn eutectic at 320°C., which was well above the melting point. Electric current from a rectifier was passed through a capillary which was connected at either ends with reservoirs. It was found that movement of lead or tin occurred under the electrical field. It is now proposed to investigate this phenomenon extensively in binary eutectic systems above their melting point.

51.0 Effect of Thermal-cycling of Cu-Pb Alloys

The investigation was undertaken with a view to study the possibility of causing separation of copper from mixed white metal scrap by physical methods. The segregation of lead during thermal cycling in the Cu-40 per cent Pb alloy was greatly masked by the gravity segregation of the immiscible liquids. In order to study the effects of thermal cycling of copper, Cu-10 per cent Pb alloy was chosen in the range of liquid immiscibility. Metallographic examination was undertaken to study relevant solidification features.

52.0 Effect of Hydrogen on Overheating and Burning of Steels

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

Specimen (3/4 in. dia.×3/4 in. long) from steels listed in Table 28 were soaked in the higher temperature regions of austenite stability for 30 min.; the temperature of soaking was raised progressively by steps of about 25°C., until the specimens showed signs of overheating or burning based on metallographic examination. One set of specimens was soaked in an atmosphere of nitrogen and the other in hydrogen so that hydrogen was picked up by steel. Although hydrogen content of the specimen was not determined, the hydrogen-soaked specimen should retain hydrogen as the entire heat-treatment was done in an atmosphere of hydrogen. The specimens were subsequently etched (i) by 10 per cent nitrosulphuric acid and (ii) electrolytically using ammonium nitrate as electrolyte. The nitrosulphuric acid etch indicated overheating by exhibiting a polygonal network of black boundaries against a light background, and burning by white polygonal boundaries against a dark background. On the contrary, the electrolytic etch indicated overheating by white boundaries and burning by black boundaries.

Results of the investigations are recorded in Table 28. It will be noticed that distinct lowering of the overheating and burning temperatures was observed only in three steels; in the two other steels, partial melting at the grain boundary occurred in presence of hydrogen. Two steels could not be overheated or burnt up to a soaking temperature of 1375°C. In the remaining four steels, hydrogen did not affect the overheating and burning temperatures.

These observations, however, do not establish the effect of hydrogen unequivocably vis-à-vis overheating and burning; nevertheless they do indicate that hydrogen may lower overheating and burning susceptibility temperatures in certain steels. These observations also confirm the peculiarity of hydrogen that its effect does not affect the susceptibility to embrittlement or the formation of hairline flaking or the gamma-to-alpha transformations in all the steels to the same extent.

Since two etching techniques do not always give identical results, much reliance cannot be placed on the metallographic detection of overheating of the steels. It was felt that the nitrosulphuric acid etch is more sensitive to detect burning and electrolytic etch for overheating.

53.0 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 of combined carbon present either as

Table 28 — Results of Overheating and Burning of Steel Samples

Sl. No.	Elements (percentages)									Overheating temp., °C. environment of		Burning temp., °C. environment of	
	C	S	P	Mn	Si	Cr	Ni	Mo	V	Hydrogen	Nitrogen	Hydrogen	Nitrogen
1	0·64	0·025	0·10	0·7	0·06	—	0·004	—	.	1325	1325	1350	1380*
2	1·47	0·05	—	0·25	0·28	0·53	0·07	0·02	—	1250†	1275	1275	1300
										1275**	1300	1325	1325
3	0·50	0·036	0·02	0·82	0·18	0·60	—	—	—	1375	1400	—	—
‡4	0·78	0·046	—	0·34	0·25	0·04	0·13	0·02	—	1380†	1380	1350	—
										1350**	1350		
‡5	0·90	0·031	0·03	0·27	0·11	0·07	—	—	—	1350†	1350	1375	1375
										1325**	1325	1375	1375
6	0·44	0·014	0·02	0·73	0·21	—	—	—	—	No overheating detected up to 1375°C.			
7	0·43	0·012	0·026	1·50	0·23	0·03	—	—	—	do	do	do	—
8	0·29	0·01	0·011	0·44	0·20	0·69	0·29	0·35	0·28	1400	1400	—	—
9	0·70	0·022	0·014	1·8	0·36	—	—	—	—	—	—	1380	1380
10	0·40	0·01	0·14	1·48	0·16	1·00	—	—	—	1375	1375	—	—
11	0·59	0·022	0·15	0·6	0·15	1·17	—	—	—	1380†	1380	—	—
										1350**	1360		

*Unless otherwise stated, the two etchants gave identical temperatures.

**Temperatures obtained by etching the specimens electrolytically.

†Temperatures obtained by etching the specimens in 10 per cent nitrosulphuric acid.

‡Partial melting at the grain boundary was observed in presence of hydrogen.

cementite or as carbides of alloying elements, and (iii) the amount and nature of solutes dissolved in the ferritic matrix.

Alloying elements are known to influence the above three factors to different extents. Mn, Ni, Cu, Al, Cr, Mo, V and Ti are the commonly added alloying elements in iron. Of these Ni, Mn and Cu stabilize austenite, but excercise diverse effects on the formation of graphite. Similarly all the carbide formed do not have similar effect on the formation of graphite. Although titanium is a strong carbide former, its low contents in the cast iron act as graphitizing agent, such as nickel or silicon. Neither the effect of higher percentages of titanium nor the mechanism of the effect of titanium on cast iron is fully known. This project was undertaken in view of the abundance of titanium in India. So far several heats containing varying amounts of titanium and aluminium were made using the ferro-titanium made in the National Metallurgical Laboratory.

54.0 Mar-ageing of Steels

The conventional process of hardening and toughening steel is through the formation of martensite and subsequent tempering. During tempering, different carbides are precipitated. 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. The 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. So far heats containing aluminium between 1 and 4 per cent were made with iron-20 per cent nickel alloy. Kinetics of ageing at 500°C. are being investigated.

55.0 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 and technology of grain refinement of steels or aluminium are well known, those of magnesium and its dilute alloys are known somewhat on an empirical basis and no generalizations are as yet possible. 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 treatments is not completely understood. In particular, the following points remain obscure:

(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;

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

The investigation has been undertaken with a view to fully understand the mechanism of grain refinement of magnesium. It is proposed to study the mechanism of grain refinement of magnesium and its alloys by the following techniques: (i) It is possible that the grain refinement occurs through the formation of embryos of the solid phase in the liquid metal. Their formation and temperature dependence could be investigated with the help of the liquid metal centrifuge. As this technique has already been developed at the National Metallurgical Laboratory, it is felt it can be applied towards investigating this problem. (ii) Identification of the nuclei and development of the technique of inoculation: The effect of different metal additions on grain refinement will be investigated by conventional techniques. Attempts will be made to identify the nuclei through the technique developed by Cibula.

56.0 Measurement of Carbon Activity in Iron and Its Alloys

Under the C.S.I.R. Research Fellowship Scheme work was taken up on the equilibrium study between carbon dissolved in iron and definite mixtures of carbon monoxide with carbon dioxide which was of considerable importance in order to gain exact knowledge of the phase boundaries that existed in Fe-C system, activity of carbon in different regions of the system, heat of solution of carbon in different regions of the system, heat of solution of carbon in different solid solutions, heat of transfer of carbon from the gas mixture to gamma iron, heat of transformation of the different forms of iron and nature of the crystal structure, etc.

Measurements of the activity of carbon in austenitic iron at 800° and 1200°C. were completed. For measurements at 800°C., no special modification of the original set-up was needed whereas for performing experiments at 1200°C. the reaction tube utilized for experiments at 800° and 1000°C. was replaced by an impervious thermal alumina tube. Both the ends of the thermal alumina tube were fitted up with standard joints with the help of silicon compound and connected to the rest of the system at both the ends with two male joints. Tables 29 and 30 give some of the typical results.

Table 29 — Experimental Results of Equilibrium of CO-CO_2 Mixtures with Austenite at 1200°C.

Sl. No.	$\gamma = \frac{p^2CO}{pCO_2}$ in atmospheres	Final carbon content % by weight
1	118·62	0·35
2	190.05	0·59
3	349·10	0·75
4	276·0	0·81
5	300·54	0·89
6	330·32	1·0
7	350·4	1·02

Table 30 — Experimental Results of Equilibrium of CO-CO_2 Mixtures with Austenite at 800°C.

Sl. No.	$\gamma = \frac{p^2CO}{pCO_2}$ in atmospheres	Final carbon content % by weight
1	7·76	1·36
2	7·82	1·35
3	7·43	0·87
4	7·81	0·92

57.0 The Effect of Nitrogen on the Transition Temperature

Nitrogen is a deleterious element for most steels and many of the new methods of steel-making aim at reducing the nitrogen contents in the steels. Its presence in small amounts contributes to ageing effects and formation of stretcher strains. The embrittlement due to nitrogen is one of the major causes of the failure of many structural members. It is, therefore, very important to have a clear and effective picture of the form and magnitude of its effects singly or in combination with other elements or alone on the mechanical properties of steels. The susceptibility of a steel to brittle fracture is studied by determining the transition temperature, i.e. the temperature at which its mode of failure in impact tests changes from ductile to brittle. The lower the transition temperature, the less susceptible to brittle fracture steel is considered to be. The transition temperature can be determined by any of the various criteria, such as energy absorption, ductility or fracture appearance. A review of available literature showed that the results on the effect of nitrogen on the transition temperature, particularly in combination with manganese, are not consistent. This project was taken up with a view to study the effect of nitrogen at various manganese levels on the transition temperature.

A low residual iron, with 0·03 C, 0·07 Si and without aluminium, was taken as the basic raw material. Various methods were tried to introduce controlled percentage of nitrogen at different manganese levels. It was found by experiment that this can be achieved by the addition of nitrided and unnitrided electrolytic manganese. Some preliminary heats were made in the high frequency furnace. Ingots of 2 in. sq. cross-section were forged and the impact specimens made from them after proper heat-treatment are being tested at ambient and at various sub-zero temperatures. Total nitrogen as well as the nitrogen present in solid solution was analysed for all the heats and its metallographic structures fully examined.

58.0 Phase Transformation in Iron-Manganese Systems

Phase transformations in the binary iron-manganese systems do not appear to have been fully investigated so far. The investigation was taken up with a view to study the kinetics of the phase transformations in this system, and a beginning was made with a study of martensitic transformation with resistivity measurements in iron-15 per cent manganese alloys. Alloys were prepared in the high frequency furnace from the low-carbon iron made in the Laboratory. Experimental technique is now being standardized.

59.0 Phase Transformation Study on Copper-Tin Alloys

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

Electrolytic copper-tin alloys of compositions ranging from 12 to 100 wt. per cent Cu were deposited at constant cathode potential. Alloys in certain ranges of compositions were not found to possess structures identical to their thermal counterparts.

60.0 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 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 was, therefore, undertaken to develop suitable manufacturing techniques for different types of bimetals.

Extensive studies were completed on the physical and mechanical properties of various ferrous and non-ferrous alloys to assess their suitability for use in thermo-bimetals. Fe-Ni alloys containing 36-46 per cent nickel are the most general choice for low expansion component. Austenitic Fe-Ni-Mn, Fe-Ni-Cr-Mn alloys containing nickel in the range 19 per cent to 22 per cent, alpha-brass, silicon-bronze, Al-bronze, and Mn-Cu-Ni alloys were selected for high expansion component. Work was also carried out to establish the most suitable composition of these alloys. Details of the technology of production of all-ferrous thermo-bimetals (i.e. thermo-bimetals containing ferrous alloys only as high and low expansion component) were worked out in details.

These types of bimetals are most commonly used. Main stages involved in the production of such thermo-bimetals are: (i) melting and casting, (ii) hot working of the ingots to slabs, (iii) surface preparation, (iv) bonding by hot pressing and hot rolling, (v) hot rolling of the bonded composites to strips, (vi) pickling and cleaning, (vii) cold finishing, (viii) heat-treatment, (ix) testing. Problems encountered in each stage of production were successfully tackled and a preliminary project report was prepared. The essential process details were communicated to the industrial firm to whom the process was leased out for exploitation. A flowsheet of of the process is given below:

Flowsheet of the Production Technology of All-ferrous Thermo-bimetals

MELTING THE CHARGE IN THE H.F. FURNACE AND CASTING OF INGOT → FORGING OF INGOTS TO SLABS → SURFACE PREPARATION OF THE SLABS → PREPARATION OF THE COMPOSITE BY WELDING THE PERIPHERY →

→ BONDING BY PRESSING AT HIGH TEMPERATURE FOLLOWED BY HOT ROLLING OF THE COMPOSITE TO STRIP → PICKLING AND CLEANING → COLD ROLLING WITH INTER-STAGE ANNEALING → STRIPPING AND FINAL HEAT TREATMENT → TESTING

Details of the technology of production of thermo-bimetal with brass and silicon-bronze were worked out successfully. Special bonding technique in this case was to be adopted in which brass (or silicon-bronze) was cast on a slab of invar. The composite assembly was then rolled to final thickness. Bonding of Al-bronze was quite successful, and further work is necessary. Further work is in progress to perfect the technology of production of thermo-bimetals with Mn-Cu-Ni alloys. Properties of the thermo-bimetals that have been developed so far are given in Table 31.

Future work on this project is related to perfecting the production technology of thermo-bimetals containing Al-bronze and Mn-Cu-Ni alloys as high expansion component, and development of other special type thermo-bimetals. Work is also in progress on the development of composite strips and wire for use as substitute for spring and conductor in electrical industry.

Table 31 — Properties of Thermo-bimetals Developed at N.M.L.

Type	Flexivity (30-80°C.) ×10 per °C.	Sp. resistance micro-ohm-cm.	Young's modulus 10^6 p.s.i.	Range of max. sensitivity °C.	Max. temp. of use °C.
45	28·0	75	23	20-150	300
55	27·0	77	23	20-150	300
65	25·0	74	23	20-150	300
B5	28·0	14	17·5	20-150	175
41	26·0	70	24	30-250	350
96	18·5	60	24	100-300	400
98	14·0	50	24	100-350	500

61.0 Development of Clad Metals

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

The process of cladding aluminium on mild steel and the determination of optimum condition to achieve good bond strength were determined. After standardizing the technique of bonding aluminium with mild steel, the obvious choice was to utilize the same technique for cladding aluminium-base bearing alloy with mild steel so as to work a bimetallic bearing. A suitable aluminium-base bearing alloy does not appear to have been developed which may be used for bonding, and as such work was carried out in the first instance to develop a suitable aluminium-base bearing alloy possessing good frictional properties which can be cladded to steel.

Series of heats were made using aluminium as the base metal and lead, antimony, copper and zinc as the alloying elements. After casting, the necessary mechanical and metallurgical properties of the heats were studied. The bigger heats were made of the compositions which showed good mechanical and metallurgical properties to obtain good dispersion of the alloying elements and reproducible properties.

62.0 Development of Controlled Friction Materials

This investigation was taken up at the instance of Ministry of Railways to develop antifriction material of suitable composition which will give a coefficient of friction ranging between 0·12 and 0·16 in long service under repeated sliding conditions.

Reproducible results were obtained earlier using P.T.F.E. impregnated bakelite compacts of various compositions starting from 20 per cent P.T.F.E. to 75 per cent P.T.F.E. In some experiments, the surface layers of the compacts were made with a given composition, the remaining portion being of bakelite. Some quantity of asbestos fibre was also added in some cases to toughen the compacts. The use of graphite in various proportions with bakelite powder did not prove satisfactory. It was found from the results that the compact with 20 per cent P.T.F.E. satisfied the required condition reasonably and may serve the purpose.

For testing under actual service condition, some bakelite P.T.F.E. compacts (20 per cent P.T.F.E.) of 6 in. sq. and ½ in. thick were prepared at 180-200°C. under a pressure of 820 lb./sq. in. For this purpose, a suitable hydraulic press was procured and a suitable electric furnace fabricated. Suitable moulding chamber was also fabricated. The set-up is shown in Fig. 48. Compression test of the above composite material was done and the result is given below:

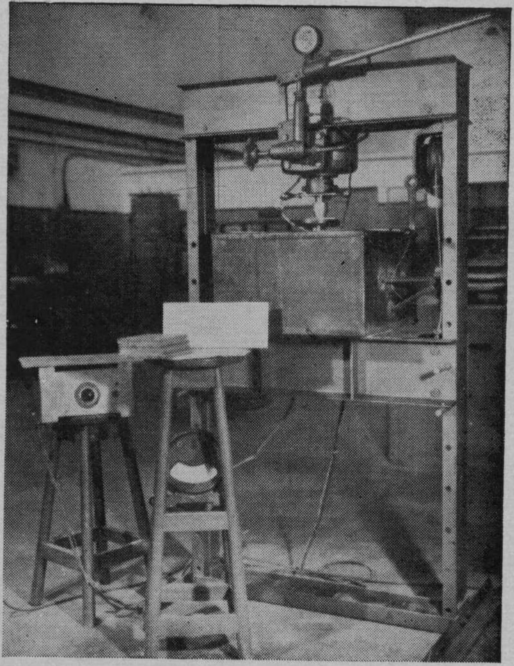


Fig. 48 — A set-up for the production of antifriction liner

Specimen	*Crushing strength tons/sq. in.*
Bakelite P.T.F.E. compacts (20 per cent P.T.F.E.)	3·6

Wear tests of both bakelite P.T.F.E. compact and fabric liner against grey cast iron were carried out with a view to compare the frictional results which are given in Table 32.

Table 32 — Comparative Wear Test of the Specimens

Specimen	Mating material	Number of hr. of service	Coefficient of friction
Bakelite P.T.F.E. compacts (20% P.T.F.E.)	Grey cast iron	10	0·14-0·16
Fabric liner	do	7	0·26-0·35

A number of slabs of the above compacts were made for sending to the Railway Research Centre, Lucknow, for conducting actual service tests. Work is in progress for preparing more antifriction compacts of the same composition with impregnation of small quantity of asbestos for improving the toughness.

63.0 Production of Phosphor-bronze

At the instance of Railway Design and Standards Organization, investigation on the production of phosphor-bronze for springs was undertaken; studies were made to obtain the specified properties for the springs.

The work involved the development of suitable melting and casting technique of phosphor-bronze ingots, hot and cold working technique to produce strips and wire samples and studies on effect of cold working and subsequent thermal treatment on elastic properties so as to impart required spring-temper to the spring alloys.

Raw materials such as suitable grades of copper, tin and phosphor-copper containing 13 per cent phosphorus were used. The melting was carried out in a gas-fired frit furnace. Atmosphere control and other precautions were taken to avoid inverse segregation and other defects. Oxidizing flux of a suitable mixture of CuO, $Na_2B_4O_7$ and clean dry sand was used. The melt was poured in a predressed permanent steel mould within a definite pouring temperature range.

The ingots so prepared were free from inverse segregation and blow holes, etc., in the macro-examination (Fig. 49). The chemical compositions of some of the heats made are given in Table 33.

Some of the ingots were hot worked at a predetermined optimum temperature. The cold workability of phosphor-bronze ingots was very satisfactory. A homogenizing

Table 33 — Chemical Composition of Heats

Sl. No.	Chemical composition %		Cu	Charge in gm.
	Sn	P		
1	6·57	0·29	Balance	CuO—3000 Sn—225 Phos-Cu—93
2	6·92	0·18	do	do
3	6·74	0·26	do	do
4	7·16	0·21	do	do

Table 34 — Results of Tests Conducted on the Different Heats

Heat No.	Casting condition	V.P.N.		Observation	Reduction per cent
		As cast	As homogenized		
19	Cast using recommended flux	138	116	No side crack was observed	10
		148	144		20
		161	174		30
		183	192		40
		210	226		50
		220	244		60
20	Cast without using recommended flux	133	158	do	10
		150	166		20
		176	180		30
		187	199		40
		209	213		50
		223	235		60

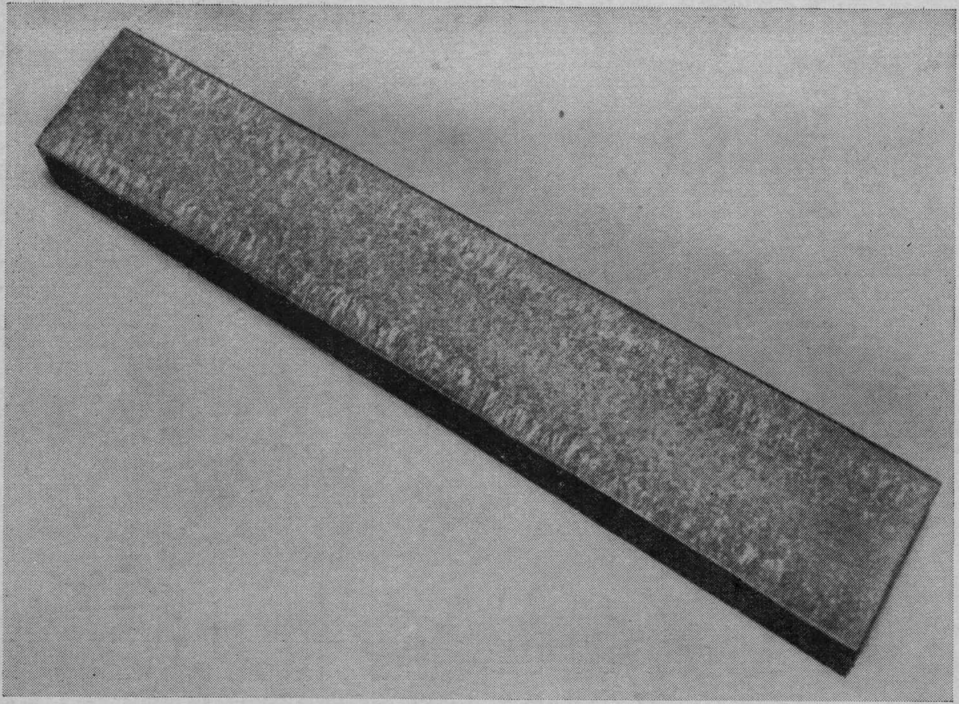


FIG. 49 — MACRO-ETCHED LONGITUDINAL SECTION OF PHOSPHOR-BRONZE CAST INGOT

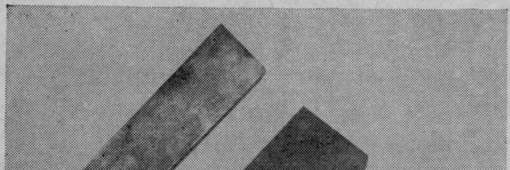


FIG. 50 — SOME OF THE FINAL PRODUCTS OF PHOSPHOR-BRONZE INGOTS AFTER COLD WORKING

treatment for a good length of time at a suitable temperature prior to mechanical working was found to improve the cold workability. Homogenized as well as cast slabs were reduced to strip and wire by cold working (over 60 per cent reduction) and intermediate annealing. Some intermediate products and the final products of strip and wire are shown in Fig. 50. Strips and wires produced from homogenized as well as cast ingots were subjected to different mechanical tests. Some of the results are shown in Table 34 and in Fig. 51. Table 34 indicates the increase of hardness due to different percentages of cold reduction and Fig. 52 shows the change in 0·2 per cent proof-stress, ultimate tensile strength and in elongation per cent due to cold reduction.

After cold working, the phosphor bronze pieces were given specific temper-annealing treatment to obtain the required properties of spring. The work has been completed and a full report is now under preparation.

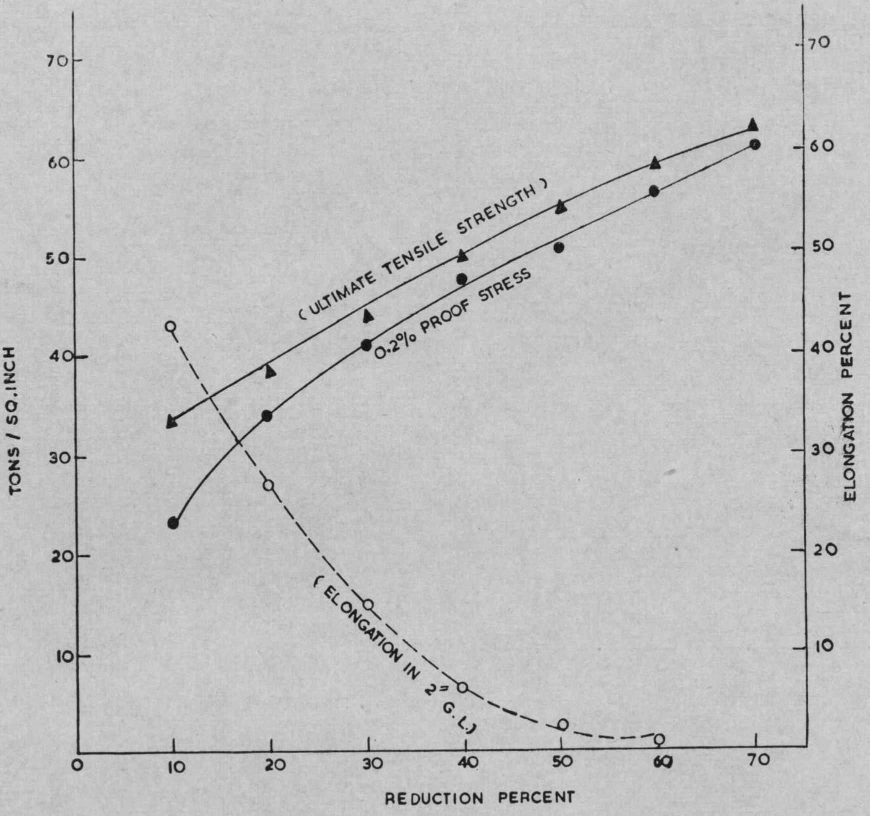


Fig. 51 — Variation of mechanical properties with percentage reduction of phosphor-bronze

64.0 Studies on Deep Drawing of Metals

The majority of commercial pressing operations represent combination of deep drawing, bending and stretch forming. To evaluate a sheet material in terms of these properties, it cannot be expected that any single laboratory test would stimulate all these properties. However, there are specific tests that attempt to produce useful quantitative data upon which sheet materials may be assessed for press work. Of these tests Olsen test is a stretch forming operation and Swift's cupping test is a deep drawing operation. Friction plays an important part in all the above methods of sheet metal forming. Work was, therefore, undertaken to study the effects of variation of surface roughness of the sheet in contact with the punch in all the different types of metal forming processes.

Commercial grades of aluminium, copper and brass were used in the tests. Blanks of two different thicknesses were prepared by rolling from the same stock material in all cases. In order to standardize the surface conditions, background surface finish of (oo) grade emery paper was given for all the blanks. Over this finish, specific roughness of different grades was imparted only on the central portion of the blanks, where they come in contact with the punch. All the deep drawing and hemispherical bottom forming tests were carried out on the Swift's cupping press, using flat bottom and hemispherical punch respectively. Lubricant (paste of graphite with soluble oil) was applied only on the annular portion of the blank under blank holder to

Table 35 — Results of Deep Drawing and Olsen Tests

Material	Blank thickness in.	Surface grade of the blanks	Olsen cup depth in.	L.D.R. values	
				Flat punch	Hem. punch
Aluminium	0·038	Grade 0	0·367	2·13	
		Grade I	0·364	2·15	
		Grade II	0·354	2·21	
	0·053	Grade 0	0·378	2·18	
		Grade I	0·373	2·18	
		Grade II	0·367	2·25	
Copper	0·030	Grade O	0·398	2·20	
		Grade I	0·387	2·25	
		Grade II	0·374	2·30	
	0·042	Grade 0	0·414	2·26	
		Grade I	0·407	2·26	
		Grade II	0·401	2·33	
Brass	0·030	Grade 0	0·466	2·25	2·25
		Grade I	0·450	2·25	2·27
		Grade II	0·424	2·27	2·32
	0·042	Grade 0	0·483	2·25	2·30
		Grade I	0·476	2·25	2·35
		Grade II	0·463	2·25	2·40

facilitate the drawing. The blank holding load was maintained at a value which just prevented flange wrinkling. In Olsen test, contrary to the normal testing practice, unlubricated blanks were used. Throughout the tests with Swift's cupping press drawing speed was kept constant at 4 ft./min.

The results of pure deep drawing tests and Olsen tests for aluminium, copper and brass and for combined forming test carried out on brass are given in Table 35.

The limiting drawing ratio recorded showed that in all cases of pure deep drawing, increase in surface roughness improved the limiting drawing ratio. The improvement was quite marked for aluminium and copper. In the case of combined forming test, the increase in surface roughness resulted in greater limiting drawing ratio values. In the case of pure biaxial stretch forming, the effects of surface roughness were just the reverse. Further work is in progress to determine the surface finish effects at several higher drawing speeds and simultaneously to find out the role of frictional forces while forming sheet metals at higher speeds.

65.0 Photoelastic Stress Analysis

Work on photoelastic stress analysis covered the study of distribution of stresses in engineering components as such a study is necessary from the point of view of understanding the behaviour of the components as well as for improving the design. Photoelastic evaluation of stress concentration factor due to multiple notches was undertaken to obtain quantitative measure of the stress-relieving effect of neighbouring notches.

The beam used was stressed in pure bending by applying the load by means of four pins. The distance between the two nearest pins was 6 in. A central notch was cut in a beam loaded in pure bending and stress concentration factor due to the notch was evaluated. Two symmetrical notches were later cut at a fixed distance on either side of the central one, and their effect on the stress concentration factor of the central one was evaluated. Several readings were taken by changing the depth and proximity of the additional notches. The results obtained are shown in the graph in Fig. 52. The graph shows the relative variation in stresses at the root of the central notch (as represented by percentage decrease in stress concentration factor) with variation of the depth of the stress-relieving notches. Three series of readings were taken with different distances between the roots of the notches. For curves I, II and III the horizontal distance between the roots of the notches was $\frac{3}{4}$, $\frac{3}{8}$ and $\frac{1}{2}$ in. respectively. It appeared that the effect of the notch distance was most prominent when the distance between the notches was $\frac{3}{8}$ in.

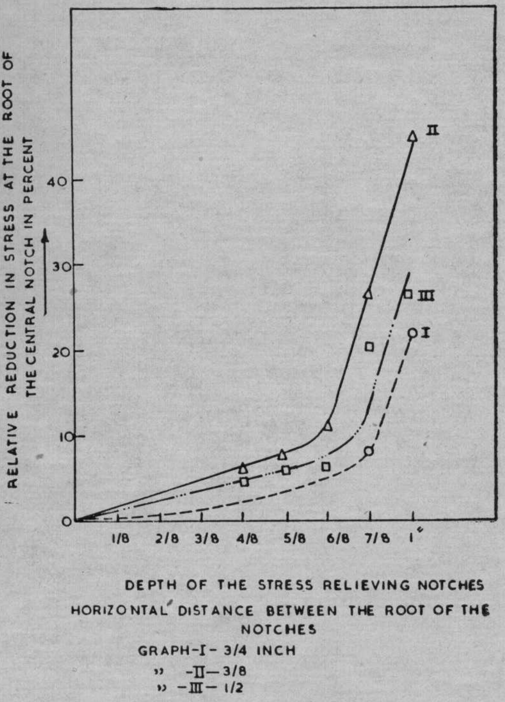


FIG. 52 — DECREASE OF STRESS CONCENTRATION WITH DEPTH OF THE NEIGHBOURING NOTCHES

66.0 Preparation of Thin Metal Foils For Transmission Electron Microscopy by Electrolytic Method

Thin foil technique for transmission electron microscopic examination was taken up with a view to know the inner details of structure and to correlate it with the mechanical properties of substitute and other alloy steels being developed at the National Metallurgical Laboratory. Thin foils show directly and faithfully into the interior of metals and alloys and suffer no loss in resolution which is encountered with replicas. The best resolution of carbon replica is of the order of 20 A.U. and beyond that it does not represent the structure faithfully and thus entails some uncertainty, whilst thin foils are completely free from all such troubles.

In order to prepare thin metal foils for direct transmission electron microscopic examination, the most widely applicable electropolishing method was followed. Electropolishing was carried out with chemical baths of different compositions. The polishing was done at a voltage on the plateau of the current-voltage curve corresponding to the specimen and composition of the bath under test. A non-conducting lacquer, ' Lacomit ', was used to coat the edges of the specimen to prevent the preferential attack of the specimen by the edges. Flat as well as pointed electrodes were used for polishing; it was noted that the Bollmann method of using pointed electrodes was better and satisfactory since it produced perforations quickly by the window technique on the specimen due to concentration of currents at points. After the perforations had progressed to a certain extent and the sides around the perforations appeared to have been sufficiently thin, the specimens were washed well immediately with alcohol. A sharp scalpel was used to cut out thin specimens from portions near the edges placed over a plastic sheet by pressing. The specimen thus prepared was ready for transmission microscopic examination. It was an additional advantage with this thin foil specimen that these thin specimens were also used for electron diffraction studies for the identification of the different phases thought to exist in the specimen. The results so far obtained are most interesting. Further work is in progress to improve the technique and to overcome some technical difficulties encountered.

67.0 Preparation of Carbon Films for Transmission Electron Microscopy

The preparation of carbon films for transmission electron microscopy was carried out with a view to study, in general, the surface structure of the different metals and alloys, and in particular the structure of substitute and other alloy steels in the course of their development in the National Metallurgical Laboratory.

The carbon films are superior to other replicas due to their greater strength and better representation of the structure of the specimens than the other plastic replicas. Moreover, much additional information is obtained about the chemical nature of the constituents by the selected area electron diffraction of the small particles extracted from the surface of the specimen in course of preparing the replica by the carbon extraction replica technique.

To make the carbon replica for transmission electron microscopic examination, the surface of the specimen was prepared by electropolishing and etched subsequently with a suitable etchant. The process of electropolishing and etching were tried out for different types of steels with different electrolytes under various polishing conditions. Amongst the different compositions of electrolytes, current density, cell voltage and temperature of the electrolytic baths, it was noted that for nickel-free austenitic stainless steels the electrolyte having a composition of 25 gm. chromium trioxide, 133 cc. glacial acetic acid, and 7 cc. distilled water gave good results. The temperature of the bath was 20°C., the voltage applied was 20 volts and the time required for polishing was ranged from 5 to 10 min. depending upon the size and finishing of the specimen surface.

The specimen after electropolishing and etching was placed inside the evaporating unit fitted with two carbon rods. One of these rods was blunt whilst the other one was very sharp and they touched each other under the tension of a spring. A clean white porcelain, with a drop of vacuum oil on it, was placed near the specimen inside the evaporating unit in order to judge by contrast the thickness of the carbon film formed over the surface of the specimen. A quick flash at a pressure below 10^{-4} mm. of mercury was given with the evaporator switch and the thickness of the carbon film was noted around the sides of the drop of oil over the porcelain piece. The carbon film thus formed over the specimen surface faithfully followed the contours of the etched sample and the contrast in the image over the screen was due to the variation of the transmitted electrons through the film. The specimens during carbon evaporation in the unit were placed at a suitable angle to the evaporating carbon source in order to improve the contrast by self-shadowing. For still better contrast, attempts were made to evaporate the carbon in the usual manner along with a piece of platinum wire embedded into one of the carbon electrodes.

After the formation of the carbon film over the specimen, the films were stripped off by the method of extraction with some chemical reagents. The films were better extracted with the help of a solution of bromine and absolute alcohol. Concentrations of bromine like 20, 15, 10, 5, 2 and 1 per cent in absolute alcohol were tried. It was observed that 1 per cent bromine solution gave good results for extracting the replica. The replicas were then washed well with alcohol and put to electron microscope for direct transmission as well as for diffraction study of the sample. Results so far obtained were satisfactory and further work is in progress.

68.0 Production of Basic Refractories from Indigenous Resources

(i) *Almorah Magnesite*

This investigation was taken up with a view to evaluate the physical and refractory properties of Almorah magnesite and to assess its suitability for the manufacture of refractories.

Investigation on a 10-ton sample of Almorah magnesite from Agar Girichchina sector was completed. It was found that the sample, as received, showed low refractoriness underload, which was attributed to the presence of lime up to 5 per cent in the calcined product, resulting in the formation of monticellite and merwinite in considerable amounts. By a process developed in this laboratory, it was possible to lower the lime content to below 2 per cent resulting in the overall improvement of refractory properties. When lime additions were made to this magnesite such that the lime and silica combined as tricalcium and/or dicalcium silicates, a highly refractory product was obtained (Table 36). The performance of bricks made from this product has to be assessed by actual use in steel plants. Laboratory scale tests indicated, however, that it possessed superior refractory properties over the conventional magnesite brick. Further work on evaluation of magnesite from Dewaldhar sector was taken up on 5 cwt. samples received from the U.P. State Industrial Corporation.

(ii) *Rajasthan Magnesite*

A sample of magnesite from Rajasthan was obtained from Messrs Ishwar Industries. Microscopical examination revealed the presence of large amounts of quartz. The chemical analysis showed 7·4 per cent silica. The silica was not uniformly distributed as indicated by visual appearance as well as calcination tests. After calcination at 1650°C. the high silica portions yielded a light porous sinter while some pieces having low silica yielded a dense sinter (Fig. 53). Few pieces showed fusion to varying degrees due to the presence of impurities. The refractory properties of the brick made from this magnesite are given in Table 37. Even though the refractoriness was high, the high silica content was not acceptable as per I.S.I.

Table 36 — Refractory Properties of Almorah Magnesite

	As received	After processing	With the lime addition		
			I	II	III
Porosity	21·8	14·38	12·7	15·8	16·00
Bulk density, gm./cc.	2·73	2·99	2·99	2·94	2·86
Specific gravity	3·60	3·55	—	—	—
Reheat shrinkage at 1650°C. for 3 hr., %	1·85	0·64	0·69	0·92	1·38
Spalling resistance (No. of cycles)	9-16	23-24	22-30	23	30
Cold crushing strength, kg./cm.^{2}	444	506	—	—	—
Refractoriness underload:					
ta	1500°C.	1550-1570°C.	1830°C.	1760°C.	1780°C.
te	1540°C.	1600°C.	—	1800°C.	1830°C.

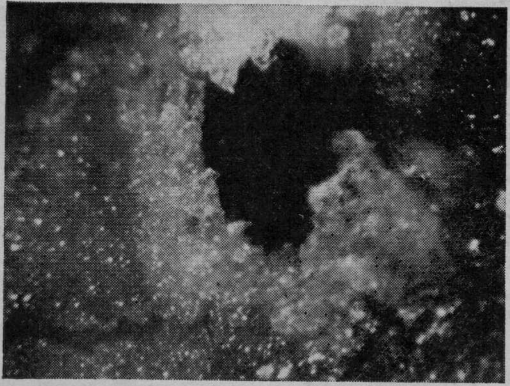


FIG. 53 — RAJASTHAN MAGNESITE FIRED TO 1600°C., SHOWING THE CONCENTRATION OF SILICON MATRIX MINERALS AROUND A PORE (×25 APPROX.)

Table 37 — Physical Properties of Fired Brick from Rajasthan Magnesite

	Without addition	With 2% iron oxide addition
Bulk density, gm./cc.	2·53	2·5
Porosity, %	24·72	27
Refractoriness underload	ta 1650°C. te 1760°C.	ta 1720°C. te 1800°C.
Reheat shrinkage at 1650°C. for 3 hr.	0·1%	0·7%

specification. Therefore, the possibility of reducing the silica content to 2 per cent by mineral beneficiation methods was examined. However, the raw material as received can also be utilized in the manufacture of forsterite refractories.

(iii) *Correlation between the Constitution and Physical Properties of Magnesite Refractories*

This investigation was started with a view to determine the effects of variations of chemical compositions, especially lime and silica, on the constitution and physical properties of magnesite. The results obtained would prove helpful in forming standard specifications for basic refractories.

During the period under review, the influence of chemical composition on the physical properties of magnesite brick having molecular lime-silica ratio up to 2·24 was studied. Twenty-four batch compositions were made at 5, 7, 9 and 10 per cent silica levels and lime was added progressively to those base compositions such that molecular lime-slilica ratio was raised from 1 to 2·24. Some of the physical properties such as apparent porosity, apparent specific gravity, apparent bulk density, true specific gravity, true bulk density, crushing strength, refractoriness underload, spalling resistance and linear shrinkage of these batch compositions were determined. Table 38 shows the physical properties of some of the batch compositions. Further studies on mineralogical composition by X-ray and microscopical methods are under progress. The effect of slag attack was also to be studied.

69.0 Development of High-alumina Refractories

High-alumina refractories are required to meet a wide variety of service requirements in metallurgical furnaces and cement kilns. The presence of high titania in many bauxite deposits made them unsuitable for manufacture of refractory bricks containing above 60 per cent alumina. Investigation work was, therefore, specifically undertaken to determine the suitability of Indian bauxites containing varying amounts of impurities, specially titania and iron oxide, for the manufacture of high-alumina refractory having 60 per cent Al_2O_3 and above and to develop suitable compositions therefrom.

(i) *Studies on Bauxite and High-alumina Clays from Kashmir*

Using a sample of diasporic bauxite, high-alumina compositions having about 70-72

Table 38 — Physical Properties of Magnesite with Silica and Lime Addition

SiO_2 level %	CaO/SiO_2 ratio	Apparent porosity	Apparent specific gravity	Apparent bulk density	True bulk density	True specific gravity	Crushing strength lb./sq. in.	Refractoriness underload		Spalling index
								ta	te	
3·1-3·41	0·38	19·11	3·56	2·94	2·89	3·623	284	1700	1780	N.D.
	0·76	14·23	3·50	3·00	3·03	3·623	484	1600	1680	12
	1·15	11·43	3·50	3·10	3·04	3·623	655	1600	1750	16
	1·53	14·16	3·51	3·01	3·01	3·604	766	1700	1800	10
	1·91	15·25	3·48	2·96	2·92	3·597	501	1680-1700	1800	30
	2·24	22·50	3·51	2·72	2·70	3·562	439	1750	1800	17
4·33-4·59	0·25	17·72	3·54	2·91	2·94	3·651	311	1680	1750	11
	0·80	15·88	3·46	2·91	2·98	3·572	546	1520	1560	19
	1·19	15·28	3·46	2·94	2·94	3·617	571	1600	1640	18
	1·69	15·76	3·47	2·92	2·94	3·659	560	1690	1730	26
	2·18	20·36	3·50	2·79	2·79	3·597	513	1720	1850	26
6·22-6·731	0·18	18·92	3·54	2·87	2·87	3·597	500	1700	1780	7
	0·80	12·81	3·39	2·96	2·97	3·657	594	1500	1560	8
	1·18	11·69	3·39	3·05	3·04	3·634	1170	1570	1600	7
	1·67	12·09	3·40	2·99	2·95	3·579	842	1640	1780	15
	2·15	18·91	3·46	2·81	2·81	N.D.	520	1780	1800	N.D.
7·88-8·85	0·15	19·98	3·54	2·84	2·84	3·572	515	1700	1810	6
	0·80	11·18	3·33	2·96	2·97	3·619	1196·5	1560	1610	19
	1·20	11·60	3·34	2·95	2·94	3·579	1090	1530	1580	18
	1·69	10·28	3·35	3·00	3·08	3·568	937	1640	1750	22
	2·18	22·67	3·49	2·70	2·68	3·584	450	1800	1800	N.D.
9·51	0·12	19·42	3·51	2·83	2·79	3·597	408	1720	1810	10

N.D.= Not determined.

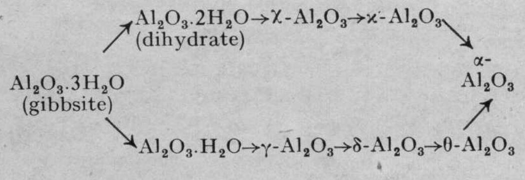
per cent Al_2O_3 were prepared with the addition of clay as bond in some cases and with chemical bonding in the others. Chemically bonded brick had a porosity of 23·5 per cent, bulk density 2·5 per cent, refractoriness underload: 'ta' 1530°C. and 'te' 1670°C.

In addition to the above, thirteen samples of bauxite, bauxitic and high-alumina clays were received from the State of Jammu and Kashmir for detailed study. These samples were collected from different pits in the Salal area of Jammu province and were obtained through the Director of Geology and Mining, Jammu and Kashmir Government. Out of the five samples of dispore, only one sample seemed to be of refractory grade and the others were high in iron oxide and also in silica. The bauxite sample comparatively low in iron oxide is under investigation for the manufacture of high-alumina refractories.

Of the remaining eight samples, four clays are bauxitic in nature and the other four are usual kaolinite type. Chemical analyses of these clays show a wide variation in their Al_2O_3 (38-59 per cent) and SiO_2 (21-45 per cent) contents as also in their Fe_2O_3 contents (0·88-2·40 per cent) and offer wide scope for the manufacture of some varieties of high-alumina refractories. A detailed study on the mineralogy of these clays by X-ray, D.T.A., T.G.A and electron microscopy as well as on the various physical and refractory characteristics is in progress.

(ii) *Development of Foamed Alumina Brick*

(a) *Dehydroxylation and phase transformation of Bayer's alumina* — A research report was prepared on the dehydroxylation characteristics and mode of phase transformations of Bayer's alumina. With the aid of X-ray powder pattern, differential thermograms and thermogravimetric studies, it is postulated that gibbsite (Bayer's alumina) on thermal treatment breaks down to monohydrate (boehemite) and dihydrate simultaneously at about 200°C. The monohydrate breaks down to ϒ-alumina at about 500°C. and dihydrate, just after its formation, again breaks into χ-alumina, which also is a dehydrated form of alumina. These ϒ alumina and χ-alumina ultimately transform to α-alumina through different stages, viz. δ-alumina, θ-alumina and κ-alumina. The mechanism may be summarized as follows:



$$\begin{tikzcd}[column sep=small] & Al_2O_3.2H_2O\ (\text{dihydrate}) \arrow[r] & \chi\text{-}Al_2O_3 \arrow[r] & \kappa\text{-}Al_2O_3 \arrow[dr] & \\ Al_2O_3.3H_2O\ (\text{gibbsite}) \arrow[ur] \arrow[dr] & & & & \alpha\text{-}Al_2O_3 \\ & Al_2O_3.H_2O \arrow[r] & \gamma\text{-}Al_2O_3 \arrow[r] & \delta\text{-}Al_2O_3 \arrow[r] & \theta\text{-}Al_2O_3 \arrow[u] \end{tikzcd}$$

(b) *Study of optimum conditions for obtaining high-alumina light-weight bricks* — In continuation of the earlier work on the development of foam alumina brick, after making a suitable sintered alumina and standardizing the foaming procedure, bricks were cast. The cast bricks after drying and firing to 1550°C. showed considerable amount of cracks. Casting in plaster moulds as well as wooden moulds was tried. But as invariably cracks were developed in these bricks when they were fired, other techniques are being tried to get rid of cracks.

70.0 Development of Calcium Aluminate Cement

High-alumina cements offer wide application in refractory castables and concretes that can withstand up to 1300°C. and above. These cements that are hydraulic setting are composed essentially of calcium aluminates and varying percentages of other compounds. These are now used in large quantities in India for various applications in the iron and steel industry as also for use in some high-temperature furnaces and the country's requirements of these cements are met wholly by import. It is expected that demand will increase in future owing to the growing trend towards the use of monolithic linings and refractory concretes, particularly in locations where special and complicated shapes made by conventional refractory-making practice, involving the employment of skilled labour and high cost, are used. In view thereof and of the availability of raw materials for their manufacture in our country, a two-pronged study was taken up on the development of high-alumina cements, namely (i) studies on pure calcium aluminates for high-temperature applications and (ii) development of molten aluminous cements of the 'cement fondu' type from indigenous raw materials.

(i) *Pure Calcium Aluminate Cements*

The effect of firing temperatures on the modulus of rupture of calcium monoaluminate cement with different aggregates was studied. The modulus of rupture of the specimens fired to 400°C. was considerably decreased. Refractory concretes with calcium monoaluminate cement and different aggregates, such as sillimanite, kyanite and calcined bauxite, were also developed. Different properties of calcium dialuminate cement with graded sillimanite as aggregates were studied. The cold crushing strength and modulus of rupture were increased by the incorporation of CA cement along with CA_2. Table 39 shows some of the properties.

(ii) *Ferruginous Calcium Aluminate Cement of 'Cement Fondu' Type*

Work was carried out on this project utilizing the bauxites from Kashmir, Lohardaga and Katni and limestones from Madras and Bisra. During the period under review work was extended using some more bauxites from Shevaroy and Kharagpur

Table 39 — Physical Properties of Calcium Dialuminate Cement

Sample No.	Composition	Cold crushing strength lb./sq. in.	M.O.R. at 800°C. lb./sq. in.	Refractoriness underload	
				ta °C.	te °C.
A	A.S. 15% CA_2	1546	338	1440	1560
B	A.S. 20% CA_2	4919	345	1420	1570
C	A.S. 25% CA_2	2150	684	—	—
D	A.S. 10% CA_2 +5% CA	4099	608	—	—
E	A.S. 14% CA_2 +6% CA	6272	675	—	—
F	A.S. 17% CA_2 +8% CA	8366	825	—	—

A.S. = Assam sillimanite.

hills as a result of which a number of good cement compositions were developed with properties equal to or even better than some of the currently imported high-alumina cements. During the course of this investigation, following observations were made on the effect of some of the minor constituents, namely SiO_2, Fe_2O_3 and MgO, on the optimum Al_2O_3/SiO_2 ratio of the molten high-alumina cements and their properties.

(a) The properties of high-alumina cements and the optimum Al_2O_3/CaO ratio are influenced to a great extent by the presence of other constituents like SiO_2, Fe_2O_3 and MgO. Good cements are obtained when their Al_2O_3/CaO ratios are between 0·9 and 1·4; it may, however, be possible to get good cements beyond a ratio of 1·4 if silica content is low.

(b) The cementing properties do not appear to be adversely affected up to 9 per cent SiO_2 in the cement, when the Al_2O_3/CaO ratio is preferably above 1·1.

(c) The presence of 7-10 per cent Fe_2O_3 in the cement assists in easy melting of the cements, whereas in higher amounts it is difficult to control the consistency of the products due to difficulties arising out of reduction of Fe_2O_3.

(d) Magnesia in the cement beyond about 2·5 per cent is harmful to the strength of the cement.

Further work is in progress on the influence of fineness of the cement on its hydraulic properties as well as on the effect of heat-treatment on the strength of cements at different temperatures. Possibilities of developing these cements by the sintering process are under examination.

70.1 Phase Relationship on the System Gehlenite, Calcium Dialuminate, Monocalcium, Monoaluminate and Spinel

While calcium aluminate cements were composed of monocalcium monoaluminate (CA) and calcium dialuminate (CA_2), in the course of investigation, it was found that the presence of impurities of silica and magnesia lead to the formation of gehlenite and spinel (MA). Even though binary relationships between CA-CA_2 are known, the quarternary relationships involving all the four phases are not fully known. Therefore, a knowledge of this quaternary system is important in studying the refractoriness and effect of siliceous and magnesium impurities in calcium aluminate cements.

The system CA-CA_2-gehlenite-MA is assumed to be a quaternary within the quaternary system CAO-MgO-Al_2O_3-SiO_2 which is perhaps one of the most well-known four-component systems. The bounding planes of the composition tetrahedron are (i) gehlenite-CA-CA_2, (ii) gehlenite-CA-MA and (iii) gehlenite-CA_2-MA, of which only the first is known. Therefore, an investigation on the liquidus relationships in the quaternary system was undertaken by quench method as well as hot stage microscopy.

The plane at gehlenite-CA_2-MA is composed of three binaries. Two of the binaries, namely gehlenite-spinel and gehlenite-CA_2, were checked and found to differ only slightly from the data of the previous investigators. The binary CA_2-MA, not reported earlier, shows an eutectic at 1700°C. The three component system is a true ternary and shows an eutectic at 1514°C. The plane gehlenite-CA-MA is also a ternary. The binary side CA-MA was investigated and the eutectic was located at 1585°C. The ternary eutectic temperature was found to be 1483°C. Further studies on the system CA-CA_2-MA and determination of the quaternary system are in progress.

71.0 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 applications in metallurgy. Such a study involved in the first instance detailed examination of their pyrolytic characteristics with and without pretreatment as well as additions which will be followed up by their graphitization characteristics. Some of the carbonaceous materials readily available in India such as low-ash coals of Assam, washed coals of the Jharia and Raniganj area and lignite from Neyveli and petroleum coke from Digboi were initially taken up for studies.

During the period under review, a working model of automatic thermal balance was fabricated and installed with a view to facilitate kinetic studies on chemical reactions which involved a gain or loss in weight, which was of particular importance on the study of pyrolytic characteristics of carbonaceous materials. Considerable progress was also made on the study of dense carbon aggregates. A successful method was worked to convert the high volatile carbonaceous material into dense aggregate without any intermediate precalcination. Similar study was done on low-ash Assam coals. The important conclusions obtained from these studies are as follows:

(i) It is possible to obtain dense carbon from carbonaceous material by a simple process of briquetting with a binder and heat-treating the briquettes. The compacts shrink considerably during heat treatment and lose weight due to pyrolysis. Loss in weight is significantly lower than what can be expected from the proximate analyses of the material. Shrinkage does not affect the soundness of the resultant compacts. Petroleum coke yields hard, abrasion-resistant compacts while Assam coals yield comparatively softer ones. The strength and bulk densities of the latter are also low. It is possible however, that with better proportioning of binder and coal powders and by control of the grain size of the latter, these properties may show considerable improvement.

(ii) Grain size of the base material has a very great influence on the physical properties of the final compacts. There appears to be relationship between the granulometric composition of the powder and the maximum bulk density of the compacts that can be made from it. The data also indicate that with a given carbonaceous material and binder, there is a higher limit to the bulk density of the carbon compact when this is achieved by increasing the fineness of the powder.

(iii) Quantity of binder is also a significant variable. Depending on the size of the powder, there is an optimum proportion of the binder that gives the best properties to the compacts.

(iv) Time/temperature schedule of heat-treatment, particularly at low temperature, up to 450°C., is another variable that needs close control. It is suggested that certain changes occurring in the constitution of the binder, and also probably in that of the base material itself, in this temperature range may be influencing the properties of the final compacts.

72.0 Study on Refractory Raw Materials from Kerala

This work under a Research Fellowship scheme of the Council of Scientific & Industrial Research, was completed during the year. The first part of the research report deals with ' Separation of rare earths based on a high temperature technique ' and the second part relates to ' Use of rare earths in the preparation of refractory articles such as sillimanite and zircon bricks and refractory crucibles '.

A new method based on the introduction of mixture of calcium oxide and calcium chloride as a flux to break up rare earths at high temperatures was adopted. Experimental work showed that the break up of rare earths was possible as expected, but the rate of reaction of individual rare earths towards the flux consisting of a mixture of CaO and $CaCl_2$ was unknown. So taking X-ray analysis as the most important criterion, individual rare earths, like CeO_2, La_2O_3, Pr_6O_{11} and Sm_2O_3 were treated with CaO and $CaCl_2$, separately and in combination in varying amounts. Chlorination of the heated mass was also investigated. It did not, however, give any means of separating them, excepting that it was seen that lanthanum hydroxide, after preheating with CaO and $CaCl_2$, on chlorinating could partly distil at 1250°C.

A special type of chlorination apparatus along with a furnace was designed and fabricated at the National Metallurgical Laboratory (Fig. 54). Rare earth, after preheating with the flux was chlorinated at 1300°C. Experiments under different conditions showed that if the rare earth chloride was preheated with the flux at 800°C. for 2 hr. and then chlorinated, it would give a disintegrated mass and on leaching it with acid, 15-20 per cent strong, the residue would be a praseodymium oxide concentrate. From the acid leachings, neodymium oxide could be separated as oxalate at a *p*H of 3. It, however, gave no hint of the possibility of preparing calcium oxide or lanthanum oxide in a pure form.

So, the isolation of cerium oxide was first attempted by oxidation to ceric oxide, which was insoluble in dilute acid. The remaining rare earth oxide, when heated with the flux and chlorinated, showed that cerium oxide which escaped first isolation could be recovered and individual rare earths like Pr_6O_{11} and La_2O_3 could be separated in a more or less pure form as could be seen in the X-ray diffractograph and spectrographic analyses. Neodymium oxide concentrate was found to contain more impurities when compared to the other rare earths. Though indication of the concentration of heavy rare earths like lutecium, ytterbium, erbium, etc., was seen, much importance was not attached thereto, as their quantity was only about 1 per cent of the total rare earths.

Special attention was given to find out a use for rare earth chloride and experiments showed that it could be used as a mineralizer in sintering refractory raw materials

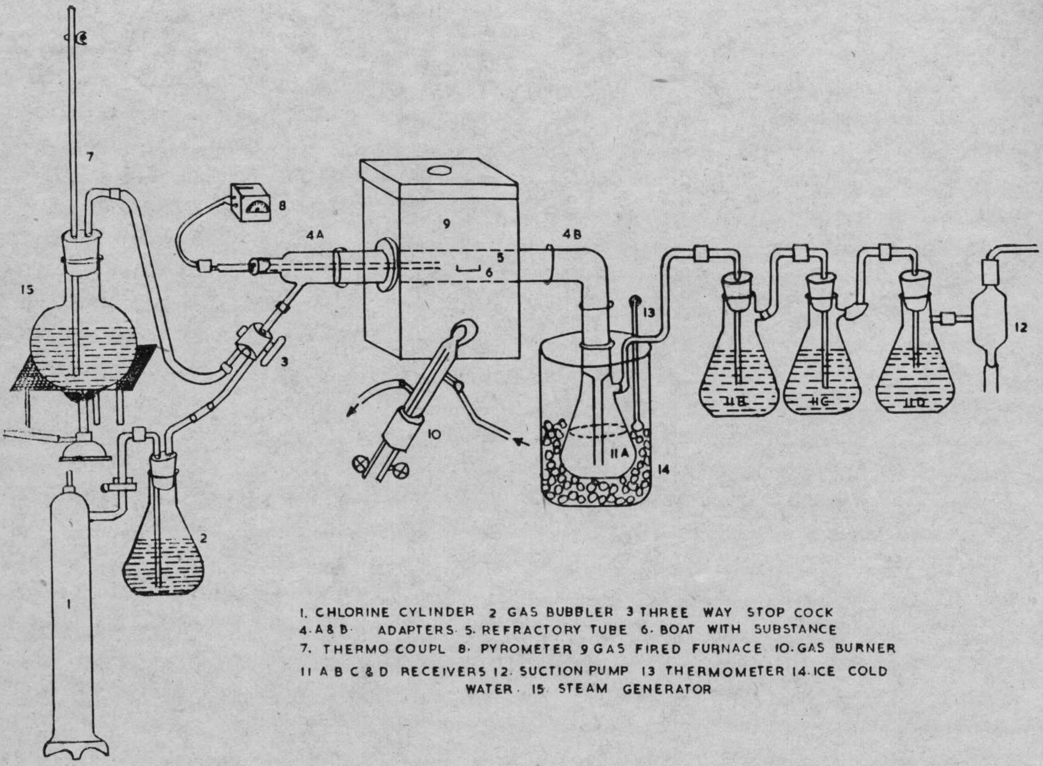


FIG. 54 — CHLORINATION AND STEAMING APPARATUS

like sillimanite and zircon sands. Different grain sizes of sillimanite with ball mill fines and grogs made out of them were mixed in different proportions along with varying quantities of rare earth chloride solution, with and without other binding agents, and pressed into bricks under different pressures and fired at different temperatures. The bricks prepared under optimum conditions possessed a porosity of 20 per cent, bulk density 2·5, crushing strength 11,700 p.s.i., shrinkage nil, and refractoriness under-load: 'ta' 1680°C. and 'te' 1750°C. It also passed 30 cycles of spalling test when heated to 1300°C. and cooled to room temperature. X-ray analyses of the brick showed that mullitization of the brick was complete at 1500°C.

Zircon was also treated in the same way as sillimanite with rare earth chloride as mineralizer and found to get sintered at 1400°C. The bricks prepared under optimum conditions possessed a crushing strength of 13,000 p.s.i., porosity 22 per cent, bulk density 4·2 and refractoriness under load: 'ta' 1580°C., 'te' 1760°C. It also passed a spalling test of 30 cycles when heated to 1300°C. and cooled to room temperature. Crucibles made with these compositions remained unaffected even after 100 cycles of heating at 800°C. for melting aluminium.

73.0 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 which are growing in importance. Of a lesser importance but of great commercial potential are materials such as sillimanite and kyanite. Experiments were, therefore, initiated with sillimanite from the beach sands of Travancore and its grindability under certain predetermined conditions was assessed.

Some preliminary trials of extruding finely ground sillimanite rendered plastic by the use of starch or flour paste as bond were made. Sound specimens could be extruded, provided water content was properly adjusted. Experimental casting slips were also made from sillimanite to study the effect of electrolyte addition on sillimanite water mixes containing large amount of sillimanite. It was found that alkaline sillimanite slips are more stable as compared to acid slips. Optimum quantities of NaOH or Na_2Co_3, Na_2SiO_3, $Na_3P_4O_7$ or phosphate or a corresponding amount of NH_4OH can properly deflocculate the slips. *p*H had to be carefully controlled for satisfactory result. The actual trend of change of viscosity and *p*H with electrolyte addition was studied.

A new series of experiments was started with bladed kyanite which was ground in a rubber-lined pot using 1 in. size steel balls for a period of 72 hr. The ratio of material/balls was varied as 1: 2, 1: 2·5, 1: 3, 1: 3·5 and 1: 4. A grind was also made using fired sillimanite balls. Table 40 gives the particle sizes of kyanite ground in this fashion.

A material/ball ratio of 1: 3·5 seems to give maximum fines in a period of 72 hr. and if ground longer will yield enough of fine material. Further studies with bladed kyanite and Assam sillimanite as the non-plastic materials are in progress.

74.0 Development of Steel-pouring Pit Refractories

Considerable difficulty is experienced in obtaining the pouring pit refractories of satisfactory quality from indigenous sources. As these refractories form a vital link in steel production, the Hindustan Steel plant authorities of Durgapur Steel Project desired that investigation to develop the same from indigenous raw materials should be taken up to meet the pressing problem.

Physical properties of some imported nozzles and inserts were evaluated and compared with the properties of indigenously made nozzles. During the period under review, an erosion test apparatus was designed and is being fabricated. In this

Table 40 — Particle Size of Kyanite

Material/ball	1: 2	1: 2·5	1: 3	1: 3·5	1: 4	1: 2·5 sillimanite balls
Microns	*Cumulative percentages*					
26	68	79	79	82	76	78
8	45	51	53	57	53	53
5·6	35	41	46	49	46	44
3·9	28	22	39	39	38	29
3·17	25	28	34	35	34	34
2·43	21	23	28	29	31	29
1·119	11	13	17	16	16	17
0·779	10	10	13	12	12	14
0·537	7	7·8	9·3	8·4	8·7	10·0

apparatus, it is intended to simulate the failure of the steel-pouring nozzles by erosion with molten steel. As molten steel flows through the nozzle, it reacts with the refractory and by its constant removal along with the metal progressively enlarges the orifice of the nozzle. If the wearing out of the nozzle is uneven and cracks also develop, the teeming of the steel will not be successful.

The erosion test apparatus will compare the extent to which various refractories get eroded by molten steel. The samples are submerged in the molten steel held in the crucible and the latter is rotated. The samples thus get eroded by the molten steel. The crucible with melt can also be made to move up and down past the refractory sample. The test will be performed for a specified time and is expected to give an effect similar to that of teeming certain quantity of steel past a given quantity of the refractory. Erosion may be expressed as reduction in area of cross-section per unit initial cross-section area.

A sketch of the apparatus is given in Fig. 55. 6×1×1 in. bars (1) are hung from the roof tile (2) of a furnace. The molten steel is contained in a graphite crucible which rests on a refractory pedestal. The pedestal is revolved by a reduction gear coupled to a motor. Coke-oven gas is used as fuel and is fed in through two tangential burners.

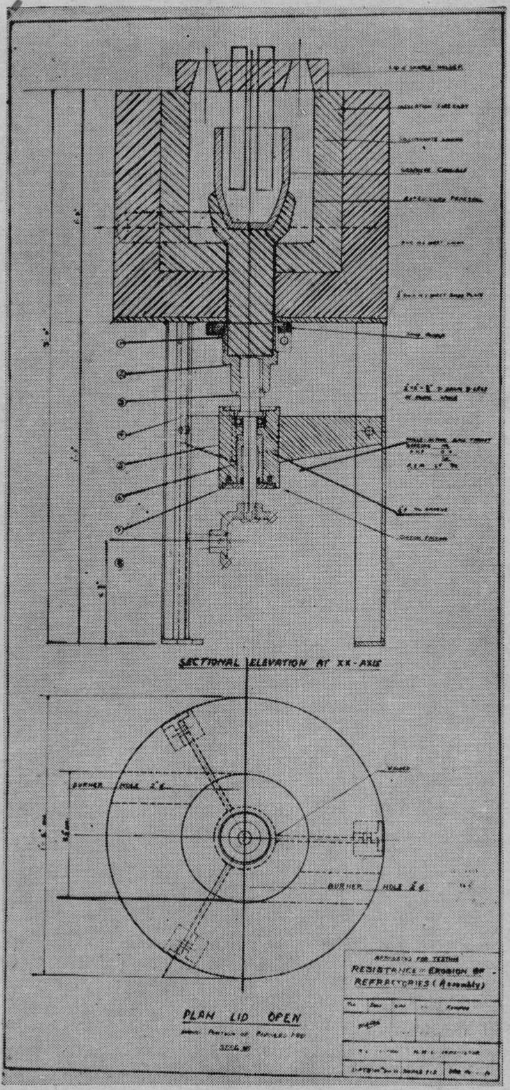


FIG. 55 — LINE DIAGRAM OF FINGER TEST APPARATUS FOR SETTING EROSION OR CORROSION OF REFRACTORIES BY MOLTEN STEEL OR SLAG

75.0 Suitability of Jainti Dolomite for Sintering in Shaft Kiln

Sintering and firing characteristics of Jainti dolomite were taken up to assess its suitability both as semi-stabilized and completely stabilized product for use in steel melting furnaces. The dolomite occurs in Jalpaiguri district of West Bengal. The reserve is said to be practically inexhaustible. The sample studied was supplied by Messrs Bengal Lime & Stone Co. Private Ltd. The dolomite was fairly pure and was found suitable for both semi-stabilization and complete stabilization as revealed by the laboratory study. Since the dolomite was contaminated with calcite, selective mining is recommended, and as the calcite occurs in isolated pockets, veins and streaks white in colour, selective mining should not be difficult.

To get a uniformly dense product, the size of the charge should preferably be between 1 and 2 in. as normally is the case for calcination in a rotary kiln. However, if the dolomite is to be calcined in a gas-fired shaft kiln and design considerations preclude the use of such small size, the alternative size would be from 2 to 4 in. It is, however, necessary to emphasize that for dolomite available in India, rotary kiln calcination is distinctly more suitable for sintering. Compared with other dolomites studied earlier for use in steel plants, Jainti dolomite appears to be superior to Hirri dolomite in quality and as good as Hardi or Baraduar dolomites.

76.0 High-temperature Mechanical Properties of Refractories

Investigation on high-temperature mechanical properties of refractories and its correlation with the service performance in iron and steel making furnaces have gained importance in recent years. With a view to initiate studies on high-temperature mechanical properties of refractories, an apparatus for studies on high-temperature torsion of refractories as well as one for high-temperature sonic modulus were designed and fabricated (Figs. 56 & 57) and these are being standardized.

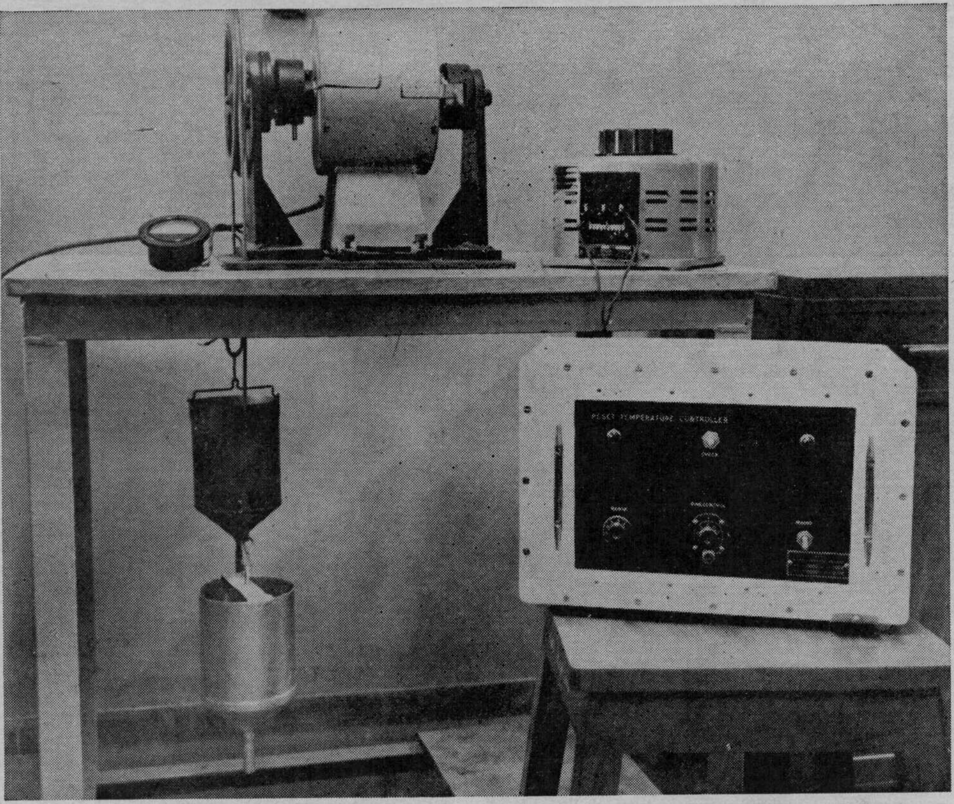


FIG. 56 — HIGH-TEMPERATURE TORSION APPARATUS

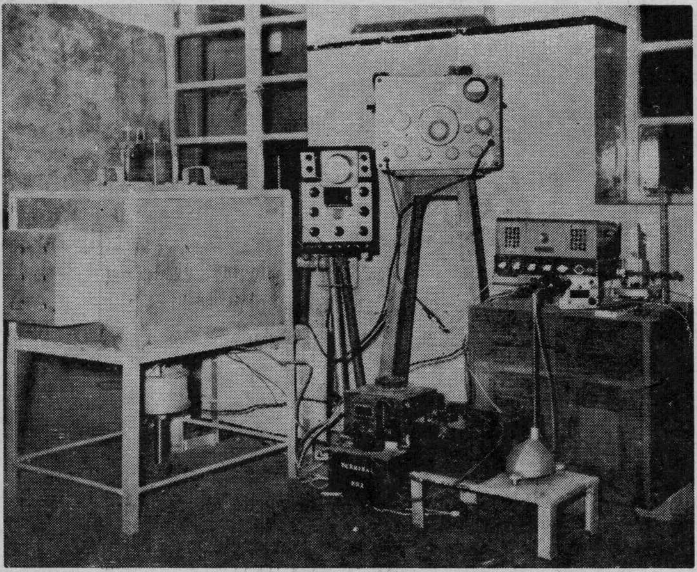


FIG. 57 — APPARATUS FOR DETERMINING MODULUS OF ELASTICITY OF REFRACTORIES AT HIGH TEMPERATURE

77.0 Mineralogy of Indian Blast Furnace and Steel Melting Slags

An understanding of the constitution of slags is of considerable importance in the metallurgical practice as well as for economic utilization of these waste products. Slags from low-shaft furnace of the National Metallurgical Laboratory and blast furnace, L.D. and open-hearth slags from Rourkela were collected for study. The highly aluminous and basic blast furnace slags from Rourkela showed the presence of spinel merwinite. No manganese-bearing mineral was observed. Further studies on the liquidus determination and mineralogy are under progress.

78.0 Development of Boron Nitrides

The project on preparation and properties of boron nitrides was taken up in view of the rapidly gaining importance of this material as a valuable refractory for a variety of applications and also as an electrical insulator at high temperatures. Boron nitride is quite stable under neutral and reducing atmospheres and hence can serve as a highly refractory material under these conditions due to its high melting point (3000°C. under high pressure of nitrogen). Boron nitride prepared at low temperatures is known to possess low bulk density and hence can be used for thermal insulation at high temperature.

In the current investigation, the aim is to prepare boron nitrides by nitriding anhydrous boric oxide. The oxide made by fusing boric acid will be glassy and is difficult to crush. Hence boric acid was dehydrated under vacuum at low temperatures to get a porous oxide mass which is easy to grind. Some isothermal dehydration experiments on boric acid in the temperature range 100-250°C. were carried out with a view to know the optimum temperature at which the rate of dehydration will be rapid without affecting the porosity of the dehydrated mass. The time taken for dehydration depends on the quantity of acid and the temperature and has to be assessed under the particular experimental conditions.

An apparatus for nitriding B_2O_3 was set up and experiments are being undertaken. An apparatus for measuring the specific surface and porosity of fine solids by adsorption of gases at low temperatures is being fabricated.

79.0 Studies on Clays

Fundamental studies were made on the effect of exchangeable cations (H^+, Na^+, K^+, Li^+, Ca^{++}, Mg^{++} and Ba^{++}) on thermal behaviour of kaolinite. Three kaolinites were taken for study and amongst them Travancore china clay was relatively more well crystallized and ball clay was a b/3 disordered kaolinite. Rajmahal ranked intermediate in crystallinity. The above conclusions were arrived at by D.T.A., X-ray, base exchange and electron microscopic studies. Electron micrographs are given in Figs. 58, 59 and 60. In the *p*H neutralization study of the hydrogen forms of these clays, Travancore china clay exhibited two inflexion points whereas others showed only one point; such difference in behaviour was examined and explained. The 'bec' of clays was determined by three different methods with a view to compare different techniques.

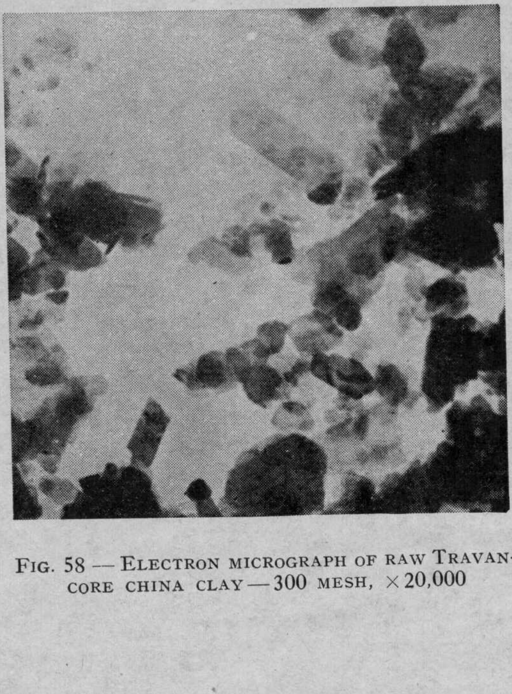


FIG. 58 — ELECTRON MICROGRAPH OF RAW TRAVANCORE CHINA CLAY — 300 MESH, ×20,000

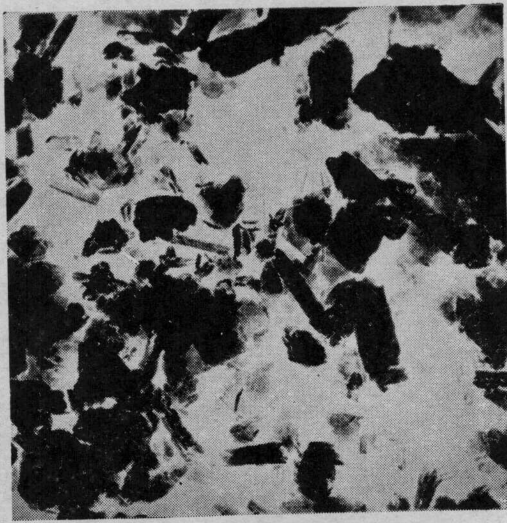


FIG. 59 — ELECTRON MICROGRAPH OF RAW RAJMAHAL CLAY — 300 MESH, ×20,000

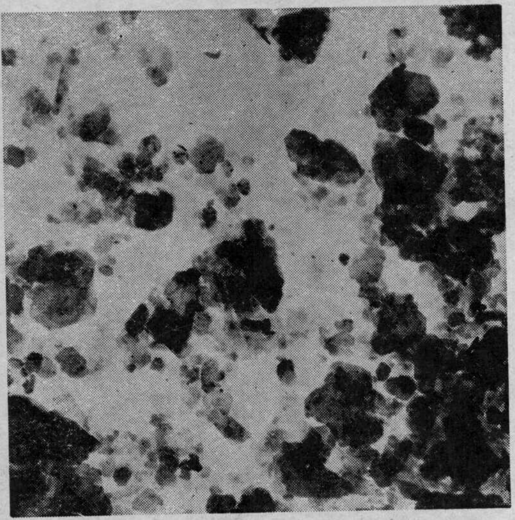


FIG. 60 — ELECTRON MICROGRAPH OF BALL CLAY — 300 MESH, ×20,000

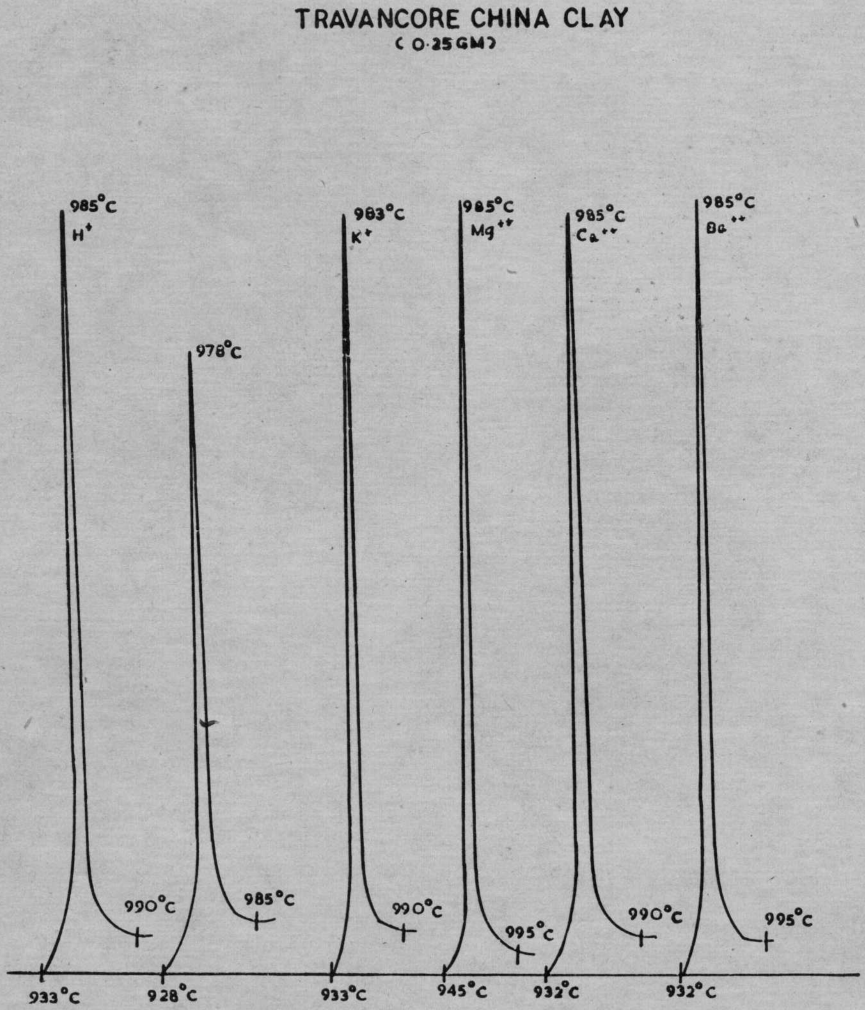
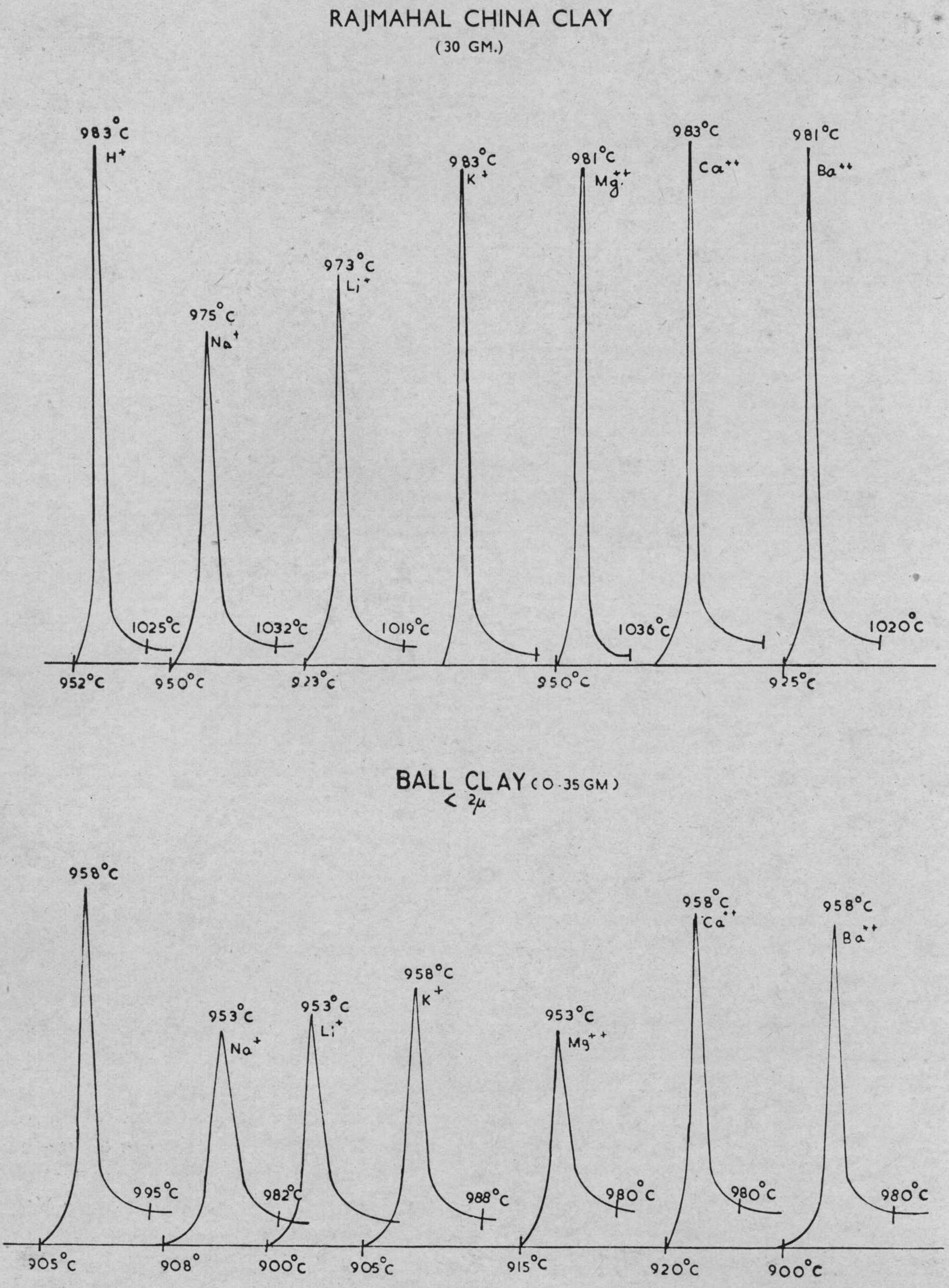


FIG. 61 — VARIATION IN SHAPE OF EXOTHERMIC PEAKS — MONIONIC FORMS

Hydrogen kaolinites gave the highest amplitude, area and slope ratio of the dehydroxylation peak in D.T.A. and barium forms the lowest values for the same factors. No rigid order prevailed between the various cationic forms in all the three kaolinites examined. The changes in peak shapes were considered to be due to differences in rate of dehydroxylation. The influence of cations on the shape of dehydroxylation peak is given in Figs. 61 and 62.

The exothermic peak in the region 950-1000°C. was greatly affected by various cations, Na^+ and Li^+ decreasing peak intensity and H and divalent cations, in general, increasing it. The reaction peak was considered to be due to rapid formation of spinel phase within a temperature range; the spinel phase was considered to be identical with the one reported by Brindley and coworkers in view of its similarity of crystal size and the nature of its formation from metakaolin. It was considered by the author that the variation in relative proportion of spinel and mullite phases in accordance with peak intensity was



RAJMAHAL CHINA CLAY
(30 GM.)

BALL CLAY (0.35 GM)
< 2μ

FIG. 62 — VARIATION IN SHAPE OF EXOTHERMIC PEAKS — MONIONIC FORMS

coincidental and may not have any direct bearing on intensity as proposed by Baker and coworkers. Figs. 63-65 represent the effect of cations on the D.T.A. curves in the range 950-1000°C. of the three clays studied.

A single exothermic peak was observed above 1000°C. in Rajmahal and ball clays and no peak was detected in the case of Travancore china clay. Evidence gathered in this study revealed that the peak is due to rapid formation of mullite and cristoballite has nothing to do with the peak, since the peak was absolutely unaffected in some cationic forms even though the cristobalite phase was completely undetectable in these samples both on quenching and prolonged heat-treatment during the peak temperature range.

A detailed study of the effect of cations on high temperature phases in ball clay revealed that the cations had no mineralizing effect on mullite formation. The cristobalite phase was very much affected by cations and the variation in intensity of the characteristic line of cristobalite in X-ray diffractograms was considered to be due to the degree of disorder caused by the various cations on cristobalite lattice. Figs. 63-65 indicate the influence of some cations, viz. H, Li and Na, on cristobalite as evidenced by the change in intensity of 4·1 Å. line of cristobalite. The work is completed.

79.1 Studies on Dehydration of Clays

Messrs. Mechanical Services (P) Ltd., Calcutta, sent 5 tons of china clay for low-temperature calcination at 700°C. for subsequent use as a base material for ultramarine blue. The analysis of the raw clay is given in Table 41. It was desired by

Table 41 — Chemical Analysis of Raw Clay from Messrs Mechanical Services (P) Ltd.

Loss at 110°C., %	2·10
Loss on ignition, %	11·91
SiO_2, %	45·28
Al_2O_3, %	34·28
Fe_2O_3, %	3·80
TiO_2	Trace
CaO	Trace
MgO, %	0·11
Na_2O, %	1·38
K_2O, %	2·21

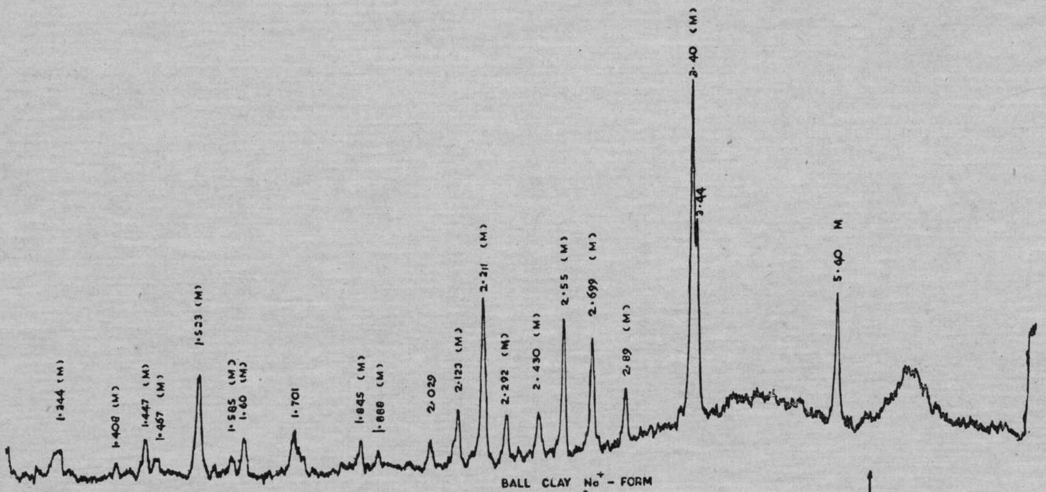


FIG. 63 — EFFECT OF CATIONS AS CRISTOBALITE

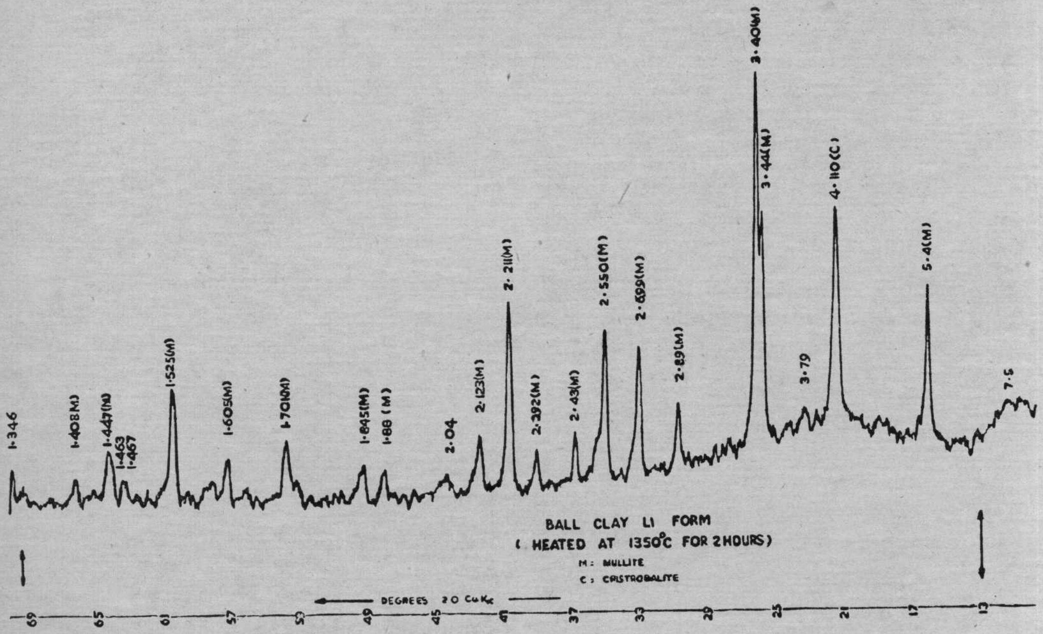


FIG. 64 — EFFECT OF CATIONS AS CRISTOBALITE

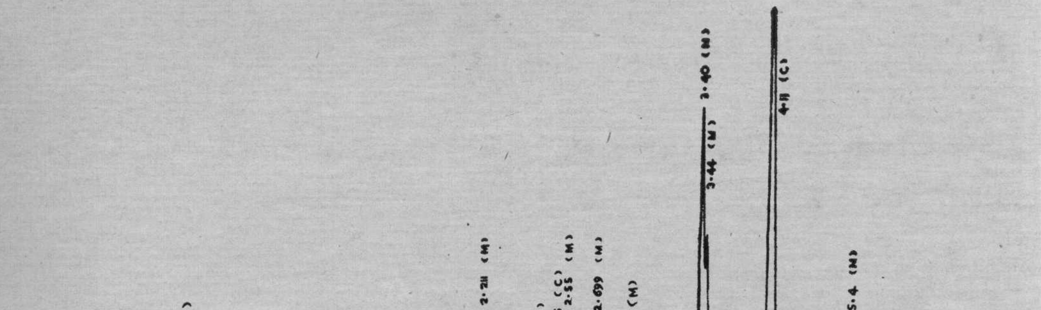


FIG. 65 — EFFECT OF CATIONS AS CRISTOBALITE

the party that the total water in the form of moisture and structurally combined should be reduced to less than 2 per cent without any transformation of alumina to α-form. As a preliminary test, the clay was sampled, crushed to below 2 in. lumps and heated in an electric kiln at 700°C. for ½ hr., 1 hr. and 2 hr. It was observed that the clay after heating for one hour or more had the desired optimum loss on ignition. D.T.A. and X-ray powder pattern confirmed that with one hour heat-treatment at 700°C., kaolinite structure completely breaks down to metakaolin, liberating all the combined water without any phase change in alumina.

To simulate the industrial conditions, a small rotary kiln (12 ft. long, 9 in. inner dia., r.p.m. ½) was put into commission. The hottest zone was kept at 700°C. and the inclination of the furnace was maintained such that the total time required for a particle to move from the charging end to the discharging end was 1½ hr. The entire lot of 5 tons of clay was passed through the rotary kiln. The product had the following chemical analysis:

	Per cent
Loss at 110°C.	0·3
Loss on ignition	1·0
Al_2O_3	36·73

D.T.A. and X-ray diffractograms confirmed that the dehydrated product is composed of metakaolin only.

80.0 Studies on Corrosion of Metals and Alloys

(i) *Studies on Atmospheric 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. In addition studies on the monthly and yearly corrosion rates of mild steel and zinc and the effects on yearly corrosion rates as influenced by the initial conditions of exposure were also undertaken.

(a) *Monthly and yearly corrosion rates of mild steel and zinc* — Samples of mild steel and samples of zinc were exposed to atmosphere on the first day of every month. Two samples of mild steel and zinc were removed after every month and the respective corrosion rate for that particular calendar month was determined. The remaining two mild steel and two zinc samples remain exposed to the atmosphere and will be removed after the completion of one year.

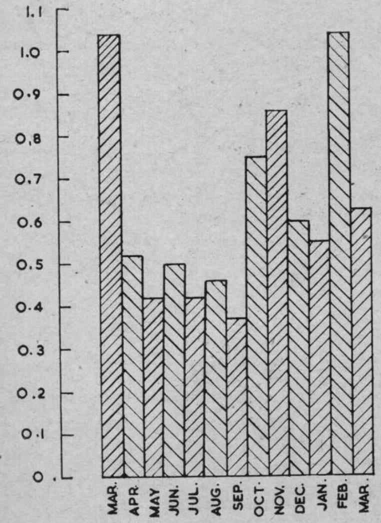
(b) *Cleaning of the corroded samples* — Samples exposed for yearly studies will be taken out after the completion of a year and corrosion rates will be assessed. This study was started since July 1963 and will continue for a period of two years.

Samples of mild steel and zinc were cathodically cleaned in 5 per cent inhibited sulphuric acid and 20 per cent chromic acid respectively to remove the corrosion product.

Studies were continued on the properties of rust on mild steel and low-alloy steel on samples removed after periods of 2, 6, 12 and 24 months and their effect on resistance to corrosion. The next set of samples will be removed in January 1965 to study the properties of rust and to analyse for total sulphate, ferrous iron, total iron and moisture content as was done in the case of earlier samples.

Evaluation studies of performance of organic and inorganic coatings in the industrial atmosphere of Jamshedpur are under progress. Periodic observations of the samples are being made on the condition of test panels. Even though slight rust developed at places where the paint was damaged by scratching and hammering, no failure of paint was observed near the damaged area.

Continuous recording of daily changes in atmospheric temperature and relative humidities are being done at the test site.



SULPHUR POLLUTION DATA DURING THE YEAR MARCH 1963 TO MARCH 1964

FIG. 66

Sulphur pollution of the atmosphere was determined every month by exposing lead peroxide candle at the test sites. Sulphur pollution data for the year 1963-64 are given in Fig. 66.

(ii) *Chromate Passivation of Aluminium*

Studies on the chromate passivation of aluminium and their corrosion resistance to salt spray test and humidity tests were earlier reported. During the period under review, further studies were carried out on the protective properties of chromate films after ageing and heating the samples at temperatures ranging from 50° to 200°C. The samples were dried in the oven at required temperature for an hour and later tested in salt spray chamber. All the samples after drying showed lower resistance to corrosion in salt spray test (compared to the unheated samples) indicating that the chromate film subjected to increasingly higher temperatures loses the protective properties. It was further noted that chromate-treated samples kept in dessicator for more than a week before subjecting them to test showed slight improvement in the corrosion resistance properties of the film.

Protective properties of chromate film were also examined under immersed condition in NaCl solution of different concentrations along with the untreated aluminium samples. For the tests, samples of aluminium were treated in two chromating solutions containing 3 and 6 mg./L. of chromic acid. In both the solutions, the amount of sodium dichromate and sodium fluoride were kept constant (3 gm./L. and 0·8 gm./L. respectively). The samples were treated for 3 and 10 min. The concentration of NaCl solution was varied between 20 and 2000 p.p.m. It was found that chromate treatment has a marked effect in increasing the resistance to corrosion even in solutions containing as high as 2000 p.p.m. of NaCl.

(iii) *Studies on Phosphatizing of Steel*

Amongst various methods of surface protection, phosphating of steel is of universal industrial importance. Most of the conventional phosphatizing baths are sludge-forming type which makes the handling of the solution and operation of the bath very difficult. In recent years, attempts have been made to develop non-sludge-forming bath though till now their use has not been extensive. Present investigation was taken up for the possibility of developing non-sludge-forming bath and also to compare the various characteristics of the phosphate coating on their protective and other properties. The efficiency of the non-sludge-forming phosphatizing bath developed in the National Metallurgical Laboratory was closely studied and its properties were compared to those of sludge-forming bath. The amount of coating formed and loss of metal due to acid attack with respect to the time of treatment showed similar behaviour in both the sludge and non-sludge-forming baths. Marked difference in acid attack and coating weight was observed for samples treated in two different ways. When the samples were treated singly in the bath one after another for different times, the curves for the coating weight and metal attack versus time of treatment showed an increase with time during initial period of treatment. With further treatment a sharp fall was observed. On the other hand, in batch process, when a group of samples were placed in the bath at a time and removed one by one after different times of treatment, the curves showed a gradual increase. To find out this difference in the two methods of treatment, further studies were made with special reference to the effects of poisoning of the solution, volume of the bath, concentration of the solution, etc. Chemical and X-ray diffraction studies on the phosphate coating were carried out. The coating from both sludge and non-sludge-forming baths was identified as $(ZnFe)_3(PO_4)_2,4H_2O$.

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

(i) *Dull Nickel Plating Salt*

Dull nickel plating salt was developed and standardized and its performance was experimented and tested in continuous production plating practice in light engineering firm. Dull nickel plating salt had established its superiority in service performance compared to imported salt.

For further development of dull nickel plating salt, it was found necessary to investigate the behaviour of the plating bath solution as derived from the salt mixed by replacing completely $NiCl_2$ by NaCl in the plating solution and further to assess the suitability of the proposed formulae with NaCl in commercial plating practice. The cost expenditure in relation to one of the costly main ingredients in the salt mixture will be thus reduced by such replacement. From the physical and metallographic studies of electrodeposited nickel under different conditions of platings, it was possible to postulate a new nickel plating composition containing NaCl in place of $NiCl_2$ which will be cheaper and economic. Its performance in commercial plating practice is under trial before its industrial scale production can be established.

(ii) *Bright Nickel Plating*

Bright niclel plating requires no buffing after plating and thus reduces the labour cost in the production plating practice. The organic reagents for bright nickel platings contain usually a suitable organic brightener, a leveller, an antistress reagent together with some other reagents which help and improve brightness in nickel plating during the course of electrolysis. A similar organic brightening compound was developed in the laboratory and exhaustive experimental trials with it were carried out to standardize the product for bright nickel plating. The organic chemicals that were used in the dull nickel plating bath to get the bright platings were organic brightener, viz. napthalene, sulphonic acid, a leveller coumarin, and an antistress reagent saccarin together with sodium salt of a weak acid. The reagents were added in minute traces in right proportions to get a mirror-like finish of nickel under the rigid control of *p*H and temperature of the nickel plating solution. The suitability of nickel plating brightener in commercial plating practice was established on performance tests on production plating trials of a light engineering works and a few defects that cropped up during the plant operation were subsequently remedied and the process was standardized for utilization in industries.

(iii) *Imparting Black Colour to ' Bidri ' Wares*

Imparting black colour to ' Bidri ' wares having the composition 96 per cent Zn and 4 per cent Cu used to be carried out by the particular type of clay available at Bidar (a small district town in Mysore State) where the ' Bidri ' handicraft industry is mainly localized. As desired by All-India Handicrafts Board, investigation was undertaken to find out a suitable substitute for the clay of Bidar for colouring ' Bidri ' wares black.

A substitute process was worked out and standardized for imparting lustrous black colour to zinc. This substitute is a mixture of a few chemicals and is soluble in water. ' Bidri ' wares with engravings of silver on the surface were polished, degreased, cleaned and subsequently immersed in the solution and imparted a shining black colour in two minutes leaving the silver portion unaffected. The colours thus obtained were blue, grey to black depending on the composition of the solution and time of immersion. All-India Handicrafts Board has been furnished full details of the process developed in this laboratory for imparting black colour to ' Bidri ' wares.

82.0 Gases in Metals

Vacuum fusion gas analysis apparatus was utilized in analysing various samples of metals and alloys. Samples were received from different research projects in the laboratory and also from other laboratories and institutes for the estimation of oxygen, nitrogen and hydrogen by vacuum fusion method.

For the determination of hydrogen in metals and alloys having low melting point and high vapour pressure, ' carrier gas ' method developed in the laboratory worked satisfactorily. The object of the introduction of this method of hydrogen determination was based on the fact that the metals of low boiling point cannot be accurately analysed by the conventional hot vacuum extraction method, as the vapour condenses on the wall of the furnace tube absorbing hydrogen and then lowers the hydrogen value. These difficulties with the low boiling metals have been completely eliminated by the practice of carrier gas method.

Hydrogen pick-up by steels during pickling with some common industrial inhibitors were studied. The aim was to find out a suitable inhibitor which can inhibit the hydrogen pick-up, but at the same time can pickle the sample of steel having different carbon composition. The acid corrosion of metals, the standard potentials of which are negative, is usually accompanied by the evolution of hydrogen. The hydrogen evolved in the atomic state during acid-metal reactions can penetrate into metallic crystal lattice to form solid solution which is inherently brittle and is known as hydrogen embrittlement or pickling brittlement. Part of the diffused

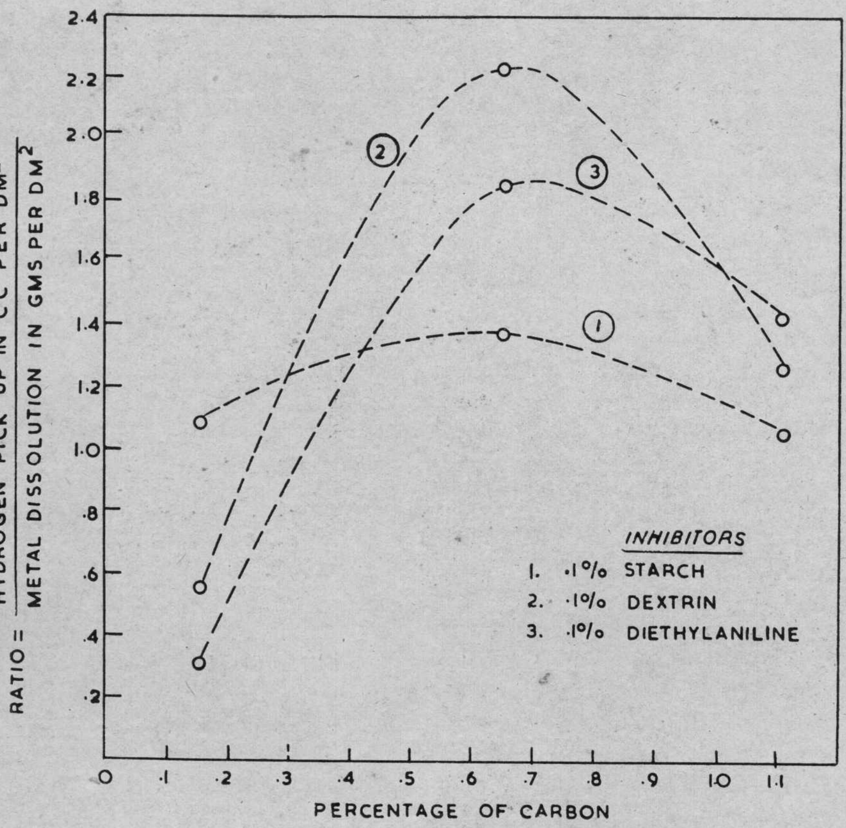


FIG. 67 — RATE OF HYDROGEN DIFFUSION DURING PICKLING IN THE PRESENCE OF INHIBITORS

hydrogen atoms in the crystal lattice forms molecular hydrogen on the walls of the numerous microscopic pores (microcavities) in the crystal lattice. Accumulation of molecular hydrogen in these microcavities is associated with high pressure which is the chief cause of formation of pickling blisters on the surface of the metals. So the study of problems associated with the diffusion of hydrogen into metals during pickling has gained importance during recent years.

Different inhibitors were studied in relation to steel having different carbon compositions in acid bath and the rate of diffusion of hydrogen on the samples was examined. The ratio of hydrogen pick-up by the metal and the metal dissolution was considered the criterion of the efficiency of the inhibitor. Lower the ratio, better is the performance.

Inhibitors that acted efficiently on mild steel in inhibiting hydrogen were found to be ineffective in the case of medium carbon and high carbon steel. Comparatively weak inhibitor like starch for mild steel acts effectively for pickling medium carbon steel and high carbon steel. The ratio of hydrogen pick-up to metal dissolution was observed to increase with increase of carbon content and was maximum at 0·65 per cent of carbon and decreased again when carbon content reached 1·11 per cent (Fig. 67). The difference in the rate of diffusion of hydrogen in steel containing different amounts of carbon is probably due to the difference of solubility of hydrogen in steel having different carbon contents. It was also observed that inhibitors which strongly retard the solution process do not always lower the diffusion of hydrogen into metals. Thiourea and its derivatives are typical examples. Potassium iodide was also found to inhibit efficiently the mild steel dissolution, but both thiourea and potassium iodide were found ineffective when used for medium carbon and high carbon steel; these retarded the metallic dissolution even in 7*N* sulphuric acid bath and prolonged the pickling process. For medium carbon and high carbon the inhibitors dextrine and diethylaniline also showed better performance as compared to starch.

Some studies on the possibility of determination of nitrogen in steel in interstitial solid solution by electrochemical method were made. Till now there is no single method except the internal friction measurement method, introduced by Fast and Verijp, for the direct determination of nitrogen which is in interstitial solid solution. When a steel contains different alloying elements, this physical method offers complications. By chemical method indirectly the solid soluble nitrogen can be estimated by determining the nitride nitrogen by Beeghly method and substracting it from total nitrogen determined by Jeldahl method or V.F. method. Recently Yu. A. Klyachko and O. D. Larina emphasized the possibility of determination of nitrogen in steel in interstitial solid solution by electrochemical method. When a steel sample is subjected to anodic solution, the metal crystal lattice is broken down and the atoms of the gaseous element present in a solid solution or in a surface compound are thereby freed. The gases are collected in anode chamber and analysed.

Various samples were analysed and the nitrogen (interstitial atom) was determined. The difficulty encountered was that the nitrogen content of the gas collected in the anodic chamber is very low. The conventional method for the determination of nitrogen in the collected gas (by difference) was not sufficiently accurate. Arrangements were made to analyse the gas sample by mass spectrometer.

83.0 Spectrographic, Polarographic and Other Analytical Work

The following projects were taken up during the period under review:

(i) *Spectrochemical Determination of Antimony in Lead-Tin Solders*

This work was taken up to develop suitable spectrographic methods for accurate and rapid determination of antimony in lead-tin solders. Porous cup technique was adopted for estimation of high amounts of antinomy up to 10 per cent and oxide arc technique for antimony up to a lower limit of 0·001 per cent. Accuracy (per cent error) varied from 3 to 6 per cent of the amount present when 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 Slag*

This project was undertaken to develop a suitable and rapid routine process to analyse steel-making slag spectrographically. Conventional chemical methods were highly time-consuming to be of immediate value during alloy and steel-making itself. With a view to improve both the speed and the accuracy of analysis, an attempt was made to use spectrograph as a tool for analysing steel-making slag.

To start with, basic open-hearth slag was taken up. The sample was converted into solution by fusing and subsequent treatment with acid. Few drops of this solution were dried on a graphite electrode and excited with A.C. intermittent arc taking pure carbon as counter-electrode. The spectrum was recorded on Kodak B_{10} plate using Hilger large quartz spectrograph. Neutral filter having 10 per cent transmission of radiation was used to reduce the intensity of the prominent analytical lines of the elements to be measured by microphotometer to prepare the working curve. Attempts were made to prepare the working curves for Ca, Mg, Si, Mn, and Fe using standards prepared synthetically taking different spec. pure oxides of the elements. Further work in this line is being continued.

(iii) *Spectrographic Analysis of Residual Elements in Steel*

This work was taken up to develop an accurate and suitable spectrographic method to analyse trace elements such as As, Sb, Sn, and Ti in steel. Chemical methods of analysis of these trace elements are time-consuming and cumbersome. Attempts were made to develop a suitable method for accurate analysis of trace elements.

Due to very low concentration of these trace elements in steel, oxide arc technique was adopted for estimation. Both samples and standards prepared synthetically were converted into oxide and few exposures under standardized conditions were taken using Hilger large quartz spectrograph to prepare the working curves. Further work in this line is being continued to study the reproducibility of the method and accuracy of the results.

(iv) *Spectrophotometric Determination of Chromium and Silicon in Ferrochrome and Slag*

This investigation was taken up for developing rapid methods of analysis of ferrochrome and slag during the production of the alloy. Work was carried out for the determination of chromium. It was found that chromium in slag and alloy could be determined by measuring the intensity of the colour of chromate in alkaline solution. Further work for the determination of silicon is in progress.

(v) *Polarographic Analysis of Zinc Ore Concentrate for the Determination of Lead, Copper, Cadmium and Zinc*

The work was taken up to find out a rapid and reliable method for the determination of the constituents of a large number of samples of zinc ore concentrates and tailings received during the beneficiation of the zinc ores.

The procedure developed consisted of separating the lead as sulphate after decomposing the sample with aqua regia and recording the polarogram of cadmium, zinc and copper from ammonical medium in the filtrate. The polarogram of lead was obtained from the solution of lead sulphate in ammonium acetate. Results obtained compared favourably with those obtained by classical methods.

(vi) *Determination of Fluoboric Acid in Spent Solution after Precipitation of Cryolite*

For recycling the solution obtained after precipitation of cryolite, it was necessary to determine free sulphuric acid and fluoboric acid content in the spent solution. This work was started to find out a suitable method for the determination of the free acids in the solution as no methods are available for this purpose.

For the determination of fluoboric acid null-point potentiometric method was tried. It was observed that though small amounts of fluoboric acid can be determined in pure solution the method was not applicable in the presence of boric acid and aluminium. Its reference of boric acid and aluminium had also been studied.

(vii) *Tannin Method for the Estimation of Vanadium and Its Separation from Phosphorus*

Work on this investigation was started with the objective that tannin precipitation method may prove to be a quicker and easier method for the estimation of vanadium than the existing lead vanadate or mercurous vanadate method. It was also considered worth while to find out if it can be extended for separation of vanadium from phosphate, which also forms a white precipitate with tannin in the presence of an ammonium salt.

A gravimetric method for the estimation of vanadium from a solution of alkali vanadate, using tannin as a precipitant under different conditions, e.g. in the presence of ammonium acetate, ammonium chloride, ammonium sulphate at varied *p*H, under different dilution and temperature conditions, was studied. It was found that most effective precipitation of vanadium could be obtained by carrying out the estimation at *p*H 6 from 200 ml. of 10 per cent ammonium chloride by slow addition of 20 cc. of 10 per cent tannin for every 50 mg. of V_2O_5, when the solution was heated just to boiling condition. The filtration could be made as soon as the beaker cooled down to room temperature. This method was extended on to the separation of vanadate from phosphate without previous reduction of vanadium from pentavalent to trivalent state. It may be mentioned that prior reduction of vanadium is necessary for the separation of phosphate in the molybdate method.

(viii) *Routine Analysis*

During the period under review 5600 samples were analysed for 17,000 constituents.

84.0 Preparation of Slag Cement from Low-shaft Furnace Granulated Slag

Investigations were completed on the subject of utilizing of granulated L.S.F. slags, containing less than and more than 5 per cent MgO respectively, for portland blast furnace slag cement-making. Studies were undertaken to determine the optimum ranges of MnO content in the slag which will not adversely affect the hydraulic properties of slag. The other direction of investigation was to define the extent to which the slags obtained from the iron and steel plants in India vary in respect of major constituents such as SiO_2, Al_2O_3 and CaO as well as in MgO and MnO. So far as Al_2O_3 and CaO were concerned, Indian slags were higher in Al_2O_3 and fairly low in lime as compared to corresponding American and British slags, since Indian iron ores and coke ash contained high alumina. Since an increase in the alumina content increases the viscosity of the slag, it was normal to add dolomite in the burden to increase the MgO content in the slag to achieve improved viscosity and requisite fluidity. In Indian blast furnace slags, MgO content ranged up to 9 per cent. Previously, the Indian standard specifications stipulated a maximum of 8 per cent MgO in the granulated slag for portland slag cement and this was subsequently revised to a maximum limit of 17 per cent. Current U.S.S.R. practice accepts MgO contents of the blast furnace slag up to 18 per cent for use as blast furnace slag cement.

An important question relates to the influence of MnO on the hydraulicity of the slag. A reference to the following equation which gives the relation between basic and acidic oxides of the slag and indicates the hydraulicity of the slag where *F* denotes the hydraulic index, will show

$$F = \frac{CaO + \frac{1}{2}MgO + CaS + Al_2O_3}{SiO_2 + MnO}$$

that any increase in the MnO content, when other contents are constant, would adversely affect the hydraulicity of the slag. Investigations were undertaken to determine the optimum range of MnO content in the slag which would not adversely affect the hydraulic properties of the slag.

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

(i) *Insulation Concrete*

Thermal conductivity values of light-weight concrete proportioned on volume as well as weight basis using expanded low-shaft furnace slag aggregates and portland cement were determined by the Blakely & Cobb apparatus.

Concrete blocks made with thirty different proportions showed thermal conductivity values from 0·9 to 1·5 B.t.u./(hr.)(sq. ft.)(in.)(°F.) in comparison to 3·55 B.t.u./(hr.)(sq.ft.)(in.)(°F.) of that of gravel-sand concrete. From these investigations, it was concluded that the slag with basicity varying between 1·1 and 1·35 and remaining sulphur above 0·4 per cent when expanded at high temperature can yield light, porous and strong aggregate. The concrete made by these aggregates was light, strong and more insulating, and such light-weight concrete can be used in the construction of houses, air-conditioned buildings and cold storages, etc., to provide thermal insulation. For acoustic insulation also such concrete can be used in cinema houses, theatres, public halls and other sound-proof buildings.

Expansion of low-shaft furnace and blast furnace slag on pilot plant basis is being taken up.

(ii) *Structural Concrete*

Studies on the suitability of expanded L.S.F. slag for light-weight structural concrete has been taken up as per the A.S.T.M. Specification No. 330-53T. As a part of broad based research and development programme on indigenous slag utilization, investigations are also planned to be undertaken on the use of blast furnace slag as soil conditioner as well use of air-cooled slag as railroad ballast. Preliminary data are being collected in collaboration with the Directorate of Agriculture, Bihar, and Ministry of Railways respectively. Mineralogical studies on Indian steel plant slags have also been initiated particularly with a view to study the slag attack on refractories.

86.0 Recovery of Zinc from Scraps and Wastes

In the galvanizing plant about 40 per cent of the zinc is lost in the form of dross, ash, flux skimmings and blowings. With the exception of dross, the wastes produced as such fetch a price which is next to nothing, are not yet processed for the recovery of zinc in our country and exported abroad. Owing to the manifold increase in the consumption of zinc, non-availability of indigenous virgin metal and the most difficult foreign exchange position, producers of zinc wastes, especially those produced during galvanizing, are seriously considering the recovery of zinc values from wastes and scraps and the National Metallurgical Laboratory is receiving numerous enquiries on the subject. The present project was, therefore, undertaken based on two parts. The first part contains an exhaustive technical review report based on the information gathered from contemporary technical literature. The second part consists of the experiments performed for the recovery of zinc from galvanizers' ash. Two series of experiments were performed. In the first series, the ash was leached with caustic soda solutions of various strengths and the resulting leach liquor was electrolysed to formulate optimum conditions for zinc recovery. It was found that leaching with a solution containing 400 g./l. of NaOH for 3 hr. at boiling temperature gave best results. While electrolysis of the solution containing 40 g./l. of Zn and 180 g./l.

NaOH at a current density of 450 amp./m.^{2} gave best results, the current efficiency was about 97 per cent.

In the second series of experiments, the ash was leached with ammoniacal ammonium carbonate solutions of different strengths. The best results were obtained by leaching with a solution containing 12 gm. NH_3 and 26·4 gm. $(NH_4)_2.CO_3$ per 100 cc. of the solution for 10 min. which gave a recovery of 97 per cent. Work will be taken up in future to treat ash and other wastes by pyro-metallurgical and various other processes.

87.0 Production of Secondary Aluminium

Though India possesses abundant bauxite reserves for producing all the aluminium needed by our country, one handicap relates to the lack of adequate cheap electric power. Indian industries produce heavy aluminium and aluminium alloy scraps and by utilizing them for producing secondary aluminium and aluminium alloys, overall availability of the metal would improve. Some Indian producers are, therefore, seriously considering the production of secondary aluminium and aluminium alloys and as such approached the National Metallurgical Laboratory for the solution of their respective problems in these fields. With a view to advise them expeditiously, the current technical literature was exhaustively surveyed and a full review report was prepared. A systematic study of some of the suitable processes enumerated in the review report has been now taken up.

PILOT PLANTS

88.0 Low-shaft Furnace Pilot Plant Project

It was mentioned in earlier reports that comprehensive investigations had been conducted with iron ore received from the Punjab. Based on the feasibility report of the National Metallurgical Laboratory on the smelting characteristics and economics of production, the State Government has decided to install a 100,000 tonnes/annum pig iron plant. In the absence of coal, smelting will be conducted with surplus nut coke obtained from the nearest integrated iron and steel plant. Further investigations in the low-shaft furnace pilot plant will be conducted to find out the possibility of employing semi-anthracitic coal from Kalokote and other indigenous resources wherever available.

In view of the relatively faster throughput time in the low-shaft furnace in comparison with classical blast furnace, extensive investigations were undertaken to improve the smelting efficiency by appropriate size grading of iron ore and limestone, chemistry of the slag and adjustment of operational variables.

Programme of Research and Development Wark

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

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

Phase III

(1) Utilization of lignites after its high-temperature carbonization for iron smelting.

(2) Utilization of Salem magnetite after its beneficiation and 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 production of such operations.

Phase V

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

Phase VI

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

Extensive investigations under Phase I (1) to (4), (5) and Phase II were completed and investigation project and feasibility reports were submitted to appropriate State Governments. Based on the raw materials employed for smelting and the objectives aimed, the investigations during the period under review are grouped into the following campaigns.

Twentieth Campaign

Smelting trials with low-temperature carbonized coke (Kolsit) made from non-coking coal (Singareni Colliery), iron ore fines (Orissa), and blended fluxes with a view to produce iron of somewhat lower silicon contents.

Twenty-first Campaign

Smelting characteristics of fine-grained iron ore (Andhra), blended fluxes and low-temperature carbonized coke (Central Fuel Research Institute) made from non-coking coals (Jambad-Bhowla, New Satgram, Selected Jambad, Lower Kenda Collieries, etc.) with the objective of assessing operational behaviour, coke rate, analysis of pig iron under acid slag conditions.

Twenty-second Campaign

Smelting was conducted with a burden composed of small lumpy iron ore (Orissa — B. Patnaik), blended fluxes and low-temperature carbonized coke (Central Fuel Research Institute) made from non-coking coals [Jamuria (Koithu Seam), South Joyramdanga Collieries] with the object of studying the effect of increase of blast pressure and volume on the operational characteristics.

Twenty-third Campaign

Smelting trials with iron ore fines (Orissa), low-temperature carbonized coke made from non-coking coal (Singareni), blended fluxes for studying the effects of variations of MgO in the slag at levels of 8, 12 and 16 per cent respectively on the operational characteristics, composition of pig iron produced, partition of sulphur and fluidity of slag.

Twenty-fourth Campaign

Smelting trials employing iron ore fines (50 per cent — 3 mm.), low-temperature carbonized coke (Central Fuel Research Institute) made from non-coking coals and blended fluxes to assess the effect of progressive replacement of lumpy limestone by fines on the fuel rate and productivity.

Apart from extensive smelting trials with large varieties of substandard grade of raw materials, the replacement of highly alloyed heating elements of the recuperator by argon-arc welding and other maintenance jobs constituted another facet of intense activity.

With a view to utilize surplus 'naphtha' from the petroleum refineries as blast additive for economizing fuel rate, the incorporation of fuel injection in the low-shaft furnace is under way. As naphtha injection for iron smelting has not been conducted elsewhere, the work of the National Metallurgical Laboratory will be pioneering in this field, obviously demanding high technical skill in handling this dangerous explosive material. Painstaking and progressive research is aimed at achieving efficiency of iron smelting by the utilization of surplus products.

The oxygen supply pipeline is being laid. The oxygen-enrichment of the blast with simultaneous naphtha injection will decide the commercial possibilities. The oxygenated air blast will be employed for smelting ferro-alloys in the low-shaft furnace.

Details of Furnace Campaigns

Twentieth Campaign

In this campaign, smelting trials were conducted whilst employing a burden consisting of fine-grained iron ore from Orissa, blended fluxes and-low temperature carbonized coke (Regional Research Laboratory) made from non-coking coal (Singareni Colliery). Although low-shaft furnace process of smelting iron is characteristically suitable for the production of high silicon foundry grade pig iron, the object of the campaign was to examine the feasibility and conditions under which it may be possible to produce low silicon basic iron by adjustment of the ore/fuel ratio and the basicity degree of the slag, so that low-shaft furnace basic iron can be employed for steel making. The chemical analyses of raw materials used are given in Table 42.

The screen analysis of the raw materials employed is recorded in Table 43.

The furnace was blown under the following operational conditions:

Tuyere diameter	75 mm.
Hot blast wind volume	1900-2800 Nm.^{3}/hr.
Hot blast wind pressure	1800-2200 mm. WG.
Hot blast temperature	440-550°C.
Top gas temperature	230-500°C.

At the initial stages of the furnace operation, nut coke was employed as fuel. But

Table 42 — Chemical Analyses of Raw Materials

1. Proximate analysis of Kolsit (Singareni)

Moisture %	Ash %	Volatile matter %	F.C. %
2·4	19·3	9·2	69·1

2. Analysis of ash from Kolsit

SiO_2 %	CaO %	MgO %	Al_2O_3 %	Fe %	S %	P %
66·3	0·7	2·2	23·3	4·2	0·06	0·028

3. Analysis of iron ore (Orissa mineral)

Fe %	SiO_2 %	Al_2O_3 %	CaO %	MgO %
64·14	3·28	4·57	Trace	Trace

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	0·055	0·46

Table 43 — Screen Analysis of Raw Materials

Raw materials	Above 50 mm. %	−50 mm. +25 mm. %	−25 mm. +12·5 mm. %	−12·5 mm. +6 mm. %	−6 mm. +3 mm. %	−3 mm. %
Orissa minerals:						
Iron ore	—	—	19·9	50·0	26·6	3·8
Kolsit	17·2	53·8	28·0	0·5	0·15	0·35
Limestone (Madras)	—	44·4	34·6	10·5	4·8	5·7
Limestone (Andhra)	—	20·8	64·2	9·3	2·1	3·6

after the furnace became regular, nut coke was replaced by low-temperature carbonized coke (Kolsit) which was screened to remove −6 mm. material. The ore rate was 1·43 tonnes and the flux rate at about 0·9 tonnes/tonne of pig iron, while the slag volume of about 0·95 tonne/tonne of pig iron. As high silicon in pig iron is generally due to high smelting temperature, the ore/fuel ratio was carefully adjusted, but beyond an optimum value the furnace showed evidence of chilling. In order to reduce the activity of SiO_2 in the slag, the basicity degree was also increased. However, the analytically determined basicity was always lower than the basicity in accordance with burden calculation presumably due to the contamination of limestone with clayey matter and its loss in the form of flue dust. Silicon content of pig iron averaged 2·3 per cent, although silicon contents below 2 per cent were well achieved. The compositions of the pig iron and slag are shown in Tables 44(a) and 44(b) respectively.

Table 44(a) — Composition of Pig Iron

Composition	Percentage
Total carbon	2·7-3·5
Silicon	1·6-3·0
Sulphur	0·06-0·185
Phosphorus	0·09-0·28
Manganese	0·5-0·8

Table 44(b) — Composition of Slag

Composition	Percentage
CaO	33-42
SiO_2	34-37
FeO	1-4
Al_2O_3	15-22
MgO	4-7

Towards the latter part of this campaign, the MgO content of the slag was increased to a calculated figure of approximately 10 per cent by increasing the amount of dolomitic limestone (Andhra) in the burden. The lime basicity of the slag was maintained at a calculated figure of about 1·2. The presence of higher amounts of MgO in slag did not alter the silicon content of the pig iron.

The appropriate adjustment of the ore/fuel ratio within certain limits and the basicity degree of slag enabled the production of pig iron with further lower silicon contents, but the sulphur content was somewhat on the high side due to lower hearth temperature.

Twenty-first Campaign

Smelting trials were conducted with the burden composed of a mixture of fine-grained iron ore from Andhra, blended fluxes consisting of limestone and dolomite with low-temperature carbonized coke from Central Fuel Research Institute. The campaign was carried out to examine the effects of changes in the nature of fuel on the silicon contents of the pig iron smelted. Besides, investigations were conducted under acid-slag conditions to reduce the fuel rate. The higher fuel rate in low-shaft furnace necessitated higher addition of flux for producing normal basic slag, which cumulatively increased the fuel rate. As iron smelting in the low-shaft furnace is primarily meant for the exploitation of substandard grades of raw materials, lowering of the basicity without sacrificing the quality of the metal may lead to economy in flux and fuel consumption. However, the use of the fuel, which had degraded considerably in storage, caused severe operational problems and often resulted in a cold furnace. The inadequate hearth temperature caused the high sulphur content and low carbon content of the pig iron produced. In the latter half of this campaign, investigation was conducted with a view to examine the effects of changes in lime basicity ratio on the fuel rate, quality of pig iron, analyses of top gas, direct and indirect reductions, degree of desulphurization and particularly the technological aspects of operational performance. For this purpose, the physical and chemical characteristics of the raw materials were kept identical excepting that the basicity of slag was maintained at different levels based on calculated $CaO/SiO_2 = P_1$ values of about 1·0, 0·8, 0·75, 0·65, 0·6 and 0·55 respectively. Chemical analyses of the raw materials used in this trial are given in Table 45.

The furnace was blown in under the following conditions:

Tuyere diameter	75 mm.
Hot blast wind volume	1950-2300 Nm.^{3}/hr.
Hot blast wind pressure	1500-1900 mm.WG.

Table 45 — Chemical Analyses of Raw Materials

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

Moisture %	Ash %	Volatile matter %	F.C. %
8·5	25·9	4·1	61·5

2. Analysis of coke ash

CaO %	SiO_2 %	MgO %	Al_2O_3 %	Fe %	P %
5·68	53·8	1·88	28·0	4·6	0·8

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 iron ore (Andhra)

Fe %	SiO_2 %	Al_2O_3 %	S %	P %
63·7	3·8	2·0	0·03	0·06-0·12

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	—
Dolomitic limestone (Andhra Pradesh)	32·20	25·0	0·3	0·56	0·55	N.D.

Table 46 — Screen Analyses of Raw Materials Employed

	−50 mm. %	−50 mm. +25 mm. %	−25 mm. +12 mm. %	−12 mm. +6 mm. %	−6 mm. +3 mm. %	−3 mm. %
Low-temperature carbonized coke	14·1	33·2	37·3	8·8	2·9	3·7
Andhra iron ore	6·2	16·5	35·8	15·2	11·2	15·1
Madras limestone	17·6	43·7	20·6	9·0	4·3	5·5
Andhra limestone	—	18·3	60·0	12·5	4·1	5·7

Hot blast temperature	450-550°C.
Top gas temperature	300-500°C.

The high fuel consumption in the campaign was partly due to high ash in the coke but also due, in some measure, to deterioration of the L.T.C. fuel during long storage and to atmospheric high humidity during the rainy seasons. Furthermore, fuel consumption was high due to very high moisture contents of the burden. The deterioration of physical properties on long storage of the fuel led to operational difficulties, particularly smoothness of burden descent. The ore rate throughout this campaign was 1·48 and the flux rate was maintained at about 1·3 in the initial stages of the campaign and reduced to as little as 0·76 towards the latter parts of the campaign. The operational data under various acid-slags are summarized in Table 47.

A critical examination of the data established that the silicon contents in the metal did not appreciably increase with the lowering of the basicity degree and increase in silica activity of the slag. The somewhat poor carbon saturation in low-shaft furnace pig iron further deteriorated under acid smelting conditions. The smelting characteristics were satisfactory up to a CaO/SiO_2 ratio of 0·60, below which the slag became very viscous and consequently was not free-flowing. The flux and fuel rate decreased substantially on decreasing the CaO/SiO_2 ratio, but the partition of sulphur was adversely affected due to poor sulphur capacity of acid-slag, which will require ladle desulphurization of pig iron. The slag showed characteristic black colour due to the presence of 4-7 per cent FeO. The recovery of iron and manganese decreased with increase in activity of the slag.

Table 47 — Effect of Decreasing the Basicity Degree of Slag on Smelting Characteristics

Slag basicity degree (CaO/SiO_2)	1·0	0·87	0·70
Slag volume tonnes/tonne of pig iron	1·94	1·81	1·60
Average analysis of metal, %:			
C	3·0	2·75	2·40
Si	3·4	3·80	3·90
S	0·08	0·10	0·17
Mn	0·40	0·38	0·20
P	0·14	0·11	0·09
Average analysis of slag, %:			
CaO	37·80	32·50	25·20
SiO_2	38·20	37·50	35·40
FeO	0·70	3·60	7·60
Al_2O_3	18·10	19·80	25·42
MgO	4·40	6·20	6·40
CO/CO_2 ratio in top gas	6·40	6·70	6·80

Twenty-second Campaign

In the twenty-second campaign, trials were conducted with a burden composed of a mixture of small lumpy iron ore from Orissa, low-temperature carbonized coke from the Central Fuel Research Institute, blended fluxes consisting of limestone and dolomite. The object of the smelting trial was to evaluate the effects of increase of blast pressure and volume on furnace operations and particularly on the iron output. Chemical analyses and screen analyses of raw materials used in this trial are given in Tables 48 and 49 respectively.

The furnace was blown in under the following operational conditions:

Tuyere diameter	75 mm.
Hot blast wind volume	1800-3000 $Nm.^3$/hr.
Hot blast wind pressure	1000-2600 mm.WG.
Hot blast temperature	410-550°C.
Top gas temperature	200-450°C.

The rate of descent of the burden generally depends on the rate of combustion of coke in the tuyere zone. A greater amount of wind at higher pressure will accelerate

Table 48 — Chemical Analyses of Raw Materials Employed

1. Analysis of small lumpy iron ore (B. Patnaik, Orissa)

Fe %	SiO_2 %	Al_2O_3 %	P %	S %
63·2	2·88	5·6	0·05	—

2. Proximate analysis of low-temperature carbonized cokes

Location	Moisture %	Ash %	V.M. %	F.C. %
C.F.R.I. (Jealgora)	5·80	28·00	4·60	61·60
R.R.I. (Hyderabad)	2·30	21·20	8·90	65·67

3. Ash analysis of low-temperature carbonized coke

SiO_2 %	MgO %	Al_2O_3 %	CaO %	Fe %	S %	P %
66·3	2·2	23·3	0·7	4·2	0·06	0·028

4. Analysis of fluxes

Fluxes and location	CaO %	MgO %	SiO_2 %	Al_2O_3 %	S %	Fe %
Limestone (Madras)	54·31	1·0	0·88	1·2	0·027	—
Dolomitic limestone (Andhra Pradesh)	32·20	25·0	0·3	0·56	0·55	0·46
Dolomite (Assam)	31·3	20·7	0·4	—	0·6	—

Table 49 — Screen Analyses of Raw Materials Employed

Raw materials	Above 50 mm. %	−50 mm. +25 mm. %	−25 mm. +25 mm. %	−12·5 mm. +6 mm. %	−6 mm. +3 mm. %	−3 mm. %
Low-temperature carbonized coke	21·3	45·1	31·2	1·6	0·3	0·5
Small lumpy ore (B. Patnaik)	—	—	1·5	65·2	31·5	1·8
Limestone (Madras)	—	30·5	41·5	15·9	6·8	5·3
Dolomitic limestone (Andhra)	—	7·9	59·5	23·2	5·8	3·6

the combustion of coke producing a larger volume of bosh gas. In order to fully utilize the thermal and chemical energies of the ascending gas, satisfactory interaction with descending ore should occur to obtain the optimum fuel efficiency. The rate of blowing has to be suitably adjusted. On increasing the blast pressure and volume, it was found that the number of charges per hour was increased which gave increased production of pig iron. The increase in blast volume by 8 and 12 per cent over the base period increased the production substantially. However, it was not possible to quantitatively assess these results over a long period as the harder rate of driving led to irregular descent of the burden. The monsoon weather conditions during this campaign caused high humidity of the air blast and high moisture contents in the burden, both of which adversely affected the fuel consumption. At a flux rate of 1·5 tonnes and an ore rate of about 1·5, the slag volume amounted to about 2 tonnes/tonne of pig iron. Slag basicity was maintained between a calculated CaO/SiO_2 ratio of 1·0 and 1·2. The compositions of the pig iron and slag produced are given in Table 50.

Table 50 — Composition of Pig Iron and Slag

1. Composition of pig iron

Total carbon %	Si %	S %	Mn %	P %
2·9-3·7	1·60- 2·80	0·05- 0·12	0·13- 0·35	0·11- 0·33

2. Composition of Slag

CaO %	SiO_2 %	FeO %	Al_2O_3 %	MnO %	S %
32-41	32·36	0·80- 1·60	17-21	0·30- 0·60	0·33- 0·50

Twenty-third Campaign

In order to define the effects of increasing amounts of MgO in the slag on smelting characteristics further trials were conducted with a burden consisting of iron ore fines from Orissa, low-temperature carbonized coke from the Regional Research Laboratory, Hyderabad, and blended fluxes. In the later half of the campaign, low-temperature carbonized coke (Kolsit) was replaced by L.T.C. coke from the Central Fuel Research Institute, Jealgora. Keeping other variables fixed, the MgO content on slag was maintained initially between a calculated figure of 9 and 10. In the second stage of the campaign, the MgO content was increased to 13-15 per cent, and finally the MgO content of slag was increased to 17-19 per cent. Generally throughout the experiment, the lime/slag basicity was maintained at a calculated figure of between 1·15 and 1·25, but the replacement of a part of CaO by MgO was also undertaken. Chemical analyses of the raw materials used in this trial are given in Table 51.

Increase in the MgO content of the slag to about 15 per cent did not appreciably affect furnace operation and the fluidity of the slag. The slag/metal separation was also satisfactory. In the later part of the trial, the MgO content was increased and L.T.C. fuel was substituted for Kolsit. It was found that the furnace operation became difficult and slag/metal separation became poorer. It is felt from a study of the results that increasing the MgO content of the slag to about 16 per cent had no appreciable effects on furnace operations or on the slag fluidity. Increase in fuel consumption during the later part of the trial was primarily due to inferior properties of L.T.C. to Kolsit as a fuel. Analyses of pig iron and slag produced are given in Table 52.

The effect of variation of MgO contents in the slag at levels of 8, 12 and 16 per cent

Table 51 — Chemical Analyses of Raw Materials Employed

1. Proximate analysis of low-temperature carbonized coke (Kolsit)

Moisture %	Ash %	Volatile matter %	F.C. %
2·4	19·3	9·2	69·1

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

SiO_2 %	MgO %	Al_2O_3 %	CaO %	Fe %	S %	P %
66·3	2·2	23·3	0·7	4·2	0·6	0·028

3. Proximate analysis of a typical sample of L.T.C. (Central Fuel Research Institute)

Moisture %	Ash %	Volatile matter %	F.C. %
8·5	25·9	4·1	61·5

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	0·46

5. Analysis of iron ore (Orissa minerals)

Fe %	SiO_2 %	Al_2O_3 %	S %	P %
64·14	3·28	4·57	0·01	0·02

Table 52 — Analyses of Pig Iron and Slag

1. Analysis of pig iron

C %	Si %	S %	Mn %	P %
3·0-3·6	1·75-4·0	0·01-0·13	0·40-0·50	0·086-0·16

2. Analysis of slag

	CaO %	SiO_2 %	MgO %	FeO %	Al_2O_3 %	S %
(a)	38-42	32-35	8·5-9·5	0·5-2·0	14-16	0·35-0·40
(b)	32-41	34-37	11·5-13·5	1-5		
(c)	32-37	30-37	15·5-17·5	1-8		

in the slag with lime basicity degree around 1 : 2 was found to improve the carbon saturation in pig iron while unfavourably affecting the fluidity and replacement of CaO by MgO decreased the sulphur capacity of the slag. Non-alteration of CaO/SiO_2 ratio increased the slag volume which thereby adversely affected the fuel rate, demonstrating the futility of higher MgO contents therein beyond the optimum value established earlier.

Twenty-fourth Campaign

Based on earlier results relating to improvement of smelting efficiency with the use of fine-grained iron ore (Campaign XIX), extensive investigations were conducted to assess the effect of progressive replacement of lumpy limestone by fines on the fuel rate.

In this campaign, the burden was composed of iron ore fines (Orissa), low-temperature carbonized coke made from non-coking coals (Central Fuel Research Institute, Jealgora), dolomite and lumpy limestone which was progressively replaced by small chips and fines. The smelting trials consequently were carried out in stages with the replacement of lumpy limestone (50-75 mm.) with 25, 50 and 75 per cent fine-grained limestone (97 per cent below −6 mm.).

The chemical and screen analyses of the raw materials used in the campaign are given in Tables 53 and 54.

Table 53 — Chemical Analyses of Raw Materials Employed

1. Proximate analysis of L.T.C. (C.F.R.I.)

Moisture %	Ash %	Volatile matter %	F.C. %
8·5	25·9	4·1	61·5

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

SiO_2 %	MgO %	Al_2O_3 %	CaO %	Fe %	S %	P %
66·3	2·2	23·3	0·7	4·2	0·06	0·028

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	0·46
Bisra limestone (Orissa)	44·8	3·27	6·96	1·6	—	—

Table 54 — Screen Analyses of Raw Materials

	Above 50 mm. %	−50 mm. +25 mm. %	−25 mm. +12·5 mm. %	−12·5 mm. +6 mm. %	−6 mm. +3 mm. %	−3 mm. %
L.T.C.	6·8	66·4	21·5	3·7	0·7	0·9
Iron ore fines	—	—	2·3	26·1	26·2	45·4
Limestone (lumpy variety)	22·7	62·5	6·8	3·3	2·1	2·6
Dolomite	—	5·3	60·5	21·8	5·2	6·2
Limestone (chips and fines)	—	—	—	2·6	40·9	56·5

The furnace was operated under the following conditions:

Tuyere diameter	75 mm.
Hot blast wind volume	2300-3200 $Nm.^3$/hr.
Hot blast wind pressure	1300-2300 mm.WG.
Hot blast temperature	450-620°C.
Top gas temperature	210-450°C.

In the first stage of the trial employing lumpy limestone, the furnace operation was smooth and regular. The progressive replacement of lumpy limestone by fines did not involve any serious operational difficulty, but the exclusive use of fines caused irregular descent of the burden due to its poor permeability. Consequently, the first stage with limestone fines exclusively was abandoned due to extreme difficulties in furnace operation. The ore rate was 1·5 tonnes/tonne of pig iron which was associated with a slag volume of about 1·2-1·6 tonnes per tonne of pig iron depending on fuel rate. The slag basicity CaO/SiO_2 was maintained at a calculated figure of about 1·25, but the analytical slag basicity ranged between 0·95 and 1·1. Analyses of pig iron and slag produced are given in Table 55.

It was observed that replacement of lumpy limestone (50-75 mm.) progressively with 25, 50 and 75 per cent fine-grained limestone (97 per cent below —6 mm.) decreased the fuel rate by 10, 19 and 30 per cent, increased the daily output by 29, 41 and 42 per cent and increased the flue dust by 8, 40 and 62 per cent respectively. The decrease in the burden permeability and increase in dust make limited the employment of fine-grained ore or flux.

Table 55 — Analyses of Pig Iron and Slag

1. Analysis of pig iron

C %	Si %	S %	P %	Mn %
2·6-3·0	2·4-4·0	0·04-0·15	0·55	0·20-0·45

2. Analysis of slag

CaO %	SiO_2 %	FeO %	MgO %	Al_2O_3 %
32-38	31-38	0·5-0·8	6-7	19-21

88.1 Investigations on Briquetting

Briquetting of Beneficiated Limestone for Steel-making Purpose

The specifications for the open-hearth grade of limestone limit SiO_2 at 5 per cent max. It has become increasingly difficult to obtain such a grade of limestone and the lower available lime increases the slag volume. Beneficiation of limestone (45·5 per cent CaO, 10·5 per cent SiO_2) yielded a concentrate with 46 per cent CaO, 2-3 per cent SiO_2. As the beneficiated product is —200 mesh, it was agglomerated by briquetting with the addition of 6 per cent molasses as the binder. The briquettes were aged at room temperature for about 24 hours followed by heat-hardening to develop a thin, hard skin, which was adequate to prevent degradation during handling.

Several trial melts for different grades of steel were conducted in 250 tonnes basic open-hearth furnace of the Tata Iron & Steel Co. Ltd., Jamshedpur. The employment of beneficiated limestone briquettes (or pellets) enabled to attain higher slag basicity which would facilitate dephosphorization. The flux consumption was significantly decreased.

88.2 Malleabilization Characteristics of Low-shaft Furnace Pig Iron

The malleabilization characteristics of inherently low phosphorus pig iron produced in low-shaft furnace was further investigated based on the additions of boron with a view to shortening the malleabilization cycle. From earlier experiments, it was found that the addition of ferro-boron and aluminium in the melt shortened the annealing cycle. It was, however, experienced that the introduction of ferro-boron was associated with considerable difficulties and was costly. The commercial boric acid is cheaper than ferro-boron and the boron content of the boric acid is 17·5 per cent which is more than ferro boron. Requisite amount of boron was introduced by the addition of boric acid and aluminium to molten pig iron prior to casting.

Several castings were made containing 0·01-0·03 per cent boron and residual aluminium. In other heats with 0·03 per cent boron, the amount of aluminium was raised to 2·0 per cent. The presence of these elements has significantly lowered the heat-treatment period.

88.3 Pneumatic Method of Steel-making in Side-blown Converter

It is well known that Indian pig irons containing about 1-1·2 per cent Si and 0·3-0·4 per cent phosphorus cannot be refined into steel by either acid or basic Bessemer converter. The basic oxygen steel-making process can be applied with or without modifications, but this will necessitate the installation of a tonnage oxygen plant, the installation of which in a regional steel plant of limited capacity or in foundry for making steel casting will involve heavy capital and foreign exchange cost structure. Extensive investigations were conducted in the basic lined side-blown converter which was earlier designed and fabricated at the National Metallurgical Laboratory, employing hot metal of low and high silicon contents, with or without addition of ferro-silicon or ferro-manganese. Indian pig irons containing medium phosphorus were refined into commercial grades of steels by a pneumatic process without employing any oxygen during steel-making at all. Investigations were conducted under different operational conditions. It was fully practicable to oxidize the carbon to below 0·04 per cent, silicon to 0·02 per cent and phosphorus to 0·05 per cent. The nitrogen content of the blown metal was reasonably low. The chemical analyses of molten pig iron, blown metal, and corresponding slag are summarized in Tables 56 and 57 respectively.

88.4 Experimental Miniature Furnace for Studying Injection of Auxiliary Fuels

Injection of auxiliary fuels through the tuyeres, such as natural gas, furnace oil, oil-coal slurry and pulverized coal, etc., in the blast furnace has significantly reduced the coke rate with simultaneous increase in iron output. In order to properly understand the thermodynamics and kinetics of reactions involved in the reduction of iron ore during the use of such auxiliary fuels, an experimental miniature blast furnace was designed and fabricated at the National Metallurgical Laboratory with provisions for injecting auxiliary fuels, such as gaseous, liquid and oil-coal slurry. The effects of prepared and agglomerated burdens, viz. sintering, pelletizing, and hot-briquetting of beneficiated iron ore, on smelting can also be studied in the miniature shaft furnace. The furnace shell is cylindrical and divided into two parts to facilitate relining, with a drop bottom type. The effective height of the furnace is 130 cm. with hearth dia. of 25·4 cm., bosh dia. of 33 cm. and throat dia. of 23 cm. It has been provided with two rows of tuyeres connected to two separate and independent bustle pipes. The normal tuyeres are suspended from one bustle pipe whilst the tuyeres for injection of auxiliary fuels are connected to the other bustle pipe. The auxiliary tuyeres are set at a height of 7·6 cm. above the normal air blast tuyeres and the axes are inclined at an angle of 45° so that the air blast and the injected fuel are injected into the furnace hearth. The main air blast and auxiliary bustle pipes encircle the furnace as hexagons which are made out of round pipe of

Table 56 — Details of Experimental Blows

Heat No.	Wt. of the hot metal blown kg.	Tempe-rature of hot metal °C.	Time of blow min.	Tempe-rature of blown metal °C.	Sample	Chemical analysis (percentages)						Basicity CaO/SiO$_2$ in slag	Remarks
						C	Si	P	S	Mn	N		
23	400	1180	15	1680	Hot metal	3·19	2·28	0·28	0·065	0·51	—	1·23	Double slagging
					B. metal	0·6	0·15	0·069	0·04	Trace	0·008		
24	520	1180	35	1720	Hot metal	4·05	0·887	0·28	0·06	0·64		1·11	Double slagging — 4 kg. ferro-silicon was added after the 1st slagging. 4 kg. ferro-manganese was added before pouring in ladle
					B. metal	0·04	0·149	0·11	0·047	0·82			
25	540	1180	35	1700	Hot metal	4·00	0·73	0·28	0·06	0·33	—	1·20	Double slagging
					B. metal	0·04	0·02	0·07	0·05	0·08	0·004		
28	500	1100	25	1575	Hot metal	3·85	0·66	0·23	0·056	0·58	—	1·4	Double slagging — 4 kg. ferro-silicon was added in the beginning and 4 kg. was added after 1st slagging. 1 kg. mill-scale and 1 kg. iron ore were added in the end
					B. metal	0·57	0·41	0·05	0·026	0·90	0·005		
30	500	1190	35	1600	Hot metal	0·85	0·66	0·23	0·06	0·58	—	1·6	Double slagging — 4 kg. ferro-silicon was added after the 1st slagging. 2 kg. mill-scale added
					B. metal	0·27	0·16	0·03	0·024	0·17	0·006		

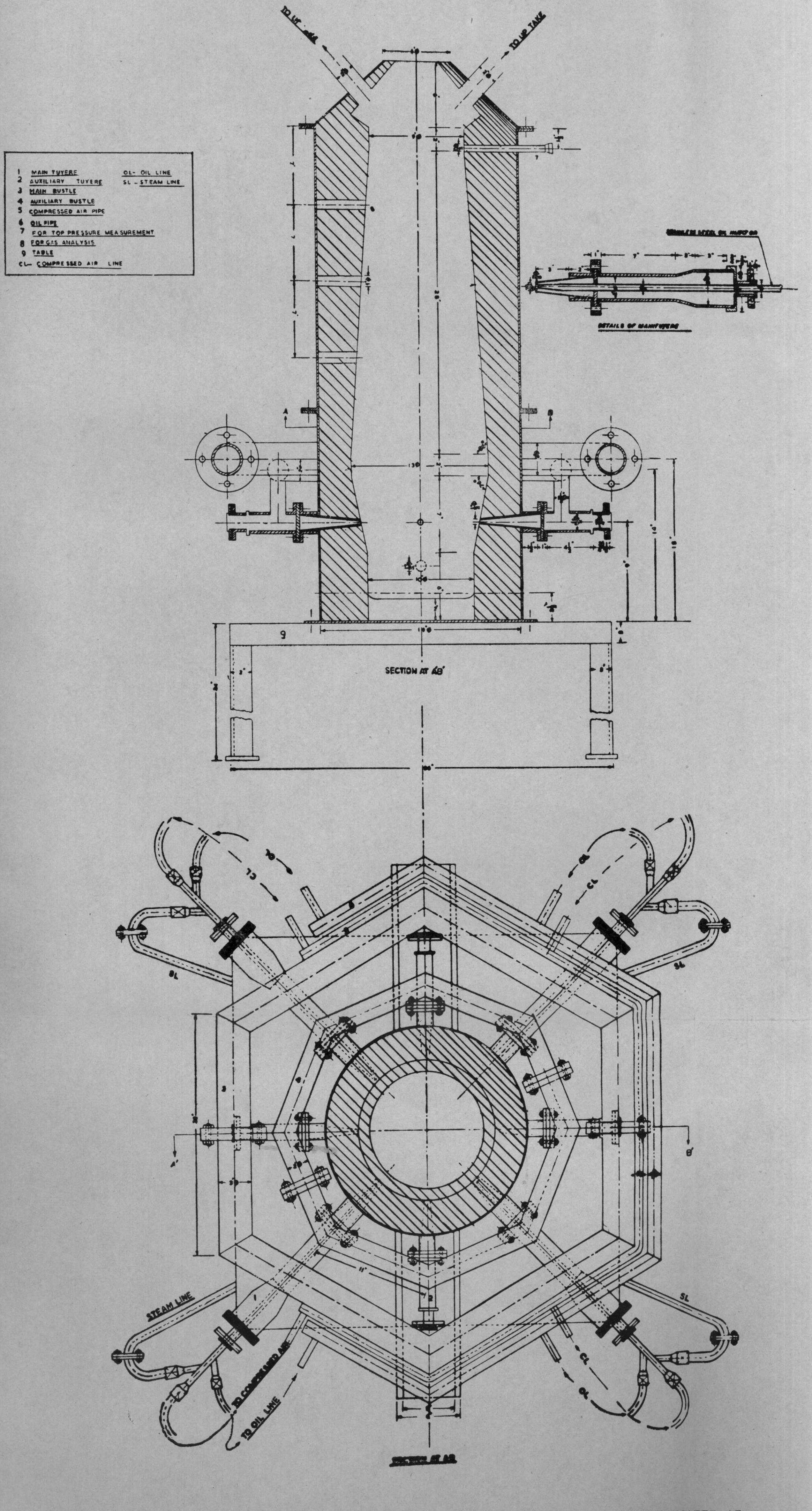


FIG. 68 — EXPERIMENTAL BABY BLAST FURNACE FOR FUEL INJECTION, DESIGNED AND FABRICATED AT THE NATIONAL METALLURGICAL LABORATORY

Table 57 — Slag Analysis

Heat No.	CaO %	SiO_2 %	MgO %	P_2O_5 %	Fe_2O_3+FeO %	Basicity CaO/SiO_2	Remarks
23	11·6	8·9	9·3	2·0	64·6	1·3	Flush slag after 10 min. blow
	9·9	7·8	10·9	1·4	79·0	1·26	Final slag
24	25·9	23·2	13·5	2·5	32·30	1·11	Final slag
25	9·02	15·7	18·8	1·71	48·35	0·58	Flush slag after 10 min. blow
	10·8	9·0	21·5	4·70	49·60	1·20	Final slag
28	13·0	30·48	15·26	3·2	35·35	0·42	Flush slag after 10 min. blow
	33·3	23·60	12·46	2·9	25·50	1·40	Final slag
30	9·82	17·44	9·48	0·75	59·44	0·56	Flush slag after 10 min. blow
	18·76	9·96	9·41	4·39	55·46	1·60	Final slag

25·4 mm. dia. and compressed air pipe is of 12·5 mm. dia. The furnace is lined with a monolithic basic refractory lining; stack of the furnace is provided with arrangements for determining the temperature gradient and distribution and sampling of the gas. The injection mechanism consists of 1·3 cm. dia. stainless steel tube placed axially in the main tuyeres, the dia. of the injection jet is 2·3 mm. The auxiliary tuyeres are, however, meant mainly for injecting either a synthetic gas mixture or oil-coal slurry. The injection mechanism is provided with facility for purging the lance with steam and atomization of oil by compressed air. The furnace assembly is shown in Fig. 68. The outer shell is cooled by spraying water.

The air blast for the operation of the furnace will be heated in a metallic tube recuperator which has also been designed and fabricated at the National Metallurgical Laboratory. The recuperator heating elements consist of 3·8 cm. dia. mild steel pipes of suitable grade. The expansions of heating elements are accommodated by corrugated type leak-proof expansion joints fastened to each element. The recuperator is lined with fire-bricks and top of it is removable to facilitate examination and relining of the recuperator. The recuperator is normally heated by an oil burner, but provision exists for utilization of the top gas for heating the air blast.

As the experimental miniature blast furnace is primarily meant for studying the fuel requirements under various operational conditions, such as oxygen-enrichment of the blast, injection of gaseous and liquid fuels, etc., the coke rate for the base 'parameter' with operations normal air blast was ascertained in the first instance. For this purpose, the furnace was operated with iron ore fines (Orissa), nut coke employing blended fluxes containing limestone and dolomite. The chemical ands creen analyses of raw materials are recorded in Tables 58 and 59.

The air blast was heated in the mild steel tube recuperator to a temperature of 240-250°C. The blast pressure was initially kept at a 25 mm.Gh which was subsequently increased to 55 mm.Gh for the desired smelting rate. Under these raw materials and operating conditions, fuel consumption amounted to 1·43 tonnes of F.C./tonne of pig iron. Analyses of pig iron and slag are recorded in Table 60.

Investigations in the miniature blast furnace employing oxygenated air blast and auxiliary fuel injections are in progress.

88.5 Utilization of Alternative Fuels for Foundry Coke for Melting in Cupola

In view of the well known acute shortage of foundry coke, investigations were

Table 58 — Analyses of Iron Ore, Limestone and Dolomite, Nut Coke and Coke Ash

1. Analysis of iron ore, limestone and dolomite

Material	Fe %	SiO_2 %	Al_2O_3 %	CaO %	MgO %	S %	P %
Iron ore (Orissa)	64·14	3·28	4·57	Trace	Trace	0·01	0·02
Limestone (Madras)	—	0·88	1·2	54·31	1·0	0·027	—
Dolomite (Assam)	—	0·4	0·6	31·3	20·7	—	—

2. Analysis of nut coke

Material	F.C. %	V.M. %	M %	Ash %	S %
Nut coke	75·1	1·8	3·0	20·1	0·5

3. Analysis of coke ash

CaO %	SiO_2 %	MgO %	Al_2O_3 %	Fe %	P %
3·8	52·08	2·12	33·0	6·0	0·69

Table 59 — Screen Analyses of Raw Materials

Materials	+50·8 mm.	−50·8 mm. +25·4 mm.	−25·4 mm. +12·7 mm.	−12·7 mm. +6·35 mm.	Below 6·35 mm.
Nut coke (Tisco)	70·5	27·6	1·85	0·35	0·60
Iron ore (Orissa)	—	4·10	23·10	31·35	40·50
Limestone (Madras)	—	1·20	51·0	46·10	1·6
Dolomite (Assam)	—	3·3	50·85	16·25	29·5

Table 60 — Analyses of Pig Iron and Slag

1. Analysis of pig iron

C %	Si %	S %	P %	Mn %
2·66	0·22	0·12	0·30	0·20

2. Analysis of slag

CaO %	SiO_2 %	Al_2O_3 %	MgO %	FeO %	S %
35·00	32·80	18·50	6·50	5·30	0·46

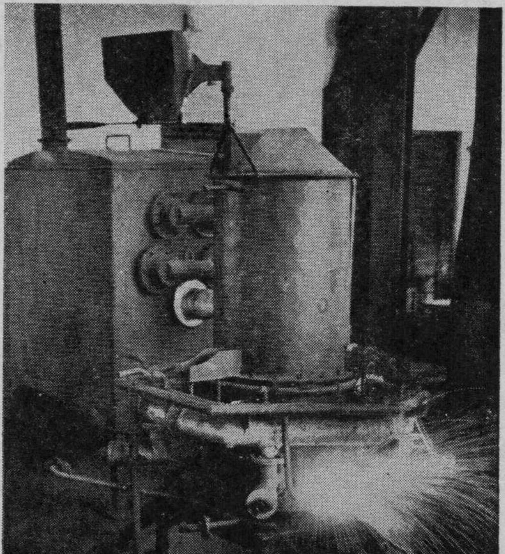


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FIG. 69 — EXPERIMENTAL BABY BLAST FURNACE FOR STUDYING THE EFFECTS OF BLAST ADDITIVES ON THE IRON SMELTING DESIGNED AND FABRICATED AT THE NATIONAL METALLURGICAL LABORATORY

conducted to assess the possibility of utilizing (i) Jhama coal (naturally burnt coke); (ii) low-temperature carbonized coke made exclusively from non-coking coals made in (a) externally heated ovens (Central Fuel Research Laboratory, C.S.I.R.) and (b) internally heated ovens,' Kolsit ' (Regional Research Laboratory, Hyderabad); (iii) coke breeze bricks, and (iv) raw lignite briquettes for melting iron in cupola. The chemical analyses of the fuels employed, their ashes and physical properties are given in Tables 61 to 63.

The cupola bed was made up with foundry coke. Foundry coke in the individual charge was progressively replaced by 25, 50 and 100 per cent by each of the different fuels mentioned above. On the basis of favourable results with Jhama coal in the 1 tonne/hour experimental cupola, industrial trials were successfully conducted in a 5 tonne/hr. cupola employing the molten pig iron for 9-12 ft. long and 6-12 in. dia. pipe and other commercial castings, which demonstrated that Jhama coal can be successfully substituted for foundry coke in individual charges.

It was also found that the low-temperature carbonized coke (Central Fuel Research Institute) in size grading of 75-100 mm. can replace a part of hard foundry coke, whilst 25-40 mm. size Kolsit was unsuitable for foundry iron smelting in cupola. Coke breeze and raw lignite could not be utilized due to their disintegration in upper regions of the cupola leading to operational difficulties and inadequate heat in melting zone.

88.6 Appraisal of Raw Materials for Iron Making

With a view to correlate the coke rate and slag volume for smelting iron in the low-shaft furnace, appraisal of raw materials consisting of determination of (i) porosity, (ii) reducibility characteristics, (iii) shatter index, (iv) abrasion index, (v) crushing strength and (vi) decrepitation behaviour of different iron ores and dissociation characteristics of several limestones collected from different parts of India was continued.

Standard shatter and abrasion tests were performed in the normal manner employing the apparatus fabricated in the laboratory. Abrasion tests were performed with iron ores of different size gradings such as (i) −25+12 mm., (ii) −12+16 mm. and (iii) −6+3 mm. It was generally found that as the particle size of ore was reduced the abrasion index also decreased. Cold crushing strength of iron ores were determined on 18-25 mm. cubes by subjecting the specimen to a compression test in a universal testing machine. Hot crushing strength of iron ores was determined by using similar cubes of iron ore which had been heated to a temperature of 400°C. A furnace was specially designed and fabricated at the National Metallurgical Laboratory, which was fitted to the universal testing machine. Extra care was taken to prevent the radiated heat to affect the oil container of the machine. Arrangements were made for determining the temperature of the iron ore specimen during the experiment. The determination of crushing strengths at room and elevated temperatures indicated their behaviour in the stack of blast furnace. These experiments were repeated by varying the temperatures of the ores from 400° to 550°C. Effects of decrepitation of ores on their crushing strength at elevated temperatures were determined.

Decrepitation characteristics of ores are important from more than one angle. The ore decrepitation characteristics can be determined in a stationary atmosphere, or one with a slight flow of air or in gaseous environments simulating the blast furnace stack atmospheric conditions.

The porosity test, which consists of successively filling the voids with known volumes of mercury or gases and noting the difference in pressure, provides a measure of available pores of the ores. The value of porosity thus

Table 61 — Chemical Analyses of the Different Fuels

	Moisture %	Ash %	V.M. %	Fixed carbon %	T.S. %
Hard coke	1·15	23·9	2·5	72·45	0·4-0·6
Jhama coal	2·5	15·7	10·8	71·0	0·5
L.T.C. (C.F.R.I.)	8·5	25·9	4·1	61·5	0·23
Kolsit (R.R.L.)	2·4	19·3	9·2	69·1	—

Table 62 — Ash Analyses of the Fuels

	SiO_2 %	Fe %	Al_2O_3 %	CaO %	MgO %	P %
Hard coke	54·0	5·0	28·7	3·04	1·95	0·30
Jhama coal	48·8	4·1	34·2	3·4	4·70	0·14
L.T.C. (C.F.R.I.)	53·8	4·6	28·0	5·7	1·9	0·80
Kolsit	66·3	4·2	23·3	0·7	2·2	0·03

Table 63 — Physical Properties of the Different Fuels

Data on	Hard coke	Jhama coal	F.R.I. (L.T.C.)	Kolsit	Coke breeze agglomerate	Raw lignite briquettes
Shatter index % on +12·5 mm fraction	99·4	89·70	91·1	83·1	94·0	97·5
Bulk density	1·28	1·35	—	—	—	—
Abrasion index on +3 mm fraction	76·9	65·0	70·8	64·1	—	70·1
Porosity %	40·0	10·0	—	—	—	—
Reactivity to CO_2	145	138	168	—	—	—
Coal value cal/kg. (air-dried basis)	6380	6890	5890	6330	—	—

Table 64 — Chemical and Physical Properties of Iron

Iron ore	State	Visual examination	Sp. gr.	Chemical analysis %							Crushing strength cold T/sq. in.	Hot T/sq. in. 400°C.	Shatter strength %+½″ fraction
				Fe	SiO_2	Al_2O_3	CaO	MgO	S	P			
Barajamda (O.M.)	Orissa	Mostly laminated	4·2	64·1	3·28	4·57	—	—	0·01	0·02	4·0	2·6-10·1	89·50
Mohindergarh area	Punjab	Massive and hard	4·7	62·6	6·65	2·1	—	—	0·03	0·13	4·8	1·7	97·40
Guntratimargoo area	Andhra	Massive and also laminated	3·9	59·8	4·0	5·9	—	—	0·05	0·12	1·52	—	96·70
Megboodabad area	Andhra	Mostly porous	5·6	63·7	3·8	2·0	—	—	0·03	0·06	3·7		98·00
Phulchoki	Nepal	Massive and hard	4·6	60·00	9·0	2·8	0·7	1·0	0·038	0·01	3·08	2·9	97·23
Chomu Moreja area	Rajasthan	Massive and hard	4·7	61·6	9·9	1·2	0·3	0·9	0·20	0·50	2·8	1·2	97·60
Barbil area	Orissa	Massive and hard	3·7	63·2	2·9	5·6	Trace	Trace	0·01	0·05	4.6	—	90·50
Barajamda (N.V.)	Orissa	Mostly laminated	3·7	61·4	1·52	3·9	0·04	Trace	0·02	0·02	3·3	1·9	96·40
Barajamda (S.L.)	Orissa	Massive and hard	4·1	61·1	0·72	1·9	Trace	Trace	—	—	5·5	2·6	98·40
Kiriburu area	Orissa	Massive and hard	4·2	64·2	1·89	1·1	—	—	—	—	—	—	95·60
Meghatuburu area	Orissa	Massive	4·2	63·2	0·52	8·48	Trace	Trace	0·016	—	—		94·17
Rajhara area	Madhya Pradesh	Massive	4·1	63·0	3·12	5·92	0·2	Trace	—	Trace	—	—	96·90
Barsua area	Orissa	Massive	4·1	52·18	4·8	11·34	0·42	0·84	0·48	0·07	—	—	96·70
Taldih area	Orissa	Massive	4·2	47·5	4·32	19·6	Trace	Trace	0·09	Trace	—	—	95·70
Gua area	Bihar	Massive	4·0	44·9	4·96	19·29	Trace	Trace	0·052	Trace	—	—	95·43
Bolani area	Orissa	Massive	4·15	56·89	5·23	7·10	0·20	0·40	0·03	0·05	—	—	95·16
Nilancha area	Warrangal Dt., Andhra Pradesh	Massive and hard	4·4	—	—	—	—	—	—	—	—	—	99·01
Bayaram area	Khammam Dt., Andhra Pradesh	Massive and hard	4·2	—	—	—	—	—	—	—	—	—	98·50
Tomka area	Cuttack, Orissa	Massive and hard	5·2	—	—	—	—	—	—	—	—	—	98·00

Ores

Abrasion strength %+¼″ fraction			Decrepitation temperature °C.	Reducibility					
				Size −½″+¼″				Size +¼″+⅛″	
				At 900°C. and 800 cc./min. of hydrogen flow		At 800°C. and 800 cc./min. of hydrogen flow		At 900°C. and 800 cc./min. of hydrogen flow	
−2+1″	−1″+½″	−½″+¼″		% Reduction	Time in min.	% Reduction	Time in min.	% Reduction	Time in min.
71·00	—	—	410/415	91·70	32	83·50	42	92·10	32
89·80	—	—	450/470	80·90	62	70·70	76	75·50	60
89·40	92·50	—	460/470	95·60	74	95·90	82	94·80	68
79·30	95·35	—	465/470	96·90	139	92·80	95	97·10	130
93·40	95·10	—	450/460	74·70	34	59·40	47	83·20	32
94·2	89·80	78·40	465/490	91·70	32	83·50	42	92·10	32
88·30	95·00	92·70	415/425	87·30	130	84·20	110	89·10	90
87·70	92·45	90·60	410/420	80·20	95	67·70	110	84·90	85
93·42	96·50	95·30	480/490	80·6	85	60·00	110	85·00	84
85·20	87·50	—	360/380	—	—	—	—	—	—
74·40	—	—	405/435	64·60	75	64·60	90	—	—
74·40		—	380/400	—	—	—	—	—	—
72·10	—	—	—	—	—	—	—	—	—
72·00		—	490/500	79·60	90	71·50	115	—	—
78·30	—	—	440/460	70·10	85	66·40	110	—	—
78·57	—	—	800	81·70	80	70·00	90	—	—
—	81·80	—	420/425	—	—			—	—
—	88·20	—	380/440	—	—	—	—	—	—
93·60	90·00	89·20	440/420	—	—	—		—	—

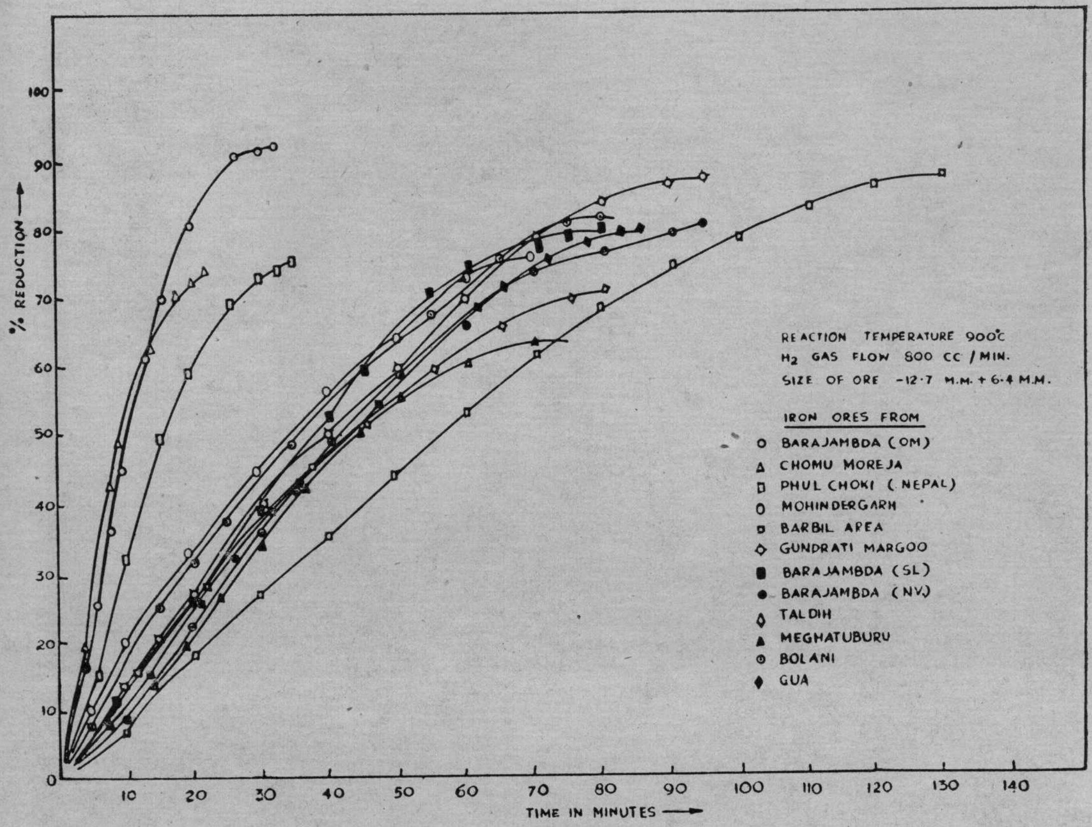


Fig. 70 — Reducibility characteristics of iron ores

determined is relative to the fluids used. Initial experiments were performed by using mercury and moisture-free iron ores (—3+4 mesh sizes). The porosity of the ore was related to the reducibility characteristics of the iron ores. The chemical analyses and physical characteristics of the ores are summarized in Table 64. The reducibility characteristics of several ores are given in Fig. 70.

It was determined that (i) as the particle size of iron ores was reduced the abrasion index decreased whilst the shatter index remained unaltered; (ii) the shape of ore particles controlled the abrasion and shatter indices, angular grains decreased the indices; (iii) ores possessing poor abrasion index possessed superior reducibility characteristics and decrepitation, and (iv) reducibility characteristics were related to porosity.

88.7 Investigation into Ingot Structures by Casting Stearine-naphthalene Ingots

This study covered the examination of small steel ingots cast by botton pouring technique which is to be preferred for casting studies of small steel ingots obtained by pneumatic steel-making processes. In laboratory studies, ingot structures were investigated by casting stearine and naphthalene ingots which simulated the structure of cast steel ingots as shown in Fig. 71. Effects of the rate of pouring, narrow and big end up pouring, top and bottom pouring, on the ingot structures were thus studied.

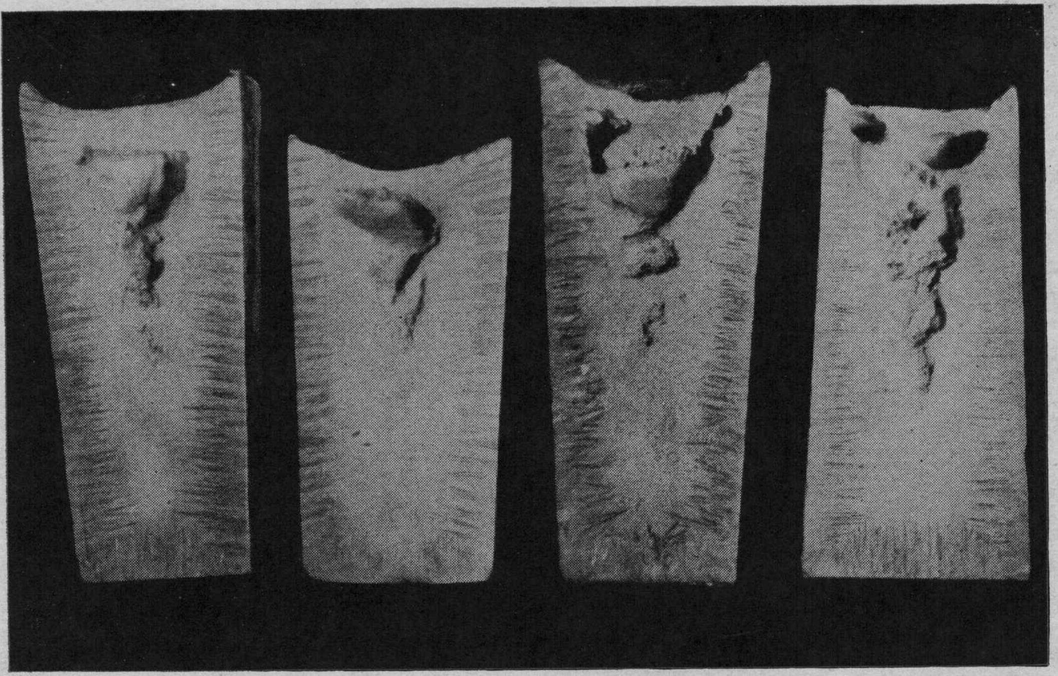


FIG. 71 — STEARINE-NAPHTHALENE INGOT STRUCTURE

88.8 Thermal Conductivity of Blast Furnace Raw Materials

In order to assess the efficiency of heat transfer and thermal exchange between the descending burden and the ascending gases in a blast furnace, the determination of coefficient of thermal conductivity of iron ores is important. The temperature distribution patterns follow somewhat identical patterns irrespective of the furnace contours which indicates that the heat transfer in the blast furnace stack is governed by known fundamental laws. It is necessary that the thermo-physical characteristics of the heated materials, i.e. its 'coefficient of thermal conductivity', is well known in addition to the formulation of requisite parameters.

Such information is not readily available in general and more so for the Indian raw materials. An apparatus for determining the coefficient of thermal conductivity was designed and fabricated in the National Metallurgical Laboratory.

The comparative method for determination of thermal conductivity is based on a one-inch cube sample. A uniform heat flow is established through two or more samples. The temperature drop, heat flow and the thermal conductivity depend on the equation:

$$Q = \frac{K_1 A_1 \Delta T_1}{\Delta L_1} = \frac{K_2 A_2 \Delta T_2}{\Delta L_2}$$

where Q = heat flow; K = thermal conductivity; A = mean cross-sectional area; ΔT = temperature gradient; and ΔL = distance between the thermocouples on the specimen.

Knowing the conductivity of one, conductivity of the other is determined. In this method, errors are introduced mainly by lateral heat flow and interfacial resistance between the samples.

Lateral heat flow is reduced by employing a heated guard cylinder around the samples and the two end heaters. The temperature of the guard cylinder is controlled by five individual heating elements. The temperature gradient in the tube is adjusted to match the temperature gradient in the specimens, thus reducing heat flow to a minimum. Interfacial resistance is lowered by polishing the surface of the samples and introducing a thin silver foil in between the samples, thus forming a good interfacial contact.

The apparatus was standardized by using samples of known thermal conductivity. Further investigations on the subject are currently in progress.

88.9 Distribution of the Burden in the Stack of Low-shaft Furnace

Even distribution and descent of raw materials in the blast furnace stack ensure uniformity of gas flow and improves the fuel rate. The distribution of different burdens was mapped in a half-scale model of the low-shaft furnace vertically sectioned. The nature of burden distribution in the stack depends upon the character of the raw materials, e.g. variations in size, shape, density, moisture content, charging arrangement vis-à-vis furnace contour, distributor, and equalizer. Such variables were first studied individually and later in groups, following which distribtuion patterns were plotted. The form of the stockline contour, which can be deduced from a knowledge of the trajectories, largely determines the initial burden distribution, which generally persists throughout the furnace shaft.

Firs tset of experiments showed one side-slope type contours by keeping the rotation of the charging drum in one direction. The clockwise and anticlockwise rotations of the sluice drum gave a uniform 'M' type contour. The typical distribution of burden using Orissa iron ore, nut coke (Durgapur) and limestone (Madras) is shown in Fig 72. The blowing of air as done in a normal furnace depicted a more realistic and clear picture of the M' type contours.

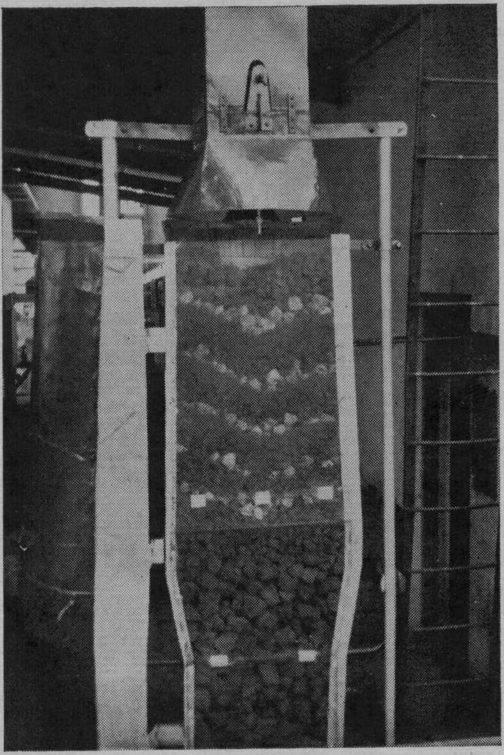


FIG. 72 — HALF-SCALE MODEL OF 15-TON PER DAY LOW-SHAFT FURNACE FOR STUDYING DISTRIBUTION CHARACTERISTICS OF THE BURDEN

89.0 Electrolytic Manganese Metal Pilot Plant

During the period under review, regular production of manganese metal was continued on pilot plant scale for meeting the requirements of different research and development projects underway at the National Metallurgical Laboratory, such as the development of thermostatic bimetals, development of

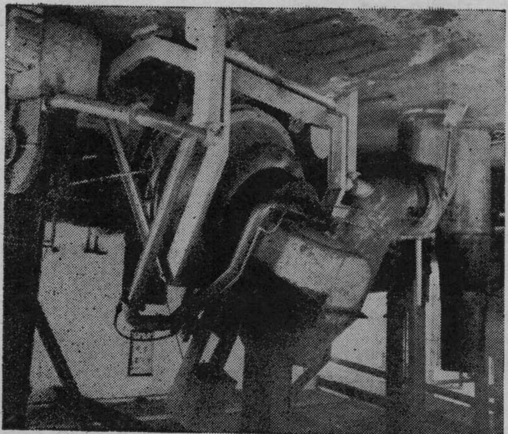


FIG. 73 — ROTATING FURNACE FOR IRON-MAKING FROM IRON ORE FINES, LIMESTONE DUST AND COKE BREEZE

magnetic materials, nickel-free coinage alloy, manganese brasses, nickel-free austenitic stainless steel, etc.

The 100 lb. per day manganese metal pilot plant was also put into intermittent operations during the year. It was found that the overall design of the plant including the regeneration plant and electrolytic cell was excellent; the pilot plant operated smoothly and most satisfactorily. Circulation of the cold water from the water-chilling unit through the lead coils inside the cells maintained the cell electrolyte constantly at room temperature during continuous operations of the pilot plant. The rectifier also, which had earlier given endless trouble, now gave good service. During pilot plant operations, a current efficiency of the order of 60 per cent was generally obtained.

Enquiries from outside parties for supply of manganese metal were continued to be received and, wherever possible, after meeting the research needs of the laboratory, some electrolytic grade manganese metal was supplied. Besides regular production of manganese metal in the pilot plant, studies on the structure of electrodeposited manganese and the influence of addition agents on the structure and current efficiency of deposition were taken up.

89.1 Electrolytic Manganese Dioxide Pilot Plant

The electrolytic manganese dioxide pilot plant of 100 lb. daily capacity was successfully intermittently operated. The regeneration plant for the leaching of reduced ore in the spent liquor and purification of the resultant solution is common for both the manganese metal and dioxide pilot plants. The electrode steam boiler continued to give good service. With steam passing at 30 lb. per sq. inch pressure through the lead coils inside the cell, the cell electrolyte could be heated to 90°C. in about half an hour and well maintained at that temperature range during continuous operations of the electrolytic cell. Lead-antimony alloy anodes were used for the production of electrolytic manganese dioxide which contained traces of lead. With increase in the time of deposition on each anode, the lead content of the deposit decreased. A current efficiency of the order of 90 per cent was generally obtained during regular production cycles.

90.0 Pilot Plant for Hot-dip Aluminizing of Steel

Atmospheric Corrosion Resistance of Aluminized Steel Wire in Saline Conditions at Visakhapatnam

Field tests of aluminized steel wire at Visakhapatnam have been in progress since December 1960. Comparative loop resistance values in drums on aluminized 150 lb./mile wire and galvanized 300 lb./mile wire erected side by side on 30 December 1961 are as follows:

	20-12-61	3-7-62	31-12-62	28-8-63
Aluminized wire	44	58	60	70
Galvanized wire	22	27	55	30

One and a half ft. long bits of the aluminized and galvanized line wire exposed to saline atmosphere at Visakhapatnam since 30 December 61, cut on 24 August 1963, were received for examination.

Representative samples were boiled in distilled water to estimate soluble corrosion products, stripped in 20 per cent NaOH solution to determine the weight of the coated metal and the amphoteric oxide, and finally treated with conc. HCl to evaluate the weight of alloy layer. Results are given below:

	150 lb./mile A.I. wire	300 lb./mile G.I. wire
1. Wt. of soluble corrosion products	51 mg./ft.	130 mg./ft.
2. Wt. of Al or Zn and their soluble oxides	75 mg./ft.	78 mg./ft.
3. Wt. of intermediate alloy layer	468 mg./ft.	1004 mg./ft.

Detailed chemical analysis report on the sample is given below:

Table 65 — Chemical Analysis of the Sample

mg./ft.	150 lb./mile A.I. Wire	300 lb./mile G.I. Wire
Fe_2O_3	419	561
Al	2·8	—
Al_2O_3	47·4	—
Zn	—	11·51
ZnO	—	125·5

Microstructure of transverse sections shown in Fig. 74 clearly indicated that galvanized wire had deteriorated 3 times more than the aluminized wire in terms of penetration of corrosion pits. Alloy layer still present in the aluminized wire at the end of 18 months had also fully disappeared by then.

Aluminizing of High Carbon Patented Wire

The work was carried out for Messers Usha Martin Black (Wire Ropes) Ltd., Ranchi. 0·0546 in. dia. wire of 58 TSI strength on aluminizing had a dia. of 0·056 in., coating thickness of 0·0007 in., tensile strength of 53 TSI and successfully passed the specified wrapping test. The surface was a bit rough and the wire could not be drawn for further improvement in the surface smoothness.

Aluminizing of Hardware for Railway Electrification

Both steel and malleable cast iron samples were aluminized for EIC (OME Design), Railway Electrification, Indian Railways, Calcutta, in July 1963.

Aluminizing of Bunsen Burners

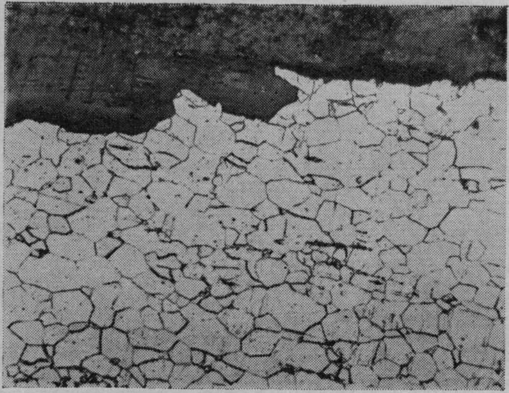
Experiments on aluminizing of bunsen burner tubes were carried out for Department of Industries, Government of the Punjab. Aluminized steel burners were also given a performance test under usual chemical analytical laboratory atmosphere in comparison with the electroplated steel and brass burners. After only 70 hr. operations, the electroplated burners were fully oxidized at the flame end whereas there was no effect whatsoever on the aluminized burners. The brass burners showed signs of dezincification only after 200 hr. of test. The trial was continued to complete the 1000 hours' test cycle.

Fig. 75 shows microstructure of the burners used in the investigation.

Aluminizing of Auto-mufflers in EME Workshops

Capital estimates of the plant, equipment and operational costs break-up were prepared and communicated to the EME Workshops as detailed below:

Auto-muffler, silencer and exhaust systems were aluminized piece by piece only as far as the current designs were concerned, i.e. the outer shell, inner passage tube, and covers and nipples, etc., had to be aluminized separately. The assembly should be done



Aluminized wire after 20 months' exposure at Visakhapatnam. Etched in nital ×450 ⟶

Galvanized wire after 20 months' exposure at Visakhapatnam. Etched in nital ×450 ⟶

FIG. 74 — SHOWS THE DEPTH OF CORROSION IS THREE TIMES MORE IN THE GALVANIZED WIRE IN EQUIVALENT PERIOD UNDER IDENTICAL CONDITION

by welding with the argon-arc process. If auto-muffler designs are modified so as to permit draining of degreasers, water, acid, flux, and molten aluminium, it may be possible to minimize the welding if not avoid it altogether.

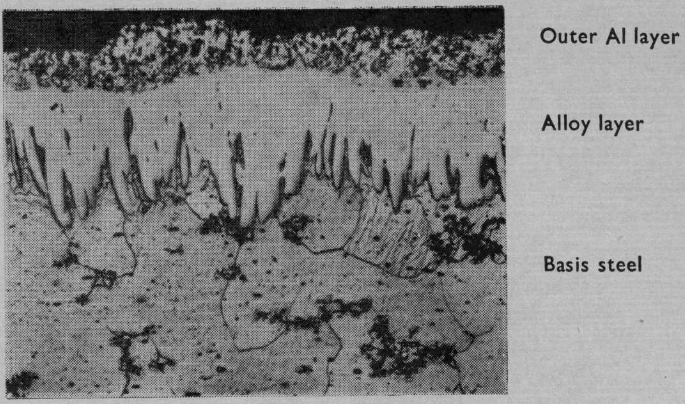
The aluminizing plant would consist of the following essential processing units:

1. Degreasing tank 6 × 4 × 5 ft. M.S. G.I.	Rs. 1,000
2. Two wash tanks (cold water), M.S.	Rs. 3,000
3. Rubber-lined pickling tank, M.S.	Rs. 2,000
4. Rubber-lined copper sulphate tank, M.S.	Rs. 2,000
5. Linseed oil tank, M.S.,	Rs. 1,500
6. Drying oven, M.S.	Rs. 2,000
7. Oil-fired aluminium melting furnace, M.S.	Rs. 10,000
8. Temperature controller, M.S.	Rs. 10,000
9. Centrifuge	Rs. 5,000
	Rs. 36,500

Cost break-up of aluminizing one 3-ton Dodge truck auto-muffler would be as follows:

	Paisa
1. Degreasing	10
2. Washing	2
3. Pickling	30
4. Copper precoat	5
5. Linseed oil	20
6. Aluminium	160
7. Fuel for aluminizing	10
8. Electric power centrifuging	5
	Rs. 2.42

Argon-arc welding is somewhat expensive; the cost of argon gas consumption and stainless steel filler rods may amount to Rs. 1.50 per muffler. The argon-arc welding unit itself may cost about Rs. 25,000.



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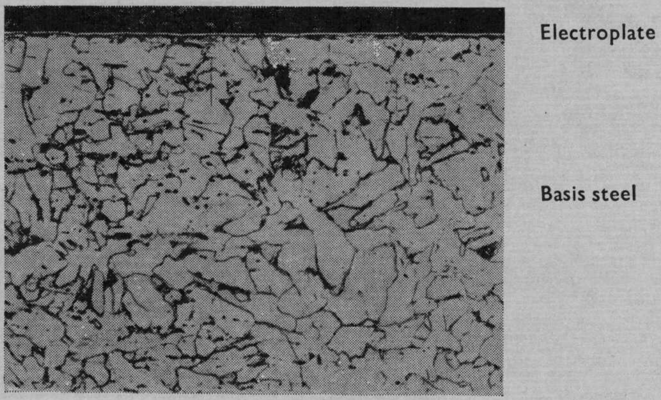

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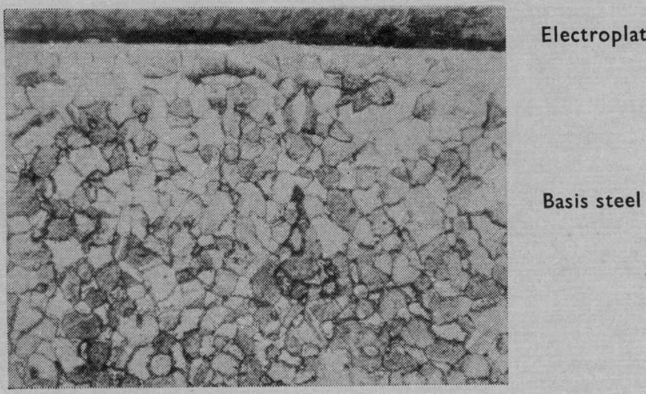

Etched in $FeCl_3$ ×250

FIG. 75 — MICROSTRUCTURE OF HOT-DIP ALUMINIZED (TOP), ELECTROPLATED STEEL (MIDDLE) AND ELECTROPLATED BRASS (BOTTOM) BUNSEN BURNER TUBES IN TRANSVERSE SECTION

Hot-dip Aluminizing of M.S. Angles

Mild steel angles analysing C, 0·25; Si, 0·11; Mn, 0·6; S, 0·03 and P, 0·035 per cent were aluminized during the operations of the aluminizing pilot plant. Mechanical properties before and after aluminizing the steel angles are given below. Coating was carried out with a bath temperature range of 750-800°C. varying the dipping time from 1 to 6 min. One and a quarter in. angles of 8 in. length were used. Section thickness was 0·136 in.

Sample	Dipping time min.	Yield stress TSI	Max. stress TSI	Elongation %
Uncoated M.S. angle	—	21·53 L.Y. 21·90 U.Y.	31·38	28·1
Aluminized M.S. angle	1	20·07 L.Y. 21·11 U.Y.	29·27	21·85
do	2	20·07 L.Y. 21·11 U.Y.	29·58	25·75
do	3½	18·55 L.Y. 19·49 U.Y.	26·38	25·75
do	4	19·28 L.Y. 19·95 U.Y.	28·06	21·85
do	5½	18·70 L.Y. 19·81 U.Y.	28·95	21·85

Aluminizing of Tubes

One hundred and fifty-two steel tubes were aluminized successfully for heat-resistant applications on behalf of Messrs John Miles & Partners (London) Ltd. for I.C.C. Ltd., Ghatsila, which was indeed the first large-scale assignment for steel tube aluminizing at the National Metallurgical Laboratory and which was successfully completed.

The pilot plant for hot-dip aluminizing of steel wire and other products was constantly in operation for undertaking aluminizing trials and for training of personnel of industrial firms to whom this process has been released by the National Metallurgical Laboratory.

91.0 Pilot Plant for Refractories

Facilities for determining physical properties, such as apparent porosity, bulk density, etc., were provided in the pilot plant and a Ro-tap sieve shaker was installed for sieve analysis. The installation of a twelve-point temperature recorder and thermocouples for recording temperature of a 7-ton capacity down-draft kiln in the pilot plant was completed. Arrangements were being made to install an 18-point temperature indicator for the tunnel dryer. Design of the ceramic-cum-metal recuperator for the down-draft kiln was completed and fabrication work was taken up.

(1) *Comparative Study of Some Methods of Compaction of Mixes of Dry Press Consistency*

This work has now been completed and a full report prepared on the subject containing the results of research investigations on the comparative study of some methods of compaction of mixes of dry press consistency. Experiemntal procedure and certain properties of the compacts justified the following conclusions:

(i) Under various methods of compaction, the increase in bulk density of dry press mixes followed the relationship

$$\frac{dBD}{dP} = K(BD-BD_P)$$

where dBD was the increment in the bulk density for increment in the compacting work factor dP; BD was the maximum possible increment in bulk density; BD_P was the increment in bulk density under pressure P, and K was the proportionality constant.

(ii) In different methods of compaction studied, two mechanisms A and B appear to govern the increase in bulk density.

(iii) When the mix is compacted under its own weight, the influence of mechanism A predominates over that of mechanism B; its influence decreases, however, the increase in the height of the compact.

(iv) When the mix is compacted under light blows, mechanism B is slightly more predominant than mechanism A which increasesas the height of the compact increases.

(v) In one-stage pressing, the preponderance of mechanism B over mechanism A is very marked; mechanism B is responsible for nearly all the increase in the bulk density. The results, however, suffer from the fact that the assumed initial B.D. may not be the true initial B.D.

(vi) When the mix is tamped under high intensity blows, most of the increase in B.D. appears to be due to mechanism A; this aspect, however, needs verification and is also subject to the same discrepancy regarding initial B.D. as mentioned in the previous conclusion.

(2) *Comparative Study of Hydraulic Press and Pneumatic Hammer as Brick Forming Machines*

The comparative study of various methods of compaction of mixes of dry press consistency showed that there was a definite relationship between the increase in compacting work factor and increase in the B.D. of the mix. It was shown that under similar experimental conditions, bumping under the mixes' own weight gave the least increase and compacting under heavy hammer impacts gave the highest increase in bulk density; the latter gave the maximum of 25 per cent increase in B.D. while one-stage pressing yielded a maximum of 19 per cent increase. The other advantages of tamping under pneumatic hammer are the low capital cost of the hammer and its effective practical utilization for making blocks of large dimensions. Comparative study of the two methods of fabrication (1) under one-stage pressing and (2) employing a pneumatic hammer for press work was, therefore, considered well worth while.

Experimental procedure — Two series of experiments were accordingly planned. In the first series electrical energy consumed by the two modes of pressing for the fabrication of $9\times4\frac{1}{2}\times3$ in. compacts was measured. In the second series, $9\times4\frac{1}{2}\times3$ in. compacts made by the two methods were tested for their homogeneity, density, strength and granulometric compositions.

Consumption of Electrical Energy

(a) *One-stage pressing* — The 500-ton hydraulic press and its attachments were used. The pressing cycle starting from the conveyance of the mix and ending up with the ejected compact was divided into 4 stages for experimental convenience, these stages being (i) conveyance of the mix (charging box) from the charging end to the brick die, which coincided with the down travel of the plunger until it just touched the surface of the mix in the die, (ii) the actual application of the pressure on the mix, (iii) upward travel of the plunger coinciding with the back travel of the charging box, and (iv) ejection of the compact from the mould. The time and electrical energy consumed in each stage were determined with a stop watch, reading up to 0·1 second and a kilowatt-hour meter reading up to 4·2 watt-hours. Ten compacts of $9\times4\frac{1}{2}\times3$ in. were pressed at various pressures in the range of 5500 p.s.i. to 13,400 p.s.i.

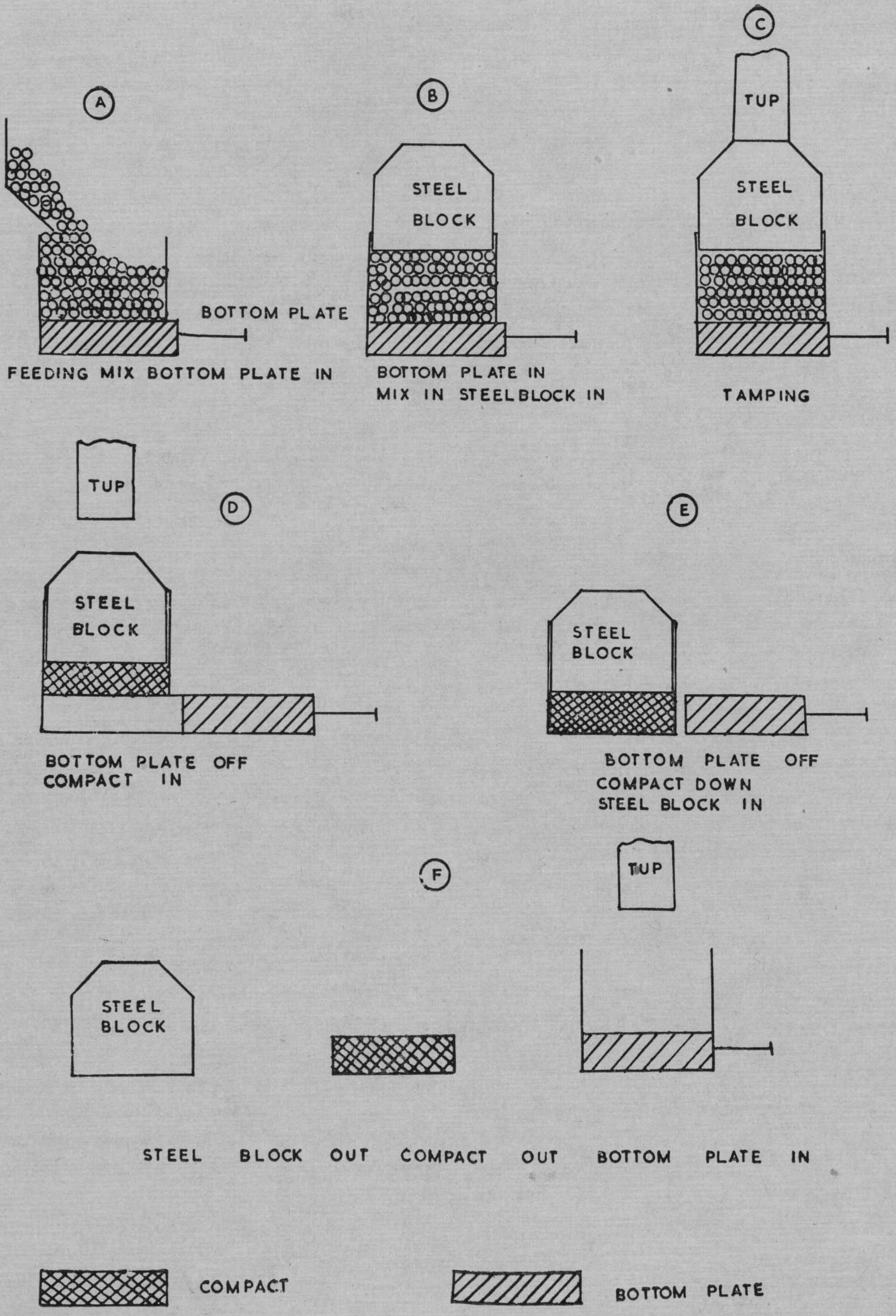

Fig. 76 — Tamping cycle of compact under hammer

(b) *Hammer* — In the pneumatic hammer and mould set-up, the mould was held on a bottom support in which a manually operated sliding steel block with polished surface formed the bottom of the die; this block was actuated by a threaded rod running in a threaded support. At the commencement of the tamping cycle, the hammer was kept running in idle position, the bottom block was slid in and a weighed quantity of mix was introduced into the die. Another hardened steel block having a sliding fit in the die was placed on the mix. The hammer was then actuated to deal blows on the top of the block at a preset intensity. After a definite number of blows had been given, the hammer was brought to the idle position, the bottom block was retracted to free the compact and a light blow from the hammer was given to release the compact which was later pushed out by the sliding bottom block. The entire cycle is shown schematically in Fig. 76.

For determining the energy consumption, the entire cycle was divided into 3 parts: sliding of the bottom block, introduction of mix and placing the top block, the retraction of the bottom block and giving the ejection blows. Ten compacts of $9 \times 4\frac{1}{2} \times 3$ in. were tamped at various intensitities 2a, 2b and 2c marked on the dial. The time and electrical consumption at each stage were measured with a stop watch reading up to 0·1 second and a kilowatt meter reading up to 5·5 watt-hours.

Physical properties of the bricks made by the above two methods were studied as follows: The green and dry compacts were observed for lamination, if any. Green compacts were cut into sections as shown in Fig. 77, weighed and kept in container and dried at 110°C. The percentage loss in weight in each case was calculated. The cut sections were subjected to wet sieve analysis using B.S.S. sieves to observe the ultimate granulometric composition. Compacts dried at 110°C. were cut into $4{\cdot}5 \times 2{\cdot}5 \times 2{\cdot}5$ in. size approx. and tested in the Avery universal testing machine for crushing strength. Modulus of rupture of the specimens $9 \times 2{\cdot}6 \times 1{\cdot}5$ in. dried at 110°C. was determined under Riehle automatic testing machine, keeping a span of 6 in.

Energy consumed for forming compacts, crushing strength and modulus of rupture are shown in Table 66. Bulk density of the compacts pressed under hydraulic press with various pressures and tamped under hammer at intensities 2a, 2b and 2c are shown in Table 67.

The study of the results indicated that the hammer and hydraulic press were fully comparable as brick forming plant machines. When very high pressures and high intensity tamping were compared, the hammer appeared to have advantage over the press in energy consumption. The compacts made under hammer appeared to possess lower compression strength than those made under the hydraulic press and with the same granulometric composition; hydraulic press compacts possessed higher bulk density than the hammer compacts.

The hammer pressing appeared to be an entirely satisfactory equipment for producing large refractory blocks. Further study to find out a rational formula covering the size of the hammer with the optimum size of the furnished pressed product is in progress. Steps were taken to see if the steel block between the tap and the mix in the mould could be avoided to increase the overall efficiency of the process.

(3) *Forsterite Project*

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) Bricks were made at various pressures and tamped under hammer with different grain sizes of rocks by incorporating dead burnt, raw and light calcined magnesite. The bricks were fired to 1500° and 1600°C. with 5 hours' soaking.

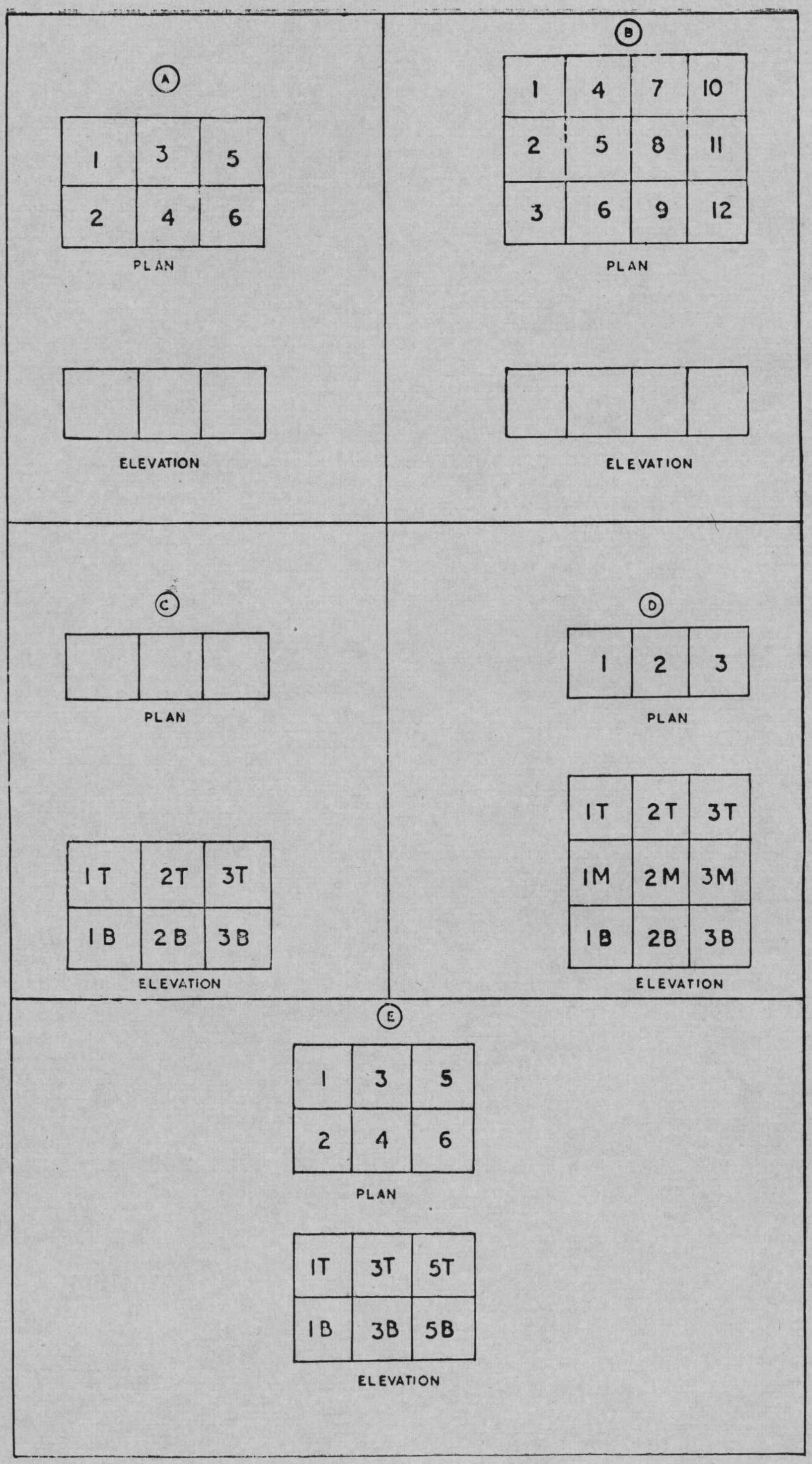

Fig. 77 — Brick cutting section

Table 66 — Time and Electrical Energy Consumed in Forming Compacts and Compression Strength and Modulus of Rupture of the Compacts Dried at 110°C.

(a) *One-stage Pressing*

Pressure in tons	Pressure in p.s.i.	No. of compacts pressed per hour	Time and energy consumption at each step								Energy in idle running kWh.	Total energy consumed per compact kWh.	Modulus* of rupture p.s.i.	Compression** strength p.s.i.
			Step I		Step II		Step III		Step IV					
			Time in sec.	Energy kWh.	Time in sec.	Energy kWh.	Time in sec.	Energy kWh.	Time in sec.	Energy kWh.				
100	5,500	40	37·5	0·0315	7·3	0·0084	13·2	0·03	8·83	0·010	0·02	0·10	n.d.	n.d.
120	6,670	40	37·4	0·030	9·0	0·010	13·8	0·033	9·4	0·010	0·017	0·10	44	203
150	8,335	42	38·0	0·030	11·6	0·0126	12·7	0·031	9·3	0·092	0·017	0·10	n.d.	n.d.
180	10,000	40	37·0	0·0298	10·6	0·013	11·75	0·028	10·26	0·0118	0·018	0·10	46	213
200	11,000	43	37·2	0·0294	11·2	0·0146	13·1	0·031	10·00	0·011	0·014	0·10	n.d.	n.d.
240	13,340	40	37·7	0·0300	15·3	0·019	13·9	0·0325	10·35	0·010	0·019	0·11	54	219

(b) *Hammer*: 20 *blows wielded uniformly on each compact*

Tamping position	No. of compacts made per hour	Time and energy consumed at each step						Total energy consumed per compact kWh.	Modulus* of rupture p.s.i.	Compression** strength p.s.i.
		Step I		Step II		Step III				
		Time in sec.	Energy kWh.	Time in sec.	Energy kWh.	Time in sec.	Energy kWh.			
2a	60	28·1	0·018	6	0·015	15·9	0·087	0·12	42	111
2b	60	39·4	0·036	6	0·014	14·6	0·080	0·13	45	115
2c	60	39·0	0·034	6·1	0·014	14·9	0·082	0·13	58	136

*On specimens 9×2 in. (broad)×1·6 approx. depth — effective span 6. in.

**On full brick standing on 4·5×2·5 in. face.

Table 67 — Bulk Densities of Compacts Dried at 110°C.

Hydraulic press			Hammer press			
				Tamping intensities		
Pressure imposed tons	Pressure in p.s.i.	Bulk density gm./cc.	No. of blows	2a B.D. gm./cc.	2b B.D. gm./cc.	2c B.D. gm./cc.
100	5,500	1·91	2	1·67	1·65	1·69
120	6,670	1·95	3	n.d.	1·76	n.d.
150	8,335	1·98	5	1·83	1·84	1·86
180	10,000	1·99	10	1·94	1·96	1·98
200	11,000	2·00	15	1·99	2·03	2·06
240	13,340	2·03	20	2·04	2·08	2·10
			25	2·07	2·10	2·14
			30	2·10	2·13	2·16
			35	2·11	2·15	2·13
			40	2·14	2·17	2·20
			45	n.d.	2·19	2·21
			50	n.d.	2·20	2·21
			55	n.d.	2·20	2·22

(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 successfully 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, attempts were undertaken to improve the bulk density and other characteristics of the finished products by incorporating certain percentage of forsterite grog and increasing MgO content in the batch compositions.

Results

1*st series* — Bricks were pressed under various pressures of 5000, 10,000 and 14,000 p.s.i. and fired at 1500° and 1600°C. The properties of the bricks pressed under 14,000 p.s.i. and fired at 1600°C. have been given in Table 68. 'a' denotes batches with the

Table 68 — Properties of Forsterite Bricks Pressed at 14,000 p.s.i. and Fired at 1600°C.

Properties	Batch numbers											
	a^1	a^2	a^3	a^4	b^1	b^2	b^3	b^4	c^1	c^2	c^3	c^4
App. porosity, %	35	32	30	31	34	33	37	32	32	34	23	24
Bulk density, gm./cc.	2·18	2·2	2·31	2·42	2·35	2·22	2·23	2·27	2·26	2·19	2·25	2·47
Weight loss, %	15	15	14	14	29	29	29	30	23	24	23	24
Volume shrinkage	11	13	12	12	14	14	13	14	13	13	13	—
Crushing strength, lb./sq. in.	870	1792	1548	3045	1068	1901	n.d.	4212	910	834	3041	n.d.

Table 69 — Physical Properties of Forsterite Composition with Dead Burnt and Light Calcined Magnesia

Specimen No.	Volume shrinkage %	App. porosity	Bulk density	Cold crushing strength	Modulus of rupture	Spalling	General conditions after firing
M1	—	28	2·38	—	—	—	Fair
M2	12	28	2·40	2300	1170	4	Fair
M3	12·5	29	2·38	2100	777	3	Fair
M4	12·5	28	2·39	1800	712	4	Good
M5	13	28	2·39	2700	785	3	Good
M6	13	28	2·39	2100	847	4	Good
M7	14	27	2·40	2700	849	3	Fair

addition of dead burnt magnesite, 'b' denotes batches with the addition of raw magnesite, and 'c' denotes batches with the addition of light calcined magnesite. Superscripts 1, 2, 3 and 4 denote the mesh size of serpentine rock —5 mesh, —10 mesh, —20 mesh and —48 mesh B.S.S.

In the second series, packing density of the mixes was determined, which varied from 1·79 gm./cc. to 2·09 gm./cc. The batches with bulk density of 2·00 gm./cc. and above were selected for making bricks with the additions of dead burnt magnesite, sulphite lye and water. These bricks were pressed under 14,000 p.s.i. and fired at 1550°C. The apparent porosity of the specimens varied from 28 to 32 per cent and bulk density from 2·24 to 2·35 gm./cc.

3rd series — Physical properties of the bricks are given in Table 69.

Physical properties of the bricks of the 4th series are given in Table 70.

These pilot plant scale trials indicated that forsterite bricks of acceptable physical and chemical characteristics could well be made from serpentine by blending proper grain sizes of the rock and adequate quantities of magnesite, compacting the mix at a reasonably high pressure and firing them, following a controlled heating schedule, to 1600°C. Incorporation of magnesia should preferably be done in the form of a mixture of light and dead burnt magnesite. Pilot plant scale production for subsequent *in situ* operational trials in the Bhilai Steel Plant of Hindustan Steel Ltd. awaits requisite supplies of raw material from different parts of the country.

Table 70 — Physical Properties of Forsterite Bricks Made with Grog and Additional MgO

Batch No.	Weight loss %	Volume shrinkage	Apparent porosity	Bulk density gm./cc.	Crushing strength p.s.i.
F1	N.D.	N.D.	25	2·52	N.D.
D-1	N.D.	N.D.	—	—	N.D.
X-2	N.D.	N.D.	—	—	N.D.
Y1	15	10	35	2·14	1300
Y2	13	9	35	2·19	1200
Y3	21	28	32	2·3	2800
Y4	19	20	29	2·39	3000
Y5	18	13	33	2·23	1000
Z1	—	—	34	2·23	2000
Z2	—	—	32	2·27	2100
Z3	—	—	33	2·25	2200
Z4	—	—	31	2·31	2200
Z5	—	—	29	2·40	2800

92.0 Pilot Plant for Production of Magnesium Metal

The investigation was taken up with a view to produce magnesium metal by silicothermic reduction of calcined dolomite. The pure magnesium so obtained will find varied applications, particularly for the production of light structural alloy, coinage alloys and for defence and ordnance requirement.

An oil-fired furnace was installed having an effective heating zone of four feet length with three feet width and two and a half feet height and capable of reaching a maximum

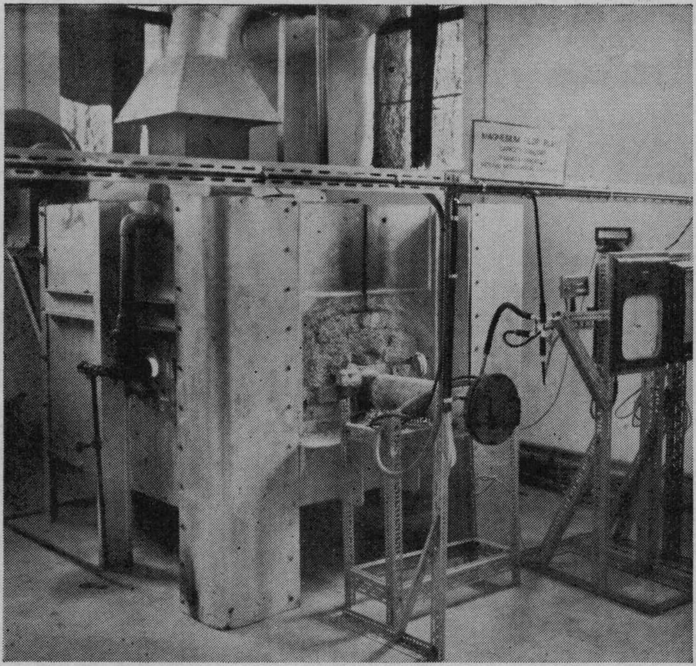

FIG. 78 — A VIEW OF THE PILOT PLANT FOR THE PRODUCTION OF MAGNESIUM METAL (CAPACITY 25 LB./DAY) INSTALLED AT THE NATIONAL METALLURGICAL LABORATORY. THE PLANT HAS BEEN DESIGNED AND FABRICATED AT THE NATIONAL METALLURGICAL LABORATORY

temperature of 1300°C. It was complete with a blower, oil tank, oil preheater and two burners on the sides.

A double conical blender of 3 c.ft. capacity for intimately mixing ground calcined dolomite, ferro-silicon and fluorspar was also installed as the charge required to be well mixed prior to briquetting.

A speedivac oil-sealed rotary mechanical pump of capacity 450 litres per minute was used for evacuating the retort chamber. Vacuum lines were fabricated with standard $\frac{1}{2}$ in. copper tubes. Vacuum valves were provided wherever necessary. A four-point temperature recorder was installed to record the temperatures at different points, such as the furnace temperature, charge temperature, condensation temperature and retort temperature. Vacuum gauges were provided to note the pressures at different points in the system. A compressor type of pump capable of providing a pressure of about 30 mm. of Hg at its suction end was connected in parallel to the vacuum retort for initial removal of moisture and other volatiles, while the charge of briquettes was at a lower temperature of about 600-700°C.

A tablet-making press was installed for pressing the dolomite mix into compacts of 1 in. dia. and $\frac{1}{2}$ in. thick. Tablets were charged into the vacuum retort made of high nickel alloy with the help of long-handled scoops. The charge was then pushed with a pusher for closer packing. The contents were discharged at the end of the experiment by using the same scoop and pusher.

It was initially observed that the flames from the burners impinged directly on the retort thereby producing hot spots and weakening the walls of the retort and its life. Baffle wall was built to eliminate the possibility of the flame directly striking the retort.

The sample of dolomite obtained from Messrs Bengal Lime & Stone Co. from their quarries was taken up for pilot plant production of magnesium metal. The dolomite sample analysed as follows:

		Per cent
CaO	...	29·92
MgO	...	22·20
L.O.I.	...	45·01
Acid-insolubles	...	2·20
Alkalies	...	0·10

Samples were received in $\frac{1}{2}$-$1\frac{1}{4}$ in. size and were calcined in a loaboratory type shaft kiln to 1000°C. to eliminate moisture and carbon dioxide. The calcined product was crushed to —100 mesh (B.S.S.) size in a laboratory type Raymond impact pulverizer. Ferro-silicon and fluorspar were also crushed to fine size (—200 mesh B.S.S.). Weighed quantities of calcined dolomite, ferro-silicon and the catalyst were mixed in the proportion of 100: 20:1 in a blender and briquetted.

The mixed charge was initially pressed into bricks and broken to a size of $\frac{1}{2}$-$1\frac{1}{4}$ in. Fines were pressed into bricks again. Reactions were carried out between 1100° and 1150°C. for 4 to 6 hours at pressures better than 100 microns. The temperature of magnesium deposits was observed to lie in the temperature zone of 480-320°C. A separate alkali condenser was placed beyond the magnesium condenser and alkalies were found deposited at the zone recording temperatures below 250°C.

It was observed that with pressures better than 100 microns in the system, magnesium metal appeared as massive deposits and was free of any alkali contamination. With higher pressures obtaining in the system, alkali metals were found to codeposit with the magnesium metal and tarnish its surface immediately after atmospheric exposure. Argon gas was employed as an inert gas for breaking the vacuum in the system before removing the magnesium condensate as the presence of inert gas eliminated the chances of any fire hazard.

Further experiments are under progress to study the briquetting properties of different types of indigenous dolomites (micro and macro-crystalline varieties) in relation to the calcination temperature as the calcination temperature range depended on the dolomites' moisture contents and carbon dioxide absorption rates.

SUMMARY OF RESEARCH THESES SUBMITTED FOR THE AWARD OF DOCTORATE DEGREE BASED ON THE WORK UNDERTAKEN AT THE NATIONAL METALLURGICAL LABORATORY

(i) *Low Nickel and Nickel Substantive-Free Stainless Steel*

Although the development of fully austenite Cr-Mn-N stainless steels has been reported, no data are available to show the effects of the manganese content on the properties of such substitute stainless steels. This subject has been studied in detail and the optimum manganese content necessary to yield a stable 17 per cent chromium was established. The beneficial effects of small additions of nickel and copper on the work hardening of these steels on cold working were also examined. As the manganese content was lowered, the tendency of steel to undergo martensitic transformation was noticed and conditions were then investigated which could produce low-nickel steels comparable in mechanical properties to the 17-7 chromium-nickel precipitation hardening stainless steels. The equation

$$0{\cdot}12(\%\ \mathrm{Cr}-13{\cdot}8) = (\%\ \mathrm{C}+\%\ \mathrm{N})+0{\cdot}05\%\ \mathrm{Ni}$$

derived from phase data has been shown to be related to the quantities of ferrite-forming element chromium with the austenite forming elements carbon, nitrogen, nickel and copper, to give the phase relationship at 1050°C. It has further been shown that the excess of the ferrite-forming element over austenite-forming elements can be quantitively related to the amount of ferrite present in the steel at 1050°C. by the equation

$$\%\ \text{delta-ferrite} = 95[0{\cdot}12(\mathrm{Cr}-13{\cdot}8)]-[(\%\ \mathrm{C}+\%\ \mathrm{N})+0{\cdot}05\%\ \mathrm{Ni}]$$

The mechanical properties of steels containing varying amounts of manganese were determined. The coefficient of thermal expansion for a steel containing 12 per cent manganese was determined and found to be of the same order as 18-8 Cr-Ni steel. The deep drawing properties of several steels were determined in Mohr and Federhaff and in Swift cupping testing machines. The beneficial influence of nickel on deep drawing properties has been reported. The corrosion resistance of several steels, with varying manganese contents, was determined in 65 per cent boiling nitric acid and it has been shown that both the manganese content and the presence of delta-ferrite in small quantities have no significant effect on corrosion rate. The effect on hardness after cold working delta-ferrite free steels containing varying amounts of manganese showed that as manganese decreased from 12 per cent to 6 per cent, hardness increased for the same amount to cold working. The effect of the quantity of delta-ferrite in reducing hardness obtained after cold working is shown for 8 per cent manganese steels. It is further shown that, though ferrite decreases the hardness after cold working 6 per cent manganese steels, no clear relationship exists between the amount of delta-ferrite and the hardness obtained in cold working as in the case of 8 per cent manganese steels. The effect of nickel and copper on decreasing the hardness on cold working and on conferring resistance to tempering after cold working was noticed. The superiority of 8 per cent manganese steel over 6 per cent manganese steel, considering all the mechanical properties, in cold rolled condition has been shown. It was noticed that no significant increase in strength occurred on liquid air quenching solution treated steels, though some increase occurred in 30 per cent cold reduced steels after this treatment. This shows that sub-zero treatment to increase the strength of this type of steels in annealed condition will not be effective. On tempering the 30 per cent cold reduced and liquid air quenched specimens of 6 per cent manganese steels the elongation was found to drop to 0 per cent at 500°C. On ageing solution treated steels containing 6 per cent manganese at 750°C. for 14 hours, the steels were found to have excellent properties. For this treatment to be effective, about 6 per cent ferrite and 1 per cent nickel were found to be essential. The isothermal transformation curve for a 12 per cent manganese steel was determined and the pearlite-like structure obtained was noted. Welding trials and sensitization tests showed that steels having more than 0·03 per cent carbon are subject to weld decay, but those having carbon content of this order or less were not subject to this defect. Metallographic study of the various structures obtained during the course of this investigation has also been made. X-ray diffraction study of two 18 per cent manganese steels after solution treatment and 30 per cent cold working was carried out. 18-8 Cr-Ni steels were also tested in similar conditions for purposes of comparison. It has been shown that while 18-8 Cr-Ni steel did partially transform to martensite on cold working, no such transformation was observed in the Cr-Mn-N stainless steels.

(ii) *Ion-exchange of Clays with Special Reference to the Effect of Exchangeable Cations on Thermal Behaviour of Kaolinite*

This work has covered the study of the effect of exchangeable cations on thermal changes of kaolinite based on differential thermal analysis. Exchangeable cations significantly influence the low temperature properties of clays, viz. plasticity, workability, viscosity of clay slips and drying behaviour of clay-water bodies, but little work has been reported on the thermal behaviour of clays. Three kaolinites of varying crystallinity and base exchange capacity were chosen and the effects of some monovalent and divalent cations such as Na^+, K^+, Li^+, Ca^{++}, Mg^{++} and Ba^{++} were studied on their thermal behaviour. The thermal reactions of the three kaolinites were followed up to 1350°C. by differential thermal analysis; changes in various reaction peaks were corroborated, wherever necessary, with thermogravimetric and X-ray diffraction data.

Of the three kaolinites examined, Travancore china clay was relatively most well crystallized and ball clay was a b/3 disordered type kaolinite. Rajmahal ranked intermediate in crystallinity. The three kaolinites varied in crystalline impurities and the mineralogical composition of the clays was calculated by Norm method keeping in view the X-ray data on some crystalline impurities. In the *p*H-neutralization study of the hydrogen forms, Travancore china clay exhibited two inflexion points whereas others showed only one point; such difference in behaviour has been examined. The 'bec' of the clays was determined by three different methods with a view to compare different techniques.

There was no appreciable change in the peak temperatures of the dehydroxylation reaction with ion saturated, but the cations affected the peak shapes. Hydrogen gave the highest amplitude, area and slope ratio of the peak and barium gave the lowest values for these factors. Even though no rigid order existed between the various cations, closer examination revealed that Na^+, especially of monovalent cations, tended towards hydrogen and divalent cations appeared to group together. The changes in the peak shapes were considered to be due to differences in rate of dehydroxylation.

The exothermic peak in the region 950-1000°C. was greatly affected by various cation, Na^+ and Li^+ decreasing peak intensity and H^+ and divalent cations in general increasing it. The reaction peak was considered to be due to rapid formation of spinel phase within a temperature range; the spinel phase was considered to be identical with the one reported by Brindley and co-workers in view of the similarity of its crystal size and the nature of its development from metakaolin as revealed by electron microscopic studies. The change in peak intensity with the various cations was considered to be due to particle flocculation or deflocculation brought about by cations in the original clay. With a view to explain the changes in peak intensity with ion saturated, relative proportions of spinel and mullite phases in the various samples heated at 1000°C. for 20 hours were assessed semiquantitatively in a manner similar to that of Baker and co-workers. It was considered that the variation in relative proportion of spinel and mullite phases in accordance with peak intensity was coincidental and may not have any direct bearing on peak intensity.

A single exothermic peak was observed above 1000°C. in Rajmahal and ball clays and no peak was observed in the case of Travancore china clay. The peak was considered to be due to rapid formation of mullite on the presumption that kaolinites which gave rise to rapid formation of mullite would exhibit exothermic peaks above 1000°C. whereas the peak may not be detected if there was progressive development of mullite without rapid formation within a particular temperature range. The peak was not due to cristobalite formation since there was absolutely no change in the peak shape of Na^+ and K^+ forms of ball clay even though the phase was completely undetected by X-rays in these cationic forms.

A detailed study of the effect of cations on high temperature phases in ball clay revealed that the cations had no mineralizing effect on mullite formation. The cristobalite phase was very much affected by cations and the variation in intensity of the characteristic lines of cristobalite in X-ray diffractograms was considered to be due to the degree of disorder brought by the cations on cristobalite lattice. Perhaps, differences in the degree of disorder caused by the various cations may lead to changes in the X-ray intensity reflected by disordered cristobalite. Even though further work was needed to arrive at positive conclusions on the subject, the possiblity of glass formation in the case of Na^+ and K^+ forms, and hence complete non-appearance of cristobalite phase, cannot be ruled out.

(iii) *Studies on the Phase Transformation in Alloys by X-ray Diffraction and other Techniques*

These studies relate to mechanism and kinetics of precipitation reactions involving phase transformations in alloy steels when subjected to various heat-treatments and/or deformation under severe stresses.

Detailed investigation carried out on the transformation characteristics of carbides in low chromium steels, based on X-ray structural analyses and metallographic examination of the isothermally treated steel samples revealed the following features.

It has been established that Fe_3C is the initial carbide formed during isothermal transformation at all temperatures ranging from 250° to 700°C., which has been shown to transform to Cr_7C_3 and then to $Cr_{23}C_6$; the reaction continues even after the completion of the decomposition of austenite. The type of the final carbide formed has been shown to be the function of both chromium content and chromium/carbon ratio in the steels. On the basis of changes observed in the lattice parameters and chemical composition of carbides, it has been shown that transformation of Fe_3C to Cr_7C_3 or Cr_7C_3 to $Cr_{23}C_6$ is by general enrichment of Fe_3C or Cr_7C_3 lattice by chromium. The maximum chromium content in Fe_3C has been shown to be nearly 27 per cent and that of Cr_7C_3 to be nearly 65 per cent.

The other parts of the studies relate to the effects of subcritical ageing and cold working on the stability of austenitic phase and/or formation of other phases in chromium-manganese-nitrogen-carbon steels. It has been established that as in 18 per cent chromium and 8 per cent nickel austenitic stainless steels, austenite to ferrite transformation takes place during cold working in chromium-manganese-nitrogen-carbon steels. The crystal structure and the lattice parameter of the precipitated phase have been examined and were found to be the same as those of ferrite. During the subscritical ageing treatment, Cr_2N and $Cr_{23}C_6$ types of precipitates were identified, depending on the temperature of ageing and carbon and nitrogen contents of the steel studied. The relation between C/N ratio in steels and the different equilibrium precipitates (i.e. Cr_2N or $Cr_{23}C_6$ or both) at different temperatures of ageing has been determined. Intersolubility of various alloying elements (i.e. Fe and Mn) in the Cr_2N type of precipitate with time and temperature of ageing have been determined by X-ray spectrochemical analysis. The amounts of these various alloying elements in the precipitate have been shown to be a function of time of ageing at any particular temperature. The activation energy of the formation of Cr_2N type of precipitate in chromium-manganese-nitrogen steels during ageing has also been determined.

(iv) *Studies on Age-hardening*

Studies were carried on ageing of industrially important aluminium-copper and iron-nickel-aluminium permanent magnet alloys.

It has been shown that natural ageing of aluminium-copper alloys is conditioned by the concentration of quenched-in vacancies rather than quenching stresses or dislocation channels as postulated by other workers. The greater the concentration of the vacancies retained during quenching, the greater will be the acceleration and extent of first stage of ageing in which Guinier-Preston zones develop. Any process which reduces the number of quenched-in vacancies will also retard zone formation and, therefore, hardening of the alloy. On the basis of these investigations, it has been possible to rationalize the effect of quenching medium, specimen size, interrupted quench and plastic deformation on the ageing and formation of Guinier-Preston zones. Increasing specimen size, quenching medium of poor heat-abstracting capacity and interrupted quench will invariably lead to slow rate of ageing as most of the vacancies are likely to be lost during slow cooling of the specimens. Slow rate of ageing after cold deformation has been explained on the basis that extra dislocations produced during deformation act as skins for vacancies. The vacancy hypothesis has further been able to clear many controversies connected with dissipation of hardness acquired during ageing, on reversion treatment. It has been shown that such dissipation or softening takes place in two stages. The first stage of softening is associated with evaporation of G.P. zones through trapped vacancies and takes place between 150° and 170°C. The second stage of softening is due to dispersal of dislocation loops formed from quenched-in vacancies and takes place between 180° and 200°C.

The investigation on iron-nickel-aluminium alloys showed that such alloys also decompose and harden by the same mechanism as aluminium-copper alloys. First iron atoms separate out from the matrix in the form of clusters or zones, which subsequently give rise to intermediate and stable phases. Some evidence of presence of clusters and intermediate aligned phases is provided by the electron-microscopy of these alloys carried out at the laboratory. It has also been shown by X-ray diffraction studies that the solid solution of iron-nickel-aluminium alloys decomposes into B.C.C. stable phases in a way iron does through formation of γ-iron at intermediate temperatures. Hardening and coercive face changes of the alloys as a function of cooling rates and tempering temperature and time were also studied and explained on the basis that precipitation of critical size is the dominant factor for obtaining good magnetic properties.

(v) *Phase Transformation in Electrolytic Alloys with Special Reference to Copper-Cadmium Alloys Deposited at Constant Cathode Potential*

A comparison of the X-ray powder structures of the thermal and electrolytic copper-cadmium alloys deposited at constant cathode potential over a range of 1·27 to 85·6 per cent copper by weight was made. The phase-boundary limits for the existence of electrolytic alloy phases at ordinary temperatures do not correspond to the thermal equilibrium diagram. The existence of the metastable electrolytic alloy phases in the as-deposited state and their subsequent transformation to the stable structures have been explained in the light of the codeposition of hydrogen into the alloy composition and the rearrangement of the discharged atoms at the cathode from the amorphous unordered state to the stable or metastable phases. These rearrangements are influenced by the cathode potential and are facilitated by the disappearance of the primarily codeposited hydrogen thus leaving vacant gaps in the lattice. The formation of the electrolytic metastable phase is considered in the light of the excess energy associated with the unordered atoms. For each electrolytic phase there is a tolerance limit where it can exist beyond its equilibrium range, if not disturbed otherwise, and this observation has some practical implications.

(vi) *Chemical and Metallurgical Factors Affecting Failure of Metals and Alloys under Combined Action of Stress and Corrosion*

All metal systems contain certain alloy composition ranges where they are susceptible to stress corrosion cracking under proper environmental conditions. This type of failure occurs when alloys are subjected to the simultaneous action of static tensile stresses and of an electrochemical corrosion process. The cracks developed are propagated in a direction perpendicular to the acting stress and are intergranular or transgranular and sometimes both.

In this work, the objective has been to study the effect of variations in type and amount of alloying elements, viz. zinc, manganese and aluminium, added to copper on stress corrosion cracking susceptibility of resulting alloys. Effects of variations in corrosive conditions, anodic and cathodic polarization, cold working and annealing on cracking time and mode of cracking have been determined for different homogeneous copper alloys. Role of electrochemical factors and stacking fault parameter of alloy systems on the mechanism of cracking has been investigated.

Survey of literature shows that electrochemical factors controlling corrosion of copper alloys have not been studied very extensively. This is due to the fact that in ammonia solution the cracking takes place much more easily in the gaseous phase than in the aqueous phase. Recent studies by Mattsson on the stress corrosion cracking of Cu-Zn alloy containing 37·2 per cent zinc in ammoniacal copper sulphate solution of varying *p*H values between 3·9 and 11·2 have initiated a more elegant approach to the underlying made.

In this investigation more detailed studies on effect of change in *p*H and composition of solution, effect of cold working, annealing, etc., on cracking susceptibility of Cu-36 per cent Zn and Cu-(2-6) per cent Mn alloys have been made.

It has been shown in this investigation that with increase in the amount of copper present in Mattsson's solution, the cracking susceptibility increases. It has been further established that the state in which copper is present in solution, i.e. as Cu^{2+} or $Cu(NH_3)^{2+}4$, and the case of formation of oxide layer are important in controlling the stress corrosion cracking susceptibility. In the maximum susceptible range of *p*H, copper is present predominantly as $Cu(NH_3)^{2+}4$ and is also associated with the formation of a sufficient amount of cuprous oxide on the surface.

It has also been shown that cracking can be prevented by cathodic polarization. Anodic polarization has been found to increase slightly the cracking susceptibility for Cu-Zn alloy. On the contrary in the case of Cu-Mn alloys anodic polarization at lower current densities increased the cracking time which, however, became constant with further increase in the current density.

Cold working and annealing have been found to have a marked influence on cracking. For Cu-36 Zn alloy, it has been shown that with increase in degree of cold drawing, the cracking time first decreases and then increases with further increase in degree of cold drawing. The minimum in the percentage reduction versus cracking time curve has been shown to lie between 30 and 35 per cent reduction. In Cu(2-6) per cent Mn alloys, however, no such minima were observed at reductions above 16 per cent, which was the range of study in this investigation.

In Cu-Zn alloys, transition from inter to transgranular cracking occurs above 18-20 per cent zinc and when plastically deformed. In Cu-Mn and Cu-Al alloys also similar (dependence of composition) transition has been shown to occur between 6 and 10 per cent and at 5 per cent respectively.

The results obtained have been explained in the light of reactivity of structural sites, stress concentration at these sites and stacking fault energy that defines the mode of deformation and creation of barriers in a lattice. Mode of cracking has thus been shown to be dependent on the stacking fault parameter of the alloys studied; alloy composition having lower parameter failed intergranularly and those having higher parameter failed transgranularly.

RESEARCH GROUPS

THE following Research Groups have been formed at the National Metallurgical Laboratory to promote coordinated research work on rather specialized fields and projects requiring intensive integrated work amongst different metallurgical disciplines. The work of the Research Groups is reviewed from time to time in the weekly meetings of the scientific staff when the working of the entire research, development and pilot plant projects is discussed at length, often supplemented by visits to different divisions and sections by the scientific staff.

Research and Development Groups

1. High Temperature Creep Research Group.
2. Radio-active Isotope Research Group.
3. Metallic Surface Coating Development Group.
4. Gases in Metals Research Group.
5. Standard Metals Development Group.
6. Metallurgical Slag Research Group.
7. High Temperature Furnace Development Group.
8. Secondary Metals Refining Research Group.
9. Non-metallic Inclusions Research Group.

The working of these Research Groups is proving very useful indeed in many ways. Electric Furnace Development Group takes care of the maintenance and repair of all the electric furnaces in the National Metallurgical Laboratory, introduces specialized furnace designs and undertakes fabrications of the furnaces to meet the specialized research needs, such as for temperature control, programmed heating and cooling, etc. The design and fabrication of nichrome, kanthal, platinum-molybdenum wound furnace, oil and gas-fired furnaces, their repair and maintenance are undertaken on a fairly extensive scale. A graphitization furnace to attain 3000°C. is under design and fabrication stage. A full inventory and record of the furnaces designed and fabricated are kept for coordinating the demands and requirements of different research projects. 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 in large numbers for a two and 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 the maximum effective use of indigenous alloying elements.

With these objectives, a high temperature creep research laboratory has initially been equipped with a dozen 5-ton creep testing units; a multi-specimen creep testing machine with provisions for twelve test points in a single machine will shortly be installed for creep stress-rupture tests. In the high temperature creep research laboratory, normal facilities for protective devices have been provided; provision for protected power supply by installing an

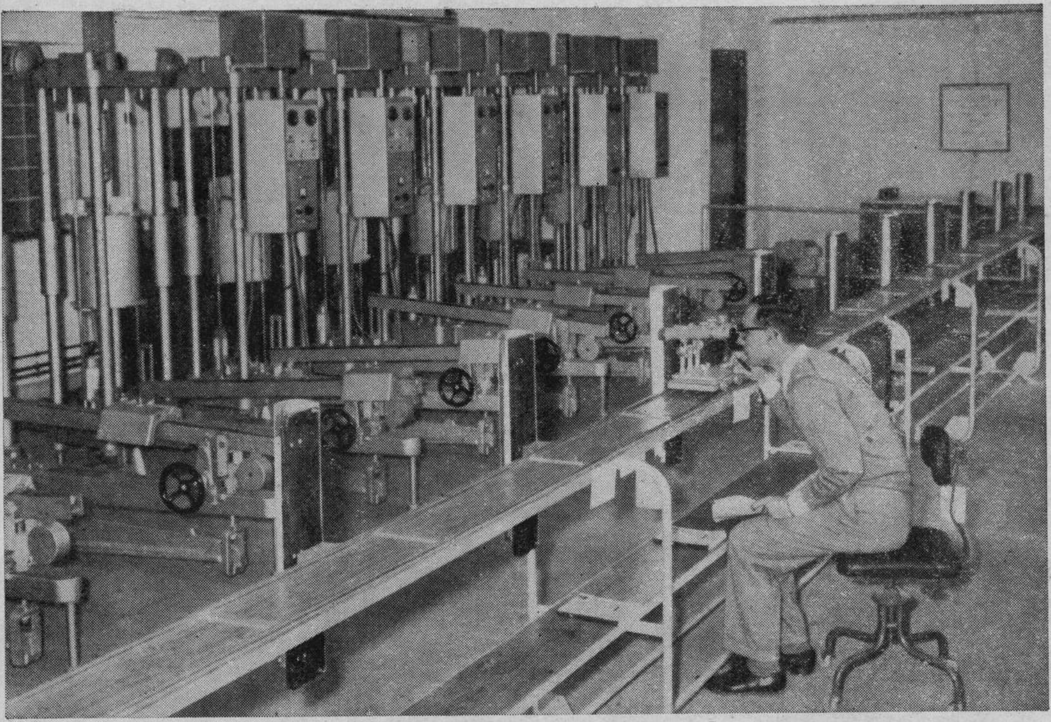

FIG. 79 — A VIEW OF THE BATTERY OF CREEP TESTING MACHINES INSTALLED AT THE NATIONAL METALLURGICAL LABORATORY

automatic stand-by diesel generator has been made. Voltage stabilizers and constant temperature and humidity controls have been incorporated in the creep research laboratory.

In view of the total absence of nickel in this country, the development of high temperature creep compositions has been based on the nickel-free Cr-Mn-N austenitic stainless group of alloys, work on which has successfully been pursued at the National Metallurgical Laboratory for the last few years on the formulation of nickel-free austenitic stainless steels possessing optimum deep drawing and corrosion resistance properties. High temperature creep characteristics of Cr-Mn-N austenitic steels have been taken up as the basic test material during the first phase of the work. This work has been formulated to determine the creep properties at 650°C. and 15 kg. per sq. mm. stress with expected rupture life of over a thousand hours. These alloys will also be subjected to different thermal and mechanical treatments, viz. as forged, solution treated (at temperatures in the range of 1050-1200°C.), single and double aged, and warm worked at various temperatures after the solution treatment. The prior mechanical and heat-treatments are expected to define optimum conditions for mechanical and heat-treatment cycles to impart adequate good temperature creep strength while ensuring the required creep rupture ductility required for various high temperature creep applications.

Radioactive Isotope Research Group

The uses and value of radioactive isotope as metallurgical research tool are today well established. A radioactive isotope research section is being established at the

National Metallurgical Laboratory. Adequate laboratory space has been provided in full consultation with the Atomic Energy Commission of the Government of India, proper room design, ventilation, air-conditioning and requisite safety precautions have been introduced. Requisite equipment have been ordered. Some laboratory equipment have been received whilst glove boxes etc. are under fabrication.

The work of the Radioactive Isotope Research Group will in due course cover a fairly wide field. Initial work will relate to grain boundary and surface segregation studies on alloy systems, diffusion studies, radioactivation analysis, metallic wear and lubrication studies, etc. In due course, the use of radioactive isotope will be made for studying refractories wear in the low-shaft furnace, slag-metal studies on the side-blown pneumatic steel making process developed by the National Metallurgical Laboratory. For tracer studies, the requisite samples will be irradiated in the atomic reactor of the Atomic Energy Commission whose cooperation is greatly appreciated.

The layout of the Radio-active Isotope research laboratory is given in Fig. 80.

Metallic Surface Coating Development Group

Chromium Plating

Electrodeposition of chromium on cold rolled mild steel samples was carried out under the following conditions:

Chromic acid	. . .	250 gm/1
Sulphuric acid	. . .	2·5 gm/1
Temperature	. . .	60°C.
Volume of the bath	. . .	1 lit.
Time of treatment	. . .	10 min.
Current density	. . .	25 amp/dm^2

The weight of chromium deposit was found on average 130 mg./dm.^{2}. The adhesion of coating was tested by bending the plated samples. It was found possible to bend it through 90° when the samples were broken, but no peeling of the deposit could be detected.

Corrosion tests on the chromium plated samples with and without lacquer finish were carried out in the following media:

(a) One per cent boiling sodium chloride solution.

(b) One per cent citric acid.

The tests were carried out for the duration of one hour. From visual observation it was apparent that the chromium plated lacquer coated samples give better performance as compared to lacquer coated mild steel samples.

Vapour coating process was also tried and uniform chromium coating of matt finish was obtained by hanging cleaned mild steel specimens in a container containing chromium chloride and ammonium chloride in suitable proportions. The container was covered so as to avoid oxidation of the surface and allow the ammonium chloride fume to act as cleaning agent. The thickness of the coating was found to be proportional to the time of contact with the vapour and also temperature of the container.

Efforts are now being made to directly pass vapour of chromium chloride over heated steel samples and to compact the coating by subsequent rolling operations.

Lacquer Coating

Three varieties of lacquers were used for coating of tin cans. A layer of acid resistance lacquer was applied on the plated samples by passing them through hand pressed rubber sheets wetted with the lacquer. The lacquer coated samples were heated at high temperature for 15 minutes. In order to get a uniform thin coating of lacquer, a rubber lined roller was designed and the same was fabricated in the N.M.L. workshop.

Plastic Coating

Plastic coating has gained a great prominence as it resists abrasion and corrosion

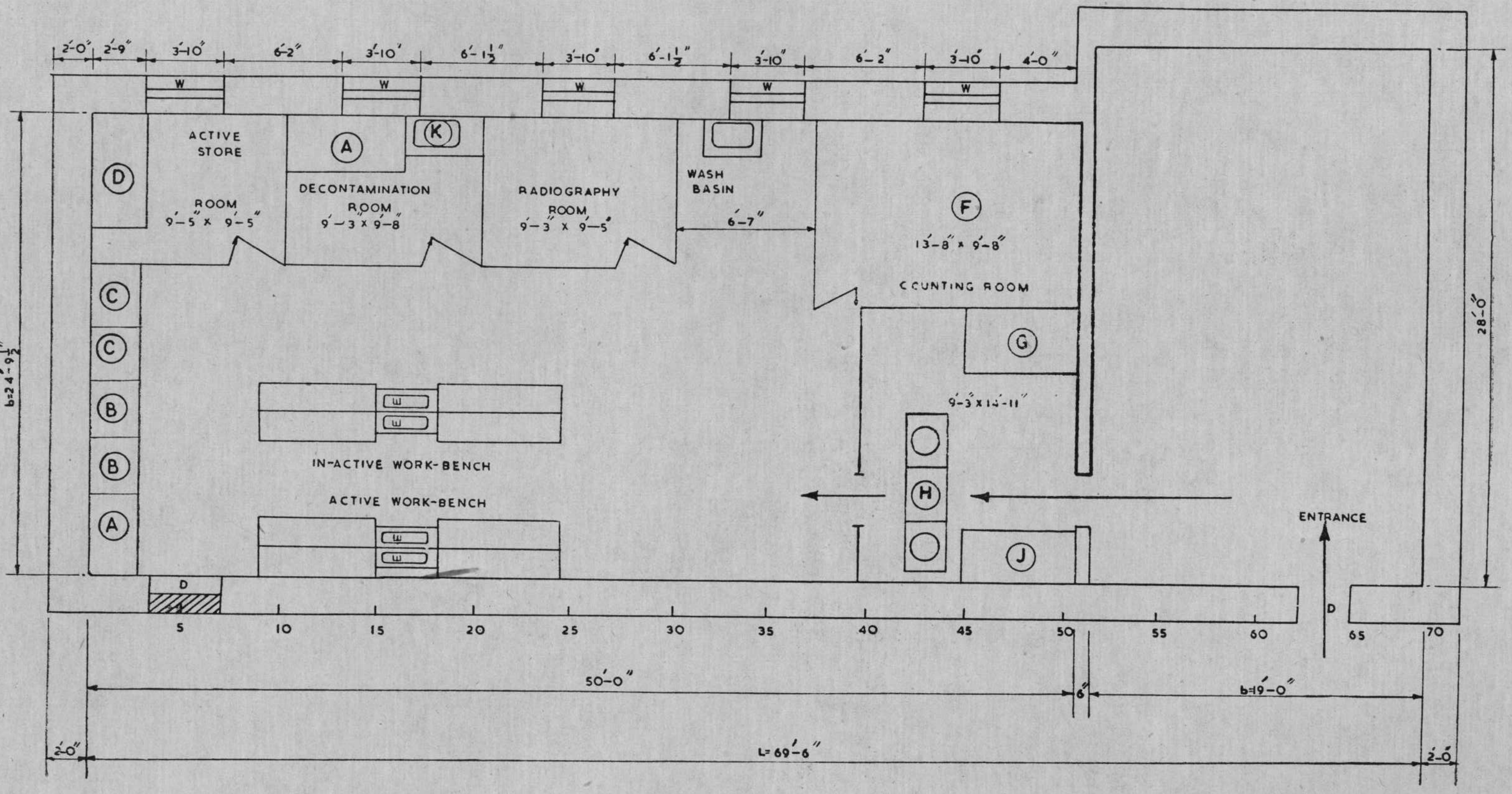

FIG. 80 — THE LAYOUT OF THE ISOTOPE LABORATORY OF THE NATIONAL METALLURGICAL LABORATORY

due to moisture and chemicals better than paint or enamel. Nylon coating provides additional wear resistance along with low friction to those parts where normal lubrication is not possible. Such coatings can be given to jigs and fixtures, rollers, bushings, loading hooks, gliding surfaces and household appliances, etc. India has now a good plastic industry and as such use of plastic in the field of corrosion prevention needs attention. Study has already been taken up at the National Metallurgical Laboratory to develop suitable methods of bonding plastic to steel.

Initial experiments were concentrated on the finding of a suitable adhesive to bond P.V.C. plastic film to steel base. Films of suitable thickness were procured from the plastic film manufacturers and all-purpose adhesives were used as bonding agent. Thermosetting adhesives have also been tried. Results so far obtained indicate that it is possible to achieve the objective provided a suitable adhesive which quickly sets by heating to suitable temperature could be found.

ENGINEERING SECTIONS

Design Engineering

Some of the major design projects completed and in progress for different pilot plants, research and development projects are given below:

(i) *Strip Aluminizing Plant*

The detailed engineering designs of the pilot plant for the continuous hot-dip aluminizing of steel strip were completed. The pilot plant equipment is now being fabricated at the National Metallurgical Laboratory or procured from outside to N.M.L. design specifications as the case may be.

(ii) *Recuperator for High Temperature Refractories Furnace*

The engineering design of the recuperator system was completed; procurement/fabrication has been taken up for early installation and commissioning.

(iii) *Patenting Furnace*

Design work has been taken up for a multistrand furnace for continuous patenting of steel wire in a lead bath.

(iv) *Vanadium Pentoxide Plant*

A project report was prepared for an industrial scale unit with a capacity of 100 tonnes/year of vanadium pentoxide, processing approximately 10,000 tonnes of raw material per year.

Electronics Engineering

(i) *Automatic Thermogravimetric Balance*

An automatic thermogravimetric balance was developed, designed and fabricated at the National Metallurgical Laboratory. Changes in the intensity of light falling on a small mirror fixed on the beam of an analytical balance are detected and measured by a dual gas phototube in a bridge circuit. The out-of-balance photo-current is amplified by a differential cathode-follower d.c. amplifier and passed through a solenoid in which a permanent magnet hangs on the left side of the balance. The current through the solenoid moves the permanent magnet and balances the beam. The solenoid current passes through a substandard resistance, the voltage drop across which, measured by a millivolt recorder, is proportional to the changes in the weight of the sample hung on the right side of the balance and kept in a controlled furnace. Electronic dampingy has also been provided. An Indian patent application has been filed.

(ii) *Precision Temperature Controller*

A precision temperature controller for use with high temperature electrical resistance furnaces (up to 1600°C.) was designed and fabricated at the National Metallurgical Laboratory. The furnace winding itself forms one arm of the bridge. The imbalanced voltage is stepped up by two microphone transformers, the primaries of which are connected in parallel; secondaries of the transformers are connected to the grid of two high mu-triodes connected in phase opposition. The phase and magnitude of the resultant voltage control the timer circuit of a power triode, the plate circuit of which contains a relay. The relay switches on and off the furnace in proportion to the deviations in the temperature. The accuracy of the

FIG. 81 — A VIEW OF THE ENGINEERING DESIGN DIVISION OF THE NATIONAL METALLURGICAL LABORATORY

controller was determined to be within ±½°C. up to 1600°C. for short period of one hour and within ±1°C. over long periods.

(iii) *Photo-transistor Temperature Controller*

Several temperature controllers were designed and fabricated at the National Metallurgical Laboratory for different research projects and needs. Performance of these controllers proved to be fully satisfactory except in ambient temperature of more than 30°C. Work on the design of compensatory circuits for higher ambient temperatures (above 30°C.) is under progress.

(iv) *Proportional Temperature Controller Using an Adjustable Electronic Timer*

A synchronous motor-driven micro-switch actuates a time delay vacuum switch, the timing of which can be adjusted by a potentiometer in the filament circuit of the vacuum switch, which switches the furnace current on and off. The on/off time can be varied as desired.

Amongst the major maintenance installation and calibration work handled by the Electronic Section, mention may be made of Norelco autrometer, electron microscope, electron diffraction apparatus, vibrophore, deltatherm, D.T.A. apparatus, rolling mill control systems, etc. Instrumentation of refractories pilot plant furnace was also completed during the period under review. 'Preventive maintenance' programme of different electrical and electronic instruments has been drawn up, with a full record of the spares likely to deteriorate under Indian temperature and humidity conditions and needing replacement. Action to procure such spare is taken well in advance so that, as far as possible, the work is not held up.

Mechanical Engineering

Maintenance work for the whole Laboratory was looked after from minor repairs to major break-down jobs. In addition, specialized research equipment and components were designed and fabricated. A brief review of the jobs completed is given below for some of the important research projects:

(i) *Camera Copying Set-up*

A camera copying set-up was developed for taking macrophotographs with the help of 'Practika' camera. The camera fixed up in this set-up can be adjusted in any suitable position for taking photographs.

(ii) *Liquid-Metal Centrifuge*

A modified liquid-metal centrifuge was designed and fabricated to study the constitution of liquid alloys. The centrifuge was designed for a high speed and due consideration was given to the high centrifugal forces generated in formulating the detailed designs. The rating of the new centrifuge was up to 1000 r.p.m. whereas an earlier centrifuge was designed and fabricated at the National Metallurgical Laboratory for a maximum speed of 350 r.p.m. The centrifuge has the following design features.

A 3 H.P. motor drives a horizontal shaft mounted on bearings. A vari-speed pulley is attached to the motor shaft so as to give different speed ranges for the whole unit. The vertical shaft driving the centrifuge is connected to the horizontal shaft through a system of bevel gears. The bearing housing for the vertical shaft has robust constructional features so as to withstand high centrifugal force. The open type furnace carrier attached to the vertical shaft has two electrical furnaces. The elements inside the furnaces can be fitted up electrically by a system of slip-rings and power collectors. Adequate cooling arrangements for the furnaces have also been provided. For minimizing the air resistance, a closed guard is installed around the centrifuge. A bigger guard encircles the whole set-up to take care of hazards at the time of operation.

(iii) *Viscosity Measuring Apparatus*

An apparatus for measuring the viscosity of molten slags by torsion method was designed and fabricated at the National Metallurgical Laboratory. The entire set up consists of a furnace containing the slag kept vertically inside whilst its position in any direction can be adjusted by a set of

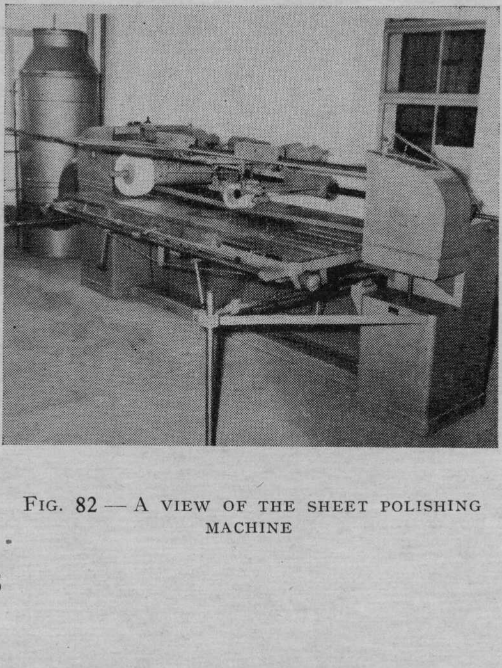

FIG. 82 — A VIEW OF THE SHEET POLISHING MACHINE

screws. A small known torque is applied to a small sphere dipped in the molten slag through metallic connections and the corresponding deflection is then measured; such deflection directly gives the viscosity of the slag after suitable calibration therefor.

(iv) *Fabrication of Tumbler*

A tumbler for the purpose of testing pellets was designed and fabricated. The 3 ft.-0 in. dia. tumbler rests horizontally on a frame supported by plummer blocks. The r.p.m. of the tumbler shaft can be varied by a system of pulleys and reduction gear attached to the motor shaft.

(v) *Remodelling of Plate-bending Machine*

The new plate bending machine in the workshop was remodelled by introducing three auxiliary rolls in addition to the existing rolls for the bending of small-sized bars and pipes.

Electrical Engineering

(i) *Installation of Electrical Equipment in the Extension of the Laboratory*

Several electrical items of equipment and components for various research projects and activities were installed and commissioned. Some major jobs handled are outlined below:

(a) For the erection of three high frequency furnaces, two air melting and one vacuum melting type, the design of layout, electrical power and control wiring, and water cooling and recirculation system were completed. One 20 kW., 5000 cycles motor-generator set and its control panel consisting of protection relays, measuring instruments, and power-factor correction equipment were installed; the above furnaces were connected to the high frequency generator in such a way that only one furnace out of the three could be switched on at a time. Motor generator, transformer, furnace coils and furnace connection leads were connected to water cooling system. Cooling system is electrically interlocked with the power supply and provided with water failure relays and alarms.

(b) One 50 kVA. arc furnace, indirect type, equipped with automatic rocking and motorized metal tapping arrangement, was installed and commissioned.

(c) The installation of electrical equipment in Mineral Beneficiation Pilot Plant: was commissioned by providing a temporary power substation. The installation of the permanent scheme of power supply is now in progress. The substation consists of one 1000 kVA. 6600/415 volts transformer, three high tension oil circuit breakers and three low tension oil circuit breakers. Electrical equipment and components for pilot plant scale agglomeration, pelletizing, magnetic and electrostatic separation studies of ores were installed and commissioned. Electrostatic separation equipment utilizes 50,000 volts for producing electrostatic charge and 101 volts d.c. motors for variable feed.

(ii) *Graphitization Furnace*

As reported earlier, one graphitization furnace to operate in the range of 2000-2500°C. is under fabrication. Special power transformer was fabricated for this unit. Design and engineering drawings for the furnace proper were completed.

(iii) Steady repair and maintenance of electrical equipment were undertaken on a short-term and long-range basis.

Civil Section

The Civil Section of the National Metallurgical Laboratory is responsible for all civil building and installation work, laying foundations of heavy plant equipment and their erection including heavy furnaces, preparing relevant layout drawings, etc. All estimates for new building works, erection of heavy equipment for pilot plants are

FIG. 83 — A VIEW OF THE MULTISTOREYED FLATS FOR N.M.L. STAFF

prepared in the Civil Section which also invites the tender and later supervises the actual execution of building projects. Likewise Civil Section collaborates with the civil staff at the C.S.I.R. Head Office in the construction of houses and residential colonies for the staff of the National Metallurgical Laboratory. The Civil Section has also to attend to all upkeep and maintenance work of the buildings. Another facet of Civil Section's active work relates to collaborative work on slag cement development project and related fields. The Civil Section has to maintain close collaboration with different research projects in executing their building works, the services and auxiliary facilities such as in the case of Marine Corrosion Research Station at Digha, Regional Foundry Research Stations at Howrah, Batala, Madras and Ahmedabad.

Safety First and First Aid Section

Safety First and First Aid Section takes care of all safety measures for different pilot plants, operations of which in some cases are quite hazardous, for different research projects, in the foundry, workshops, handling of gases, electrical substation and distribution system etc. Apart from rendering first aid, the cult of 'Safety First' is impressed upon all workers in their respective spheres of duties by lectures, talks and demonstrations, by organizing 'Safety First' month and by audio-visual means and devices. In the case of heavy pilot plant operations, the introduction of safety first measures are highly important, such as in the case of Low-shaft Furnace Pilot Plant, Mineral Beneficiation Pilot Plant, etc. Effective liaison is maintained with the research groups and divisions and their views taken into account in introducing safety-first measures in different fields. Work of this section is being intensified to prevent the occurrence of accidents and minimize the danger to life, limb and laboratory equipment and property.

Important Scientific Equipment Designed and Fabricated at the National Metallurgical Laboratory during the Period under Review

1. Experimental baby blast furnace with full provision for the injection of gaseous, liquid and oil-coal slurry fuels along with preheated air blast passing through a metallic recuperator.
2. High speed liquid metal centrifuge with accessories to work on speeds varying from 150 r.p.m. to 900 r.p.m.
3. Pelletizing disc for pelletizing of ore fines.
4. High temperature torsion apparatus for testing refractory materials.
5. Apparatus for determination of high temperature elastic modulus of refractories by sonic method.
6. Apparatus for determination of viscosity of molten slags by torsion method.
7. One camera copying set-up was developed with ' Practika ' camera for taking micro-photographs.
8. A number of ' kanthal ', ' nichrome ' and ' molybdenum ' wound tubular furnaces along with energy and temperature control equipment.
9. Recuperator set-up for the gas-fired down draft kiln.
10. One 7 ft. dia. hot plate made of 10 G. mild steel.
11. High temperature dilatometer with the necessary stand and loading frame.
12. Automatic thermogravimetric balance.
13. Precision temperature controller.
14. Photo-transistor temperature controller.

INDUSTRIAL LIAISON, OPERATIONAL RESEARCH, INFORMATION AND EXTENSION SERVICES

DURING the period under review, closest possible contacts and collaboration were maintained with the industry, scientific societies, professional associations and technical institutions to ensure effective coordination of research efforts and fruitful utilization of the research results. Technical data and research resluts of the processes developed by the National Metallurgical Laboratory were disseminated in the form of technical papers, bulletins, brochures, press-releases, etc. Practical demonstrations were also arranged. Industrial firms to whom the N.M.L. processes had been licensed were invited to attend the demonstration so as to impart the technical know-how of the processes for commercial exploitation.

Consultancy Work

The impact of increasing emphasis on dynamic growth of mineral and metallurgical industries in the country, the growth of integrated iron and steel complexes during the successive Five Year Plans has brought in its wake justified unceasing and increasing load of work for the National Metallurgical Laboratory in the form of technical consultancy, preparation of project reports and technical aid which the N.M.L. is effectively meeting to the fullest extent possible.

National Metallurgical Laboratory has conducted extensive research and pilot plant investigations on the beneficiation and agglomeration of the iron ores to determine their suitability for using in Indian iron and steel complexes as well as for export purposes. Investigations on Indian iron ores and other raw materials were undertaken and project reports were prepared and submitted to various States as well as to public sector organizations. Comprehensive work on the iron ores from Kiriburu mines were undertaken for their projected use in the Bokaro Steel Plant and currently for export to Japan. Similar investigations were conducted on coastal iron ore deposits such as in Goa for home iron smelting and for export. Beneficiation and agglomeration of Bolani iron ore were aslo completed for the Durgapur Steel Project of Hindustan Steel. The data obtained through investigations conducted on beneficiation and smelting in the submerged arc Pilot Plant furnace on Salem Magnetite iron ore was supplied to the Govt. of Madras in the form of a full report on the subject. Full report on sintering of the Tisco iron ore fines and blue dust based on the investigations undertaken at the National Metallurgical Laboratory was issued to Tata Iron & Steel Works. As regards fluxes used in iron and steel industry, National Metallurgical Laboratory conducted exhaustive pilot plant scale investigation on beneficiation and pelletization of limestone from Purnapani and about 100 tons of pellets made from concentrates were supplied for trials in steel melting shops in Rourkela Steel Plant. Successful steel melting trials were also made in Tisco steel melting shops with beneficiated limestone pellets prepared at the National Metallurgical Laboratory and a report has been submitted to Tata Iron & Steel Works for establishing 300 tons/day capacity limestone beneficiation plant. Likewise, identical research and pilot plant investigations were conducted for non-ferrous industries complex, such as for Khetri copper ore, etc.

The quantum of consultancy work rendered to industry in assessment of raw materials, causes of major failures and breakdown of plants maintained a steady upward curve. Staff members of the Laboratory were also deputed to carry out on-the-spot study of the cases.

Collaborative 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 has undertaken investigations on samples of bentonite obtained from different countries of the world. Besides the above, the National Metallurgical Laboratory is actively collaborating with the Indian Standards Institution in formulating Standards, 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.

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 are 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 Industries

Technical advice rendered to various industrial organizations, semi-Government and Government bodies has increased several folds both in the quantum of technical advice rendered as well as in the number of ad-hoc short-term and long-range research investigations besides specification tests undertaken. About three hundred technical enquiries were promptly attended to and about a hundred short-term investigations were conducted during the period under review for the industry.

Practical Demonstration and Training

Practical demonstrations of the N.M.L. processes were arranged so that the licensee firms can acquire the technical 'know-how' of the N.M.L. processes for operation of their plant engaged in exploiting N.M.L. processes. Effective training was also imparted to trainees from the industry, students from technical institutions and universities in the various disciplines of mineral and metallurgical industries.

Operational Research

Work has been initiated to prepare a comprehensive literature report on O.R. techniques as applied to problems of the iron and steel industry so that O.R. studies can be formulated in a phased programme.

Attempts have also been made to study the research problems taken up by the different research groups of the Laboratory, etc. Illustrative of such work is the problem which aims at investigating whether the eutectic lead-tin liquid consists of lead-lead or tin-tin clusters. Chemical analysis showed that possibly such clusters do form and that the solution is not ideal.

During the course of investigations, it was felt that the time of centrifuging had probably certain significant bearings on the cluster formation of lead-lead atoms. Since a detailed study in this respect could not be taken up through chemical analysis, investigations have been taken up to study this problem through statistical analysis.

Colloquia

During the period under review, colloquia activity was effectively maintained where research staff spoke on different research and technical themes. Lectures were also arranged from distinguished scientists and metallurgists from home and overseas.

REGIONAL FOUNDRY RESEARCH STATIONS AND CORROSION RESEARCH STATION

THE first Regional Foundry Research Station set up by the National Metallurgical Laboratory in the Industrial Estate, Batala, Punjab, was declared open early in 1964. Speaking on that occasion, Dr. S. Husain Zaheer, Director-General, Scientific & Industrial Research, said that one of the objectives would be to assist the industries — new industries and old industries — by bringing to them the latest improvement in science and technology pertaining to their particular industry. The National Metallurgical Laboratory, Dr. Zaheer said, has established a very high international reputation, indeed, for the high standard of researches undertaken. While the work being done in the different research centres is very good, it is necessary, Dr. Zaheer pointed out, that

FIG. 84 — DR. B. R. NIJHAWAN, DIRECTOR, NATIONAL METALLURGICAL LABORATORY, WELCOMING THE DISTINGUISHED GUESTS AT THE INAUGURATION OF THE REGIONAL FOUNDRY STATION, BATALA

technicians and scientists should go out with the latest knowledge and improvements deep into the centres of industry and sell their knowledge so that it is fully and effectively applied and utilized by the industry.

Dr. B. R. Nijhawan, Director, National Metallurgical Laboratory, in his address of welcome, thanked the distinguished guests, foundry executives and foundrymen for their full cooperation, and also expressed his gratitude to Sardar Partap Singh Kairon, Chief Minister of Punjab, for the keen interest shown by him, and to Mr. Mohan Lal, Home Minister, Punjab, and Dr. S. Husain Zaheer, Director-General, Scientific & Industrial Research, for participation in the function. He also complimented the staff of the Regional Foundry Station for the hard work put in by them under the active guidance of Mr. R. M. Krishnan, Assistant Director of the National Metallurgical Laboratory. He announced that the testing facilities will further be expanded and would also include melting units to enable the station to undertake experimental work on metal melting. The Regional Foundry Station of the National Metallurgical Laboratory, said Dr. Nijhawan, will be at the service of the foundries in Punjab for providing technical assistance on site as well as for introducing scientific control methods in their foundries.

Equipment and Facilities

The Regional Foundry Station at Batala has been 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 use for foundry moulding purposes. The Regional Station has also been equipped to undertake the carrying out of chemical analysis of both ferrous and non-ferrous metals, moulding sands, etc. In the second phase, the Regional Foundry Station will expand the service facilities with a view to undertake metallographic examination and mechanical testing of metals and alloys; it will also be equipped with the necessary melting units for undertaking experimental work on problems relating to metal melting. The Regional Foundry Station has a small library which will gradually be expanded to provide facilities for technical reference on diverse fields of foundry technology.

Programme of Work of Regional Foundry Station

The programme of work at the Regional Foundry Station will cover the following:

(i) Periodical testing of foundry sands, bonding clays used in foundries, and raw materials used for melting like pig iron, limestone, coke, etc., as well as finished products.

(ii) To tender technical advice regarding selection and application of indigenous foundry sands and other

FIG. 85 — DEFECTS IN A CASTING BEING EXAMINED IN A LOCAL FOUNDRY AT BATALA BY THE FIELD STAFF OF THE REGIONAL FOUNDRY STATION, BATALA.

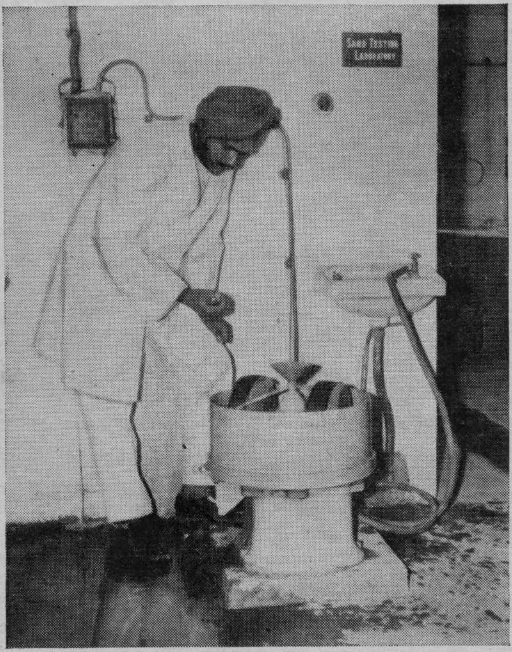

FIG. 86 — AN EXPERIMENTAL SAND MIX BEING PREPARED AT THE REGIONAL FOUNDRY STATION, BATALA

moulding materials, including selection of metal charges for the production of various types of castings.

(iii) To undertake ad-hoc investigations on the problems faced by individual foundries and to suggest suitable remedial measures thereof.

(iv) To conduct a regional survey of the moulding materials available in those regions, and to conduct a complete study of the chemical and physical properties of the moulding materials.

(v) To conduct periodical refresher courses to the staff sponsored by the foundries.

(vi) Standardization of melting and moulding techniques in underdeveloped foundries.

(vii) To advise the operational staff on modern foundry technology and to issue technical bulletins periodically.

(viii) To carry out research and development work, standardization and simplification of moulding, melting and other technological procedures to achieve quality as well as productivity.

Similar Regional Foundry Stations at Madras, Howrah and Ahmedabad are under establishment and should be in full operation in the near future.

Marine Corrosion Research Station, Digha

The Marine Corrosion Research Station has been set up this year, at Digha in West Bengal, with a view to undertake scientific studies on multiple aspects of corrosion under marine conditions, e.g. marine atmospheric corrosion, sea-water corrosion under partially and fully immersed conditions, soil corrosion, corrosion due to microorganism, etc. The climatic condition of coastal Digha location is truly marine and free of any industrial pollution.

Current work will relate to atmospheric corrosion studies of non-ferrous metals and alloys, e.g. copper, brass, aluminium, Al-Mg, Al-Mn, Ni, monel, mild steel, etc., which were exposed with effect from January 1964 for both long and short-term studies. Test panels were exposed on the roof of the laboratory building, built within 100 yd. from the sea coast under different exposure conditions, such as vertically, horizontally and at 45° to the horizontal. Regular analysis for the environmental climatic conditions, such as recording of daily atmospheric temperature and humidity, collection and analysis of rain and dew, measurement of salinity, etc., are carried out.

Provisions for carrying out other types of corrosion research including impingement corrosion, microbiological corrosion, soil corrosion, etc., have also been made at the Marine Corrosion Research Station at Digha of the National Metallurgical Laboratory. Arrangements are underway to start both

FIG. 87 — A VIEW OF THE MARINE CORROSION RESEARCH STATION OF THE NATIONAL METALLURGICAL LABORATORY AT DIGHA, WEST BENGAL

laboratory and field studies on the following topics of corrosion:

(i) Corrosion under constant immersion in sea-water.

(ii) Corrosion under alternate immersion in sea-water.

(iii) Impingement corrosion with special reference to condensers handling sea-water.

(iv) Soil corrosion of coastal places.

The work of the Corrosion Station will be undertaken in close collaboration with the Defence Research Laboratory and Ministry of Transport and Communication.

LIBRARY, DOCUMENTATION, TRANSLATION AND REPROGRAPHIC SERVICES

Library and Documentation Service

With the dynamic expansion of the National Metallurgical Laboratory and growth of its research activities, necessity for project-oriented documentation has increasingly been felt during the last few years. A programme was, therefore, launched in collaboration with the Indian National Scientific Documentation Centre in Delhi to enlist the full cooperation of the scientific staff in scanning through the technical journals received by the Library, and maintaining a full record of such technical documentation scrutiny. The arrangement has led to the orientation of the abstract to meet specifically the needs of research and development projects underway at the National Metallurgical Laboratory. The technical articles marked by the research workers are abstracted and typed in quadruplicates on standard 5×8 in. cards, in place of the earlier practice of making one copy of 4×6 in. card and then printing the cards on sheets making the IOTA Bulletin.

These cards are closely classified and chain indexed according to UDC. One copy of the card is kept in the Central Information File in the Library and the remaining cards are sent to the original scanners and interested technical personnel. It is thus hoped that such technical decumentation will ensure the compilation of an abstract file of interest to each division as well as a Central Information File and clearing agency of the Library providing abstracts covering the direct interest of the whole Laboratory, and forming a comprehensive bibliography for future references. Similar technical scanning and documentation of the patents taken in India and overseas were undertaken and the patent abstract cards were channelized on an identical basis.

With the growth of the scientific activities of the Laboratory in multiple technical disciplines, it has become imperative now to extend the range of laboratory acquisition in respect of technical subjects allied to metallurgical technology. The usual services of the Library were fully maintained and effectively extended to suit the changing pattern of research and technical needs.

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 efficiently. About 300 technical papers published in French and German were translated during the period under review, each paper being of an average of 4000 words.

Reprographic Service

Photostat, reflex prints and microfilms are made of the scientific and technical papers of interest to the research workers and are widely circulated. Documentary films were taken of important technical functions of the Laboratory as well as outstanding processes and techniques developed. Coloured transparencies and microphotographs, X-ray photographs, etc., are taken of the various research and investigation projects.

INTERNATIONAL SYMPOSIA

WITH a view to focus attention on multitudinous aspects of recovery of by-products and metallurgical wastes, an International Symposium on ' Utilization of Metallurgical Wastes ' was organized by the National Metallurgical Laboratory from 10th to 13th March 1964 to exchange technical ' know-how ', examine interrelated problems and discuss effective utilization of metallurgical wastes based on scientific and economic lines.

The symposium drew a large gathering and more than 200 delegates from all over the world including top-ranking scientists, metallurgists and technologists participated in its deliberations.

The following thirty-seven technical papers covering research and development work on utilization of metallurgical wastes were presented and discussed in six technical sessions relating to (i) Treatment of fines and other low grade by-product materials produced during mining and by mineral beneficiation plants, (ii) Utilization of by-products and wastes produced in pyro-hydro-, and other extractive metallurgical, smelting and refining operations, (iii) Utilization of waste gases and flue dusts produced during roasting, smelting and refining and economics of recovery operations, (iv) Utilization of metallic scraps, drosses, ashes and skimmings, swarfs and residues resulting from ferrous and non-ferrous metallurgical refining and processing operations, (v) Utilization of spent pickle liquors and recovery of metallic values from waste solutions, (vi) Theoretical considerations of recovery of by-products and utilization of metallurgical wastes.

1. The Rational Utilization of Fine Ores from the Lorraine Iron Ore Deposit.
2. Utilization of Important Mining and Metallurgical Wastes Employing Mineral Beneficiation Techniques.
3. Utilization of Fine Grained Raw Materials by Briquetting for Smelting of Ferro-manganese.
4. Utilization of Metallurgical Wastes in Ferro-manganese Industry.
5. Research and Development Work on the Utilization of Metallurgical Wastes at the National Metallurgical Laboratory.
6. Recovery of Unburnt Gases from Oxygen Blown Converters.
7. Energy Recovery from LD Converters — A Descriptive and Thermodynamic Comparison.
8. The Recovery and Utilization of Waste Gases in Iron and Steel-making.
9. Recovery of Waste Gas from Oxygen Converter by the O.G. Process.
10. Utilization of Waste Heat and Gas in Small Integrated Iron and Steel Making Unit.
11. Nickel Recovery from Low Nickel-Copper Ores.
12. Chloride Volatilization by Salt Roasting.
13. S.L. Direct Reduction Process and its Application for Indian High and Low-grade Ores Utilizing Non-coking Coals.
14. Utilization of Coke Breeze for Production of Pig Iron in Small Scale.
15. The New Stora Process for Iron Making.
16. Possible Economies which could be effected in the Recovery of Metallics from Steel Mill Wastes.

FIG. 88 — DR. B. R. NIJHAWAN, DIRECTOR, NATIONAL METALLURGICAL LABORATORY, WELCOMING THE DELEGATES TO THE SYMPOSIUM

17. Utilization of Metallurgical Wastes on the Indian Railways — Ferrous Metals.
18. Production of Cement from Blast Furnace Slags and Utilization of Fly Ashes by the Sintering Process.
19. Investigations on Granulated Low Shaft Furnace Slag for the Production of Slag Cements.
20. Tisco Blast Furnace By-products.
21. Scope for Utilization of Slags and Related Wastes from Indian Iron and Steel Plants.
22. Utilization of Low Shaft Furnace Slag as Light Weight Aggregate for Insulation Concrete.
23. Utilization of the Indian Blast Furnace Slag.
24. Utilization of Blast Furnace Slag in the Manufacture of Slag Wool and its Products.
25. Utilization of Metallurgical Wastes on the Indian Railways — Non-ferrous Metals.
26. Utilization of Red Mud.
27. Production of Secondary Aluminium— A Review.
28. Metallurgical Wastes from Aluminium Smelters.
29. Bayer Plant Liquors — A Source for Vanadium.
30. Short Rotary Furnace and its Application in the Treatment of Battery Scrap.
31. Recovery of Lead from Scrap.

32. Recovery of Metallic Zinc from Galvanizers' Dross.
33. Recovery of Zinc from Scraps and Wastes.
34. Pickle Liquor Regeneration by Vacuum Crystallization.
35. New Method for Treatment of Waste Pickling Acid.
36. Method for Regeneration of Waste Pickle Liquors.
37. Treatment and Recovery of By-products from Pickling Liquor Wastes.

The symposium was inaugurated by Prof. Humayun Kabir, Union Minister for Petroleum and Chemicals. Sir Jehangir Ghandy, Chairman, Executive Council, National Metallurgical Laboratory, presided at the inaugural function.

Dr. B. R. Nijhawan, Director, National Metallurgical Laboratory, in welcoming the delegates from India and abroad, said that such international symposia offer active stimulation of thought and provide an ideal forum for exchange of technical 'know-how' and 'know-why' of diverse aspects of the subject.

Dr. Nijhawan pointed out that extractive metallurgical techniques are aligned to meet the exacting requirements of not only obtaining maximum metallic recoveries but also of ensuring maximum utilization of resulting by-products and metallurgical wastes. Such metallurgical by-products

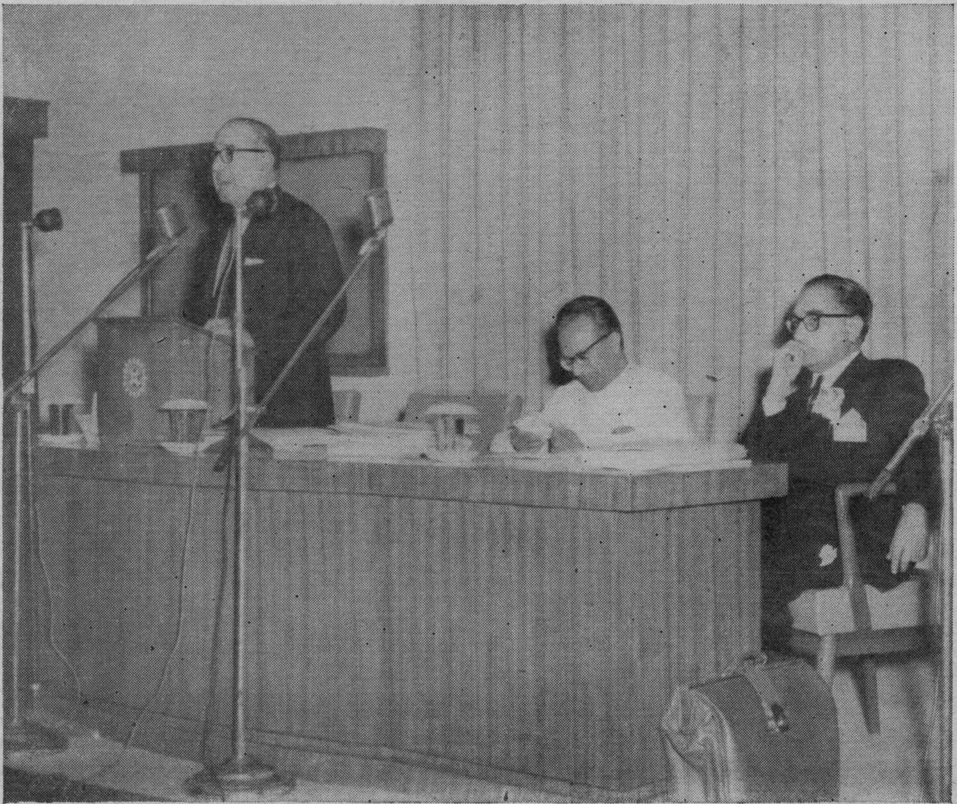

FIG. 89 — SIR JEHANGIR GHANDY, CHAIRMAN, EXECUTIVE COUNCIL, N.M.L., DELIVERING THE PRESIDENTIAL ADDRESS AT THE SYMPOSIUM

tended to deteriorate in quality in terms of their recoverable values, more so as the basic ores become increasingly leaner.

Such a situation was particularly pronounced and equally significant in India since optimum metallurgical techniques for the utilization of metallurgical wastes in India would have to be basically indigenous in character and execution. As such, the metallurgical wastes and by-products whilst following a general established pattern did pose certain characteristic features typical of the indigenous starting raw materials and processes. These developments, Dr. Nijhawan said, would require assessment of metallurgical wastes resulting during the extraction of metals and fabrication of finished end-products in the studies of which the National Metallurgical Laboratory had been actively engaged for several years besides undertaking what may be termed 'technological audit' of metallurgical techniques.

Sir Jehangir Ghandy, Chairman, Executive Council of the National Metallurgical Laboratory, said that in the wake of latest advances in scientific research and technology, many processes had been developed to utilize economically the waste products or the by-products. The National Metallurgical Laboratory, he added, had done exceedingly valuable work on the utilization of metallurgical wastes, e.g. mine discard like iron ore fines, the recovery of zinc from dross and related non-ferrous fields, etc. 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 highly valuable research and development work underway at the National Metallurgical Laboratory of direct interest and benefit to Indian mineral and metallurgical industries.

Inaugurating the symposium, Prof. Humayun Kabir, Union Minister for Petroleum and Chemicals, said that the response to the symposium was, in more than one sense, a recognition of the splendid research work the National Metallurgical Laboratory had undertaken within a relatively short span of life. The credit for this would undoubtedly go to Dr. B. R. Nijhawan, Director of the National Metallurgical Laboratory, and his colleagues who could all take due and justified pride in the Laboratory's achievements. He further paid his compliments to Sir Jehangir Ghandy, Chairman, Executive Council, National Metallurgical Laboratory, for his active support to the Laboratory.

Continuing, Prof. Kabir said that with the development of metallurgical industries and secondary processing plants under the successive Five Year Plans, it had become essential to focus attention on the optimum utilization of various metallurgical wastes. The major task before the mineral and metal industries today was to utilize, to the fullest possible extent, their different by-products and waste materials. In the industrially developed countries during the last fifty years, the major advances had been linked with the effective utilization of possibly all the by-products. The utilization and exploitation of metallurgical wastes and by-products presented a challenge to the Indian metallurgists and scientists, and in this task the National Metallurgical Laboratory had already played and would continue to play a very important role.

Prof. Kabir illustrated the utilization of metallurgical by-products by the example of the once heavily surplus molasses in India which today are exceedingly in short supply owing to the multitude of applications and also of the once waste and surplus naphtha which finds today several uses. He referred to the utilization of slags in which the National Metallurgical Laboratory had made valuable contributions. In concluding, Prof. Kabir congratulated Dr. Nijhawan and his colleagues on organizing such international symposia and on the splendid work in diverse fields — he expressed the hope that the annual symposia of the National Metallurgical Laboratory would not only develop international scientific collaboration but would also lead to the consolidation of brotherhood and mutual help between all scientists of the world.

In a résumé of the symposium at the end of the technical sessions Dr. B. R. Nijhawan, Director, National Metallurgical Laboratory, observed that the papers submitted covered multitudinous aspects of utilization of metallurgical wastes which represent a vast and challenging field. The seriousness of some of the problems encountered was reflected by the practical steps and measures suggested in most of the papers which stimulated very lively and constructive discussions of immense practical value. Viewed in the context of national economy and self-sufficiency, the N.M.L. symposium on 'Utilization of Metallurgical Wastes' was most opportune in focusing the attention of India's industrialists, — both in the public and private sectors — Government authorities and research workers both at home and abroad on the importance and urgency of expanding and consolidating work in this vast field. The international character borne by the symposium is convincing proof that it did succeed in rousing the interests of all concerned and thus fulfilled the very objective for which it was held. The symposium concluded with conducted visits round the National Metallurgical Laboratory

FIG. 90 — PROF. HUMAYUN KABIR, UNION MINISTER FOR PETROLEUM AND CHEMICALS, DELIVERING THE INAUGURAL ADDRESS AT THE SYMPOSIUM

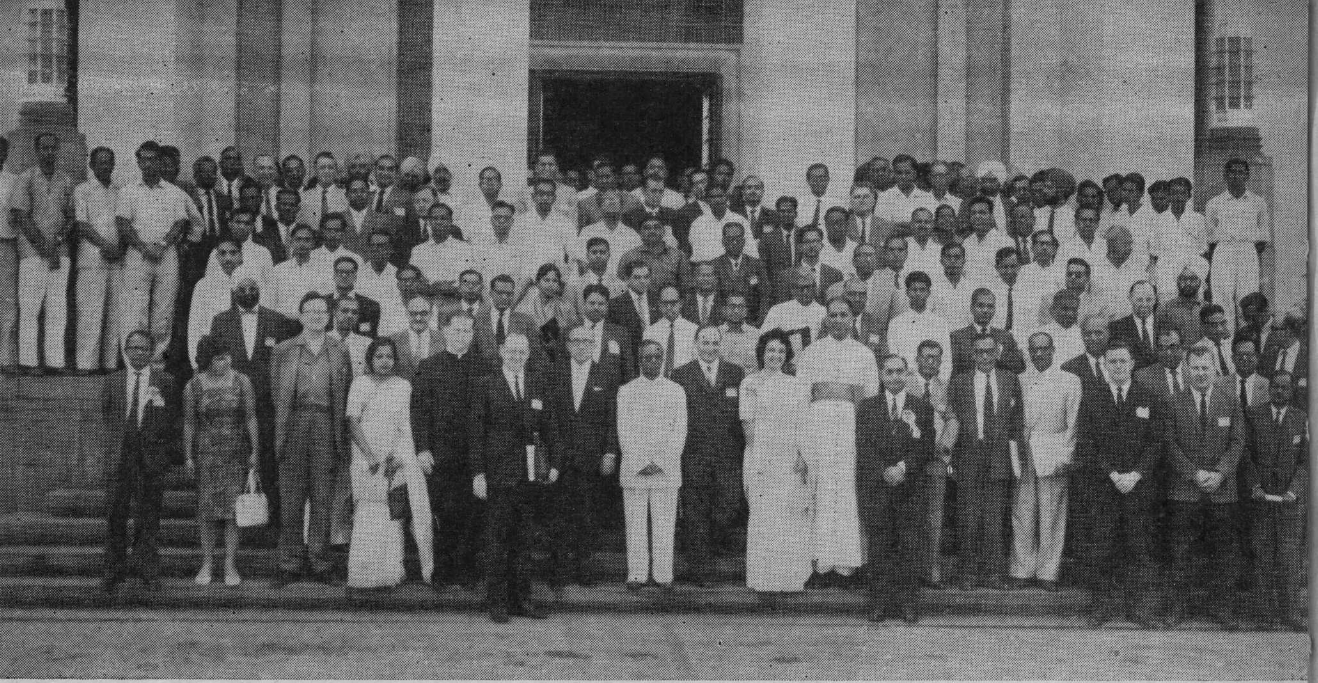

FIG. 91 — GROUP PHOTOGRAPH OF SOME OF THE DELEGATES WHO PARTICIPATED AT THE SYMPOSIUM

and its pilot plants, including the Low-shaft Furnace and the Mineral Beneficiation Pilot Plant.

The Symposium Supplement

On the inaugural day of the symposium, a special supplement was brought out in a masterly fashion by the *Statesman*. The Supplement contained leading papers from eminent scientists and technologists. Also *Economic Times* and *Financial Express* brought the supplements out during the symposium. Excellent coverage of the day-to-day proceedings was made by leading newspapers.

During the year under review, the National Metallurgical Laboratory participated in the following International Symposia by deputing scientific staff to attend the meetings and present technical and research contributions:

(i) Symposium on 'Application of Modern Technical Practices in Iron and Steel Industry in Developing Countries' held under the auspices of United Nations at Prague and Geneva.

(ii) Symposium on 'Testing and Evaluation of Materials' organized by the Govt. Test House, held at Calcutta.

(iii) Symposium on 'Corrosion' held under the auspices of the Commonwealth Defence Science Organization at Delhi and Kanpur.

(iv) Symposium on 'Powder Metallurgy' held under the auspices of Indian Institute of Metals at Bombay.

(v) Symposium on 'Steel Plant Refractories' held under the auspices of Indian Ceramic Society at Delhi.

PUBLICATIONS

NML Technical Journal

The *NML Technical Journal*, a quarterly publication, has stepped into the sixth year of its publication. The number of subscribers has gradually multiplied. The journal has also established an international recognition with a wide exchange programme with many Indian and foreign technical journals and nearly 200 journals are obtained as ' free exchange basis '. The papers published in the *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*; the contents of the *Annual Report* of the National Metallurgical Laboratory are likewise abstracted by leading abstracting establishments.

Papers Published

During the period fifty-seven scientific papers were published and presented as per Appendix I.

Brochures, Booklets, Reports, etc.

A brochure on ' Foundry Regional Station, Batala ', which has been set up by the National Metallurgical Laboratory, was brought out on its inaugural day, detailing the scope and function of the Regional Station.

A brochure on ' The National Metallurgical Laboratory ' outlining the scope, function and research activities was also brought out during the period.

Symposium Proceedings

The proceedings of the Symposium on ' Recent Developments in Iron and Steel Technology ' were brought out in a masterly fashion. The proceedings contain 15 authoritative papers from leading scientists and technologists of the world in the field of iron and steel. The symposium was organized jointly by National Metallurgical Laboratory, Iron & Steel Institute, U.K. and Indian Institute of Metals and was held during February 1963.

During the year under review, sixty-one investigation and research reports were prepared. The details are furnished in Appendix II.

PATENTS

Patents Filed during the Period

1. *Indian Patent No.* 91134 — Improvement in or relating to a precision temperature controller for use with electrical resistance furnaces up to 1600°C. (4.12.1963).
2. *Indian Patent No.* 91827 — Improvements in or relating to an apparatus for automatically recording progressive changes in weights with special reference to thermogravimetric determinations (21.1.1964).

Patents Accepted during the Period

1. *Indian Patent No.* 79597 — Improvements in a continuous vertical counter-current solid-gas reactor (11.7.1963).
2. *Indian Patent No.* 79598 — An improved device for the continuous vapour phase decreasing of metallic wire and strip (22.4.1963).
3. *Indian Patent No.* 81402 — Improvements in or relating to electro-deposition of metals particularly manganese by direct current electrolysis of aqueous solutions containing metal ions (31.8.1963).
4. *Indian Patent No.* 81403 — Improvements in or relating to devices for the conversion of pig irons into high grade steels (1.7.1963).
5. *Indian Patent No.* 82191 — An improved jacketted electrolytic cell for the electro-deposition of metals and metallic oxides in general and manganese dioxide in particular (26.6.1963).
6. *Indian Patent No.* 83652 — Improvements in or relating to magnesite refractories (19.9.1963).
7. *Indian Patent No.* 83968 — A method for reconditioning the coated magnesium powders (19.9.1963).
8. *Indian Patent No.* 84670 — Improvements in or relating to electrolytic cells (10.1.1964).

Patents Sealed during the Period

1. *Indian Patent No.* 79597 — Improvements in a continuous vertical counter current solid-gas reactor (21.1.1964).
2. *Indian Patent No.* 79598 — An improved device for the continuous vapour phase degreasing of metallic wire and strip (23.10.1963).
3. *Indian Patent No.* 81402 — Improvements in or relating to electro-deposition of metals particularly manganese by direct current electrolysis of aqueous solutions containing metal ions (14.2.1964).
4. *Indian Patent No.* 81403 — Improvements in or relating to devices for the conversion of pig irons into high grade steels (6.1.1964).

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 welding flux.
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.

GENERAL

THE building of the three side wings of the Main Building Laboratory was completed during the period under review. Requisite units of machinery and research equipment were procured and their installation undertaken; the main items of equipment in these wings relate to vacuum and air melting furnaces and related equipment, Magnesium Pilot Plant, Electronic Research and Instrumentation Division, X-ray fluorescence spectroscope, Vibrophore pulsator set-up, the Radioactive Isotope Research Laboratory, etc. The side wings now house the Extraction Metallurgy Division, the Iron and Steel and Alloy Steel Divisions, Mineralogical and Petrological Sections, work on slag projects and Refractories Divisions' fundamental research section, Fundamental Corrosion Research Laboratory, Metallic Coating and Non-metallic Inclusions Research Groups, whilst the central wing contains the expanded Library, the Liaison, Information and Operational Research Division and Design Engineering Division on the top floor.

New bays have been constructed for the expanded foundry with a hot blast cupola and related equipment, Cryolite Pilot Plant and pattern shop in the Technological Block. Installation of the hot blast cupola in the foundry will be completed in the near future. Work relating to the integrated Mineral Beneficiation Pilot Plant, such as the

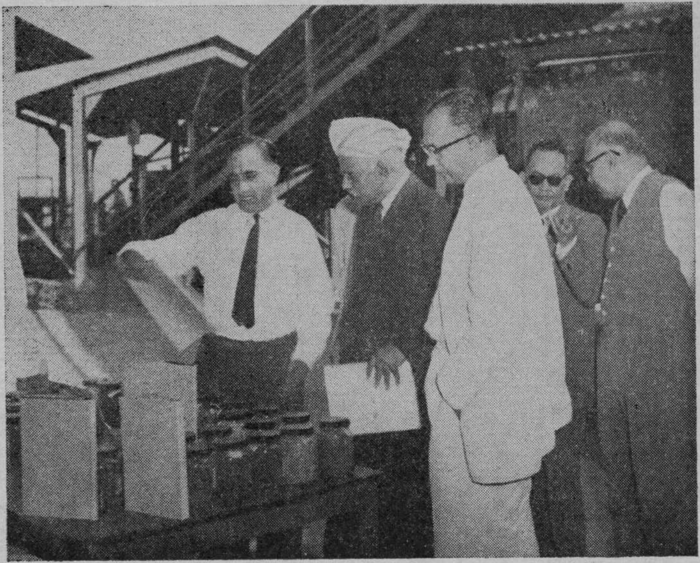

FIG. 92 — DR. B. R. NIJHAWAN, DIRECTOR, NATIONAL METALLURGICAL LABORATORY, EXPLAINING TO SIR RAMASWAMI MUDALIAR, CHAIRMAN, REVIEWING COMMITTEE, SOME OF THE PRODUCTS AND PROCESSES DEVELOPED AT THE NATIONAL METALLURGICAL LABORATORY

FIG. 93 — DR. HUSAIN ZAHEER, DIRECTOR-GENERAL, SCIENTIFIC & INDUSTRIAL RESEARCH, ADDRESSING AT DIRECTORS' CONFERENCE OF NATIONAL LABORATORIES HELD AT THE NATIONAL METALLURGICAL LABORATORY IN OCTOBER 1963

installation of the gas-holder, gas-producer, gas-linkage lines was completed during the period under review.

The construction of the Regional Foundry Stations at Batala, Madras and Howrah was completed. The Regional Foundry Station at Batala has started functioning in full swing; the Regional Foundry Stations at Madras and Howrah will also soon start functioning. The staff for these Regional Foundry Stations were trained for industry-oriented at site work in the field. The Marine Corrosion Research Station at Digha has been completed. The staff quarters at Digha are under construction.

The construction work relating to two C-II type residential quarters and 64 F, G and H residential type quarters was completed for the staff of the National Metallurgical Laboratory. Construction is underway for a further 30-unit multi-storeyed E type flats.

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 N.M.L. budget expenditure are furnished elsewhere in this report. Cooperative Banking Society of the National Metallurgical Laboratory continued its good work, indeed, throughout the year. An important highlight of the year's review was the visit to the National Metallurgical Laboratory of the C.S.I.R. Reviewing Committee appointed by the Government of India, under the chairmanship of Sir Ramaswami Mudaliar, and containing world-famous and distinguished scientists such as Sir Charles Goodeve, O.B.E. D.Sc., F.R.S., Director, British Iron and Steel Research Association, London, U.K., etc. Distinguished scientists and metallurgists, eminent dignitaries and important visitors who visited the National Metallurgical Laboratory were shown round and the research and development work and programme of the Laboratory explained to them at length.

A three-day Conference of the Directors and Heads of the National Laboratories and Cooperative Research Associations under the Council of Scientific & Industrial Research was held at the National Metallurgical Laboratory, from 30th October to 1st November 1963, under the chairmanship of Dr. S. Husain Zaheer, Director-General, Scientific & Industrial Research. In his inaugural address Dr. Husain Zaheer stressed the vital role of the National Laboratories in the scientific and industrial development of the country and pleaded for efficient coordination and greater effort in achieving the objectives set forth for each laboratory.

Dr. B. R. Nijhawan, Director, National Metallurgical Laboratory, welcomed the delegates to the Conference, which was attended by more than thirty Heads of National Laboratories and Cooperative Research Associations of the Council of Scientific & Industrial Research.

Exhibitions

During the period under review, the National Metallurgical Laboratory participated in the 14th German Industries Exhibition, West Berlin and Frankfurt, and the New York World Fair, New York.

BUDGET FIGURES FOR THE NATIONAL METALLURGICAL LABORATORY FOR THE YEAR 1963-64

(Figure in lakh of Rupees)
One lakh = 10^5 *rupees*

RECURRING

P-1	Pay of Officers	7·057
P-2	Pay of Establishment	9·118
P-3	Allowances	2·876
P-4	Contingencies	5·389
P-6	Maintenance	0·340
P-7	Chemicals, Apparatus and Equipment for Research	9·004
F.S.S.—	Fellowships and Scholarships	0·320
	Scientist Pool	0·244
	TOTAL	34·348

CAPITAL

P-5	(1)	Works	2·647
	(2)	Services	0·834
	(3)	Apparatus and Equipment	3·158
	(4)	Miscellaneous	0·385
		TOTAL	7·024

PILOT PLANT

PP-1	(i)	Equipment and Installation	4·405
	(ii)	Building and Services	2·071
PP-2		Provision for Staff	4·391
PP-3		Purchase of Raw Materials	0·502
PP-4		Miscellaneous and Contingencies	1·805
		TOTAL	13·174

LOW SHAFT FURNACE PROJECT

PP-1	(i)	Equipment and Installation	0·710
	(ii)	Building and Services	0·003
PP-2		Provision for Staff	2·752
PP-3		Purchase of Raw Materials	3·309
PP-4		Miscellaneous and Contingencies	3·669
		TOTAL	10·443

CONSTRUCTION OF STAFF QUARTERS	7·342
DEFENCE PROJECT	1·581
TOTAL	8·923
GRAND TOTAL	73·912

APPENDIX I

SCIENTIFIC PAPERS PUBLISHED

1. An X-ray Study of the Reaction of Calcium Oxide and Calcium Chloride in the Decomposition of Monazite — K. N. Kartha, *NML Technical Journal,* V (2), (4-9), 1963.
2. Symposium on 'Recent Developments in Iron and Steel Making with Special Reference to Indian Conditions'— A Review — R. M. Krishnan, B. R. Nijhawan & R. D. Lalkaka, *NML Technical Journal,* V (2), (10-16). 1963.
3. Dissolution of Steel and Absorption of Hydrogen by Steel during Acid Pickling in Presence of Inhibitor — A. K. Lahiri, N. G. Banerjee & T. Banerjee, *NML Technical Journal,* V (2), (33-37), 1963.
4. Atmospheric Corrosion Resistance of Aluminized M.S. Wire Exposed to Saline Atmosphere at Visakhapatnam, A.P.— S. M. Arora, A. N. Kapoor, P. K. Gupte & B. R. Nijhawan, *NML Technical Journal,* V (3), (5-9), 1963.
5. Observations on Oxygen Melting of Stainless Steel — R. K. Dubey, *NML Technical Journal,* V (3), (10-17), 1963.
6. Corrosion of Metals and Alloys Exposed at Jamshedpur — S. Rao Addanki, K. P. Mukherjee, A. K. Lahiri & T. Banerjee, *NML Technical Journal,* V (3), (18-24), 1963.
7. Determination of Hydrogen in Metals and Alloys by Carrier Gas Method — P. C. Debnath, N. G. Banerjee & B. C. Kar, *NML Technical Journal,* V (3), (25-27), 1963.
8. Tannin Method for the Estimation of Vanadium and its Separation from Phosphorus — M. N. Misra & H. Bhattacharya, *NML Technical Journal,* V (3), (28-31), 1963.
9. Effect of Copper on the Annealing of White Iron — T. A. Beck, Jatinder Mohan & P. K. Gupte, *NML Technical Journal,* V (4), (11-16), 1963.
10. Production of High Carbon Ferro-chrome — T. L. Srikrishna & P. P. Bhatnagar, *NML Technical Journal,* V (4), (17-21), 1963.
11. Mercury Hydrometer for Volume Determination — Ashimesh Dutt, Prithwis Chandra Sen & H. V. Bhaskar Rao, *NML Technical Journal,* V (4), (22-23), 1963.
12. Some Observations on Electrical Power Distribution System of Heavy Integrated Metallurgical Pilot Plants — H. Singh, *NML Technical Journal,* V (4), (24-28), 1963.

13. Thermal Beneficiation of Low Grade Chrome Ore from Kankauli Deposits, Ratnagiri Dist., Maharashtra — R. N. Misra & P. P. Bhatnagar, *NML Technical Journal,* V (4), (29-32), 1963.

14. Research and Development Work on the Utilization of Metallurgical Wastes at the National Metallurgical Laboratory — B. R. Nijhawan, *NML Technical Journal,* VI (1), (50-59), 1964.

15. Studies on Indian Refractories Clays — Part IV, Some Clays from Madras State — H. P. S. Murthy, A. V. Subrahmaniam & H. V. B. Rao, *Trans. Indian Ceramic Society,* **22** (3), (81-82), 1963.

16. Investigation on Suitability of Indigenous Asbestos for the Manufacture of Asbestos Cement Products — H. V. Bhaskar Rao & S. C. Jain, *Trans. Indian Ceramic Society,* **22** (4), (109-115), 1963.

17. A Study on the Suitability of Some Indian Dolomite for Use in Steel Plants — M. R. K. Rao, P. C. Sen & H. V. Bhaskar Rao, *Trans. Indian Ceramic Society,* **22** (4), (137-145), 1963.

18. A Study of the Hot Extrusion Characteristics of an Aluminium Alloy — B. N. Das & R. Choubey, *Trans. Indian Institute of Metals,* **16**, 1963.

19. Reaction of Titanium with Titanium Tetrachloride — S. R. Srinivasan & P. P. Bhatnagar, *Trans. Indian Institute of Metals,* **16**, 1963.

20. Studies on the 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,* **16**, 1963.

21. Role of Research in Foundry Technology, B. R. Nijhawan & R. M. Krishnan, *Foundry No., Indian Journal of Engineers,* 1964.

22. The Role of National Metallurgical Laboratory in Research on Minerals and Metals in India — B. R. Nijhawan, Wadia Commemoration Volume — 1964, Mining, Metallurgical & Geological Institute.

23. Some Problems of Iron and Steel Making in the Hindustan Steel Plants — B.R. Nijhawan & P. H. Kutar, *Proceedings of the Symposium on Recent Developments in Iron and Steel Making with Special Reference to Indian Conditions* — 1963, pp. 99-116.

24. Iron Production in Low-shaft Furnace Plants with Indian Raw Materials — A. B. Chatterjea & B. R. Nijhawan, *Proceedings of the Symposium on Recent Developments in Iron and Steel Making with Special Reference to Indian Conditions* — 1963, pp. 76-98.

25. Beneficiation, Sintering and Processing of Raw Materials for Iron and Steel Industry — P. I. A. Narayanan, G. V. Subramanya & G. P. Mathur, *Proceedings of the Symposium on Recent Developments in Iron and Steel Making with Special reference to Indian conditions* — 1963, pp. 63-75.

26. Some Aspects of Standardization in Ultrasonic Flame Detection — R. Choubey, B. N. Das & B. R. Nijhawan, *ISI Bulletin,* **15** (4), 1963.

The following paper was presented at the United Nations Symposium on 'Iron & Steel Making' held in Geneva and Prague, November 1963:

27. Growth Pattern of Iron and Steel Industry in India's Economic Development — B. R. Nijhawan.

The following papers were presented at the 17th Annual Technical Meeting of Indian Institute of Metals held in January 1964:

28. Some Studies on Sheet Metal Forming — K. N. Ghosh and B. N. Das.
29. Electrical Conductivity of Aluminium Silicon and Aluminium Silicon Magnesium Alloys — Manjit Singh and Rajendra Kumar.
30. Studies on the Properties of Binary Copper-Manganese Alloys — A. K. Lahiri, K. P. Mukherjee & T. Banerjee.
31. Studies on the Chlorination of Vanadium Bearing Titaniferous Magnetites — S. R. Sreenivasan, S. S. Bhoray & P. P. Bhatnagar.
32. Transformation Characteristics of Carbides in Low Alloy Steels — J. K. Mukherjee & B. R. Nijhawan.
33. Some Investigations on Basic Lined Side Blown Converter with Indian Pig Iron — A. B. Chatterjea, J. Mohan & B. R. Nijhawan.
34. Reversion on Age Hardening — Ved Prakash.
35. Some Studies for Simplifying the Manufacturing Procedure for Making Porous Bronze Bearing — S. Ranganathan & P. K. Gupte.
36. Mechanism of Sintering — P. K. Gupte, V. S. Bhandary & B. R. Nijhawan.

The following papers were presented at the Annual Technical Session of Indian Ceramic Society held in February 1964:

37. Comparative Study of Some Methods of Compaction of Mixes of Dry Press Consistency — H. P. S. Murthy, M. C. Kundra & H. V. Bhaskar Rao.
38. Comparative Study of the Hydraulic Press and Pneumatic Hammer as Brick Forming Machines — H. P. S. Murthy, M. C. Kundra & H. V. Bhaskar Rao.
39. Examination of Some Refractory Clays by D.T.A., X-ray and Electron Microscope Technique — T. V. Prasad.

The following papers were presented at the Symposium on 'Steel Plant Refractories' organized by Indian Ceramic Society in February 1964:

40. Steel Plant Refractories — Research and Development at the National Metallurgical Laboratory — H. V. Bhaskar Rao.
41. The Influence of Magnesium Oxide on the Properties of Some High Alumina Cements — B. V. S. Yadavalli, A. K. Bose, T. V. Prasad & H. V. Bhaskar Rao.
42. Some Observations on the Manufacture of Carbon Refractories in India — H. P. S. Murthy.

43. Utilization of Almorah Magnesite for Steel Plant Refractories — M. R. K. Rao, A. Dutt, P. C. Sen & H. V. Bhaskar Rao.

44. Some Recent Developments in the Production of Clay Graphite Products for Handling Steel — T. V. Prasad & H. P. S. Murthy.

45. Growth Pattern of Steel Plant Refractory Industry in India — H. V. Bhaskar Rao & B. R. Nijhawan.

The following papers were presented at the Symposium on 'Utilization of Metallurgical Wastes' organized by National Metallurgical Laboratory in March 1964:

46. Utilization of Fine Grained Raw Materials by Briquetting for Smelting of Ferro-Manganese — A. B. Chatterjea, S. R. Ghosh & J. Goswami.

47. Utilization of Important Mining and Metallurgical Wastes Employing Mineral Beneficiation Techniques — M. S. Chopra, G. P. Mathur and P. I. A. Narayanan.

48. Research and Development Work on the Utilization of Metallurgical Wastes at the National Metallurgical Laboratory — B. R. Nijhawan.

49. 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.

50. Investigations on Granulated Low Shaft Furnace Slag for the Production of Slag Cements — B. N. Singh, P. C. Debnath, B. C. Kar & B. R. Nijhawan.

51. 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.

52. Utilization of Low Shaft Furnace Slag as Light Weight Aggregate for Insulation Concrete — Narinder Singh, K. N. Srivastava, R. M. Krishnan & B. R. Nijhawan.

53. Utilization of Red Mud — C. Sankaran & P. P. Bhatnagar.

54. Production of Secondary Aluminium — A Review — M. C. Sen & T. Banerjee.

55. Recovery of Zinc from Scraps and Wastes — B. C. Mukherjee, S. C. Aush, M. C. Sen & T. Banerjee.

The following papers were presented at the Annual Convention of Indian Institute of Foundrymen held in July, 1963.

56. Break Down Characteristics of CO_2 Cores — B. V. Somayajulu, P. K. Gupte & B. R. Nijhawan, [Published in *Indian Construction News*, **12** (7), 1963].

57. Studies on the Hot Strength Characteristics of Some Indigenous Bentonite — A. T. Chirayath, V. S. Bhandary & P. K. Gupte [Published in *Indian Construction News*, **12** (7), 1963].

APPENDIX II

SCIENTIFIC INVESTIGATIONS COMPLETED AND REPORTS PREPARED

1. Moulding Characteristics of Madras Silica Sand: Part I — K. S. Vijayanarayanan, V. S. Bhandary & P. K. Gupte (I.R.265/63).
2. Electric Smelting of Salem Magnetite Briquettes in the 500 KVA Submerged Arc Furnace — Staff of Extractive Metallurgy Divison (I.R.266/63).
3. Beneficiation of Dolomite from Tisco — P. V. Raman, G. V. Subramanya, P. I. A. Narayanan (I.R.267/63).
4. Investigation on the Australian Bentonite under the International Working Group on Bonding Clays — A. T. Chirayath, Jatinder Mohan, V. S. Bhandary & P. K. Gupte (I.R.268/63).
5. Investigation Report on Electrolytic Refining of Ordnance Lead Ingot — S. K. Roychowdhury, S. C. Aush & P. K. Som (I.R.269/63).
6. Investigation on Corrosion of Rails Used on Indian Railway Tracks — B. N. Das, P. Basak & B. R. Nijhawan (I.R.270/63).
7. Pilot Plant Studies on Beneficiation of a Float Ore Sample of Low-Grade Magnetite from Salem -- G. S. Ramakrishna Rao, K. N. Rakshit & P. I. A. Narayanan (I.R. 271/63).
8. Studies on the Production of High Basicity Sinters from Noamundi Iron Ore Fines and Blue Dust — P. K. Sinha, G. V. Subramanya and P. I. A. Narayanan (I.R. 272/63).
9. Recovery of Aluminium from Aluminium Dross — G. Basak, V. S. Sampath, P. P. Bhatnagar (I.R.273/63).
10. Moulding and Core Characteristics of A.C.C. Sand — Amitava Das, V. S. Bhandary & P. K. Gupte (I.R.274/63).
11. Beneficiation and Pelletization Studies on Iron Ore Fines from Pale Mines, Goa — S. K. Banerjee, B. V. S. Yedavalli, G. V. Subramanya & P. I. A. Narayanan (I.R. 275/63).
12. Further Pilot Plant Studies on Beneficiation Iron Ore from Bolani for Durgapur Steel Plant for Hindustan Steel Limited — M. V. Ranganathan, A. Peravadhanulu, S. B. Dasgupta, G. S. Ramakrishna Rao, G. P. Mathur & P. I. A. Narayanan (I.R.276/63).
13. Reduction of Silica Content in a Magnesite Sample from Tisco Employing Flotation — P. V. Raman, G. V. Subramanya & P. I. A. Narayanan (I.R.277/63).
14. Investigation of P.B.I. Bentonite, Kashmir (Jammu & Kashmir State) — Avtar Singh, A. T. Chirayath, V. S. Bhandary & P. K. Gupte (I.R.278/63).

15. Investigation on the S.B.I. (Kashmir) Bentonite Received from Director of Mining & Geology, Jammu & Kashmir Govt.— Avtar Singh, V. S. Bhandary, P. K. Gupte (I.R.279/63).

16. Investigation on Cutch Bentonite Received from M/s. Mukund Iron & Steel Works Ltd., Kurla, Bombay — T. A. Beck, V. S. Bhandary & P. K. Gupte (I.R.280/63).

17. Investigation on the Madras Bentonite Received from Fomecs Pr. Ltd., D-38, Industrial Estate, Rajajinagar, Bangalore-10 — A. T. Chirayath, V. S. Bhandary, P. K. Gupte (I.R.281/63).

18 Moulding Characteristics of Madras Silica Sand: Part II — K. S. Vijayanarayanan, V. S. Bhandary & P. K. Gupte (I.R.282/63).

19. Moulding Characteristics of K.S. Sand (Kanhayasthan Hill Deposits) — A. Das, V. S. Bhandary and P. K. Gupte (I.R.283/63).

20. The Suitability of Jayanti Dolomite for Sintering in Shaft Kilns — P. C. Sen, M. R. K. Rao & H. V. Bhaskar Rao (I.R.284/63).

21. Reduction of Silica Content of Limestone Samples from Sirka after Calcination — P. V. Raman, P. K. Sinha, G. V. Subramanya & P. I. A. Narayanan (I.R.285/64).

22. Further Pilot Plant Studies on Beneficiation and Sintering of a Mixed Iron Ore Sample from (1) the Upper Portion of Main Haulage Road and (2) Bench 3 of Hill No. 1 of Kiriburu Mines of National Mineral Development Corporation — M. V. Ranganathan, A. Peravadhanulu, S. B. Das Gupta, G. S. Ramakrishna Rao, G. P. Mathur & P. I. A. Narayanan (I.R.286/64).

23. Beneficiation of a Fine Grained Fluorspar Sample from Ambadonagar, Gujarat State — Messrs. R. Ganesh, G. V. Subramanaya & P. I. A. Narayanan (I.R.287/64).

24. Report of the Operation of Low-shaft Furnace Pilot Plant at the National Metallurgical Laboratory for the Production of Pig Iron with Iron Ore and Limestone from Nepal (I.P.R.4/63).

25. The Operations of Low-shaft Furnace Pilot Plant at the National Metallurgical Laboratory for the Production of Pig Iron with Raw Materials from Rajasthan — (I.P.R.5/63).

26. Rapid Routine Method for the Determination of Manganese in the Nickel Free Stainless Steel — A. C. Biswas & H. P. Bhattacharjee (R.R.137/63).

27. Effect of Pressure and Influence of Bonds on the Compaction of Granular Ceramic Powders — H. P. S. Murthy, M. C. Kundra & H. V. Bhaskar Rao (R.R.138/63).

28. Effect of Copper on the Annealing of White Iron — T. A. Beck, Jatinder Mohan & P. K. Gupte (R.R.139/63).

29. Beneficiation, Sintering and Processing of Raw Materials for Iron and Steel Industry — P. I. A. Narayanan, G. V. Subramanya & G. P. Mathur (R.R.140/63).

30. Iron Production in Low-Shaft Furnace Plants with Indian Raw Materials — A. B. Chatterjea & B. R. Nijhawan (R.R.141/63).

31. Some Problems of Steel Making in the Hindustan Steel Plants — P. H. Kutar & B. R. Nijhawan (R.R.142/63).
32. Studies on the Utilization of Almorah Magnesite: Part I, Occurrence and Mineralogy — M. R. K. Rao, Ashimesh Dutt, P. C. Sen & H. V. Bhaskar Rao (R.R.143/63).
33. Studies on the Utilization of Almorah Magnesite: Part II, Effect of Heat on the Constitution, Grain Growth and Densification — M.R.K. Rao, Ashimesh Dutt, P. C. Sen & H. V. Bhaskar Rao (R.R.144/63).
34. Utilization of Low Shaft Furnace Slag as Aggregate for Light Weight Insulation Concrete — Narinder Singh, K. N. Srivastava & B. R. Nijhawan (R.R.145/63).
35. Reaction of Titanium with Titanium Tetrachloride — S. R. Srinivasan & P. P. Bhatnagar (R.R.146/63).
36. Studies on Silico-thermic Reduction of Dolomite for the Production of Magnesium — R. N. Misra, V. S. Sampath & P. P. Bhatnagar (R.R.147/63).
37. Siliconization of Basic Pig Iron — B. V. Somayajulu, R. C. Arora & P. K. Gupte (R.R.148/63).
38. Chromate Passivation of Zinc — S. Rao, Addanki & A. K. Lahiri (R.R.149/63).
39. Thermal Beneficiation of Low Grade Chrome Ore from Enuconda Deposits, West Godavari Dist., Andhra Pradesh — Miss S. Samanta, R. N. Misra & P. P. Bhatnagar (R.R.150/63).
40. Thermal Beneficiation of Low-Grade Chrome Ore from Vagda Deposits, Ratnagiri Dist., Maharashtra — Miss S. Samanta, R. N. Misra & P. P. Bhatnagar (R.R.151/63).
41. Some Studies on Sheet Metal Forming — K. N. Ghosh & B. N. Das (R.R.152/63).
42. Studies on the Chlorination of Vanadium Bearing Titaniferous Magnetites — S. R. Srinivasan, S. S. Bhoray & P. P. Bhatnagar (R.R.153/64).
43. Studies on the Properties of Binary Copper-Manganese Alloys — A. K. Lahiri, K. P. Mukherjee & T. Banerjee (R.R.154/64).
44. Transformation Characteristics of Carbides in Low Alloy Steel — J. K. Mukherjee & B. R. Nijhawan (I.R.155/64).
45. Some Studies for Simplifying the Manufacturing Procedure for Making Porous Bronze Bearings — S. Ranganathan & P. K. Gupte (R.R.156/64).
46. Electrical Conductivity of Al-Si and Al-Si-Mg Alloys — Manjit Singh & Dr. Rajendra Kumar (R.R.157/64).
47. A new Method of Modification of Aluminium-Silicon Alloys — S. S. Bhatnagar, G. G. Nair, P. K. Gupte & B. R. Nijhawan (R.R.158/64).
48. Studies on Indian Refractory Clays: Part V — Examination of Some Refractory Clays by Differential Thermal Analysis, X-ray and Electron Microscope Technique — T. V. Prasad (R.R.159/64).

49. Refractories Pilot Plant Studies: Part II — Comparative Study of Some Methods of Compaction of Mixes of Dry Press Consistency — H. P. S. Murthy, M. C. Kundra & H. V. Bhaskar Rao (R.R.160/64).

50. Studies on the Utilization of Almorah Magnesite for Steel Plants Refractories — M.R.K. Rao, Ashimesh Dutt, P. C. Sen & H. V. Bhaskar Rao (R.R.161/64).

51. Comparative Study of Hydraulic Press and Pneumatic Hammer as Brick Forming Machines — H. P. S. Murthy, M. C. Kundra & H. V. Bhaskar Rao (R.R.162/64).

52. Some Observations on the Effect of Certain Constituents of High Alumina Cements on Their Properties — B. V. S. Yedavalli, A. K. Bose, T. V. Prasad & H. V. Bhaskar Rao (R.R.163/64).

53. Utilization of Red Mud — C. Sankaran, P. P. Bhatnagar (R.R.164/64).

54. Utilization of Low-Shaft Furnace Slag as Light Weight Aggregates for Insulation Concrete — Narinder Singh, K. N. Srivastava, R. M. Krishnan & B. R. Nijhawan (R.R.165/64).

55. Recovery of Zinc from Scraps and Wastes — B. C. Mukherjee, S. C. Aush, M. C. Sen & T. Banerjee (R.R.166/64).

56. Thermogravimetric Studies on the Low Temperature Pyrolysis of Some Coals — H. P. S. Murthy & B. Chatterjee (R.R.167/64).

57. Certain Factors Influencing the Production of Dense Carbon Aggregate from Low and High Volatile Carbonaceous Substances — H. P. S. Murthy & B. Chatterjee (R.R.168/64).

58. Utilization of Important Mining and Metallurgical Wastes Employing Mineral Beneficiation Technique — M. S. Chopra, G. P. Mathur & P. I. A. Narayanan (R.R. 169/64).

59. Utilization of Fine Grained Raw Materials for Smelting of Ferro-Manganese — A. B. Chatterjea, S. R. Ghosh & J. Goswami (R.R.170/64).

60. Utilization of Coke Breeze Production of Pig Iron in Small Scale — A. B. Chatterjea, J. Goswami, S. K. Biswas, R. Santok Singh & J. S. Padan (R.R.171/64).

61. 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 (R.R.172/64).

APPENDIX III

MAJOR SPONSORED PROJECTS AT THE NATIONAL METALLURGICAL LABORATORY

Sl.	*Title*	*Sponsor*
1.	Pilot plant studies on beneficiation and sintering of Barsua iron ore for Rourkela Steel Plant of Hindustan Steel Ltd.	Hindustan Steel Ltd.
2.	Pilot plant studies on beneficiation and sintering of Rajhara iron ore for Bhilai Steel Plant of Hindustan Steel Ltd.	Hindustan Steel Ltd.
3.	Pilot plant studies on beneficiation and sintering of Bolani iron ore.	Hindustan Steel Ltd.
4.	Reducibility of Bolani iron ores and sinters.	Hindustan Steel Ltd.
5.	Pilot plant studies on beneficiation and agglomeration of a low-grade limestone sample from Purnapani Quarries, Orissa, for Hindustan Steel Ltd.	Hindustan Steel Ltd.
6.	Further pilot plant studies on beneficiation of iron ore from Bolani for Durgapur Steel Plant of Hindustan Steel Ltd.	Bird & Co.
7.	Studies on the production of high basicity sinters from the beneficiated Bolani iron ore fines for the Durgapur Steel Plant of Hindustan Steel Ltd.	Hindustan Steel Ltd.
8.	Pilot plant studies on beneficiation of limestone samples from Nandini Mines of Bhilai Steel Plant of Hindustan Steel Ltd.	Hindustan Steel Ltd.
9.	Reduction of silica content in sample No. I, II, III and IV from Purnapani Quarries of Hindustan Steel Ltd.	Hindustan Steel Ltd.
10.	A study of Hirri, Hardi and Baraduar dolomites for use in the shaft kilns at Hindustan Steel Ltd., Rourkela.	Hindustan Steel Ltd.
11.	Sintering of dolomite in shaft kiln of Hindustan Steel Plant, Rourkela.	Hindustan Steel Ltd.
12.	Beneficiation and sintering studies with an iron ore sample from Bailadilla Mines (Deposit-14) of National Mineral Development Corporation.	National Mineral Development Corporation Ltd.

13.	Batch and pilot plant flotation studies on Khetri copper ore.	National Mineral Development Corporation Ltd.
14.	Pilot plant studies on beneficiation and sintering of Kiriburu iron ore from National Mineral Development Corporation Ltd.	National Mineral Development Corporation Ltd.
15.	Recovery of diamond from diamondiferous tuff and gravel sample from Panna Area, Madhya Pradesh.	National Mineral Development Corporation Ltd.
16.	Further pilot plant studies on beneficiation and sintering of mixed iron ore sample from (1) Upper portion of main haulage road and (2) bench 3 of hill No. 1 of Kiriburu mines.	Bokaro Steel Ltd.
17.	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 Kiriburu iron ore mines.	Bokaro Steel Ltd.
18.	Electric smelting of Salem magnetite briquettes in the 500 K.V.A. submerged Arc furnace.	Salem Steel Project Ltd.
19.	Study of the distribution of hematite in the band 1, Kanjermalai magnetite deposit, Salem Dt., Madras.	Salem Steel Project Ltd.
20.	Pilot plant production of concentrate from low-grade Salem magnetite.	Salem Steel Project Ltd.
21.	Sintering studies on the magnetic concentrate produced from low-grade Salem magnetite ore.	Salem Steel Project Ltd.
22.	Reducibility of Salem magnetite ore.	Salem Steel Project Ltd.
23.	Pilot plant studies on beneficiation of a float ore sample of low-grade magnetite from Salem.	Salem Steel Project Ltd.
24.	Beneficiation and Pelletization studies on iron ore fines from Pale mines, Goa.	Chowgale & Co., Goa.
25.	Studies on the production of high basicity sinters from Noamundi iron ore fines and blue dust.	Tata Iron & Steel Co.
26.	Studies on the beneficiation of iron ores from Tisco.	Tata Iron & Steel Co.
27.	Pilot plant beneficiation studies on a limestone from Tisco and proposals for setting up a treatment plant of capacity 250 tons/day.	Tata Iron & Steel Co.
28.	Further pilot plant beneficiation studies on limestone from Tisco and proposals for setting up a 300 tons/day treatment plant.	Tata Iron & Steel Co.
29.	Pre-concentration of lead zinc ore from Zawar employing heavy media separation.	Metal Corporation of India, Calcutta.
30.	Beneficiation studies on lateritoid manganese ore from Malaya.	Eastern Mining & Metal Co. Ltd., Federation of Malaya.

31.	Beneficiation studies on a complex low-grade copper-lead-zinc ore from Bhotang Mines, Rangpo, Sikkim.	Indian Bureau of Mines.
32.	Recovery of metallic values from industrial brass dross.	Devidayal Sales (P) Ltd. Bombay.
33.	Recovery of aluminium from aluminium dross.	M/s. V. K. Paranjpe, Jamshedpur.
34.	Recovery of metallics from zinc ash.	Indian Tube Co. Ltd., Jamshedpur.
35.	Electrolytic refining of ordnance lead ingot.	Ministry of Defence.
36.	Reconditioning of Magnesium Powder.	Ministry of Defence.
37.	Suitability of Jayanti dolomite for sintering in shaft kilns.	Bengal limestone (P) Ltd., Calcutta.
38.	Pilot plant studies on a gypsum sample.	Bikaner Gypsum P. Ltd.
39.	Batch and pilot plant Beneficiation on low-grade fluorspar from Rajasthan for production of acid and metallurgical grade spare.	Directorate of Geology & Mining, Govt. of Rajasthan.
40.	Agglomeration of fluorspar flotation concentrate obtained from low-grade fluorspar from Rajasthan.	Directorate of Geology & Mining, Govt. of Rajasthan.
41.	The Operation of Low-shaft Furnace Pilot Plant at the National Metallurgical Laboratory for the production of foundry grades of pig iron with Andhra Pradesh raw materials.	Govt. of Andhra Pradesh.
42.	The operation of low-shaft furnace pilot plant at National Metallurgical Laboratory for the production of pig iron with East Punjab raw materials from Mahindergarh.	Govt. of East Punjab.
43.	The Operation of Low-shaft Furnace Pilot Plant at National Metallurgical Laboratory for the production of pig iron with raw material from Maharashtra State.	Govt. of Maharashtra.
44.	The Operation of Low-shaft Furnace Pilot Plant at the National Metallurgical Laboratory for the production of pig iron with iron ore & limestone from Nepal.	His Majesty's Govt. of Nepal.
45.	The Operation of Low-shaft Furnace Pilot Plant at the National Metallurgical Laboratory for the production of pig iron with raw materials from Rajasthan.	Govt. of Rajasthan.
46.	Development of aluminium base bearing alloys.	Indian Railways.
47.	Development of suitable indigenous Armco steel.	Indian Railways.
48.	Development of electrical contacts based on indigenous material.	Indian Railways.
49.	Development of contact springs based on indigenously available material.	Indian Railways.

50.	Development of friction fabric material for bogie, pivot, lined truck slides and slides of locomotives.	Indian Railways.
51.	Evolution of a suitable weldability test for assessing weldability of steels to IS: 226 ASW quality (at present IS: 2062-1962).	Indian Railways.
52.	Development of high speed steel ingot.	Indian Railways.