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NATIONAL METALLURGICAL LABORATORY
COUNCIL OF SCIENTIFIC & INDUSTRIAL RESEARCH
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INTRODUCTION

The National Metallurgical Laboratory, during the period under review, continued to extend its contacts with industries by undertaking large number of industrial problems and consolidating its scope of work by making steady progress in its multiple research and pilot plant projects. Pilot plant scale washing and pelletization studies were undertaken on iron ores from Donimalai on behalf of National Mineral Development Corporation who are interested in setting up a pelletization plant at Donimalai in Mysore State. The Laboratory developed flowsheet along with list of equipment needed for a 1000 tonnes/day beneficiation plant on behalf of Hindustan Copper Ltd. for the production of smelter grade copper-nickel concentrate and molybdenum concentrate from low grade copper ores from Rakha Mines. On behalf of Hindustan Zinc Ltd., tonnage quantities of phosphate concentrate was produced from phosphate rock from Udaipur for production of super phosphate. Studies made on phosphate rock from Uttar Pradesh showed that the rock can be upgraded to produce concentrate suitable for super-phosphate manufacture.

Pilot Plant trials for production of silico-chrome by single stage electric smelting process was successfully completed and the know-how obtained was furnished to the firm who sponsored the work to start production of silico-chrome. Systematic investigations are underway on the extraction characteristics of nickel from low-grade nickeliferous ores from Sukhinda, Orissa.

Studies on the development of substitute alloys have maintained steady progress. The aluminium conductor developed was found to posses satisfactory electrical, mechanical and corrosion resistance properties and the behaviour of the alloy under the 'Properzi' method of working is being studied.

The sub-merged arc welding flux produced by the licencee firms based on NML developed know-how has given excellent performance in welding pressure vessels. The aluminium based sacrificial anode developed by the Laboratory has given very satisfactory service under actual service condition. Various Port authorities and anode manufacturers have shown considerable interest in this alloy. The Laboratory acted as consultant to Calcutta Metropolitan Water & Sanitation Authority in scrutinising data and preparation of a feasibility report on the application of cathodic protection for under ground water pipe lines. Bright nickel plating process developed in the laboratory is under negotiation for commercial scale implementation. Successful work was conducted on plating of gold or its alloys on watch cases and watch straps for decorative and protective purposes. Design and fabrication of different types of apparatus and pilot plant equipment were continued during the period.

Active collaboration was maintained with Indian Standards Institution in formulation of specifications and with Research Design & Standard Organisation of Ministry of Railways on research projects of interest. Ordnance establishments were supplied with different types of special materials from time to time to meet their requirements.

A Symposium on "Recent Developments in Non-ferrous Metals Technology" was organised. The Symposium was attended by a large number of Indian and foreign delegates. Sixty two papers out of seventy five papers received were presented and discussed in seven technical sessions.

The Laboratory published during the year Proceedings of the Symposium on "Metallurgy of Substitute Ferrous and Non-ferrous Alloys" organised earlier. A monograph on "Structure of Electro-deposited Manganese" is under publication.

The various Foundry Stations of the Laboratory continued to extend their assistance to local foundries and conducted investigations on foundry raw materials available in nearby places. The Marine Corrosion Research Station at Digha studied a number of problems on corrosion under marine atmosphere.

A considerable number of short term investigations and specification tests were conducted on behalf of industries and different organization. A large number of technical enquiries were attended and relevant suggestions were furnished. Standard samples prepared in the Laboratory were supplied to a number of research and industrial organizations for analytical work.

The Laboratory imparted training to students, representatives of industrial firms etc. in various branches of metallurgy.

During the period under review the following patents were taken:

Applied for:

Indian Patent No.	Title.
116520	An improved and modified process for the manufacture of ferrite magnets.
118916	A process for stabilisation of ferro-silicon.
118902	A process for the production of electrolytically deposited manganese dioxide containing iron.
119958	Improvement in or relating to the production of aluminium alloy anodes for cathodic production.

Accepted:

106906	An improved method for removal of iron from ferruginous chrome and manganese ores.
107982	Stable castable suspensions of non-plastic alumino-silicate materials and methods of making the same.
108081	Refractory ramming, plastering and patching mixes.
108583	Chemically bonded forsterite refractories and methods of their preparation.
110834	Improvements in or relating to the production of fluoboric acid.
112574	A simple process for prehating the air blast in cupola

Sealed:

Indian Patent No.	Title.
105895	A pneumatic process for the conversion of phosphoric pig irons to steels.
106906	An improved method for removal of iron from ferruginous chrome and manganese ores.
107982	Stable castable suspensions of non plastic alumino-silicate materials and methods of making the same.
108081	Refractory ramming, plastering and patching mixes.
108583	Chemically bonded forsterite refractories and methods of their preparation.

The following processes which were earlier released were licensed to additional firms for commercial production.

1. Hot-dip aluminising of ferrous materials.
2. Production of electrolytic manganese metal.

A brief resume of the progress of various projects and other activities is furnished in the Chapters to follow.

RESEARCH AND INVESTIGATION PROJECTS

1.0 Pilot Plant Studies on Washing & Pelletization of Donimalai Iron Ores for National Mineral Development Corporation.

Donimalai iron ore samples weighing about 150 tonnes consisting of five different types of ores were sent by National Mineral Development Corporation Ltd., for investigation so as to set up a ore screening, handling and pelletisation plant at Donimalai, in Mysore State. The project report relating to the plant will be prepared by M/s. M.N. Dastur & Co. on the basis of test results from NML. The work involved crushing the sample to two different sizes viz., 100 mm and 30 mm and conducting dry and wet screening tests to produce -6 mm fines to be used as pelletising feed.

The chemical analyses of the five different types of ores and the proportions in which they were mixed to produce the composite sample as suggested by N.M.D.C. are given in Table 1.

Table 1 - Chemical Analyses of Different Types of Donimalai Iron Ore.

Samples.	Fe	SiO_2	Al_2O_3	Mixing Ratio
Type 2	64.97	1.72	2.96	4.0
Type 3	66.08	1.20	1.68	14.3
Type 4	65.52	1.0	2.56	15.7
Type 5	65.52	1.76	2.32	48.5
Type 6	64.71	1.68	2.72	17.5
Composite (Actual) sample	65.82	1.64	2.30	100.0

After selecting the most suitable method of producing the -6 mm fines, further tests on pelletisation characteristics are to be studied. Sieve analysis of as received sample was carried out on a 10 tonne sample and the products are being chemically analysed. Dry screening tests in a continuous vibrating screen after crushing the ore to -100 mm to -30 mm sizes have been completed. Wet screening, chemical analysis and other tests are in progress. A pot grate furnace specially adopted for testing the pellet heat-hardening characteristics is being fabricated for the investigation.

1.1 Iron Ore from Kiriburu North Block:

A 100 tonne sample of iron ore, prepared by mixing (1) porous laminated, (2) hard laminated ore, (3) soft biscuity ore, (4) lateritic ore and (5) laterite, was received from Kiruburu iron ore mines of National Mineral Development Corporation Ltd., with a view to study the flowsheet to be adopted after modification of Kiriburu iron ore washing plant, for increased production for supply of ore to Bokaro Steel Plant.

The mixed sample as received assayed 61.6% Fe, 1.3% SiO_2 and 4.2% Al_2O_3. A blue dust sample assaying 64.6% Fe, 1.2% SiO_2 and 3.9% Al_2O_3 was also received for mixing with the above mentioned sample. But it was observed from the size and chemical analysis of blue dust that mixing of blue dust with iron ore before washing would cause loss of about 34% by weight of high grade blue dust in the slime. Hence, it would be better to dry screen the blue dust along with the products from tertiary crusher.

The mixed sample was screened over a 4" (101.6 mm) screen. The +4" fraction which was 23% by weight, was divided into two parts; one part was crushed in a jaw crusher set at 2½" (63.5 mm) and the other at 1½" setting. The crushed products were separately washed at 1½", 3/8" and 6 mesh sizes. The -6 mesh fractions were deslimed in classifier. The -1½" +3/8" washed lumps ores obtained from +4" fractions crushed at 2½" and 1½" settings assayed 64.8% Fe, 0.8% SiO_2 and 3.2% Al_2O_3 and 64.8% Fe, 0.7% SiO_2 and 2.9% Al_2O_3 respectively. The -3/8" washed fine ores assayed 62.6% Fe, 0.9% SiO_2 and 4.0% Al_2O_3 and 62.8% Fe, 0.9% SiO_2 and 4.2% Al_2O_3 respectively.

The -4" fraction which was 77% by weight was also subjected to washing under the above set up. The -1½" +3/8" washed lump ore and the -3/8" washed fines assayed 63.3% Fe, 0.9% SiO_2 and 3.1% Al_2O_3 and 62.7% Fe, 1.2% SiO_2 and 4.2% Al_2O_3 respectively.

The +1½" washed ore obtained from these 3 wet screening tests amounting to 23.4% by weight was crushed to -1½" size and dry screened on 3/8" and 6 mesh screens. The -1½" +3/8" and -3/8" fractions obtained by the above test were mixed with the respective fractions obtained by washing. Crushing, washing and wet screening produced lump ore of size -1½" +3/8" weighing 58.2% and assayed 63.9% Fe, 1.0% SiO_2 and 3.2% Al_2O_3. Fine ore -3/8" +100 mesh weighing 27.45% assayed 62.7% Fe, 1.3% SiO_2 and 4.7% Al_2O_3 and slim weighing 14.35% assayed 50.3% Fe, 3.7% SiO_2 and 9.4% Al_2O_3.

It was observed from the material balance sheet that the total amount of -6 mesh to be fed to the classifiers would be 419.3 T.P.H. But the combined capacity of the two existing classifiers is only 200 T.P.H. So the two new classifiers should have capacity to handle approximately 225 to 250 tonnes of fines per hour. Settling rate tests were conducted with a mixed sample slimes obtained from different washing tests. The tests were conducted at different pulp densities varying from 7% to 33.3% solids.

1.2 Crushing and Wet Screening Tests with an Iron Ore Sample from Deposit No. 5 of Bailadila Iron Ore Mines of National Mineral Development Corporation.

A high grade compact and laminated variety of iron ore from deposit No. 5 of Bailadila Iron Ore Mines was received from N.M.D.C. for tests to determine the quantity and quality of fines that would be produced when the ore was crushed to -6" and -4" sizes and wet screened.

Due to hard nature of ore, only 2.8 and 3.6% by weight of -3/8" and -1/4" size washed fines, were produced from -6" and -4" size ore respectively. The fines were of a high grade, assaying 64.4% and 65.8% Fe in the two cases. Agglomeration studies with the fines were not conducted as the sample under investigation was subsequently reported by N.M.D.C. to be not a representative one and was drawn only from the massive iron ore zone of the deposit. Drop shatter tests with -6" +3/8" and -4" +1/4" washed lumps, thrice from a height of 10 M, produced only 6.5% and 4.3% of -3/8" and -1/4" fines respectively indicating that the amount of fines likely to be produced from this ore during export to Japan would be quite low.

2.0 Batch as well as Pilot Plant Beneficiation Studies with a Low-grade Fluorspar from Ambadongar District, Baroda and Detailed Proposals for Setting up of a 500 tonnes per day Beneficiation Plant for the Gujarat Mineral Development Corporation Limited.

Batch and pilot plant investigations were conducted on the development of a flowsheet and the economic evaluation of the process for setting up of a beneficiation plant of 500 tonnes per day capacity for simultaneous production of acid and metallurgical grade concentrates from a low grade fluorspar deposit from Ambadongar, Baroda Dist. Gujarat State.

The sample investigated assayed 27.99% CaF_2, 2.1% $CaCO_3$, 49.12% SiO_2, 8.25% Al_2O_3, 7.40% Fe_2O_3, 0.16% S, 0.28% Ba traces of Pb and Cu. Quartz, fluorite, feldspar and calcite were the major constituent minerals in the ore with small amounts of barite and sulphide minerals. The sample contained considerable amount of clay which could be removed by scrubbing the ore.

Laboratory scale studies indicated that simultaneous production of an acid grade concentrate assaying 98.2% CaF_2 and metallurgical grade concentrate assaying 90.9% CaF_2 with recoveries of 68.1% and 13.0% CaF_2 respectively could be achieved by employing flotation. The process consisted broadly of two stages, the first for the production of a rougher fluorspar float and the second stage for cleaning of this rougher float after regrinding and hot conditioning. No difficulty is anticipated in actual plant practice for producing 100% of the concentrate in the form of either acid grade or metallurgical grade, by employing the above method.

Pilot plant trials were conducted on a 190 tonne sample and the results more or less confirmed laboratory test results. Bond's Work Index and work input were determined for proper sizing of ball mill and regrind ball mill. Settling tests for determining thickener sizes and filtration tests for calculating filter area were also conducted.

A flowsheet with material balance (Fig. 1) and a list of equipment with complete specifications were recommended for setting up of a plant to treat 500 tonnes of fluorspar per day. Economic evaluation of the process was also furnished.

2.1 Pelletization of Metallurgical Grade of Fluorspar Concentrate from Ambadongar, Gujarat.

At the instance of Gujarat Mineral Development Corporation, detailed pelletisation studies were carried out with the metallurgical grade concentrate produced from a low grade fluorspar sample from Ambadungar, Gujarat. The concentrate assayed 87.37% CaF_2 and was a fine powder having 70.2% by weight passing 325 mesh sieve. Bulk density of the dry powder with 1% H_2O was 1.5 tonne/cu. metre and with 4.37% H_2O it was 1.13 tonnes/cu. metre.

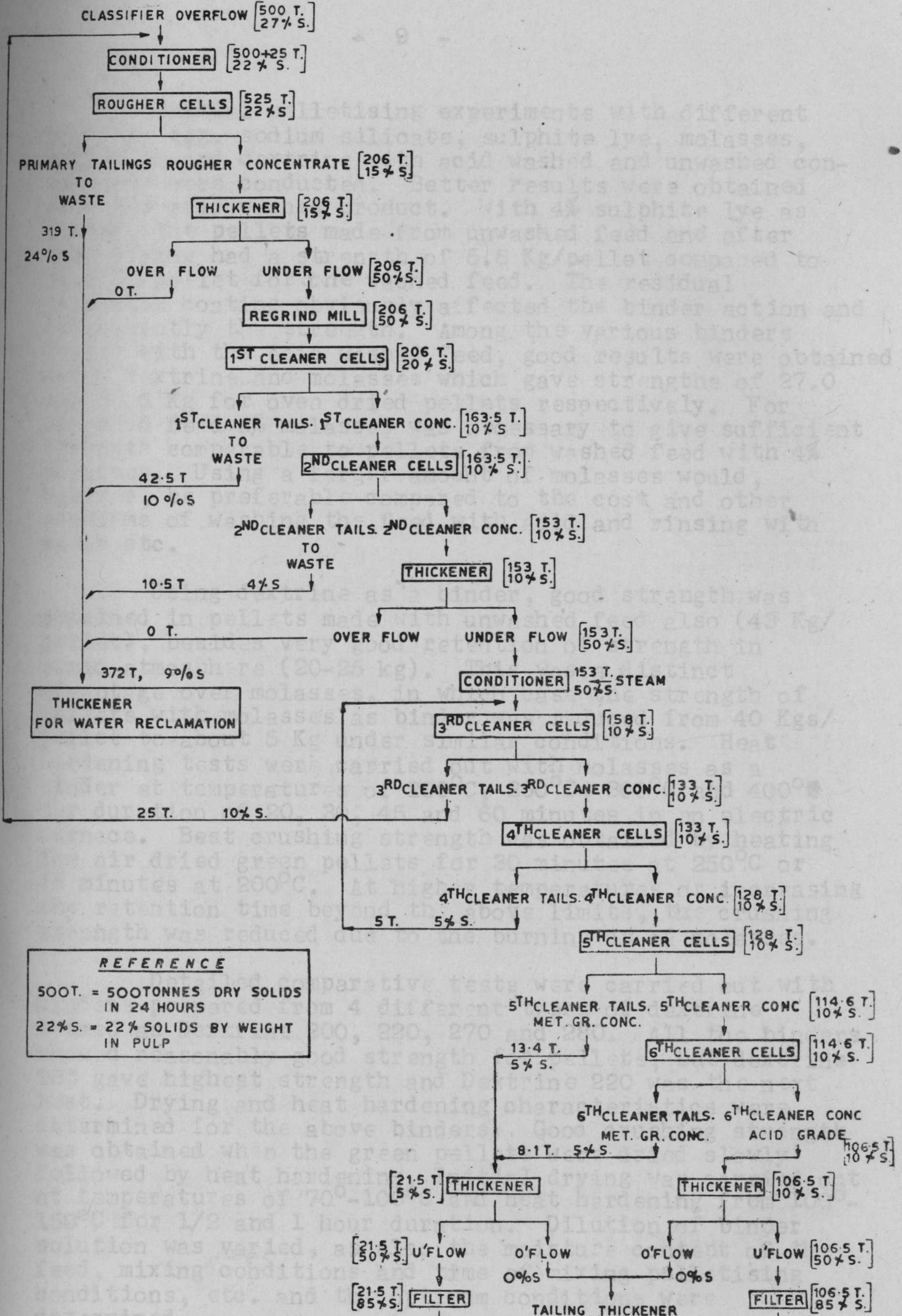


Fig. 1 Flow chart giving material balance in flotation section (Capacity-500 tonnes per 24 hr.) G.M.D.C. Fluorspar

Initial pelletising experiments with different binders, e.g. sodium silicate, sulphite lye, molasses, bentonite and dextrine with acid washed and unwashed concentrate were conducted. Better results were obtained with the acid washed product. With 4% sulphite lye as binder, the pellets made from unwashed feed and after oven drying had a strength of 5.8 Kg/pellet compared to 21.6 Kg/pellet for the washed feed. The residual collector coating obviously affected the binder action and consequently the strength. Among the various binders tested with the acid washed feed, good results were obtained using dextrine and molasses which gave strengths of 27.0 and 36.6 Kg for oven dried pellets respectively. For unwashed feed 6% molasses was necessary to give sufficient strength comparable to pellets from washed feed with 4% molasses. Using a larger amount of molasses would, however, be preferable compared to the cost and other problems of washing the feed with acid and rinsing with water etc.

Using dextrine as a binder, good strength was obtained in pellets made with unwashed feed also (43 Kg/pellet), besides very good retention of strength in humid atmosphere (20-25 kg). This was a distinct advantage over molasses, in which case the strength of pellets with molasses as binder was reduced from 40 Kgs/pellet to about 5 Kg under similar conditions. Heat hardening tests were carried out with molasses as a binder at temperatures of 200°C, 250°C, 300°C and 400°C for duration of 20, 30, 45 and 60 minutes in an electric furnace. Best crushing strength was obtained by heating the air dried green pellets for 30 minutes at 250°C or 45 minutes at 200°C. At higher temperatures or increasing the retention time beyond the above limits, the crushing strength was reduced due to the burning up of molasses.

Detailed comparative tests were carried out with binders prepared from 4 different types of dextrine termed as Dextrine 200, 220, 270 and 280. All the binders showed reasonably good strength for pellets, but dextrine 280 gave highest strength and Dextrine 220 was the next best. Drying and heat hardening characteristics were determined for the above binders. Good crushing strength was obtained when the green pellets were dried slowly followed by heat hardening. Initial drying was carried out at temperatures of 70°-100°C and heat hardening from 100°-150°C for 1/2 and 1 hour duration. Dilution of binder solution was varied, as also the moisture content of the feed, mixing conditions and time of mixing pelletising conditions, etc. and the optimum conditions were determined.

It was found that pellets with excellent physical properties and storage characteristics could be prepared from the metallurgical grade concentrate obtained from the sample of low grade fluorspar from Ambadungar, Gujarat; using dextrine based binders. Since it is a costly raw material, efforts should be made to obtain the same in a cheaper form specially if the price per dry weight is favourable when the same is obtained in a liquid or slurry form. This would also eliminate the step of bringing dry dextrine powder into solution form for use as a binder.

2.2 Settling and Filtration Tests on the Acid Grade Concentrate from a Mixed Fluorspar Sample from Dungarpur, Rajasthan.

In connection with a Techno-Economic Appraisal of the Rajasthan Fluorite Project prepared by M/s."WRIGHT ENGINEERS LTD" of Canada, the Director of Mines & Geology, Government of Rajasthan desired that settling and filtration tests to be done at the National Metallurgical Laboratory on the acid grade flotation concentrate that will be produced in the proposed flotation plant to determine the size of thickener and vacuum filter. The work involve (i) mixing of three samples in proportion (2:1:1), (ii) grinding the mixed sample to 87% -200 mesh for flotation of fluorspar and (iii) regrinding of rougher float to 92% -325 mesh and reflotation of same to produce the final concentrate for settling and filtration tests.

About 100 Kg of three low grade fluorspar sample from Ramore, Mata and Bhagat deposits occuring in Dungarpur District of Rajasthan were received. The samples consisted of lumps ranging from 62.5 mm to fines.

The three samples were separately crushed to 19 mm and mixed in the proportion of 2:1:1 respectively as desired. The mixed sample was then stage crushed to -10 mesh for flotation tests to prepare an acid grade concentrate. Flotation tests were carried out under conditions similar to those suggested by M/s. Wright Engineers of Canada in their Report.

Acid grade concentrate was obtained by flotation method and this concentrate was used for settling and filtration tests.

It was observed during the settling tests with or without flocculants, that the water separating from the settling pulp was quite clear and the thickener overflow can therefore be reused in the plant, if there is no objectionable reagent in it.

From the results of filtration tests at 50% solids and 160 seconds cycle time, the rate of filter cake formation was found to be 124.3 Kgs/hr/sq; meter. Taking this value for calculating the size of vacuum filter, an area of 3.6 sq.ft. will be required per ton of concentrate for 24 hours. For filtering 50 tonnes the required filter area will be 180 sq.ft. Giving an allowance of 25% for changing of leafs and leakages the required areas will be 225 sq.ft. The addition of flocculating agent to the pulp increased the rate of filter cake formation.

3.0 Batch as well as Pilot Plant Beneficiation Studies for the Recovery of Copper, Nickel and Molybdenum from Low-grade Copper Ore from Rakha Mines and Detailed Proposals for setting up of a 1000 tonnes per day Beneficiation Plant.

A sample of low grade copper ore weighing about 334 tonnes was received from Rakha mines of Hindusthan Copper Ltd., for beneficiation studies.

The National Metallurgical Laboratory was requested to carry out comprehensive laboratory scale and pilot plant studies and suggest a flowsheet along with a list of equipment needed for a 1000 tonnes/day capacity plant for the production of a smelter grade copper-nickel concentrate, and a molybdenum concentrate. Recovery of Uranium from the ore after the separation of sulphide minerals is being investigated by the Bhabha Atomic Research Centre.

The sample as received assayed 1.34% Cu, 10.14% Fe, 2.03% S, 65.8% SiO_2, 10.58% Al_2O_3, 2.94% MgO, 0.66% CaO, 0.053% Ni, 0.016% Mo, and 0.11% U_3O_8 (equivalent). Spectroscopic analysis of the sample revealed that Ag, Bi, Mn V and Co were present in "Trace" amounts and Sn, Pb and Zn were present in "minor trace" amounts and Au, As, Sb and Te were not detected.

Mineralogical studies indicated that chalcopyrite, violerite, and molybdenite were the principal copper, nickel and molybdenum bearing minerals respectively and were associated with quartz, chlorite, biotite, feldspar, muscovite, apatite, and tourmaline in decreasing order of their abundance. Though copper and molybdenum minerals got liberated from the associated gangue at about 100 mesh, the copper minerals were not free from the finely interlocked molybdenite even at an extremely fine size of about 200 mesh. The nickel bearing mineral was found to be finely interlocked with chalcopyrite, chlorite and quartz.

Before taking up the pilot plant studies, laboratory tests were carried out to determine the optimum flowsheet for recovering, as much as possible, of copper, nickel and molybdenum, present in the Rakha ore, in the form of a copper-nickel concentrate, and a molybdenum concentrate. Studies aimed at the recovery of Uranium from the tailings produced after the bulk sulphide flotation of Rakha copper ore are carried out separately by the Uranium Mill Project, Jaduguda.

Batch tests aimed at bulk sulphide flotation indicated that 95.7% copper, 58.6% nickel and 94.4% molybdenum present in the test sample could be recovered in a combined concentrate assaying about 13.25% Cu, 0.308% Ni and 0.150% Mo under optimum conditions.

Batch scale tests aimed at differential flotation of Molybdenum followed by four cleanings and copper-nickel flotation from the tailing from the molybdenum circuit indicated that a cleaner molybdenum concentrate assaying 33.7% Mo and 1.4% Cu with a recovery of 60.2% molybdenum and a combined copper-nickel concentrate assaying 22.02% Cu and 0.28% Ni with 87.4% and 25.3% recoveries of copper and nickel respectively could be produced out of the copper ore from the Rakha mines under optimum conditions.

Based on the above studies, the optimum flowsheet for the recovery of copper, nickel and molybdenum from Rakha ore along with material balance was recommended. The list of the equipment required for a beneficiation plant having a rated capacity of 1000 tonnes of ore per day based on operational data collected during the pilot plant run was furnished.

The capital cost of the mill is estimated at about 2 crore rupees. The cost of beneficiating the ore is estimated at Rs. 21.23 per tonne of ore.

4.4 Flotation Studies for the Recovery of Molybdenum, Copper and Nickel Minerals from Uranium Ore Samples from Jadugoda Mines.

Uranium ore sample assaying 0.17% Cu, 0.17% Ni, 0.032% Mo, 0.51% P_2O_5, 0.45% S, 77.1% SiO_2, 7.6% Al_2O_3, 0.27% Ca, 2.67% MgO, 9.2% Fe as Fe_2O_3, 0.064% O_3O_8 and 1.8% loss on ignition, was received from Uranium Corporation of India to study the possibilities of recovering copper, nickel and molybdenum minerals by mineral beneficiation techniques.

Optimum conditions for the rougher bulk sulphide flotation were established. About 0.05 Kg/tonne of potassium amyl xanthate and 0.04 Kg/tonne of pine oil were found to be optimum to float the sulphide minerals from a pulp ground to 55% -200 mesh at a pH of 7.9 thereby producing a bulk sulphide concentrate assaying 0.94% Cu, 1.84% Ni, 0.50% Mo, with recoveries of 32.6% Cu, 63.8% Ni and 92.2% Mo.

Differential flotation of the bulk sulphide concentrate, ground to about 200 mesh size after one cleaning and steam boiling, with NaCN and lime followed by 3 cleanings using NaCN yielded a Mo concentrate assaying 36.21% Mo, 2.0% Cu and 0.39% Ni with recoveries of 34.0% Mo, 0.4% Cu and 0.1% Ni in it.

No further tests were required to be carried out with this Mo concentrate for improving its grade. Instead, NML was requested to produce and supply a bulk sulphide concentrate in the pilot plant from another sample of 15 tonnes by weight from Jadugoda mines. This sample assaying 0.24% Cu, 0.28% Ni and 0.032% Mo was treated in the pilot plant under the same optimum conditions as those obtained with the second sample producing, a bulk sulphide concentrate assaying 1.16% Cu, 1.72% Ni and 0.28% Mo with recoveries of 55.1% Cu, 70.0% Ni and 99.8% Mo respectively.

About 200 Kg of this bulk concentrate was handed over to UCIL for onward transmission to BARC, Bombay, for selective flotation tests to produce Cu, Ni and Mo concentrates.

Another three samples of uranium ore from Jaduguda mines were received, from the Uranium Corporation of India Ltd., with a request to blend them in the specified proportions and develop an oprtmum flowsheet for recovering the bye-product values namely copper, nickel and molybdenum from the composite sample by mineral beneficiation techniques. The composite sample assayed 0.18% Cu, 0.2% Ni, 0.036% Mo, 7.44% Fe, 0.56% S, 1.36% P_2O_5, 66.85% SiO_2 and 12.54% Al_2O_3. In general the samples consisted chiefly of chlorite-quartz schist containing minor amounts of biotite, tourmaline and metallic minerals. The metallic minerals were chalcopyrite, pyrite, millerite, pentalandite, molybdenite, magnetite, etc. The average grain size of the economic minerals was generally very fine, 400 mesh. Batch scale flotation tests consisting of selective flotation of molybdenum from the composite sample using 0.5 Kg/tonne of fuel oil and 0.03 Kg/ tonne of cresylic acid followed by 3 cleanings of the rougher molybdenum float using NaCN as depresent for Cu-Ni minerals yielded a cleaner molybdenum concentrate assaying 30.33% Mo, 2.57% Cu, 0.6% Ni with molybdenum recovery of 47.2%.

5.0 Recovery of Nickel and Apatite from the Copper Ore of Indian Copper Corporation.

At the instance of Indian Copper Corporation, Ghatsila, beneficiation studies were undertaken at the National Metallurgical Laboratory to improve the recovery of nickel from their copper ore from the present nearly 30% and to find out whether the apatite currently lost in the tailings could be recovered profitably.

These studies were carried out (i) with the I.C.C. tailings sample assaying Cu 0.07%; Ni 0.073%; P_2O_5 1.49% as well as with (ii) the I.C.C. copper ore assaying Cu 2.2%; Ni 0.08% and P_2O_5 1.36%.

Microscopic examination showed that pentlandite and voilarite are the nickel bearing minerals and apatite is the chief phosphorous bearing mineral associated with chlorite and quartz. Apatite is liberated at about 100 mesh size, but most of the nickel bearing grains are not free even at 200 mesh size.

Nickel Recovery:

Two stage magnetic separation of the I.C.C. tailing sample yielded a magnetic fraction assaying 0.166% Ni with 28.8% Ni recovery. But this method is to be rulled out due to prohibitive cost of drying the tailing pulp containing 20% solids if apatite is to be subsequently floated. Good separation of nickel bearing minerals could not be achieved by tabling. Straight flotation of the I.C.C. tailing with 0.1 Kg/tonne sodium isopropyl xanthate and 0.08 Kg/tonne pine oil yielded a concentrate assaying 0.178% Ni, with 26.6% nickel recovery. Finer grinding to 74.9% -200 mesh followed by flotation of I.C.C. tailings improved the recovery of nickel to 43.9% (30.7% w.r.t. ore sample). Since the Cu concentrate obtained by I.C.C. carries about 30% of the total Ni the overall nickel recovery in copper concentrate and Ni concentrate will be 60.7%. Since the low recovery of nickel from the I.C.C. tailing sample is probably due to surface oxidation of the sample, further tests were carried out with the I.C.C. feed sample.

The sample was ground to 44% -200 mesh; using 0.06 Kg/tonne potassium ethyl xanthate and 0.06 Kg/tonne pine oil, the sulphide minerals were floated in 30 minutes producing a bulk sulphide concentrate assaying 0.388% Ni with 57.9% recovery of nickel. Instead of potassium ethyl xanthate, use of sodium isopropyl xanthate substantially improved the nickel recovery to 71.4%. The nickel recovery could also be improved to 72.3% Ni by grinding the sample to 70% -200 mesh, followed by flotation of the sulphide minerals using 0.04 Kg/tonne of isopropyl xanthate and 0.04 Kg/tonne of pine oil in about 6 minutes flotation time. The economics of the above two methods will have to be worked out before making the final choice.

Apatite Recovery:

About 84.9% of the total apatite currently lost in the I.C.C. tailing (76.4% w.r.t. the P_2O_5 in the ore feed) could easily be recovered by straight flotation of the tailing sample using 0.75 Kg/tonne oleic acid and 0.36 Kg/tonne sodium silicate producing an apatite concentrate assaying 8.11% P_2O_5. In practice, this could be achieved only after flotating off the sulphide minerals at 44% -200 mesh size. The rougher apatite concentrate could be further upgraded by cleaning, producing an apatite concentrate assaying 28.3% P_2O_5, with a final recovery of about 50% P_2O_5.

The test results confirm the possibility of (1) doubling the nickel recovery that is currently obtained at I.C.C. by increasing the flotation time or by grinding the ore to 70% -200 mesh and (2) recovering about 50% of apatite present in the ore by carrying out the soap flotation after the removal of the sulphide minerals.

6.0 Beneficiation of a Nickel Sample from Sukinda Area, Orissa.

The sample as received assayed 1.00% Ni, 0.62% Mn, 1.9% Cr_2O_3, 4.66% Al_2O_3, 72.8% Fe_2O_3 and 12.17% L.O.I. The sample was in the form of fines and contained 83.7% -200 mesh. Mineralogical studies undertaken on the sample could not establish the identity of the nickel mineral. Hydroclassification tabling, magnetic separation and flotation tests did not yield encouraging results. Reduction roast after balling the fines, followed by wet magnetic separation also did not produce encouraging results.

7.0 Beneficiation of a Low-grade Galenda Sample from Bandalamotta, Andhra Pradesh.

A low grade lead ore from Bandalamotta, Agnigundala area, Andhra Pradesh was received from the G.S.I. for beneficiation studies. The sample consisted of pieces of core samples and assayed 6.2% Pb, 1.87% S, 4.77% Fe, 11.55% SiO_2, 22.86% CaO and 13.82% MgO. The sample consisted essentially of dolomitic limestone and quartz with minor amounts of galenda, followed by pyrite, goethite and traces of sphalerite. A good amount of galena was free at a fairly coarse size and most of it was liberated at -100 mesh. Straight flotation using potassium ethyl xanthate as collector and cresylic acid as frother yielded a concentrate assaying 60.4% Pb with a recovery of 98.1%. One cleaning of the rougher float improved the grade of the cleaner concentrate to 73.9% Pb with a recovery of 95.2%.

8.0 Beneficiation of Phosphate Rock from Udaipur, Rajasthan.

A sample of about 163 tonnes of phosphate rock from Maton Block, Udaipur District, Rajasthan, assaying 28.1% P_2O_5 and 12.1% CaO, was received from M/s. Hindusthan Zinc Ltd., Rajasthan for beneficiation studies. Besides production of 50 tonnes of phosphate concentrate were carried out under optimum conditions to be sent to Hindusthan Zinc Ltd., for their field studies for the production of superphosphate. Petrological examination indicated that the sample contained calcite and silica as the major gangue minerals.

A few preliminary grinding tests and batch flotation tests were carried out for determining the optimum conditions for the pilot plant run. 91% -200 mesh grind was required for complete liberation of phosphate minerals. Flotation tests using oleic acid and sodium silicate indicated that 1.5 Kgs per tonne oleic acid and 2.0 Kg/tonne of sodium silicate were the optimum requirements of collector and depressant to obtained a phosphate concentrate of 35% P_2O_5 with a recovery of 70% P_2O_5 in the product. The tailings assayed 12.5% P_2O_5. Use of Katha a depressant for calcitic minerals indicated that a small quantity of Katha had no depressing effect on calcite whereas larger quantity such as 0.50 to 0.75 Kg/tonne depressed the phosphate in the tailing and the calciting fraction could be taken out as float with little loss of phosphate.

Based on the above tests, pilot plant studies for the production of phosphate concentrate were undertaken. A bulk concentrate was first obtained using sodium oleate and sodium silicate as the collector and depressant respectively.

The bulk concentrate assayed 33.5 to 34.0% P_2O_5 with a recovery of 68.5% P_2O_5 in the product. The bulk concentrate was subsequently treated for the removal of calcite with a view to improve the grade of P_2O_5. 0.75 Kg/tonne of Katha was used for depressing phosphatic minerals in the tails rejecting a highly calcitic fraction in the float. The phosphate concentrate thus obtained assayed 36.46% P_2O_5. About 50 tonnes of phosphate concentrate was prepared.

8.1 Beneficiation of Phosphate Rock from Kanpur block, Udaipur, Dist.Rajasthan.

The sample as received assayed 12.66% P_2O_5, 8.2% SiO_2, 39.6% CaO, 9.65% MgO, 24.77% CO_2, 0.64% Fe_2O_3, 27.32% L.O.I.

Soap flotation tests using various depressants did not yield encouraging results. Calcination at 1000°C and hydroclassification yielded a concentrate assaying 34.06% P_2O_5 with 53.0% recovery in it. Flotation with the calcined sand after removal of fines in the Raymond separator yielded a concentrate assaying 21.63% P_2O_5 with a recovery of 87.4% P_2O_5. Five cleanings of the rougher float using sodium silicate and tall oil, yielded a refloat concentrate assaying 29.8% P_2O_5 with a recovery of 72.6% P_2O_5. Further tests are in progress.

8.2 Beneficiation of Phosphate Rock from Marsana Block, Mussoorie Area, Uttar Pradesh.

At the instance of the Geological Survey of India, beneficiation studies were undertaken on a low grade phosphate rock from Masrana block, Mussoorie area, Uttar Pradesh, for producing a concentrate suitable for super-phosphate manufacture. The sample as received assayed 21.5% P_2O_5; 35.92% SiO_2; 26.34% CaO; 1.08% CO_2; 5.81% Fe_2O_3; 1.23% Al_2O_3; 1.84% MgO; 0.59% F and 1.01% SO_3.

Collophane was the principal phosphate mineral in the sample with quartz as the chief gangue mineral. Quartz was intimately associated with collophane and was not completely liberated even at 200 mesh size. Flotation studies were carried out using oleic acid emulsion as collector for phosphate mineral and sodium silicate as depressant for siliceous gangue. The optimum amount of reagents mecessary for rougher flotation was found to be 1.25 Kg/tonne of sodium silicate and 1.5 Kg/tonne of oleic acid emulsion at the natural pH of 7.8 after grinding the sample to 99% -200 mesh. The rougher float assayed 27.3% P_2O_5 with a recovery of 93.2%.

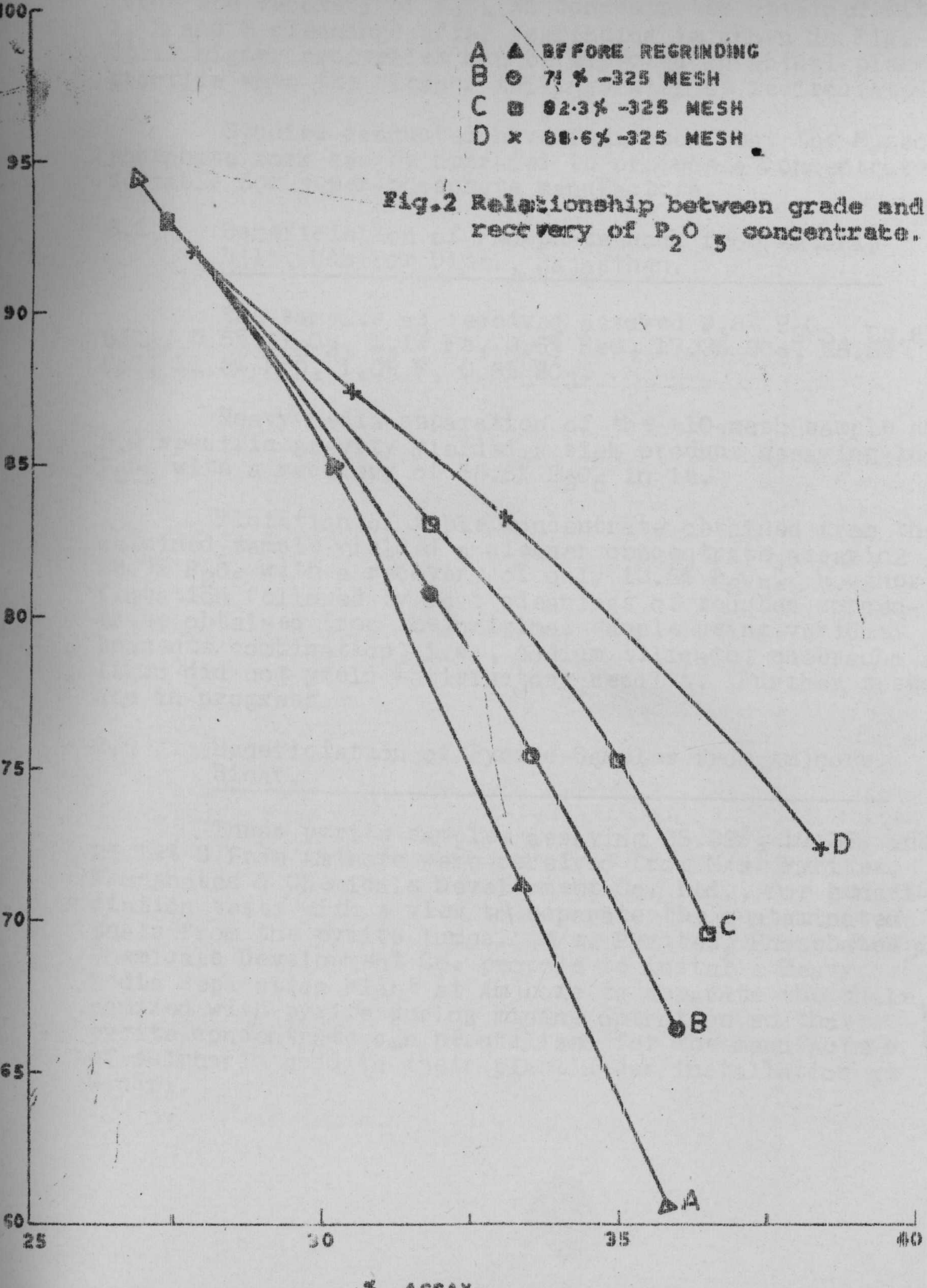


Fig.2 Relationship between grade and recovery of P_2O_5 concentrate.

Three cleanings of the rougher concentrate produced a cleaner concentrate assaying 35.8% P_2O_5 with a P_2O_5 recovery of 60.5%. Regrinding the rougher floats before cleaning yielded better grade concentrates assaying 35.7%, 36.5% and 38.4% P_2O_5 with improved recoveries of 66.2%, 69.6% and 72.4% respectively. The relationship between grade and recovery of P_2O_5 in concentrates obtained with 1, 2 and 3 cleanings after regrinding is shown in Fig. 2. Still higher recoveries can be expected in actual plant practice when the cleaner tailings will be recirculated.

Studies conducted have indicated that the Mussoorie phosphate rock can be upgraded to produce a concentrate suitable for super-phosphate manufacture.

8.3 Beneficiation of Phosphate Rock from Kataria Hill, Udaipur Dist., Rajasthan.

The samples as received assayed 9.8% P_2O_5, 23.6% SiO_2, 0.5% Al_2O_3, 1.1% Fe, 0.6% FeO, 17.3% CO_2, 28.8% CaO, 11.8% MgO, 1.0% F, 0.5% SO_3.

Heavy media separation of the +10 mesh sample at 2.9 specific gravity yielded a sink product assaying 16.2% P_2O_5 with a recovery of 58.6% P_2O_5 in it.

Flotation of table concentrate obtained from the calcined sample yielded a cleaner concentrate assaying 28.7% P_2O_5 with a recovery of only 13.5% P_2O_5. Rougher flotation followed by four cleanings of rougher concentrate obtained from the original sample using various reagents combination, i.e., sodium silicate, quebracho and liqro did not yield satisfactory results. Further tests are in progress.

9.0 Beneficiation of Pyrite Samples from Amjhore, Bihar.

Three pyrite samples assaying 25.32%, 17.36% and 25.12% S from Amjhore were received from M/s. Pyrites, Phosphates & Chemicals Development Co. Ltd., for beneficiation tests with a view to separate the contaminated shale from the pyrite lumps. M/s. Pyrites, Phosphates & Chemicals Development Co. propose to instal a Heavy Media Separation Plant at Amjhore to separate the shale admixed with pyrite during mining operation so that pyrite concentrate can be utilized for the manufacture of sulphuric acid in their plant under installation at Sindri.

Heavy Media Separation tests of the samples after crushing to -125 mm and screening out the -5 mm fines at different specific gravities indicated, that concentrates could be obtained as sink products at medium specific gravity 2.9 assaying 38.3, 34.27 and 41.0% S with recoferies of 78.9, 60.3 and 74.1% S. Heavy media separation tests when extended upto 10 mesh (1.65 mm) instead of 5 mm yielded a concentrate from the mixed sample assaying 38.7% S, 32.4% Fe and 16.03% insolubles with a recovery of 75.2% S. If the -10 mesh untreated fines mixed with -125 +10 mesh heavy media concentrate (sink at 2.9), a product assaying 37.4% with a recovery of 79.2% would be obtained. This product satisfied the grade and size specifications laid down by the acid plant at Sindri.

Jigging of the samples did not produce satisfactory grade of concentrate compared to heavy media separation.

If heavy media separation is adopted a proper selection of medium will have to be made. The use of magnetite, ferro silicon etc. for H.M.S. process is a standard practice, because of the ease of their recovery and regeneration. For low separating densities, in the range of 1.25 to 2.50, magnetite can be successfully used. But in this case, since the separating density should be kept at 2.9, either ferro-silicon alone or a mixture of ferro-silicon and magnetite will have to be used.

10.0 Beneficiation of Low-grade Limestone from Tal, U.P.

A sample of limestone from Tal, U.P. was received from Govt. of U.P. for beneficiation studies so as to make it suitable for cement manufacture. The sample assayed 38.57% CaO, 23.28% SiO_2, 0.76% Al_2O_3 and 1.1% MgO. Petrological studies of the sample revealed that silica constituted the major gangue and most of the calcite minerals was liberated at 150 to 200 mesh size. Preliminary batch flotation test to determine the grinding characteristics of the sample indicated that 81% -200 mesh was the optimum for the best grade and recovery. Flotation tests using oleic acid as collector and sodium silicate as depressant indicated that 2 Kgs/tonne of sodium silicate and 1 Kg/tonne of oleic acid would give a concentrate assaying 47.0% CaO with a recovery of 80.1% CaO in the product. Further tests using different combinations of collector and depressant to improve both the grade and the recovery are in progress.

10.1 Beneficiation Studies on an Argillaceous Limestone Sample from Dongri Limestone Quarry of Orissa Mining Corporation.

A low grade argillaceous limestone sample was received from Orissa Mining Corporation Ltd. to investigate whether it could be beneficiated to a grade suitable for cement manufacture. The sample as received assayed 33.7% CaO, 0.62% MgO, 25.70% SiO_2, 5.63% Al_2O_3, 2.70% Fe_2O_3, 0.125% S, 0.016% P and 34.30% total insolubles.

Microscopic examination of a representative -10 mesh sample indicated that quartz constituted the chief gangue with minor amounts of chlorite and muscovite. Liberation of calcite from gangue was incomplete even at 400 mesh.

Flotation was the only method investigated for separation of calcite from the gangue. Oleic acid emulsion was employed as collector for limestone and sodium silicate as gangue depressant. Optimum flotation conditions for calcite were determined by varying the feed fineness, quantity of the collector and depressant. Variation of the fineness of flotation feed indicated, that a feed having 87.0% -200 mesh was optimum. The rougher calcite float assayed 39.5% CaO, 24.2% insolubles with a recovery of 96.4% CaO in it.

The effect of sodium silicate variation on the flotation of calcite indicated that 1.5 Kg/tonne was the optimum for rougher flotation. Use of pine oil reduced the collector consumption from 1 Kg to 0.50 Kg/tonne but flotation was found to be less selective.

On cleaning of the rougher, float yielded a refloat concentrate assaying 45.3% CaO, 0.9% MgO, 11.9% SiO_2 and 14.44% insolubles with a recovery of 80.2% CaO. Recirculation of the rougher tailing back to the circuit is expected to improve the recovery of CaOin the final concentrate. This concentrate fulfills the grade requirements laid down by the Orissa Mining Corporation Ltd. for use in the cement industry. If however, three cleanings of the rougher float were done, a refloat concentrate of grade 49.03% CaO with a recovery of 73.7% CaO could be obtained.

10.2 Beneficiation of High Silica Limestone (Sample No.CG-1) from Yeotmal District, Maharashtra.

A sample of siliceous limestone marked CG-1 was received from the Director of Mines & Geology, Govt. of Maharashtra for batch beneficiation studies to investigate whether it could be beneficiated to a grade suitable for cement use. The sample was collected from Chanaka limestone deposit in Yeotmal Dist., Maharashtra and assayed 43.44% CaO, 17.5% SiO_2, 1.45% MgO, 2.0% Al_2O_3 and 34.14% CO_2.

Microscopic examination of a representative -10 mesh sample indicated that quartz constituted the chief gangue and a grind of -150 mesh was necessary for producing a cement grade concentrate.

Flotation was the only method investigated for separation of limestone from quartz. Oleic acid emulsion was employed as collector for limestone and sodium silicate as gangue depressant. Optimum flotation conditions for rougher flotation was established by varying the feed fineness, quantities of the collector and depressant. Variation of the fineness of flotation feed indicated that a feed having 94.5% -200 mesh was optimum. The rougher float assayed 45.9% CaO and 15.9% SiO_2 with 98.2% CaO recovery.

Increasing the collector beyond 0.4 Kg/tonne did not produce any float and hence 0.4 Kg/tonne of oleic acid emulsion was considered as optimum for roughing. The effect of sodium silicate variation on flotation of limestone, indicated that 0.5 Kg/tonne as the optimum for roughing. The float assayed 46.5% CaO and 15.5% SiO_2 with 98.2% CaO recovery. One cleaning of the mixed rougher float yielded a refloat concentrate assaying 48.73% CaO and 12.06% SiO_2 with 82.5% CaO recovery.

The reflotation results could be improved further if the last two scavenger floats III & IV were separately reground, mixed with floats I & II and then subjected to reflotation. Concentrates analysing 53.2% to 48.93% CaO and 5 to 11.65% SiO_2 with recoveries ranging from 69.4 to 88% CaO could be produced depending upon the number of cleanings to which the rougher float is subjected to. The concentrate obtained after one cleaning assayed 48.93% CaO and 11.65% SiO_2 with 88.0% CaO recovery and fulfills the grade requirements laid down for cement making.

The results obtained are given in Fig. 3 showing the CaO grade and recovery in concentrate for any silica content in the product. For silica content of 13.5% the grade and recovery of CaO in the concentrate will be of the order of 47.3 and 92.5% respectively.

13.3 Beneficiation of Limestone Sample (KAG-1) from Yeotmal District, Maharashtra.

A sample of siliceous limestone marked KAG-1 and collected from Yeotmal Dist., Maharashtra was received from the Director of Mines & Geology, Maharashtra, for beneficiation studies, to bring down the SiO_2 content in the sample to about 13.5%. The sample as received assayed 41.52% CaO, 0.71% MgO, 22.37% SiO_2, 1.0% Al_2O_3, 32.8% CO_2 and 33.5% L.O.I. Petrological examination indicated the limestone present was a fine grained type and the same was in intimate association with quartz, the principal gangue. Grinding the sample to about -150 mesh was necessary to produce a cement grade concentrate from the sample.

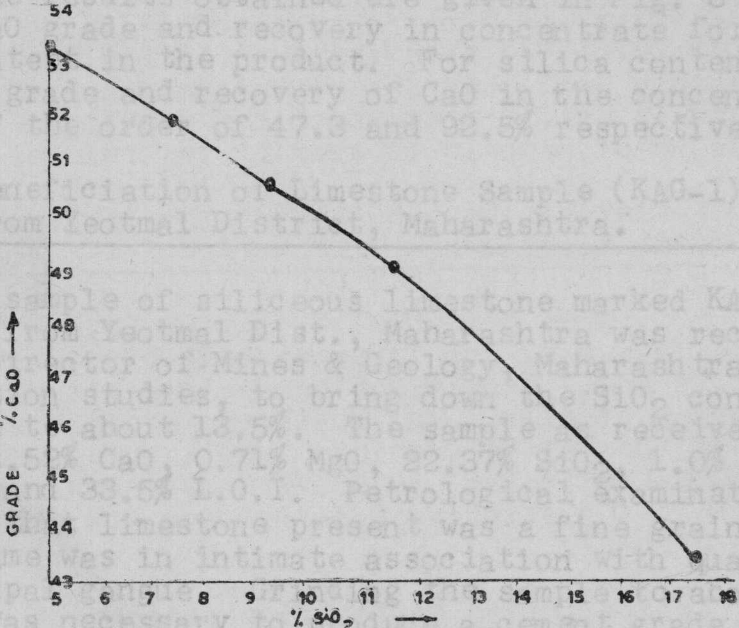
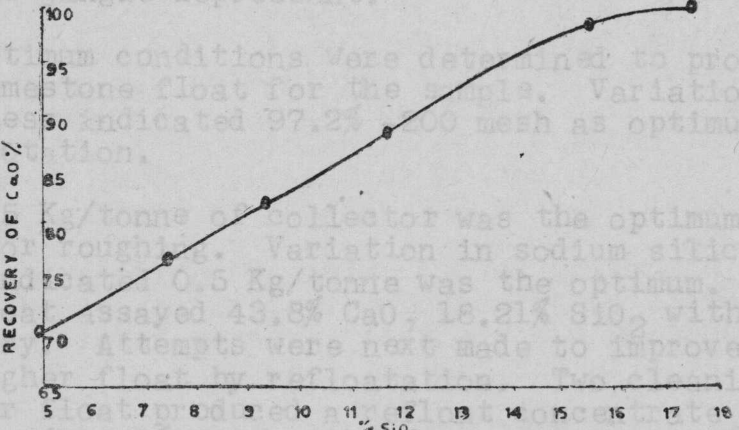


Fig. 3(a) % SiO_2 vs % CaO grade

Only flotation studies were made for separation of limestone from the siliceous gangue. Oleic acid emulsion was used as collector for limestone and sodium silicate as gangue depressant.

Optimum conditions were determined to produce a rougher limestone float for the sample. Variation in feed fineness indicated 97.2% -200 mesh as optimum for rougher flotation.

0.[illegible] Kg/tonne of collector was the optimum quantity for roughing. Variation in sodium silicate quantity indicated 0.5 Kg/tonne was the optimum. The rougher float assayed 43.8% CaO, 18.21% SiO_2 with 99.2% CaO recovery. Attempts were next made to improve the grade of the rougher float by reflotation. Two cleanings of the rougher float produced a float concentrate assaying 49.0% CaO and 10.7% SiO_2 with a recovery of 75.5% CaO in it.



Fig, 3(b) % SiO_2 vs % CaO recovery

The results could be improved further if the floats II and III were separately reground, mixed with rougher float I and then subjected to reflotation. Concentrates analysing 53.0 to 47.24% CaO and 5 to 13.3% SiO_2 with recoveries ranging from 78.3 to 89.6% CaO could be

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Only flotation studies were made for separation of limestone from the siliceous gangue. Oleic acid emulsion was used as collector for limestone and sodium silicate as gangue depressant.

Optimum conditions were determined to produce a rougher limestone float for the sample. Variation in feed fineness indicated 97.2% -200 mesh as optimum for rougher flotation.

0.5 Kg/tonne of collector was the optimum quantity for roughing. Variation in sodium silicate quantity indicated 0.5 Kg/tonne was the optimum. The rougher float assayed 43.8% CaO, 18.21% SiO_2 with 99.2% CaO recovery. Attempts were next made to improve the grade of the rougher float by refloatation. Two cleanings of the rougher float produced a refloat concentrate assaying 49.0% CaO and 10.7% SiO_2 with a recovery of 75.5% CaO in it.

The results could be improved further if the Floats II and III were separately reground, mixed with rougher float I and then subjected to reflotation. Concentrates analysing 53.0 to 47.24% CaO and 5 to 13.3% SiO_2 with recoveries ranging from 78.3 to 89.6% CaO could be produced depending upon the number of cleanings to which the rougher float is subjected to. The concentrate obtained after one cleaning assayed 47.2% CaO and 13.3% SiO_2 with 89.6% CaO recovery, and fulfills the grade requirements laid down for cement making. The results are given in Fig. 4 from which the CaO grade and recovery for a known SiO_2 could be readily determined.

10.4 Pelletization and Calcination of Lime Shells from Kerala.

500 Kg of limeshells assaying 53.08% CaO were received from M/s. The Travancore Electro-Chemical Industries Ltd., Kerala for conducting pelletisation and calcination studies. The shell contained 4.8% clay, which was removed by washing. The washed shells assayed 54.16% CaO (equivalent to 96.7% $CaCO_3$) and was found more suitable as the starting material for the manufacture of calcium carbide.

Pelletization tests after grinding the shells to 83.5% -200 mesh using 6% molasses as binder produced the best pellets having 56.4 Kg compression strength after hardening at a temperature of 250°C for 30 minutes.

Calcination studies with green, aged and heat hardened pellets indicated that heat hardened pellets did not lose much of their strength during the process and retained their shapes intact. The percentage of -6.35 mm produced during calcination was only 3.0 as compared to 47.5 for green pellets. Compression strength and shater index values of calcined pellets indicated that calcination can be successfully done in a rotary kiln without causing the pellets to break.

Hardening before calcination is essential. It was, therefore, recommended that the green pellets may be hardened on a chain belt conveyor before charging into the rotary kiln for calcination.

11.0 Sintering of Chrome Ore Fines from Orissa.

About 5 tonnes of chrome ore fines of -10 mesh size were received from the Orissa Industrial Development Corporation for agglomeration (sintering and pelletising) studies. The sample as received assayed 54.48% Cr_2O_3 and did not require any beneficiation before agglomeration.

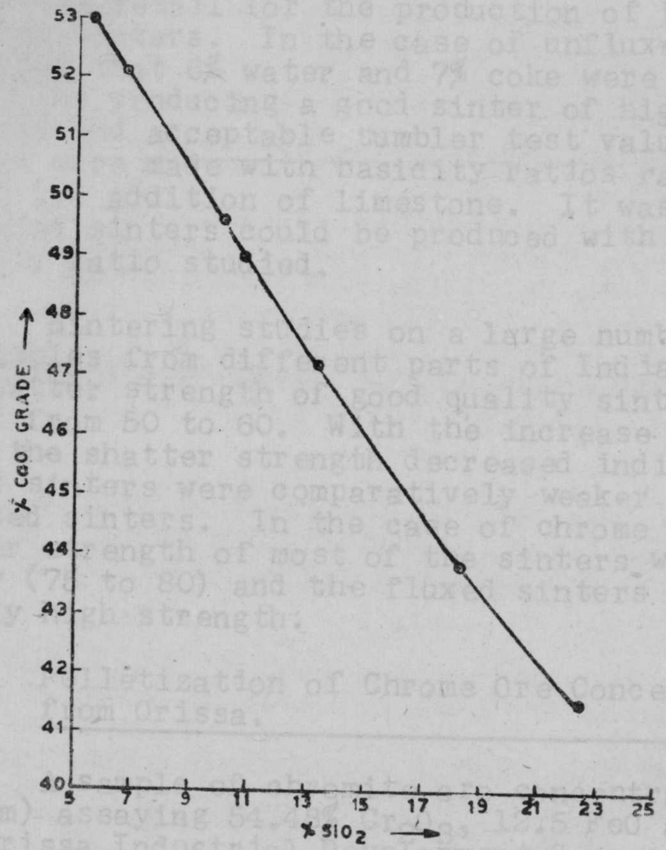


Fig. 4(a) CaO Grade vs % SiO_2

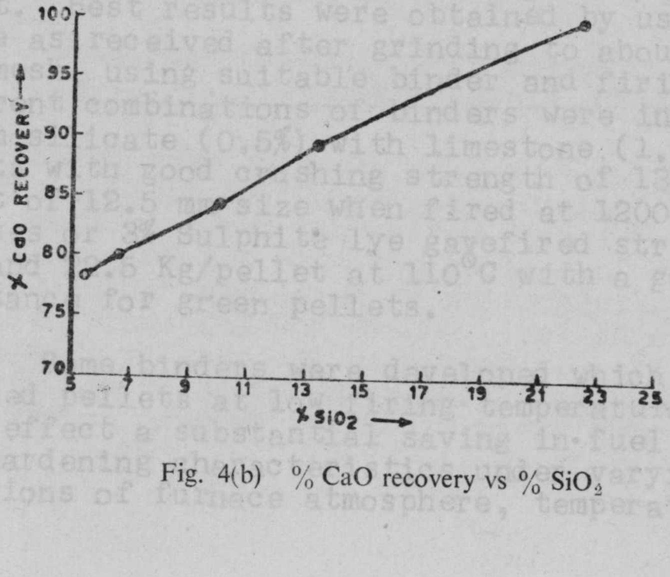


Fig. 4(b) % CaO recovery vs % SiO_2

Sintering characteristics of the sample were studied in detail for the production of both fluxed and unfluxed sinters. In the case of unfluxed sinters it was found that 6% water and 7% coke were the optimum values for producing a good sinter of high shatter strength and acceptable tumbler test values. Fluxed sinters were made with basicity ratios ranging from 1.0 to 1.6 addition of limestone. It was observed that good sinters could be produced with the range of basicity ratio studied.

Sintering studies on a large number of iron ore samples from different parts of India showed that the shatter strength of good quality sinters normally varied from 50 to 60. With the increase of the basicity ratio the shatter strength decreased indicating that fluxed sinters were comparatively weaker than the unfluxed sinters. In the case of chrome ore fines shatter strength of most of the sinters were much higher (75 to 80) and the fluxed sinters were also of equally high strength.

11.1 Pelletization of Chrome Ore Concentrates from Orissa.

A sample of chromite ore concentrate fines (-10 mm) assaying 54.48% Cr_2O_3, 12.5 FeO and 3.52 SiO_2 from Orissa Industrial Development Corporation was received for agglomeration studies. A process was developed for the first time by employing pelletising technique for chrome ore fines whereby the fines were agglomerated into pellets suitable for smelting in an Electric Furnace for ferrochrome production or for export. Best results were obtained by using the sample as received after grinding to about 80% -325 mesh, using suitable binder and firing conditions. Different combinations of binders were investigated. Sodium silicate (0.5%) with limestone (1.0%) gave pellets with good crushing strength of 138 Kg/per pellet of 12.5 mm size when fired at 1200°C. 3% Molasses or 3% Sulphite lye gavefired strength of 29.5 and 22.5 Kg/pellet at 110°C with a good shock resistance for green pellets.

Some binders were developed which gave hardened pellets at low firing temperature as this would effect a substantial saving in fuel costs. Heat hardening characteristics under varying conditions of furnace atmosphere, temperature, duration and loading; effect of storage and deterioration of properties on exposure to humid atmosphere, etc. were also studied in details.

12.0 Beneficiation of Graphite Sample from Gangavaram Mines, Mahabubabad, A.P.

A sample of low grade graphite from Gangavaram mines, Mahubabad, Andhra Pradesh, was received from M/s. S. Lal & Co., Calcutta, for beneficiation studies and for producing a graphite concentrate assaying at least 80% C and less than 3% volatile matter. The sample as received assayed 19.83% C, 71.47% ash, 1.03% S, 7.93% volatile matter, 0.77% moisture and 6.06% Fe in ash.

Quartz constituted the chief gangue, which was followed by feldspars, serpentine and micas. Siliceous gangue was finely disseminated with graphite even at -150 mesh size.

Flotation employing a feed of 62.1% -200 mesh using pine oil as frother-collector and sodium silicate as depressant for siliceous gangue, followed by 4 cleanings of the rougher concentrate after regrinding yielded a concentrate assaying 32.18% ash. Being high in ash content, this product may not be suitable even for foundry purposes. Flotation tests performed under identical conditions with the calcined sample (-10 mesh sample roasted at 925°C), yielded a graphite concentrate assaying 79.47% C, 18.24% Ash, and 2.12 V.M. This product almost conforms to the required specifications and can be used in foundry and paint industry.

12.1 Beneficiation of Graphite Sample from Errametla Mines, Mahabubabad, A. P.

A low grade graphite sample from Errametla mine, Andhra Pradesh, assaying 21.9% fixed C, 67.1% ash and 9.6% volatile matter was received from M/s. S. Lal & Co., of Calcutta to investigate the possibility of upgrading it to atleast 80.0% C and 3.0% volatile matter. Quartz and feldspar were the principal siliceous gangue in the sample. Graphite was intimately associated with the siliceous gangue and was not completely liberated even at 10 microns size.

Flotation using a feed of 84% -200 mesh employing pine oils as frother-collector and sodium silicate as oil as frother-collector and sodium silicate as gangue depressant followed by three cleanings of the rougher float after regrinding to 100% -200 mesh yielded a graphite concentrate assaying 28.5% ash. The test when repeated with the calcined sample (-10 mesh calcined at 925°C) produced a better grade concentrate assaying 74.24% fixed C, 1.45% volatile matter and 24.19% ash. The grade was slightly lower than that specified by the firm.

12.2 Beneficiation of graphite Sample from Sitapalli area, Andhra Pradesh.

The sample assaying 17.0% fixed C, 79.93% ash, 2.51% volatile matter, 0.55% moisture and 0.19% S was received from M/s. S. Lal & Co. Petrological studies showed that the gangue mineral was mainly garnet. Flotation tests using kerosene oil and pine oil are in progress.

12.3 Beneficiation of Low Grade Graphite from NEFA.

A low grade graphite sample from Lohit Dist., NEFA, was received through the Supdtg. Geologist, GSI, Assam Circle, for beneficiation studies. The sample as received assayed 2.15% fixed C, 95.75% ash, 1.54% volatile matter, 0.30% S and 5.7% Fe in ash.

Graphite was present both as crystalline and amorphous varieties. The associated gangue being muscovite, biotite, quartz, followed by minor amounts of magnetite, goethite and pyrite. Graphite grains were extremely fine, the average grain size being 1 to 2 microns and were present interspersed in the silicate matrix. The very low graphite content, its extremely fine size and nature of association with the gangue minerals indicated that a satisfactory grade of concentrate could not be produced from this sample.

A few tests were, however, carried out. Flotation tests with sodium silicate as gangue depressant and pine oil as collector-frother produced a rougher concentrate assaying 82.6% ash with a rejection of 87.2% ash in the tailing.

Reflotation of the rougher float for four times, yielded a refloat concentrate assaying 20.84% fixed carbon, and 60.48% ash with a distribution of 31.2% fixed C in it. Regrinding of the rougher float followed by four cleanings of the ground product using additional sodium silicate during each stage of cleaning yielded a final refloat concentrate assaying 45.13% F.C., 51.0% ash, 3.57% volatile matter, 5.6% Fe, in ash and 1.45% S with a distribution of 46.2% F.C. in it. The ash content was still too high in the final concentrate. Flotation tests using diesel oil and kerosene oil did not produce satisfactory results.

The very intimate nature of the graphite grains and its low content did not warrant any further beneficiation studies.

13.0 Studies on Mineralogy, Crushability and Reducibility of Iron Ore Samples from Surajgarh Deposit of Chanda District, Maharashtra.

Three iron ore samples from Surajagarh locality of Chanda Dist., Maharashtra were received for studying the mineralogy, crushability and reducibility characteristics. This ore is stated to be utilised by Govt. of Maharashtra for their proposed pig iron plant project.

Hematite was the chief iron bearing mineral associated with minor amounts of goethite and magnetite. Some siliceous and aluminous gangue were also present in the laminated sample.

From the tests carried out with the three samples from Surajgarh deposits for evaluating crushability characteristics, it was observed that the float variety offeredthe greatest resistance to compression and produced the least amount of -9.5 mm fines as compared to massive and laminated one samples. The laminated variety was found to be more friable as compared to the other two varieties and generated more fines on crushing.

Reducibility studies were carried out with closely sized fractions of each of the three samples. The rates of reduction thus determined were compared with those of Rajhara and Dalli massive iron ores which are used in Bhilai Steel Plant for iron making. Rates of reduction of laminated and massive ores of Surajagarh deposits were found to be faster as compared to Rajhara iron ore and took less time for 90% reduction. Amongst the three test samples of Surajagarh deposit, the massive ore insitu was found to be the most easily reducible followed by laminated and float ores except in the size range 4.7 - 9.5 mm where the rate of reduction of laminated variety was higher than those of the massive and float ores.

14.0 Beneficiation of Poor Grade Kyanite from Amda Lapso Mines, Bihar.

A sample of poor grade kyanite from Amda Lapso kyanite mines was received from M/s. Indian Copper Corporation, Ghatsila, Bihar for beneficiation tests. The sample as received assayed 38.2% Al_2O_3, 50.6% SiO_2, 3.1% Fe_2O_3, 1.0% TiO_2, 2.4% CaO, 1.5% MgO, 1.15% (Na_2O + K_2O). Quartz and muscovite were the main gangue minerals with minor quantities of Sericite, Chlorite, Rutile etc.

Hydroclassification followed by Tabling at -48 mesh did not give satisfactory results. Flotation with a deslimed feed at 3.8 pH employing Lactic acid and sodium sulphonate followed by four cleanings yielded a final concentrate assaying 60.4% Al_2O_3 with a recovery of 48.8% kyanite in it.

14.1 Beneficiation of a Kayanite-Sillimanite Sample received from Govt. of Maharashtra.

The sample as received assayed 40.1% Al_2O_3, 51.67% SiO_2, 0.40% Fe_2O_3, 1.36% CaO, 0.49% MgO, 0.65% TiO_2, 1.31% (Na_2O + K_2O) and 4% L.O.I. Flotation tests using lactic acid as depressant for mica and other gangue minerals and sod. sulphonate as collector for kyanite, at different pH ranges indicated that a pH of 2.5 was optimum, which produced a rougher float assaying 47% Al_2O_3 with a total recovery of 72% Al_2O_3. Cationic flotation of mica, followed flotation of mica tailing for kyanite yielded kyanite float assaying 50.95% Al_2O_3, three cleanings of the kyanite float yielded a cleaner concentrate assaying 55.4% Al_2O_3 with a recovery of 25.9% Al_2O_3 only. Further tests are in progress.

15.0 Recovery of Diamond and Concentration of TiO_2 from -20 mesh Fraction of Diamondiferous Tuff.

A sample of -20 mesh fraction of diamondiferous tuff was received from Panna diamond project, N.M.D.C. for studying the possibilities of recovering diamonds and upgrading the sample with respect to TiO_2. The sample as received assayed only 1.7% TiO_2.

Jigging and tabling tests were conducted to obtain a pre-concentrate of heavy minerals. Magnetic separation of the heavy mineral concentrates produced magnetic fractions assaying only 3.1 to 3.35% TiO_2. The non-magnetic fraction was subjected to electrostatic separation. No diamond grains could be identified in the non-conducting fraction Rutile being present in minor amounts, could not be separated by electrostatic separation. The tailing fraction carried 80.7 to 83.1% of the total TiO_2 in the product. Most of the TiO_2 in the sample was contributed by minor amounts of leucoxene as minute inclusions in the serpentine and calcite grains. Hence the TiO_2 content in the tailing could not be concentrated by electrostatic separation.

Flotation of the sample after flash grinding to about -65 mesh size using light diesel oil and pine oil could not recover any diamond grain. Flotation tests to separate TiO_2 bearing minerals from gangue did not yield satisfactory results.

16.0 Upgrading of a Silica Sand Sample from Ratnagiri District, Maharashtra.

A sample of silica sand assaying 98.52% SiO_2, 0.66% Al_2O_3, 0.21% CaO, 0.09% MgO, 0.19% Fe_2O_3, 0.14% alkalies as chlorides was received from the Director, Geology & Mining, Maharashtra, Nagpur for making it suitable for the various industries. The sample was already of a high grade and could be used as such for silica brick manufacture. Since the glassmakers prefer a sand as low in Fe_2O_3 and Al_2O_3 as possible studies were undertaken to determine the extent to which these could be reduced in the sample by adopting suitable methods. Agitation and desliming tests showed that a sand having 97.2% by wt. and analysing 98.8% SiO_2, 0.046% Fe_2O_3 and 0.35% Al_2O_3 and suitable for making good quality glass could be produced. The washing sand upon hydraulic classification, yielded products suitable for foundry use.

17.0 Production of Silico-chrome by single stage Process in the Pilot 500 KVA Sub-merged Arc Furnace.

Work was taken up to study the operating conditions in detail for the production of silico-chrome by employing the raw materials supplied by the sponsorer of the project.

Smelting trials were undertaken to produce silico-chrome by single stage process with the raw-materials supplied by M/s. Ferro-Alloys Corporation Ltd., Garividi. Silico-chrome containing Si 40 - 45%, Cr 35 - 40% and Carbon C 0.05% was produced by the direct smelting of chromite ore and quartzite with various carbonaceous reducing agents. The chemical analysis of the chrome ore, and the seive analysis are indicated in Tables 2 and 3. The chemical analysis of various reducing agents used in this investigation is indicated in Table 4. Analysis of various fluxes employed are indicated in Table 5. Typical charge burden consisting of chromite 50 kg, Garividi coke 45 kg and quartzite 50 kg was mixed and fed into the furnace at the rate of 120 kg per hour. Quartz employed was -1½" +1/8" in size and contained 98% silica. The typical operation and data in various periods of the compaign are indicated in Table 6. Based on the above studies the following conclusions were arrived at.

i) Ferro-silico-chrome containing 40 to 45% silicon can be successfully produced by the single stage process in the sub-merged arc furnace.

ii) Giridih coke when employed as the only reducing agent creates operational difficulties, such as, poor porosity of the charge, slow rate of reduction, and high chromium losses.

iii) Wood charcoal improves the smelting process and also the furnace operation conditions, when 24 per cent of Giridih coke in the charge is replaced with wood charcoal.

iv) When dolomite to magnesia to alumina ratio improved, it helped in the formation of fluid slag and improved the rate of smelting and smooth charge descent.

v) Limestone addition to the flux, improved the slag condition but the silica content in the metal dropped down to 36 per cent.

vi) The optimum MgO/Al_2O_3 ratio is found to be 1.00 below which the slag becomes low melting and viscous. When the ratio is above 1.2 the silicon content in the metal drops down.

vii) The average power consumption per tonne of the alloy produced is 7800 kWH.

Table 2 - Chemical Analysis of the chrome ores.

Constituent	Percent
Cr_2O_3	54.38
Total Fe	13.16
MgO	12.30
Al_2O_3	6.68
SiO_2	6.42
Cr/Fe ratio	2.83
$\frac{MgO}{Al_2O_3}$ ratio	1.84

Table 3 - Screen analysis of chromite.

	Weight percent
$-1\frac{1}{2}$" +1"	12%
-1" $+\frac{3}{4}$"	30%
$-\frac{3}{4}$" $+\frac{1}{2}$"	24%
$-\frac{1}{2}$" $+\frac{1}{4}$"	18%
$-\frac{1}{4}$" +1/8"	11%
-1/8"	5%
	100%

Table 4 - The Chemical analysis of the various reducing agents used in the investigation of the single stage production of silico-chrome.

%	Tisco nut coke	Tisco pearl coke	Giridih coke	Wood char-coal	L.T.C. coke from F.R.I., Jealgora.
Fixed carbon	74.4	71.35	74.12	48.12	61.50
Volatile matter	2.5	4.3	1.72	32.00	4.10
Moisture	0.2	1.1	2.2	10.18	8.50
Ash	22.9	23.2	21.94	9.70	25.90
Ash analysis:					
CaO	3.8	4.85	0.33	51.40	5.68
SiO_2	52.08	55.28	60.75	10.48	53.80
MgO	2.12	2.50	5.15	6.60	1.88
Al_2O_3	33.00	25.10	21.60	5.56	28.00
Fe_2O_3	8.57	11.20	7.60	5.64	6.57
Phosphorus	0.69	0.40	0.13	0.65	0.80
Sulphur	0.50	0.35	1.1	0.28	0.23

Table 5 - Analyses of Fluxes.

Constituent	Quartzite %	Dolomite %	Limestone %	Fluorspar %
SiO_2	98.0	--	6.44	--
Al_2O_3	1.0	--	--	--
MgO	0.05	21.37	--	--
CaO	0.60	28.43	48.81	--
CaC_2	--	--	--	75
Fe_2O_3	0.30	--	--	--
Loss on ignition	--	45.2	40.50	--
Moisture	--	0.15	--	--

TABLE 6.
OPERATION DATA ON SILICO-CHROME PRODUCTION IN 500 KVA SUB-MERGED ARC FURNACE

Period.	5	6	7	8	9	10	Remarks.
Duration hours	48	40.5	32.3	20	7.5	19.50	The carbon analysis is based on running metal Trom Tapping. It will be very much low of the order of .60% on the metal samples from ladb where it is allowed to settle on cooling.
Composition of charge (total)							
Chromite (F.A.) Kg.	2165	1700	1850	1100	350	1000	
Garidih coke kg.	1815	1495	1406	830	266	720	
Quartzite kg.	2000	1800	2095	1320	414	1125	
Charcoal kg.	--	--	410	264	84	--	
Dolomite kg.	--	--	--	195	--	--	
Limestone kg.	--	--	--	--	70	200	
L.T.C. coke kg.	--	--	--	--	--	306	
Total weight of charge kg.	5980	5001	5761	3715	1184	3351	
Charge rate $\frac{\text{Kg.}}{\text{Hr.}}$	125	123.5	178.6	187.4	158	172	
KWHr/1000 kg of charge	2542	2696		--	1646	1806	
Power input $\frac{\text{KWHr}}{\text{Hr}}$	318	333	332	348	260	301	
Weight of silico-chrome	1916	1723	1528	1065	261	837	
Weight of slag	1759	670	613	940	245	753	
Slag/metal ratio	0.92	0.39	0.40	0.88	0.94	0.90	
Silico-chrome analysis							
% Cr	34.30-46.6	37.4 -44.8	35.1-40.7	39.2-42.6	40.0	33.6 -46.7	
% Si	33.1 -41.8	35.8 -41.2	40.5-47.2	36.8-40.0	36.8	31.8 -47.7	
% C	0.14- 0.81	0.07- 0.41	0.05-0.18	0.05-0.22	0.52	0.18-0.54	

Period	5	6	7	8	9	10	Remarks.
Slag analysis							
% Cr_2O_3	1.1 - 6.4	1.8 - 6.2	0.68- 8.5	2.6 - 5.4	4.8	3.12- 8.62	
% SiO_2	45.2 -55.0	42.3 -54.2	38.0 -50.5	48.6 -54.3	50.5	46.7 -53.5	
% MgO + CaO	16.9 -24.0	19.6 -23.4	16.6 -23.4	18.8 -23.0	22.2	24.0 -24.6	
% Al_2O_3	20.3 -22.3	24.2 -33.7	22.0 -27.9	15.2 -25.6	19.0	20.9 -27.3	
% C	-	0.54- 1.76	0.29- 1.22	0.22- 3.6	5.4	4.6 - 8.5	
Operation voltage	60 - 65	62.5	60 - 65	62.5	62.5	62.5	
KWHr/Tonne of silico-chrome	7960	7720	7318	7643	7500	8125	

18.0 Fluo-Solid Roasting of Sikkim Complex Sulphide Concentrate.

Fluo-solid roasting of sulphide ores has been increasingly used for sulphide roasting as it helps in the production of high SO_2 containing flue gases, uniformity of temperature, gas composition and calcine composition throughout the bed. Systematic studies have been taken up on the roasting of complex sulphide ores.

A sample of bulk float received from Sikkim Government was taken up to recover lead, zinc, and copper. The complex sulphide concentrate analysed 14.45% Cu, 7.83% Pb, 6.65% Zn, 30.52% Fe, 29.92% sulphur, 2.64% moisture and 2.82% insolubles.

Thermo-dynamic calculations have been made to find the conditions necessary for sulphate roasting of Sikkim concentrate.

A series of continuous fluidisation experiments were carried out in a 6 cm dia stainless steel reactor using calculated amount of air to make a ratio of 50 moles of air per mole of copper content at temperatures ranging from 750°K to 925°K. The rate of feeding of the concentrate was uniform during the run.

The roasted product obtained in all the experiments was leached in 2.5% H_2SO_4 (v/v) at 60°C. The slurry was filtered and the leach liquor was analysed for copper, iron and zinc. The percentage dissolution at different temperatures is shown in the Table 7.

Table 7 - Percentage dissolution of metals in the Leached Product.

Temp. °K	% Cu in solution	% Zn in solution	% Fe in solution
750	46.7	41.8	27.2
800	55.7	51.8	25.8
850	77.4	61.1	21.2
875	88.5	70.5	10.2
900	86.2	68.4	5.6
925	74.18	62.4	4.2

Further fluidising experiments are planned in a Dorco 6 inch fluo-solid roaster (recently installed) at optimum conditions of roasting in presence of alkali salt so as to reduce the ferrite formation and promote the sulfation of copper and zinc.

19.0 Extraction of Nickel.

Several low grade nickel ores of lateritic and serpentinous origin have been brought to light during recent years by the Geological Survey of India. In view of the strategic importance of nickel, the recovery of nickel from these low grade ores is of paramount importance and systematic investigations are in progress to study the extraction characteristics of nickel from different deposits.

Light grade lateritic ore from Sukinda, Orissa.

Light grade nickeliferrous lateritics received from the Sukinda area of Orissa analysing 1.8% NiO, 47.04% total Fe 6.2% Cr_2O_3, 6.8% SiO_2, 1.1% MgO, 12.36% Al_2O_3 and 2.9% MnO with traces of coablt, was pulverised and reduced with hydrogen and leached with ammoniacal liquor in presence of guseous oxygen.

Nickel ammonium carbonate solution obtained as leach liquor containing one gram of nickel per litre was reduced with hydrogen at elevated temperature and pressure. The various factors studied include the effect of temperature of reduction, partial pressure of hydrogen and period of reduction. It was observed that 98.95% nickel could be recovered in powder form by reduction of the nickel ammonium carbonate solution at 250°C for two hours with hydrogen partial pressure of 30 kg/cm^2 by using 0.5 gram of -100 mesh (B.S.S.) size nickel powder for nucleation. The purity of nickel powder obtained was of the order of 98.77%.

20.0 Utilization of Jadugoda Ni-Cu-Mo Concentrate.

Low grade nickel,copper and molybdenum deposits are found in Singhbhum district and in view of the strategic importance of Ni,.Cu and Mo, the recovery as well as separation of the metallic values from the concentrate is of paramount importance. Systematic investigations were carried out to study the extraction characteristics of Ni, Cu and Mo; from the concentrate received from Jadugoda Mines.

Sulphation was attempted to extract nickel, copper and molybdenum from the Jadugoda Ni-Cu-Mo concentrate containing 1.56% Ni, 0.94% Cu, 0.28% Mo, 54.12% SiO_2, 9.88% Al_2O_3, 3.90 MgO, 0.56% CaO, 5.40% S and 1.40% P_2O_5.

The concentrate was roasted in air and leached with water and sulphuric acid solution to separate Ni, Cu and Mo values. The various factors studied include the effect of temperature of roasting period of reasting, duration of leaching and concentration of sulphuric acid. It was observed that complete sulphation could be carried out with a concentrate of -200 mesh (B.S.S.) size roasted at 450°C for a period of one hour. Ni, Cu and Mo could be extracted by leaching at 60° - 90°C with a 2% sulphuric acid solution for one hour. 83% Ni, 95% Cu and 95% Mo were extracted.

Preliminary pressure leaching experiments of the concentrate with ammonium hydrozide solution were also carried out to study the possibility of extraction of nickel. The results obtained show that 70% nickel can be recovered by leaching the concentrate with 10% ammonium hydroxide at 100°C for two hours with oxygen partial pressure of 20 kg/cm^2. Separation of metallic values from pregnant solution is under study.

21.0 Thermal reduction of lime for the production of Calcium metal by Aluminium Powder under Vacuum.

Calcium metal is widely used to remove non-metallic impurities in metals and alloys. This applies in particular to the removal of residual phosphorus, sulphur and dissolved gases during the manufacture of high performance steels. It is also used in the reduction of metals from their oxides, chlorides and fluorides. Indigenous production of calcium metal is necessary with the growth of nuclear power generation and other developments in the field of atomic energy.

A sample of lime stone from Salem, Madras containing 54.81% CaO, 0.44% SiO_2, 0.19% Fe_2O_3, traces of Al_2O_3 and MgO and 43.40% loss on ignation was calcined at 950°C. The calcined lime of 97% purity was powdered to -100 mesh B.S.S. and was briquetted with 200% excess by weight of the stochio-metric composition of aluminium powder of -100 B.S.S. size and charged inside the retort. The experimental arrangement and the dimension of the retort made of inconel with the assemblies are given in Figs. 5 and 6. The split type condensor of stainless steel and the circular sleeves were placed inside the retort at fixed positions. The retort was evacuated by a high vacuum pump and was simultaniously heated by a silicon carbide furnace. After completion of the experiment the retort was pulled out of the furnace and was cooled in air and the residue analysed for calcium recovery.

It was observed that calcium deposition occurs between 390°C and 400°C in a massive form. The investigation is complete and a report is under preparation.

22.0 Production of Aluminium - Titanium Alloy (10% Ti)

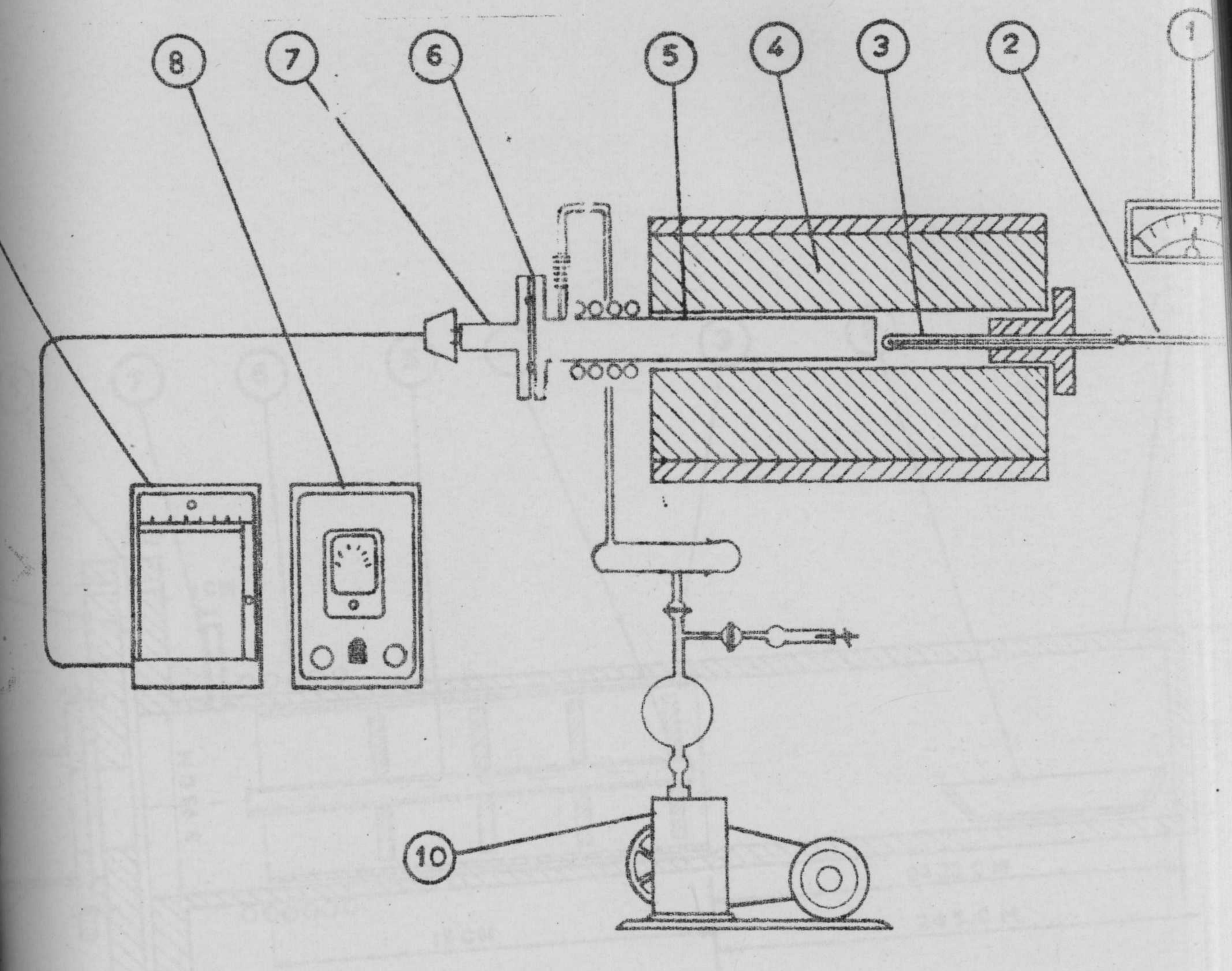
Titanium is used as an alloying element in certain weought aluminium base alloys. Its presence in these alloys to the order of 0.1% serves as a grain refiner and also imparts strength and toughness to the alloy, and such alloys are extensively used for the air-craft structural components. The work was taken up at the instance of Metal & Steel Factory (Indian Ordnance Factory), Ishapore.

Systematic studies were carried out to obtain high recoveries of titanium from titanium bearing minerals into the aluminium-titanium alloy. Exothermic mixtures with suitable composition were worked out and a recovery of 50% Ti was obtained. Further work is in progress.

23.0 Recovery of Germanium from Coal Ashes.

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

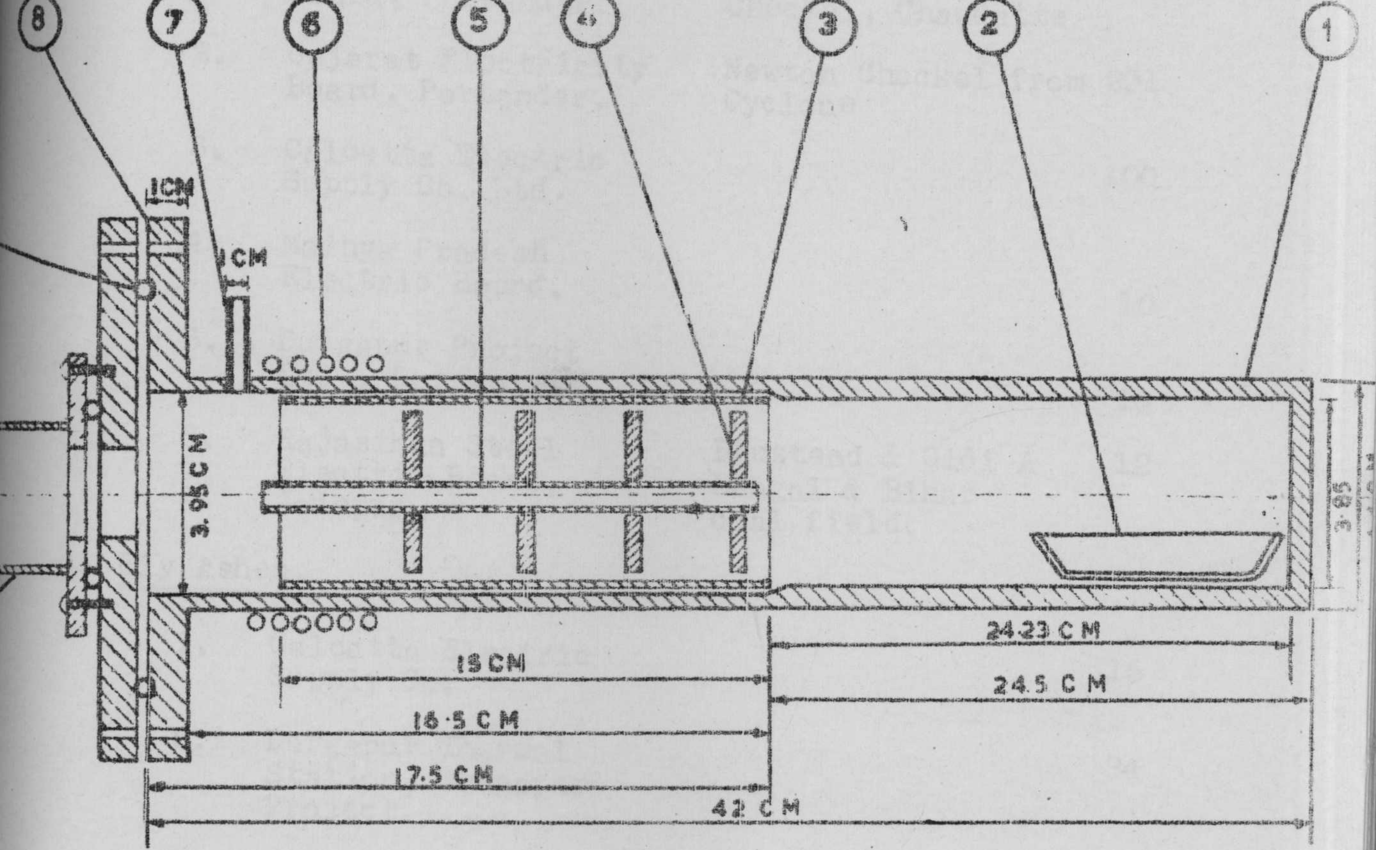
Representative samples of coals from various coal fields and fly ashes and flue dusts from various thermal power stations and gas plants were obtained and analysed for their germanium content by the X-ray fluorescence method, with the help of Autrometer. About 40 samples of coals, 22 samples of fly ashes and 14 samples of flue dusts were analysed. The coals were crushed to -200 mesh, dried at 110°C, ashed at 300°C, and were analysed. Only few samples gave positive results. Results are recorded in the Table 8.



EXPERIMENTAL SET UP FOR THERMAL REDUCTION OF LIME

1. TEMPERATURE INDICATOR 2. THERMOCOUPLE 3. THERMOCOUPLE SHEATH
4. SILICON CARBIDE FURNACE 5. INCONEL RETORT 6. FLANGE 7. THERMOCOUPLE GAUGE 8. VACUUM INDICATOR 9. VACUUM RECORDER 10. VACUUM PUMP

FIG.



CROSS SECTIONAL VIEW OF THE RETORT WITH ASSEMBLY

1. INCONEL RETORT 2. INCONEL BOAT 3. STAINLESS STEEL SPLIT TYPE CONDENSER
4. INCONEL RADIATION BAFFLE 5. STAINLESS STEEL BAFFLE HOLDING TUBE
6. COOLING COIL 7. VACUUM PORT 8. MILD STEEL FLANGE 9. 'O' RING
10. THERMOCOUPLE GAUGE.

FIG-6

Table 8 - Analysis of fly ashes and flue dusts for Germanium.

Sl.No.	Flue dusts source.	Coal Field.	Ge. ppm.
1.	Gujarat Electricity Board, Porbander.	Rahclhapar Newton Checkel, Chandatte	200
2.	Gujarat Electricity Board, Porbander.	Newton Checkel from Cyclone	201
3.	Calcutta Electric Supply Co. Ltd.		100
4.	Madhya Pradesh Electric Board.		10
5.	Durgapur Project Ltd., Durgapur		10
6.	Rajasthan Steel Electric Board, Jodhpur.	Bhustend & Gidi A Bengal & Bihar coal field.	12
Fly Ashes.			
1.	Calcutta Electric Supply Co.		15
2.	Durgapur Thermal Station, Durgapur Project.		34
3.	Trombay Thermal Station, Sholapur.		8
4.	Sindri Thermal Station, Sindri.		56
5.	Electricity Board, Faridabad.	Manoharbehal colliery Kalipahani Ghusick Jambad Kojra, Hassipur Benlee, Nags Ram, Jibanpur Pine, Sitapur.	10

1.	2.	3.	4.
6.	Rajasthan Steel Electricity Board, Jodhpur (Rajasthan).	Bengal & Bihar coal field. Bhurkumda Gidi A & Co. Saurda.	10
7.	Central Thermal Power Station, Sikka.	1. Nott Chandawetta 2. Newtonchickli 3. Ghusghen colliery 4. Saslr colliery	10
8.	Gujarat Electricity Board Porbander.	1. North Chandametta 2. Kuresia 3. Rekhi 4. Ambara etc.	20
9.	Gujarat Electricity Board, Sabarmati.	90% coal from Korea, Rewa, Penchad Chanda coal field etc.	10
10.	Beas Project, Talwar Township Dist. Hoshiarpur.		20
11.	Gujarat Electricity Board, Porbander.		145
Coal Ashes			
1.	Gujarat Electricity Board, Porbander.	Chandamatta Colliery	64
2.	Gujarat Electricity Board, Porbander.	Raman Warakhas	14
3.	Gujarat Electricity Board, Porbander.	North Cokandamatta	39
Analysis of coal for Germanium			
1.	Gujarat Electricity Board.	Newton Chikly	197
2.	-do-	Chandamatta	268
3.	-do-	Rawanwarakhas	248
4.	-do-	North Chandamatta	248

The percentage of Ge in the samples is quite encouraging in some cases. The flue dust from Porbander Power Station, Gujarat, contained 200 ppm germanium and was obtained from the Newton Checkly coal field. The coal from Newton Chickly is also being tested for germanium. The concentration of germanium in different fractions of ashes was also studied by sieve analyses of the sample.

24.0 Recovery of Selenium and Tellurium from Copper Slime.

Anodic slimes settled under the bottom of the electrolytic cells during the electrolytic refining of blister copper at Indian Copper Corporation is an important source of selenium and tellurium metals in India.

With a view to producing these rare metals indigenously to meet their ever increasing demand in modern electronic industry, a systematic study on the recovery of selenium and tellurium apart from some noble metals was taken up.

The method followed and developed consists in sulphatisation of slime in two stages i.e. digestion followed by roasting.

The samples of slime used for recovering the metallic values have the following chemical composition.

Se	8.3%	As	5.44%
Te	6.2%	Au	0.0928%
Cu	14.6%	Fe	0.51%
Ni	33.7%	SiO_2	6.5%
Ag	1.58%	S	3.76%

Initially sulphate roasting of slime was done after digesting slime at 280°C for 6 hours. The residue was found to sinter at 700°C resulting in sulphate decomposition. To avoid sintering of the residue, roasting temperature was lowered and digestion of the slime was done at 200°C with twice the quantity of H_2SO_4 in a silica boat kept in a sealed stainless steel reaction tube. A number of experiments were carried out to determine the effect of digestion time, roasting temperature and time. The results are tabulated in Table 8. The best results were obtained under conditions of the experiment No. 11. Table 10 indicates the effect of acid concentration with other experimental conditions remaining as in experiment

No. 11. The percentage of selenium recovered at 200% (by weight) acid concentration was maximum. Since excess acid is essential to decomposethe selenides completely, 200% acid by weight was maintained in the rest of the experiments.

Experiments were also carried out to find the amount of selenides and tellurides decomposed during digestion by leaching the residues with water. The results are shown in the Table 11. It is evident from the results that with increase in the digestion temperature, selenium recovery increases while tellurium recovery decreases with copper and nickel recovery being almost constant.

The optimum conditions arrived at are 200% Conc. H_2SO_4 by weight in the charge with the digestion temperature at 280°C for 5 hours and a roasting temperature of 600°C for 4 hours. About 500 gm slime was treated in a silica retort in one batch and the recovery of selenium was about 98.5%

Cementation studies are under progress to recover silver, tellurium, copper and nickel from leached liquors.

Table 9 - Effects of digestion time and roasting temperature and time.

Temperature of digestion - 280°C.

Sl. No.	Digestion	Roasting		Se %	Te %	Ag %	Cu %	Ni %
	Time hr.	Temp. °C	Time hr.					
1.	6	500	4	75.00	24.82	28.40	78.31	72.25
2.	6	550	4	77.16	45.39	32.48	82.99	89.6
3.	6	600	4	94.82	66.21	34.23	86.35	93.59
4.	6	650	4	96.77	69.3	51.48	76.08	84.36
5.	5	600	1	92.62	64.33	15.28	97.42	86.76
6.	5	650	1	81.52	38.3	9.25	90.39	89.25
7.	6	600	1	83.97	61.34	26.08	72.21	86.27
8.	6	600	3	96.65	89.78	55.14	98.49	92.72
9.	5	600	3	96.54	72.65	29.19	97.83	93.21
10.	5	650	3	95.21	67.8	17.85	96.59	96.03
11.	5	600	1	98.16	91.82	50.25	99.13	99.95

Table 10 - Effect of acid concentration

Roasting time - 4 hours.

Sl. No.		Recovery				
	Acid %	Se %	Te %	Ag %	Cu %	Ni %
1.	75	13.66	97.12	86.28	70.15	40.45
2.	100	4.85	97.17	55.37	60.30	58.69
3.	150	17.99	96.34	90.90	54.43	29.29
4.	175	12.12	92.94	95.42	13.95	15.03
5.	200	98.16	91.82	50.25	99.13	44.90

Table 11 - Recovery of Se & Te at different digestion temperature.

Digestion time - 6 hours.

Sl. No.	Digestion Temp. °C.	Recovery				
		Se %	Te %	Ag %	Cu %	Ni %
1.	200	14.73	89.74	42.84	98.97	94.62
2.	240	55.77	53.23	56.26	95.66	79.91
3.	280	64.37	36.72	53.22	96.60	88.18
4.	320	73.85	80.34	32.72	98.09	86.92

Sulphuric acid roasting of slim involves problems relating to the material of construction and hence a few experiments were carried out to recover selenium and tellurium by decomposition of selenides and tellurides of copper and silver with sulphur under vacuum. Selenium and tellurium were deposited on the baffle plates kept at a cooler zone.

Copper slime -100 mesh was mixed with 200% by weight of elemental sulphur (-100 mesh fine) and briquetted at 2000 lb/sq, inch and the briquette was heated in a stainless steel tube inside a boat made of stainless steel. One end of the retort was closed and connected to a vacuum pump. Work is under progress on the vacuum decomposition studies.

25.0 Preparation of Anhydrous Magnesium Chloride.

Anhydrous magnesium chloride is used as a fluxing material for melting and refining of magnesium and magnesium base alloys. The work was taken up with a view to examine the possibilities of preparing anhydrous magnesium chloride from indigenous raw materials and to develop the know how for a scaled up production unit thereof. M/s. Hindusthan Aircraft Ltd., have showed interest in anhydrous magnesium chloride.

For production magnesium chloride by chlorinating magnesia, briquettes were prepared from -60 mesh magnesite, calcined at various temperatures mixed with -60 mesh calcined petroleum coke together with magnesium chloride solution of 22° Be as binder. The shape and size and heat treatment of green briquettes have been standardised. The physical properties of the dried briquettes were also measured.

These briquettes were chlorinated in a (Fig. 7) 25 mm dia quartz tube heated externally by a vertical tube furnace. The lower part of the tube was packed with graphite lumps which supported the charge. Chlorine was injected at the bottom of the graphite bed, which flowed up through the graphite and the charge. The reaction product being liquid at the reaction temperature, passed through graphite bed and collected in a silica receptacle fitted at the lower end of the quartz tube. The top was open to atmosphere under an exhaust duct. Results obtained with briquettes prepared from magnesite calcined at 1050°C (MgO 93.57%) and petroleum coke, calcined at 700°C under nitrogen atmosphere (F.C. 92.55%) were very encouraging and were studied in details.

The effect of physical properties, the time and temperature and amount of carbon on chlorine efficiency are shown in Figs. 8, 9 and 10 respectively. Chlorine efficiency was calculated on the basis of total magnesium chloride formed which includes losses due to volatilisation and absorption in the graphite bed and quantity of magnesium chloride collected. The optimum conditions for chlorination of briquettes were been established.

Pellets of 7 mm dia. prepared from the same composite mix as of briquettes were prepared in a drum pelletiser and heat treated under the same standardised conditions. These pellets were chlorinated under the

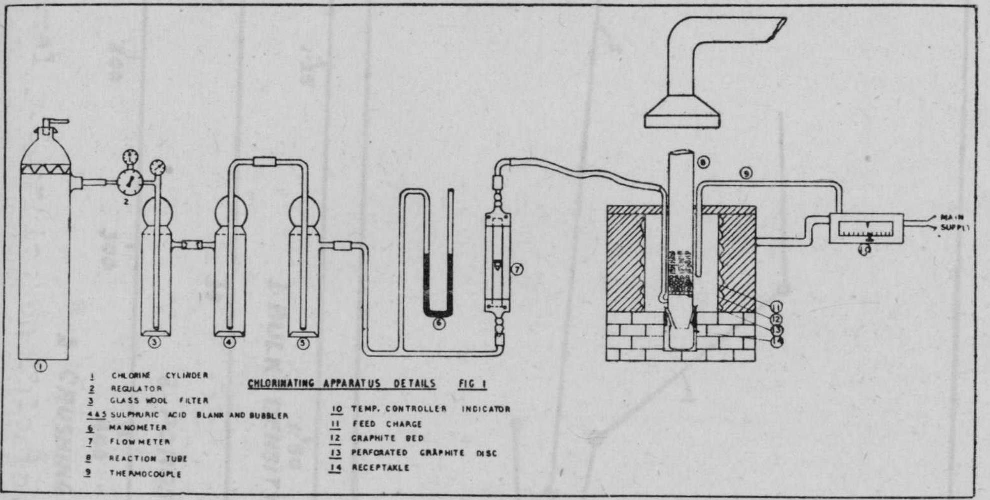


Fig. 7 Set up for Chlorination of briquettes

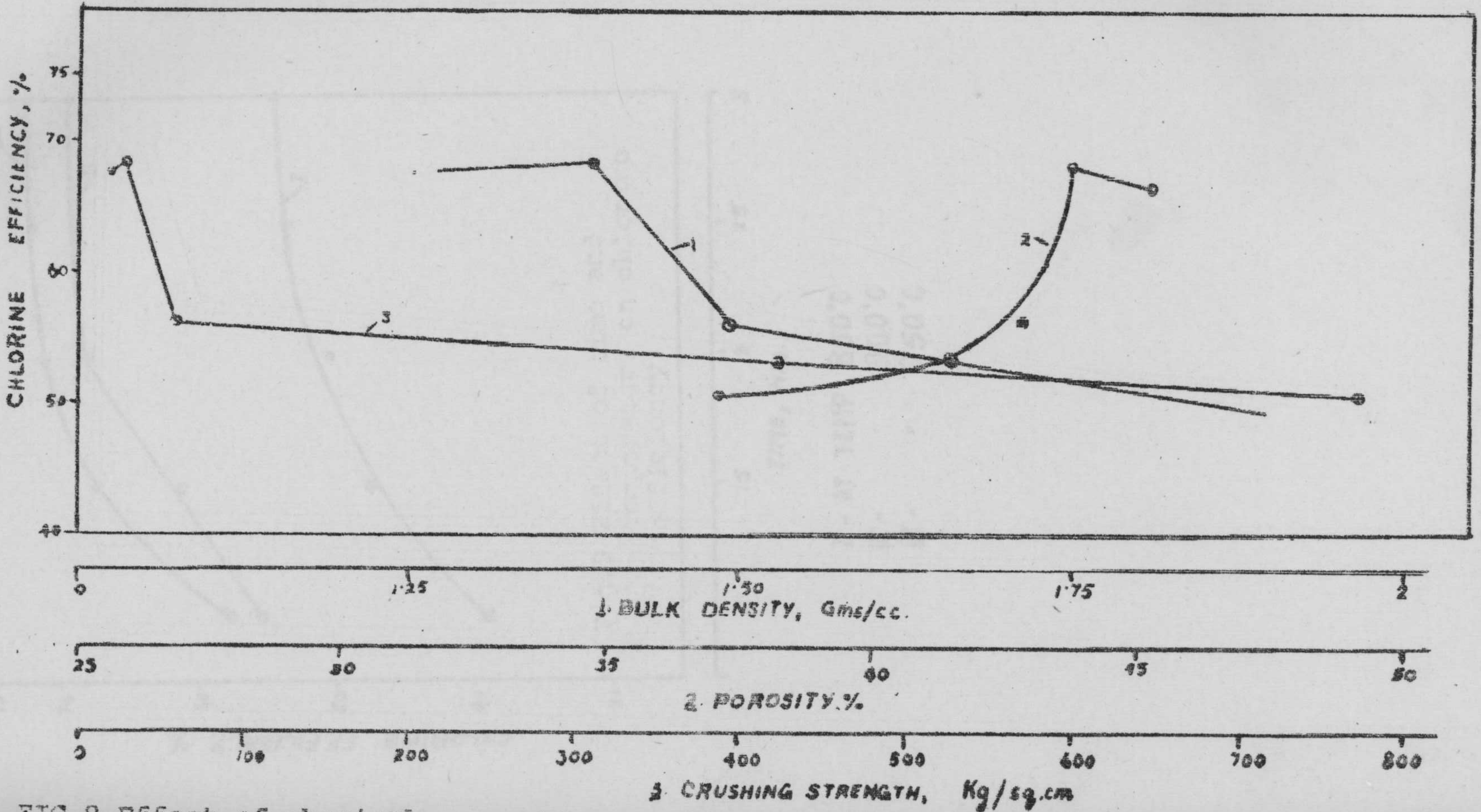


FIG.8 Effect of physical properties on Chlorine efficiency

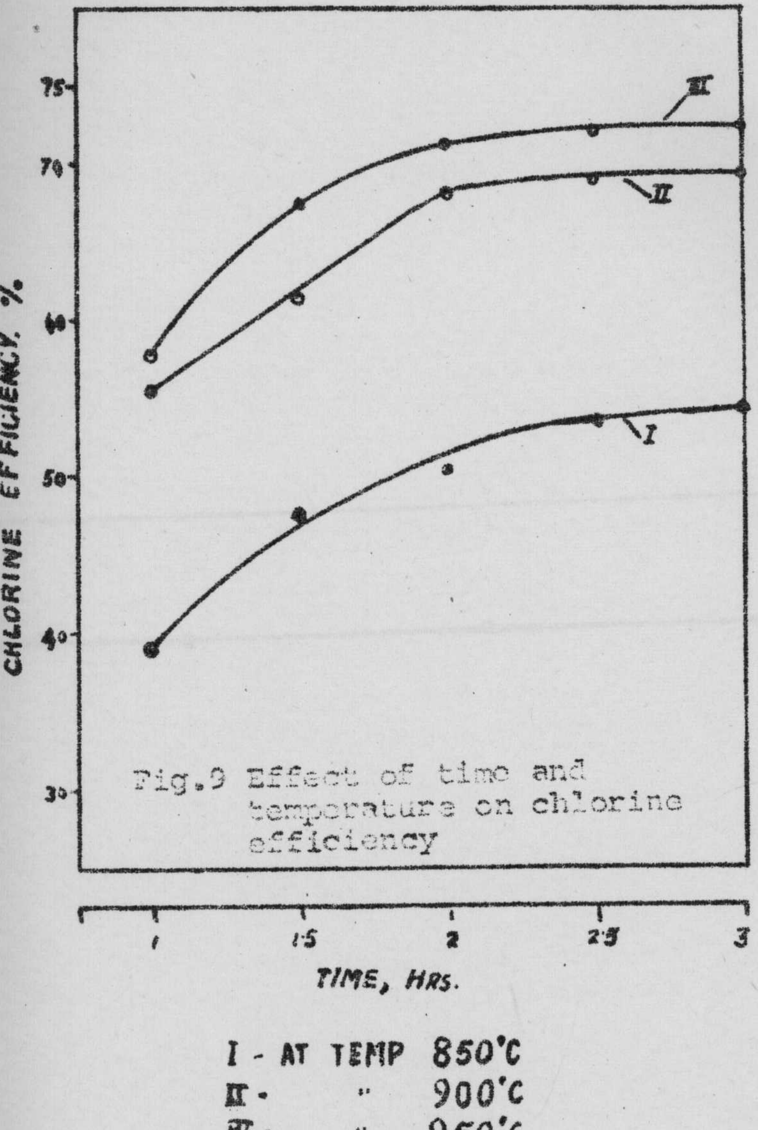


Fig.9 Effect of time and temperature on chlorine efficiency

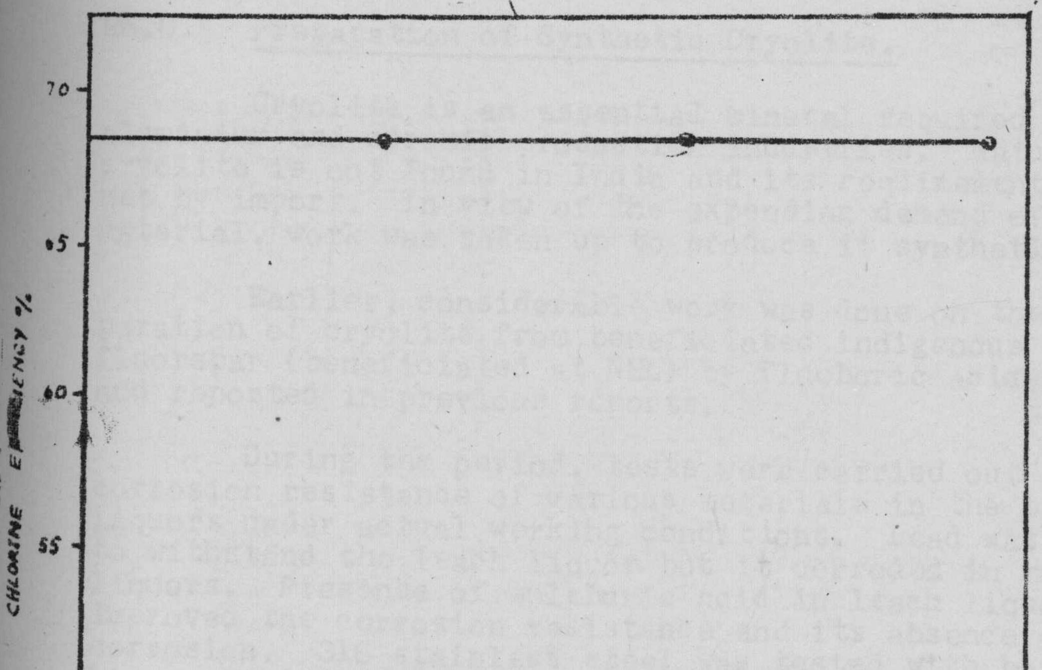


Fig.10 Effect of amount of carbon on chlorine efficiency

optimum conditions of briquettes. Results showed that the pellets were superior to briquettes as feed charge on the basis of chlorine efficiency.

On the basis of observed optimum condition proposal have been put forward to design a production unit with a capacity of 25 kg/day. The proposed unit should be internally heated.

It has also been decided to study the various variables viz physical properties, height of graphite lumps bed and chlorine efficiency etc. for both briquettes and pellets prepared from different raw materials, calcined as well as unclained.

26.0 Preparation of Synthetic Cryolite.

Cryolite is an essential mineral required in steel, aluminium and ceramic production industries. Natural cryolite is not found in India and its requirements are met by import. In view of the expending demand of this material, work was taken up to produce it synthetically.

Earlier, considerable work was done on the preparation of cryolite from beneficiated indigenous fluorspar (beneficiated at NML) by fluoboric acid process and reported in previous reports.

During the period, tests were carried out for the corrosion resistance of various materials in the process liquors under actual working conditions. Lead was found to withstand the leach liquor but it corroded in mother liquors. Presence of sulphuric acid in leach liquor improved the corrosion resistance and its absence caused corrosion. 316 stainless steel was tested with both the liquors and found resistant at room temperature. At higher temperatures 316 stainless steel was found to corrode. Ordinary 18:8 stainless steel could not withstand the liquors and corroded rapidly.

Rubber was found to be quite resistant at room temperature, but a higher temperature it softened and became completely unsuitable. Some articles coated with plastic emulsion paints were tested with the leach as well as mother liquor. Among this type of paints, epoxy paint was found quite good at room temperature. Test carried out at high temperature such as 95°C, caused the paint to peel off although the paint itself ramained unaffected. The possibility of eliminating the defect is being examined by improved surface cleaning, and better undercoat.

A few experiments were carried out for the precipitation of cryolite from fluoboric acid solution with sodium aluminate solution with a view to minimising the corrosion of lead lined tanks during precipitation as fluoboric acid is neutralised quickly by sodium aluminate. But the cryolite precipitate obtained was gelatinous and could not be filtered easily. Also the product contained traces of lead.

Table 12 - Cyclic experiment for Ammonium Cryolite.

Volume of leach liquor -- 3 litres
Boric acid added -- 360 gms
Ammonium sulphate added -- 69 gms
Sulphuric acid used -- 36 (N)
Time of leaching -- 2 hrs.
Temperature of leaching -- $95^{o} \pm 2^{o}C$.

Expt No.	Fluorspar added in gms.	Sulphuric added in c.c.	Residual sulphuric acid in leach liquor.	Calcium carbonate added in gms.	Fluboric acid found in gm.	Hydrated alumina added in gm.	Ammonium carbonate added in gm.	Ammonium cryolite obtd. in gm.
1.	600	408	17.38	17.5	313.8	238	200	386
2.	600	408	41.60	42.0	294.0	223	228	345
3.	600	408	56.90	58.0	347.0	264	250	420
4.	600	408	50.80	52.0	335.0	255	250	411
5.	600	408	7.30	7.0	335.7	255	290	420
6.	600	408	82.00	84.0	318.0	242	218	398
7.	600	408	80.19	82.0	298.0	197	218	320
8.	600	408	123.0	80.0	304.6	232	290	359
9.	600	408	165.00	80.0	294.7	224	300	340
10.	600	408	111.00	100.0	272.0	207	221	332

Leaching efficiency -- 94.6%

Overall efficiency -- 79.77%

Table 13 - Cyclic experiment for Ammonium Cryolite.

Expt No.	Fluorspar added in gms.	Sulphuric acid added in c.c.	Residual sulphuric acid leach liquor	Calcium carbonate added in gm.	Fluboric acid found in gm.	Hydrated alumina added in gm.	Ammonium carbonate added in gm.	Ammonium carbonate obtd. in gm.
1.	200	139	15.3	15.6	97.7	74.3	70	117
2.	200	139	16.9	17.2	104.9	79.7	73	128
3.	200	139	23.4	23.9	106.4	81.0	80	125
4.	200	139	34.5	35.2	108.6	82.5	80	130
5.	200	139	34.8	35.5	97.6	74.2	70	117
6.	200	139	42.1	42.9	101.0	76.8	77	118
7.	200	139	41.1	41.9	96.2	73.1	71	120
8.	200	139	46.8	30.0	90.3	68.6	90	113
9.	200	139	62.0	40.0	90.2	68.5	94	114
10.	200	139	64.5	40.0	84.2	64.0	79	103

Leaching efficiency -- 89.9%

Over fluorine recovery -- 77.0%

A few experiments were carried out for heating the process liquor with D.C. current so that external heating can be avoided. The heating was possible upto 80°C using graphite cathode and lead anode but both the electrodes suffered severe corrosion due to high corrosive nature of the liquor.

26.I Preparation of Ammonium Cryolite.

Cyclic experiments for leaching two different fluorspars with sulphuric acid and boric acid in presence of ammonium sulphate were carried out to standardise the leaching stage of the process. The results of these experiments are given in Tables 12 and 13. The results indicate that leaching efficiency ranges from 90-95% over a wide range of experiments with respect to fluorine values. In the ninth cycle, leaching efficiency dropped appreciably as is evident from residual H_2SO_4. Overall efficiency of 77-80% with respect to fluorine has been obtained. The ammonium cryolite obtained was quite pure and of high grade.

Decomposition of Ammonium Cryolite:

Ammonium cryolite obtained by the cyclic experiments were mixed and the decomposition of the mixture was studied in the apparatus shown in Fig. 11.

A pot containing ammonium cryolite was kept in the pot furnace fitted with a thermocouple and pyrometer. The outgoing gases were passed through another furnace containing alumina pellets, and three gas absorbing bublers containing boric acid solution, sodium carbonate solutions and dil sulphuric acid. The whole unit was connected to a suction pump. When the temperature was raised in the pot furnace, ammonium cryolite decomposed releasing a mixture containing ammonia, hydrofluoric acid and ammonium fluoride if any. The second furnace containing alumina was meant for reacting with HF gas to form aluminium fluoride.

The result of the experiments at various temperatures are given in table 14. Chemical analysis of the ammonium cryolite used was NH_4 17.15%; F 60.52%; Al 20.39%; Fe 0.098%; SiO_2 0.03%; SO_4 0.44%; CaO 1.24%; Moisture (110°C) 0.22%.

Table 14 - Decomposition of Ammonium Cryolite at various temperature.

Expt. No.	Wt. of Ammonium Cryolite	Temp.	Time	Residue gms.	Analysis % in Residue
1.	50 gms	300°C	4.00 hrs	45.94	F - 61.52 NH_4 - 11.19
2.	50 gms	400°C	4.00 "	40.40	F - 61.97 NH_4 - 6.88
3.	50 gms	500°C	4.00 "	34.61	F - 62.7 NH_4 - 0.25
4.	50 gms	550°C	4.00 "	33.27	F - 62.93 NH_4 - .23
5.	50 gms	660°C	4.00 "	33.16	F - 61.7 NH_4 - 0.21
6.	50 gms	650°C	4.00 "	31.15	F - 58.4 NH_4 - 0.17

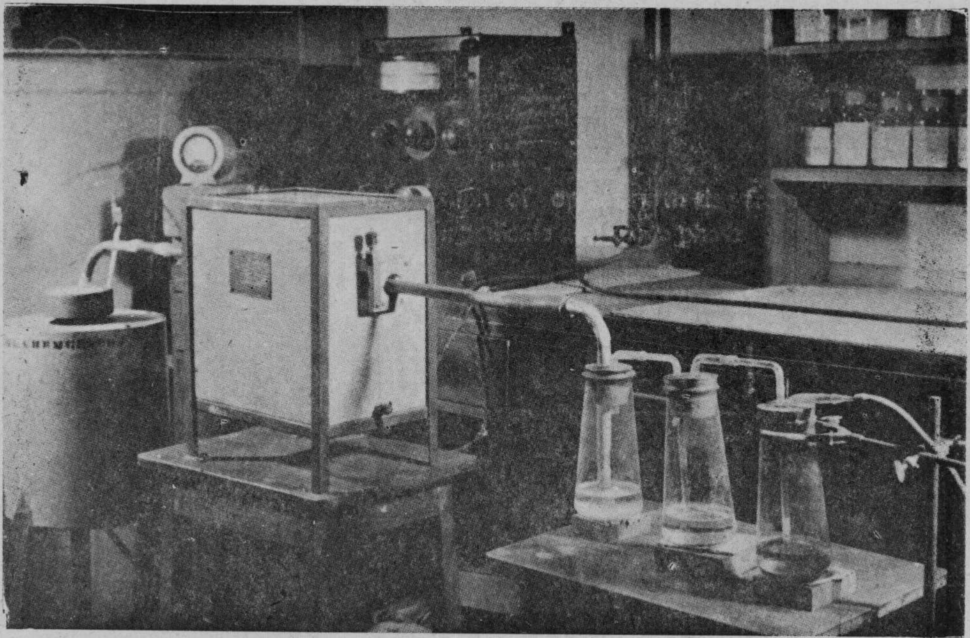


Fig. 11 Set up for studying the decomposition characteristics ammonium cryolite

Time of decomposition was kept constant for four hours. The temperature was varied from 300°C to 650°C. As evident from the table, decomposition of ammonium cryolite is almost complete at 500 to 550°C. Heating at still higher temperatures results in the loss of fluorine.

Further experiments are being continued to study the influence of time and presence of alumina on the rate of decomposition of ammonium cryolite and recovery of aluminium fluoride.

27.0 Nickel-free Austenitic Stainless Steel:

About 250 kg of electrolytic manganese was nitrided at the National Metallurgical Laboratory and another two ton heat of nickel-free stainless steel was made at the Alloy Steel Plant, Durgapur. The heat was made in a basic lined induction furnace, using low carbon ferro-chrome, nitrided electrolytic manganese and low-carbon steel scrap. The ingot obtained was soaked at 1200°C and was rolled into bloom.

The break-down plate of the first stainless steel heat made at the Alloy Steel Plant, Durgapur was analysed for its chemical composition. Tensile tests and hardness tests were carried out on the break-down plate also. Samples were taken from the break-down plate and were solution treated by quenching in water from different temperatures to determine the soaking temperatures at which the steel would develop duplex structure.

A number of heats were made in 20 lbs basic high frequency induction furnace at the laboratory to develop technique for critical control of the amount of nitrogen required, and its distribution inside the body of the ingot. A few ingots were sectioned longitudinally to study the internal structure of the ingots. Further work on the internal structure of the ingots of nickel-free stainless steel, the distribution of excess nitrogen in the steel ingot and utilisation of the hot-top technique is under progress.

28.0 Studies on Substitute High Strength Steels.

(a) Maraging type steels:

In recent years considerable interest has been shown in a new series of precipitation hardened high alloy martensitic steels known as maraging steels, developed by International Nickel Company, specially to meet the growing engineering requirements. The excellent properties obtained in these steels result from the age hardening of martensite during tempering. The exact mechanism by which the strengthening takes place is not yet properly established. The object of the present investigation is to study the exact mechanism of strengthening of martensite in these steels and to study the effect on such reactions by replacing nickel, molybdenum and cobalt by manganese and titanium etc. Studies on few such alloys containing 7-11% Ni, 4-14% Mn, 4% Mo and 6-7% Co show that excellent maraging properties can be obtained in a 11% Ni, 5% Mn, 6.5% Co and 4% Mo steel and studies on precipitation reactions etc. showed maraging takes place due to formation an ordered phase of Ni_3Mo along with Fe_2Mo. Further studies on a new series of alloys with Ti are being done using X-ray diffraction and metallographic techniques.

(b) Low cost maraging steels:

A project was taken up for the development of a low cost maraging steel with an iron-copper base composition in which precipitation of E-copper would give rise to a high order of strengthening. Chromium was added to extent of 8% in these steels to give additional strengthening and corrosion resistance. Nickel in amounts of half of the copper content was added to improve the hot working properties. The effects of small amounts of Nb and Mo on the impact properties of these steels are being presently studied.

Three 10 kg. heats of steel were made in a basic high frequency furnace, using low carbon mild steel scrap. Chemical composition of the steels are given in Table 15. The ingots were forged into 3/4" and 1/2" square bars and rolled into plates of 0.3" and sheets of 0.1" thickness. The hot working operations were carried out at 850°C to 900°C. Isothermal aging curves have been determined for these steels, ageing being carried out 485°C. The aging curves are shown in Fig. 12.

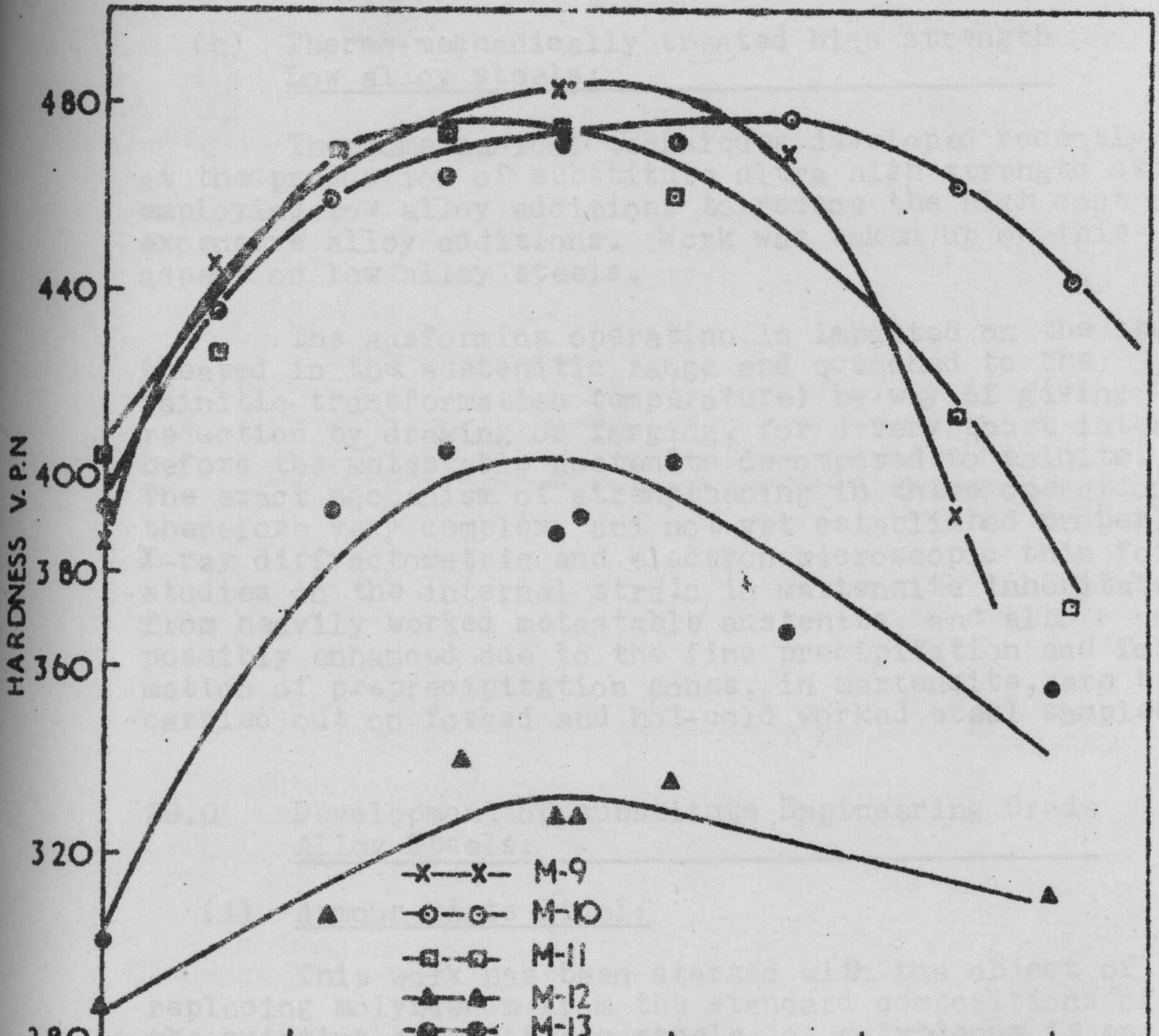


Fig.12 Precipitation hardening character-istics of the steels aged at 485°C

Table 15 - Chemical Composition weight percent.

Steel Nos.	Cu%	Ni%	C%	Cr%	Si%	Nb%	Mo%
M-9	2.80	1.48	0.07	8.82	0.28	0.05	--
M-10	2.99	1.48	0.05	8.36	0.47	--	0.50
M-11	3.20	1.55	0.07	8.30	0.30	0.05	0.50

Tensile and impact properties of these steels will be studied in detail and the optimum ageing conditions will be determined.

(c) Thermo-mechanically treated high strength Low alloy steels:

Thermomechanical techniques developed recently aims at the production of substitute ultra high strength steels employing low alloy additions to reduce the high cost of expensive alloy additions. Work was taken up on this aspect on low alloy steels.

The ausforming operation in imparted on the steels (heated in the austenitic range and quenched to the bainitic transformation temperature) by way of giving reduction by drawing or forging, for a very short interval before the metastable austenite decomposed to bainite. The exact mechanism of strengthening in these operations are therefore very complex, and not yet established properly. X-ray diffractometric and electron microscopic thin foil studies on the internal strain in martensite inheritated from heavily worked metastable austenite, and also possibly enhanced due to the fine precipitation and formation of preprecipitation zones, in martensite, are being carried out on forged and hot-cold worked steel samples.

29.0 Development of substitute Engineering Grade Alloy Steels:

(i) Armour Plate Steel:

This work has been started with the object of replacing molybdenum from the standard compositions of the existing armour plate steels, as molybdenum is an imported product. Further with the object of developing armour steel with the indigenous and more economic raw materials, work has been taken up to develop substitute steels of the Cr-Mn-V type to meet the requirements of the standard grade of steels.

As reported earlier, heat treatment was carried out extensively. Three more heats were made of the compositions given in Table 16.

Table 16 - Composition of Heats.

	H_5%	H_6%	H_7%
Carbon	0.42	0.37	0.48
Manganese	1.00	1.64	1.20
Silicon	0.35	0.30	0.30
Chromium	1.75	2.33	2.34
Vanadium	0.25	0.27	0.27

The initial hardness of the ingots as cast was observed to be too high. The ingots were therefore annealed and hardness determined (Table 17).

Table 17 - Hardness of Ingots.

Heat No.	VPN (Average)
H_3	284
H_4	329
H_5	311
H_6	335
H_7	529

Hardening temperatures for the heat series H_3 to H_5 were determined. It was observed that H_6 and H_7 varities are of air hardening type. It is proposed to compare the properties of this steel with the conventional armour plate steels. With this end in view, further heat treatment on the specimens to combine **maximum** strength with high toughness is in progress.

30.0 Carburising Characteristics of Grain-refined Steels.

In recent year, an improved variety of mild steels with residual amount of Nb additions has come into existence as an important type of structural steels. These Nb-treated steels are fine-grained steels with yield stress higher than that of mild steel. These Nb C(N) precipitates are very much resistant to growth and therefore it is believed that even after long carburising treatments the fine-grained structure with the associated toughness would be retained. A fine-grained steel which is capable of maintaining the fine grain-size during carburising has the advantages of maintaining a tough core and does not require any post-carburising heat-treatment for core-refining. The reduction in the number of heat treatment cycle reduces the cost of the process, the risk of distortion and decarburisation.

The object of work is to develop a suitable economic carburising grade steel.

In continuation to previous work, the properties of the experimental Nb-treated steels were compared with that of AISI 4520, AISI 4620, AISI 4820, AISIE 3310, AISIE 9310. The comparative case depths and case hardness values are indicated in Table 18. Variations of hardness from surface to centre of 1/2" round carburised bar of Nb-treated steel and mild steel are given in Fig. 13. A comparative values of tensile properties are indicated in Fig. 14.

Future work is to study Ti-treated grain-refined steel and mass-effect of different grain-refined steels.

Table 18 - Case depths for 1/2" round of standard steels after direct quenching.

Steels	Case depth in inch.	Case harness (Rc)
AISI 4520	0.054	64
AISI 4620	0.075	45.5
AISI 4820	0.039	60.0
AISIE 3310	0.047	59.95
AISIE 9310	0.039	59.5
Steel No. 1	0.065	67.7
Steel No. 2	0.051	67.5
Steel No. 3	0.059	67.7
Steel No. 4	0.061	67.5

31.0 Development of Substitute Die and Tool Steel.

This project was taken up with a view to develop substitute die and tool steel eliminating as far as practicable imported alloying elements like molybdenum, tungsten, etc. Such steels through judicious combination of indigenous alloying elements and optimum heat treatment should conform to the requirements of physical properties and specific service performance characteristics of standard types of steel.

i) Development of Die Steel:

The effect of quenching temperature, quenching media, tempering temperature and tempering time on hardness were studied on steel DS_4, composition of which is given in Table 19.

Table 19 - Composition of steel DS_4 in percentage weight.

Steel	C%	Si%	Mn%	Cr%	V%	Mo%	W%
DS_4	1.01	0.32	0.75	4.82	0.27	0.15	--

The hardness results of the experiments done on steel DS_4 are given in Tables 20, 21 and 22.

Table 20 - Effect of Quenching Temperature after Heating for 30 minutes.

66666

Quenching temperature °C	Hardness (VPN)
850°C	870
900°C	905
950°C	840
1000°C	790

Table 21 - Effect of Quenching Media after Heating for 30 minutes.

Quenching media.	Hardness (VPN)
Water	920
Oil	905
Air	835

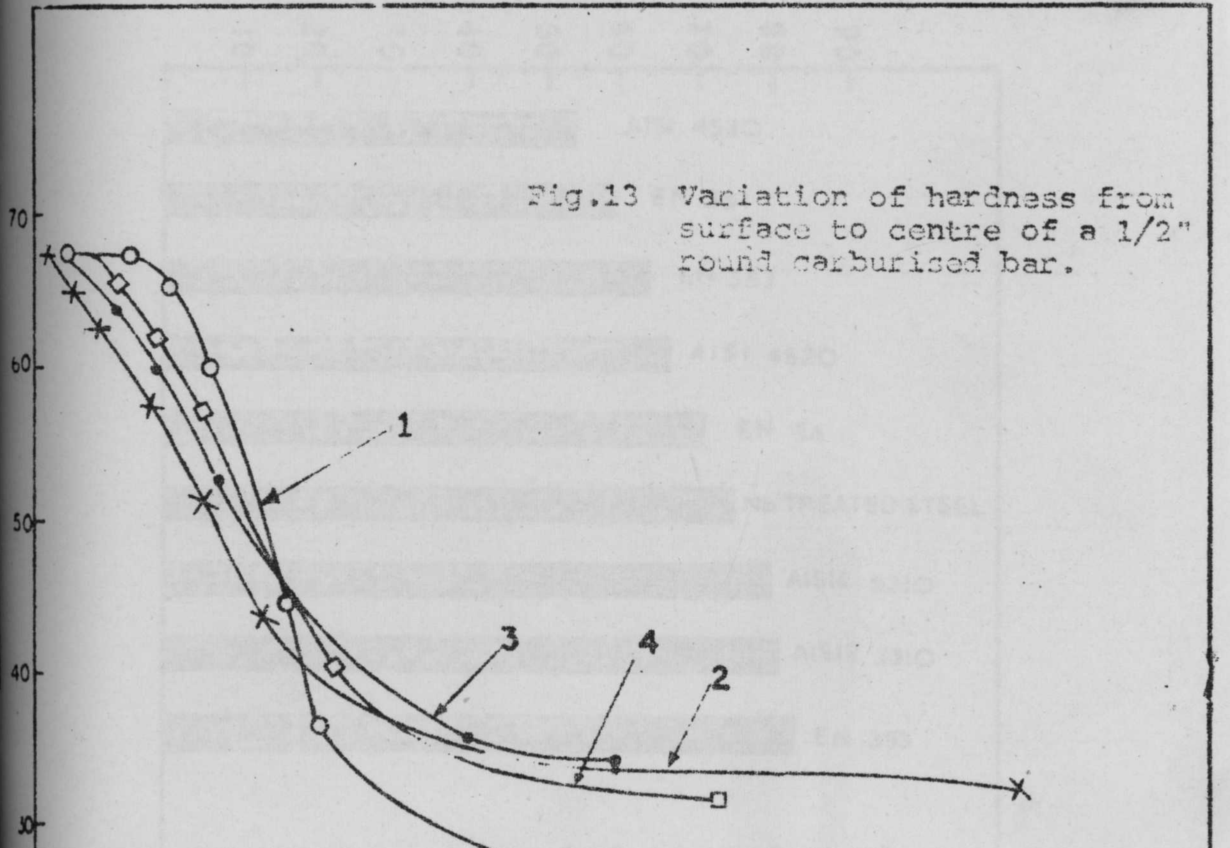


Fig.13 Variation of hardness from surface to centre of a 1/2" round carburised bar.

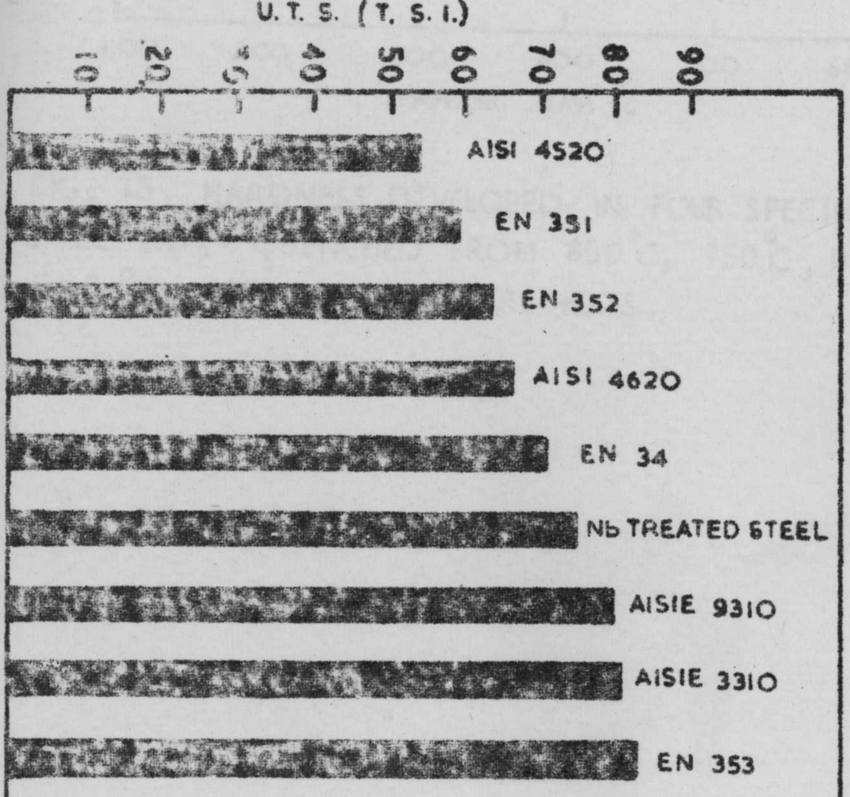


Fig.14 Comparison of Nb treated steel with alloy carburising steels.

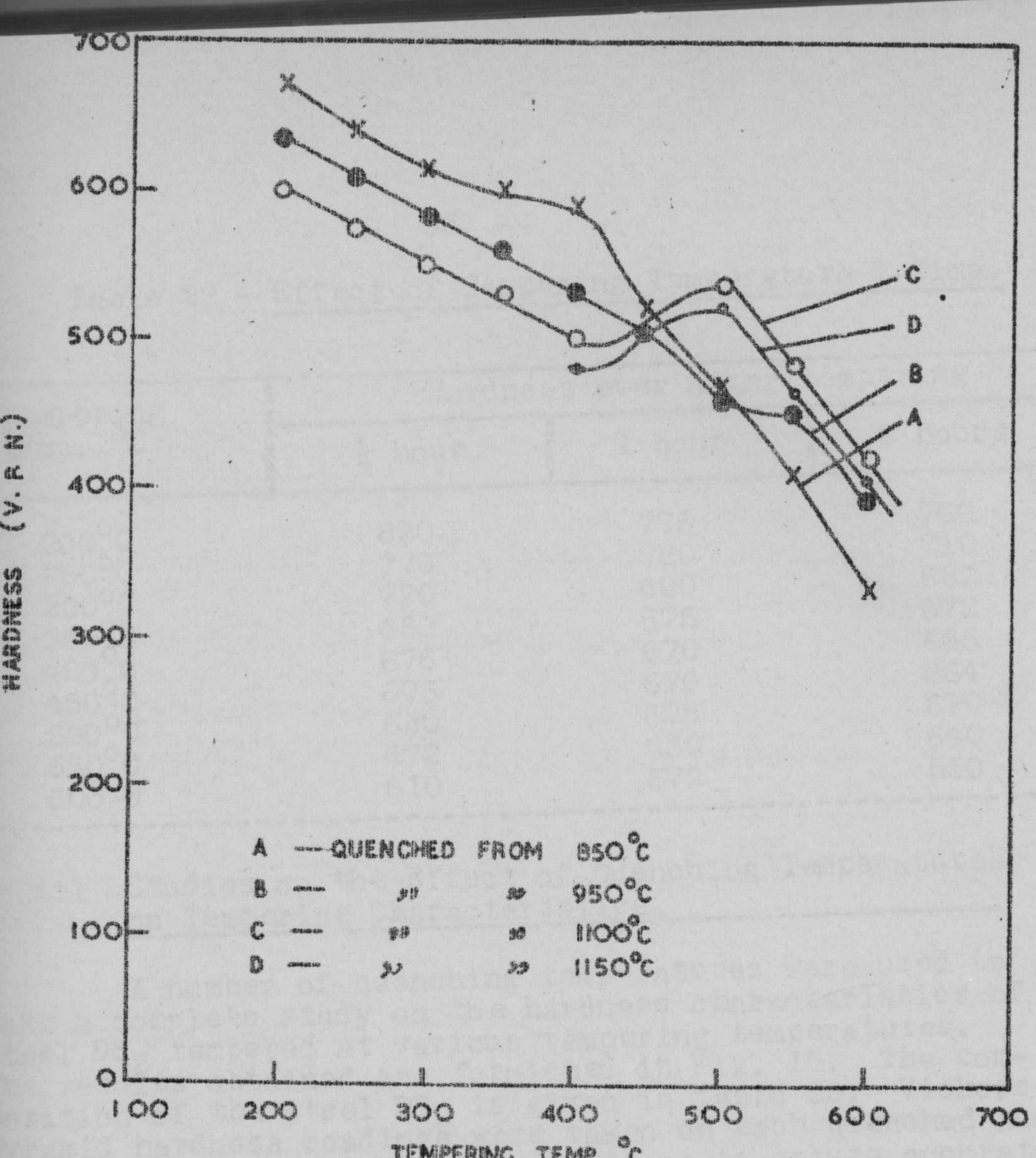


FIG: 15. HARDNESS DEVELOPED IN FOUR SPECIMENS OF STEEL DS_2 QUENCHED FROM 850°C, 950°C, 1100°C & 1150 °C AFTER VARIOUS RE-HEATS.

Table 22 - Effect of Tempering Temperature & Time.

Tempering temp. °C	Hardness ever after tempering		
	½ hour	1 hour	2 hours
200°C	820	775	760
250°C	770	720	710
300°C	720	690	682
350°C	680	675	672
400°C	676	670	665
450°C	675	670	664
500°C	680	676	670
550°C	672	650	640
600°C	610	572	550

ii) Studies on the Effect of Quenching Temperatures on Tempering Characteristics.

A number of quenching temperatures were used to make a complete study on the hardness characteristics of steel DS_2 tempered at various tempering temperatures. The results obtained are furnished in Fig. 15. The composition of the steel DS_2 is given in Table 23. Vickers Pyramid hardness readings were taken on each quenched and tempered specimen with all precautions to ensure accurate results.

Table 23 - Composition of Steel DS_2.

Steel	C%	Si%	Mn%	Cr%	V%	Mo%	W%
DS_2	1.09	0.51	0.88	2.46	0.11	0.09	--

iii) Cast Tool Steels:

Several heats of 18-4-1 type high speed steel were made in 25 lb H.F. induction furnace and were cast into cutting tools in specially made graphite moulds. Composition of the cutting tool castings are given in Table 24.

Table 24 - Composition in percentage weight.

Steel	C	Si	Mn	Cr	W	V
18-4-1	0.75	0.24	0.45	3.74	16.71	1.08

The cutting tools in the as cast condition were heat treated, i.e. sub-zero treated after quenching from different quenching temperatures and then single, double and triple tempered at 550°C. The hardness values obtained after different treatments are given in Table 25.

Table 25 - Hardness values of Tool Steel Heats after Different Treatment.

Quenching temperature °C	Graphite mould								
	Oil quenched	Sub-zero -180°C	Tempering			Sub-zero -120°C	Tempering		
			1st	2nd	3rd		1st	2nd	3rd
	(VPN)	(VPN)	(VPN)	(VPN)	(VPN)	(VPN)	(VPN)	(VPN)	
1200°C	786	915	794	786	-	823	741	735	-
1250°C	804	920	798	792	763	830	750	740	-
1300°C	886	919	860	804	786	834	782	760	-

32.0 Improved Mild Steels for Structural Purpose.

The object of the investigation is to improve the properties of mild steels by adding residual quantities of alloying elements which while contributing little to the increase in the cost of production would impart pronounced strengthening effect. With this purpose in view additions of niobium, vanadium, titanium and nitrogen were made in minute quantities to low carbon steel base. Manganese was added upto 1.5% to some of the steels.

Previous work on this project was related to the heat treatment variables affecting the mechanical properties of Mn-Nb-Ti steels. Current work was aimed at determination of the precipitation characteristics of these steels as influenced by various heat treatment processes. Samples of steels under different conditions of treatment were subjected to electron microscopic study. Work is being done on the correlation of the precipitate type with the mechanical properties of the steels.

33.0 Development of Low Alloy High Tensile Structure Steel.

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

The previous experiments were made to bring improvement in the properties of the existing low alloy steels in use through (i) Judicious modification in the percentages of the alloying elements, so that the amount of pearlite is reduced while the strength is increased (ii) Refinement of grain size by the addition of grain refining elements like vanadium, titanium and aluminium (iii) Dispersion of fine precipitates like nitrides of vanadium, aluminium etc. which go into solution at higher temperatures and precipitate on subsequent cooling.

A number of experimental heats were made and the tensile properties, impact resistance at room temperature and sub-sero temperatures, weldability and atmospheric corrosion resistance of most of the steels were determined and reported earlier. Based on the results obtained from the experimental heats a few bigger heats were also made in the 0.8 ton arc furnace. Some of the properties of these steels were also reported earlier.

During the period under review attention was directed to improve the grain size of some of these steels and some standard steels in use, following the technique of controlled temperature rolling.

Steels selected for this work were 0.2% C mild steel, Admiralty Grade 'B' steel and a vanadium bearing low alloy steel developed here. In the first phase of this work, rolling experiments were carried out with mild steel. Pieces of suitable lengths were cut from ¾" thick flat. These were homogenised at 1000°C and rolled by a single pass from the pre-selected temperature with pre-selected drafts. Table 26. The rolling temperatures, in this experiments, were considered as the finish rolling temperature as the rolling was carried out very quickly.

Table 26 - Markings on the specimens showing the details of finish rolling temperature and draft percentage.

Proposed draft %	Finish Rolling Temp.			
	950°C	900°C	850°C	800°C
10	A1	B1	C1	D1
20	A2	B2	C2	D2
30	A3	B3	C3	D3
40	A4	B4	C4	D4

From these rolled plates tensile and impact test pieces were made and tested in the as-rolled condition. The results are given in Table 27. The microstructure corresponding to each rolling condition was observed by optical microscope. The grain sizes as determined by liner intercept method are also given in Table 27.

The test results showed no appreciable change in the U.T.S. values. They gave an average value about 33.38 T.S.I. The lower yield point irrespective of finishing temperature within the range of 800° to 900°C showed a general tendency to increase with the increase of draft order upto 40%, increase in yield point were obtained only when finished above 900°C or at 900°C. The room temperature impact properties of the specimens rolled and finished at 950°C, 900°C, 850°C in general showed an improvement with higher drafts. Impact properties determined below the ambie temperature were found to fall in the usual manner as the test temperature was lowered. The specimen No. A4 showed a significant drop in the transition temperature than the others. This was rolled at 950°C with 40% reduction.

The properties of the Admiralty Grade 'B' steel and the low alloy steel mentioned above are being determined. Further work will be carried out in the same direction.

34.0 Development of Electrical Resistance Alloys for Heating Elements.

The aim of the project is to develop electrical resistance alloys indegeniously, since nichrome and kanthal group of heating elements are imported and contain large propertions of nickel and cobalt, both of which are rare in India.

As reported earlier, the major work on the development of the alloy composition and its processing techniques has already been completed and the process has been released for industrial production. During the period under review, the work was chiefly related to study the welding characteristic of the alloy with a view to evolving optimum conditions for resistance butt welding of the wire-samples. When the wire breaks during wire-drawing operation, it is necessary to butt weld the end of the wires for continuous production. Welding of the Fe-Cr-Al alloys is generally known to difficult due to excessive grain coarsening tendency and oxidation of the constituents, like chromium and aluminium, which

TABLES 27 - Tensile & Impact values of the Specimen

Sp. No	Tensile Properties			Charpy Impact Values (ft. lb.)						Average grain dia x 10^{-4} cm.
	Y.S.(L) T.SI	U.T.S. T.SI	E%	R. Temp. 30-40^{o}C	20^{o}C	10^{o}C	0^{o}C	-10^{o}C	-20^{o}C	
A1	20.5	33.5	34.3	58	-	-	40	10	8	17.8
A2	19.69	33.3	37.5	88	-	-	50	40	10	1 7
A3	20.76	33.3	34.37	48/46	34	28	16	-	-	14.24
A4	21.44	33.59	35	100	-	-	44	40	40	13.98
B1	19.44	33.5	34.3	50	-	-	40	10	8	17.2
B2	19.69	33.3	37.5	88	-	-	42	18	10	15.5
B3	20.30	34.01	31.25	112	42	24	10	-	-	10.75
B4	21.22	33.13	33	78	48	18	14	-	-	-
C1	20.16	33.5	37.5	52	-	-	42	38	8	16.6
C2	20.30	33.35	43.75	60	28	10	8	-	-	14.6
C3	21.84	33.79	37.5	130	48	24	22	-	-	12.23
C4	21.65	33.01	37.5	64	62	22	14	-	-	8.8
D1	19.51	33.5	39	36/48	-	-	8	6	-	-
D2	20.76	33.3	43.75	80	28	12	8	-	-	-
D3	21.84	34.37	37.5	42/46	38	26	10	-	-	-
D4	21.09	33.59	37.5	70	44	14	12	-	-	-

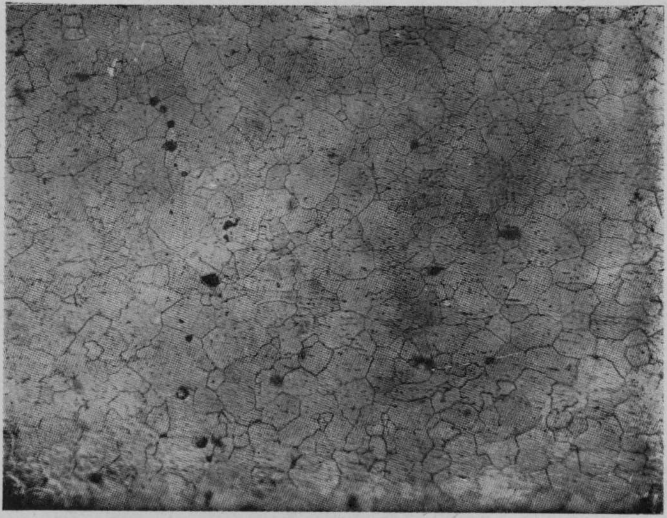


Fig. 16 Photomicrograph of weld zone

Fig. 17 Photomicrograph of weld zone

lead to the formation of weak and brittle weld zone resulting failure of the joint during wire drawing operation. To over come this difficulty, a systematic study was under taken to evolve a suitable technique for butt welding the alloys. For this purpose a resistance wire butt welding machine was used.

Work on welding of the alloy was mainly carried out in two phases. In the first phase of work, the wire samples with flat as well as tapered ends were welded under varying conditions of pressure and current. In some cases, fluxes were also used to minimise the oxidation. But satisfactory results were not obtained since most of the failures occured in the weld zone during wire-drawing showed brittle fracture. The above samples could not be drawn under heat treated condition also.

The photomicrographs (Figs. 16 and 17) of the weld zone of the wire sample welded with flat end in open atmosphere revealed very coarse grains as compared to the adjacent small parent grains.

In second phase of the work, welding was carried out in argon atmosphere with suitable quenching and heat treatment. Several wires of 10 SWG were welded with flat and tapered ends in presence of argon atmosphere under optimum condition of current and pressure. Special care was taken during preparation of wire-samples. The wire ends to be joined should be perfectly flat and there should not be any over heated zones on the flat end due to grinding. The welded wires were then heated at about 1000°C and immediately water quenched. The welded portion of several wires was successfully drawn upto 14 SWG.

As seen from the micro-structure of the heat treated wire samples, the weld zone was occupied with uniform and small grains as the parent grains which are desirable features for a good weld. Welding experiment with smaller gauges wires was also carried out under optimum condition of pressure and current and the wires were heat treated and successfully drawn up to 18 SGW. In all the cases, pressure and current were so adjusted as to produce bulging at the weld which is slightly bigger than the original diameter of the wire.

Some tests were also conducted to study the effect of nitrogen atmosphere on the quality of the weld. Several wires were welded in nitrogen atmosphere under the same conditions as was followed in case of wire treated with argon gas. All the wires failed from the weld zones during drawing. Metallographic examination of the welded samples will be carried out to evaluate the cause of failure.

Effect of trace elements:

As reported earlier, some heats were made in high frequency furnace and the ingot surface, hardness, and hot workability of each heat were all studied. Metallographic examination of the cast as well as forged pieces are being carried out to see the effect of individual element on grain refinement.

35.00 Development of Magnetic Materials.

(i) Isotropic ceramic magnets from mixed oxide:

During the period under review the effect of lubricants and binders on the density of ferrite magnets was further examined. For the present study methyl cellulose and polyvinyl alcohol were taken in concentrations varying from 0.01 to 0.3%. The powders mixed with lubricants were compacted under pressures of 470 to 2000 kg/sq.c. The densities after compacting and sintering at 1200°C for 2 hours were determined. It was found that the values of densities were maximum for 0.1 to 0.2% concentration of polyvinyl alcohol for pressures upto 1500 Kgs. At higher pressures the densities decreased with increasing concentration. In case of methyl cellulose the density changes were insignificant with increase of concentration.

(ii) Magnetic properties of cobalt-aluminium alloys:

Cobalt-aluminium alloys in the range of 14 to 15% aluminium were investigated for their magnetic properties. The alloys were quenched from 1380°C in water and tempered at 500, 550, 600, 650, 700°C for durations upto 100 hours. The magnetic properties of the quenched alloys were poor but increased manifold on tempering between 500-600°C and showed an energy product between 1.5 to 1.8 m.g.o. X-ray diffraction analysis and electron microscopic analysis of quenched and tempered specimens showed the presence of Co-Al superlattice of b.c.c. structure and elongated cobalt particles.

The effect of Ni, Fe, Mn on the magnetic properties of these alloys is under study. Preliminary results have shown that nickel has the effect of increasing the coercive force and that iron accelerates the tempering reaction.

(iii) Correlation of magnetism with crystal structure:

The correlation of magnetism with crystal structure suggested that in certain system like Nb-Al, Ta-Al in which sigma phase in appearing, ceasium chloride structures are likely to be presented and alloys of these systems made by courtesy of Bhabha Atomic Research Centre are being examined for the purpose.

36.0 Production Technology of Nickel-Silver for Electrical Purposes.

Large quantity of nickel-silver springs are used by Telephone Industries and other electrical industries in their relays and electrical controls. The technology of production of nickel silver is difficult and none of the Indian industries are producing the item uptil now. Requirements of nickel silver is being met through import. The objective of this project is to develop the technical know-how to study the foundry technology as well as mechanical working of this material by hot and cold rolling.

From the literature survey sufficient information on the subject was not available.

Few heats were made of the alloy in gas fired crucible furnace. The use of small crucible furnace has the advantage of better control of pouring conditions especially when casting a small quantity of metal.

Crucibles of magnesite and siliminite were used in order to avoid possible carbon pick up but in most of the cases it was observed that the crucible cracked. The small quantity which could be produced using these crucibles were poured in the all the mould. The chemical analyses of the product conformed the specified composition.

Hardness studies, and microstructures of the samples from such heats were made. Workability of the material, however, was not very good. Study of the metallurgical quality of the melt is being made to find out the cause of its poor workability.

A few more heats were made using high grade graphite crucibles. The possibility of carbon pick, if there is any and its extent in the nickel-silver alloy is under study. Further work will comprise of the complete study of the production and foundry technology of the nickel-silver alloy and to obtain a schedule of cold rolling and annealing.

37.0 Development of Aluminium Base Bearing Alloy.

The investigation was taken up with view to develop a suitable aluminium base bearing alloy which will replace the convensional bearings alloy i.e. white metal etc.

Standardisation of the chemical compositions and the mechanical properties has been reported in the previous annual reports. As the Indian Railways are interested for the service trials of these bearings, work was carried out to manufacture fault free floating bushes according to drawing supplied by the railways.

As various casting diffects i.e. blow holes and shrinkage cavities were experienced while casting the floating bushes, attention was given to use various degassers and to adjust the pouring temperature which will give best castings. Castings were made in sand mould. Suitable risers and gates were also provided with the mould. Pouring was done either side, top or bottom. The pouring temperature was kept within the optimum limits. Much improvement in casting was observed as a result of bottom pouring. It considerably reduced the blow holes and shrinkage cavity to the minimum. Further castings are being made to completely eliminate the shrinkage cavity and the blow holes. In all the cases the hardness of the cast floating bushes were measured and found to be within the range of 50-58 B.H.N. In antiseizure test, frictional properties under marginal lubrication conditions were found to be of the same order as those of standard bearing bronzes upto a load of at least 1480 lb. per sq. in. Metallographic structures were also determined. Further work is in progress.

38.0 Investigation on Lubrication for Cold Working of Stainless Steel.

In cold working processes of stainless steel e.g., wire drawing and press forming, where severe reductions in cross sectional area are being made and work hardening tendency is high, it is necessary to have a suitable lubricant and coating which will withstand the great pressure during the processing. The work was undertaken to study the efficiency of various lubricants and precoats on cold working behaviour of stainless steels.

Wire draw:

Four pieces of stainless steel wires (10 SWG) were pickled first in H_2SO_4 and then in a mixture of HNO_3 and HF. After cleaning thoroughly in water, three wires were given coating of lime, exalate and coating x respectively. One wire, without any coating, was drawn with E.P. Grease and graphite as lubricant. The coated wires were drawn using soap solution and stearic acid as lubricants. The wires were drawn upto 14 SWG without any intermediate annealing. The surface characteristics of the drawn samples are being studied.

Simulated cold rolling:

Four stainless steel plates (4" length x 1" width x 1/8" thick) of hardness 180 V.P.N. were solution treated to reduce the hardness to 150 V.P.N. The plates were descaled and cleaned. Three plates were given the coating of lime, oxalate and coating x respectively. The uncoated plate was given different percentage of deformation in Ford Compression Test apparatus using E.P. Grease and graphite as lubricant. The other plates were also given similar deformations using soap solution as lubricant.

The effect of various precoat and lubricant combinations on wire drawing and cold rolling can be summarised as follows.

For wire drawing, oxalate coating and soap solution as lubricant were observed to give more efficient draw even upto high percentage of reduction. For cold rompressiong lime and soap solution combination as well as precoat x and soap solution gave good results for smaller percentages of deformations; but for higher percentages od deformations, oxalate coating with soap solution gave better result. Further work is in progress.

39.0 Light Metals and their Alloys.

(i) Aluminium-magnesium alloys:

Work on the development of high magnesium-aluminium alloys was continued, and the effect of addition of small quantities of misch metal and chromium was studied. Investigations were mainly directed towards obtaining hot and cold workable alloys, and study of precipitation hardening characteristics.

Alloys having a nominal composition Al-8% Mg-2% misch metal and 0.1 to 0.2% Cr showed a Vickers Pyramid Hardness number of 77.9 immediately after solution treatment. The hardness values rose to 92.7 after 50 minutes. But aging beyond that time resulted in much lower hardness values which varied between 80 and 90 VPN, the hardness after 12500 minutes was 86.4 VPN. Similar results were obtained repeatedly in alloys having a composition within the above nominal composition.

Attempts to hot forge and hot roll these alloys did not give encouraging results. Ingots could be only partially hot forged, after which failure occurred with the production of edge cracks. Time and temperature of solution treatment and also the intermediate soaking time between two consecutive forging operations were varied to obtain better forgeability. Metallorgaphic studies were carried out in order to investigate the cause of failure. It was concluded that the ingot structure was reasonably good for hot working. It was felt that use of a heated anvil, which would reduce the rate of cooling of the work piece, may result in successful hot forging of the ingots. Fresh heats are being made to investigate this possibility.

ii) Aluminium alloys for aircraft industry:

In order to develop indigenously aluminium alloys generally required for the aircraft industry, three commonly used alloys with Y-alloy, duralumin 14ST and R-R 53, were chosen for a detailed investigation with regard to their founding, working and age-hardening characteristics. Alloys of the following nominal compositions were melted in a gas-fired furnace and cast into ingots in limecoated permanent moulds:

1) Alloy 1 Cu -4% Ni -2%, Mg -1.5% (Similar Y alloy)
2) Alloy 2 Cu -4% Mn-0.5%, Mg -0.5% (" durulumin 14ST)
3) Alloy 3 Cu -2% Ni-1% Fe -0.8%,Mg-1%(" RR-53 alloy)
Ti -0.1%, Si-0.5%.

The hardness of these alloys in the as cast condition was found to be 92.3, 42 and 83.1 VPHN respectively. The microstructure showed the presence of several binary, ternary and quartarnary intermetallic compounds such as $CuAl_2$, $Cu(Mg)Al_2$, $NiAl_3$, etc. distributed in the aluminium matrix. Results of heat treatment of the as cast specimens are given in Table 28.

Table 28 - Results of heat treatment of alloys:

Alloy	Treatment	Max stress	R.A.%	Elong.%
Alloy 1	Heated for 510°C for about 6 hrs and then quenched in boiling water and subsequently age-hardened for about 65 hours.	7.78	-	2
-do-	Same treatment except holding for 6 hrs and quenched in boiling water.	15.91	5	5
Alloy 2	Heated upto 500°C for about 2 hrs quenched and then age-hardened at 240°C for 3 hrs and then cooled in the furnace.	6.3	86	42
	Heated upto 512°C for 3 hrs followed by quenching in water and then ageing at room for 6 days.	7.4	84	40
Alloy 3	Heated upto 520°C for about 4 hrs followed by water quenching and then age-hardened at 160°C for about 27 hrs and then cooled in the furnace for about 18 hrs.	10.68	--	3
-do-	Heated upto 510°C for about 4 hrs followed by water quenching and then age-hardened at 160°C for about 27 hrs and then cooled in the furnace for about 42 hrs.	11.10	--	4

Ingots of alloy No. 1 were forged and rolled down to nearly 2 mm thick sheet, those of alloys Nos. 2 & 3 were extruded to 3/4" dia. rod and also forged and rolled down to 2 mm thick sheet. Heat treatment of these wrought alloys was carried out in a similar manner and the mechanical properties obtained are summarised below:

When a sheet of alloy 2 was quenched in water and aged at room temperature, the tensile strength and hardness increased with an increase of the quenching temperature to a point just short of actual melting of the $CuAl_2$ - aluminium eutectic, but the elongation fell off with increasing quenching temperature above 480 to 490°C (Fig. 18). The best average properties were obtained by using a quenching or 'solution' temperature of about 512°C, but actually in practice, this temperature was too high owing to osftening which renders it difficult to satisfactorily handle the material.

Fig. 19 shows that the period of "soaking" at the "solution" temperature prior to water quenching and ageing at room temperature is relatively unimportant and that for sheet, a soaking time of half an hour is sufficient. However, the best properties are not realised only if the soaking time is kept below this figure.

Fig. 20 clearly indicates that the strength and hardness rise rapidly at first and then more gradually until the maximum improvement of properties is obtained after about 72 hours.

The effect of altering the ageing period at 100°C, as shown in Fig. 21 shows that the tensile strength and related properties improve with increase in ageing time and thereafter slightly decrease.

iii) Duralumin type aluminium alloys:

Two heats were made of the duralumin type aluminium alloys having the following composition:

	I	II
Cu	3.98%	2.73%
Mg	0.67%	0.57%
Mn	0.18%	0.13%
Si	0.72%	0.63%
Fe	0.52%	0.49%

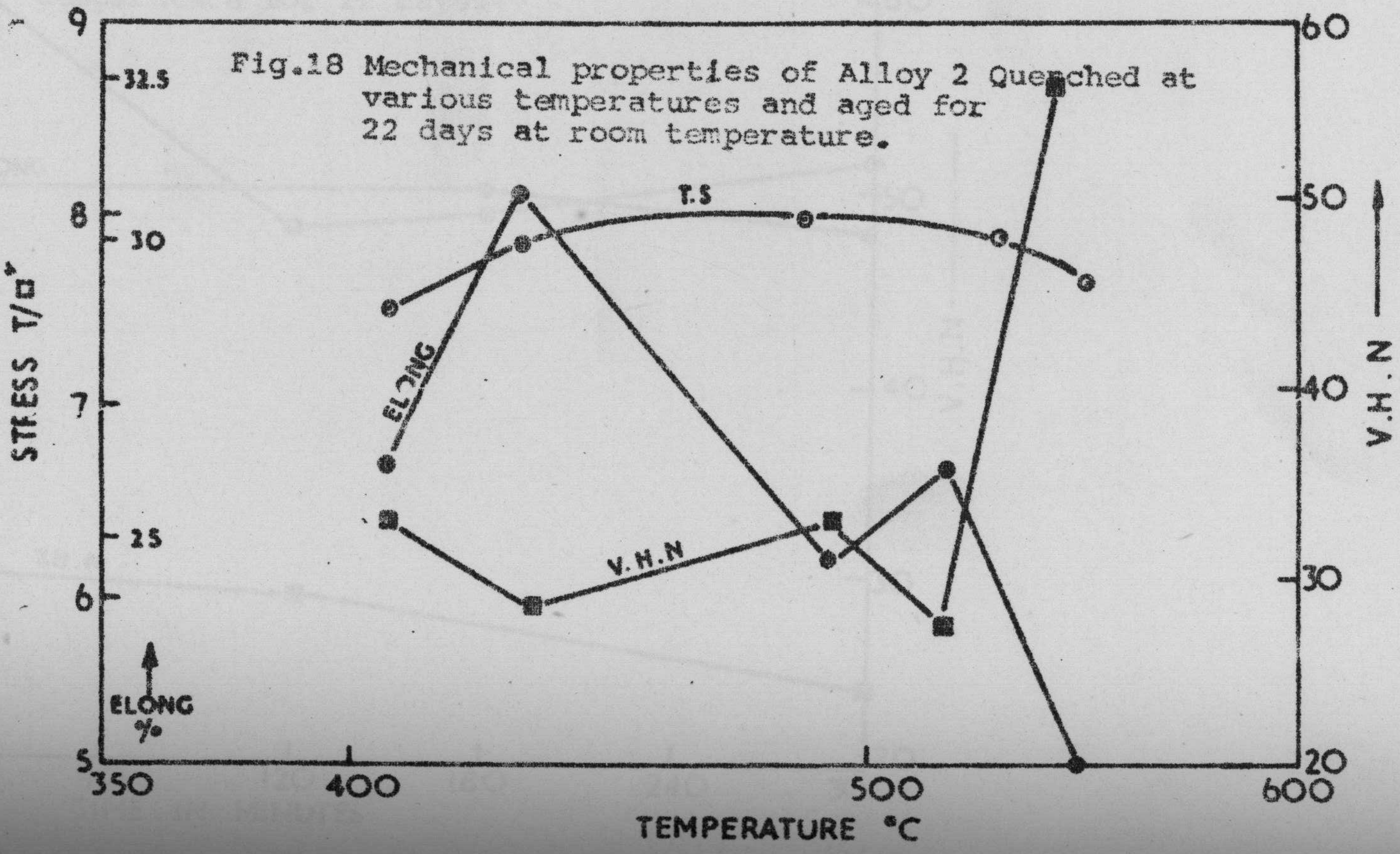


Fig.18 Mechanical properties of Alloy 2 Quenched at various temperatures and aged for 22 days at room temperature.

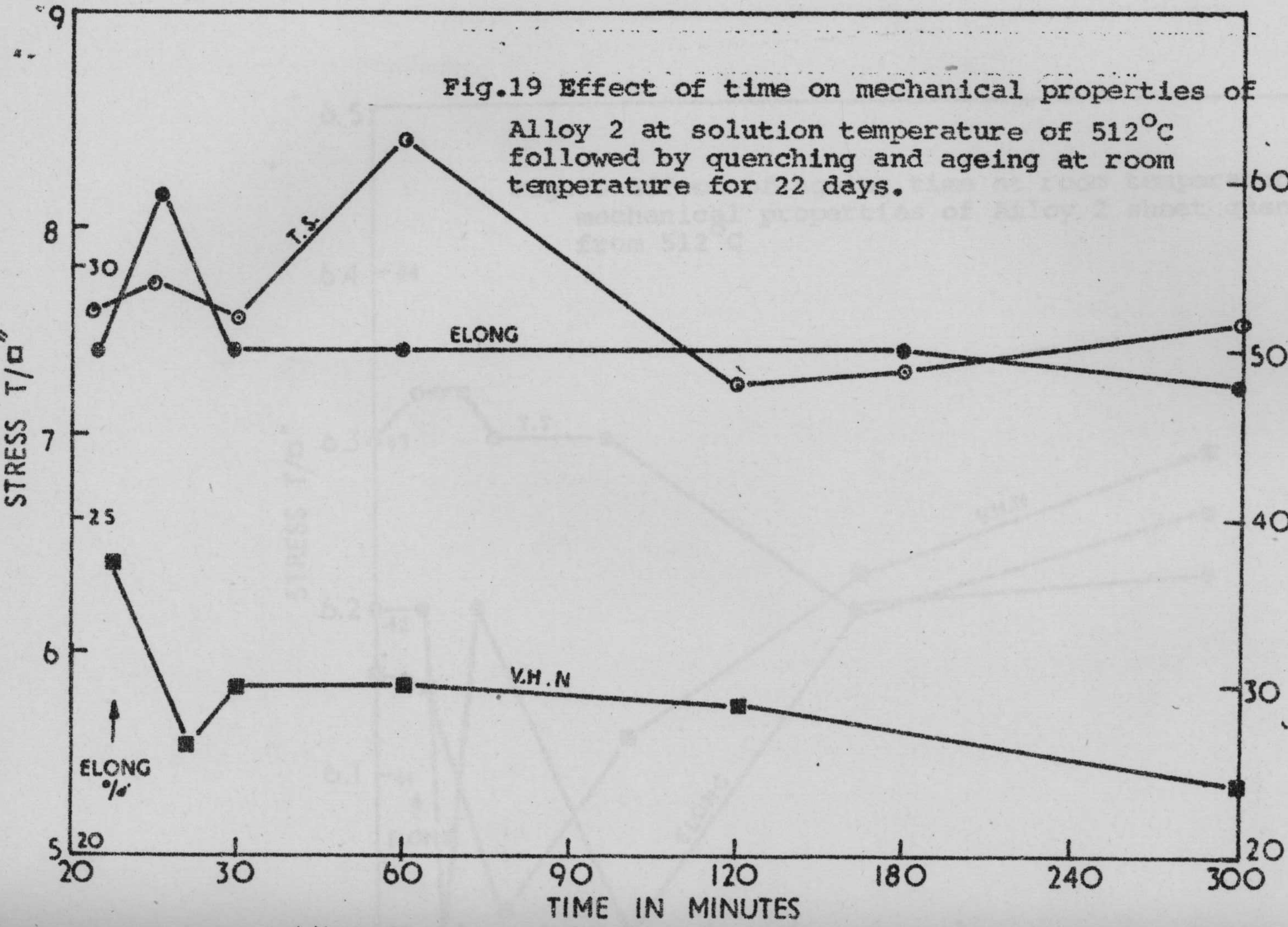


Fig.19 Effect of time on mechanical properties of Alloy 2 at solution temperature of 512°C followed by quenching and ageing at room temperature for 22 days.

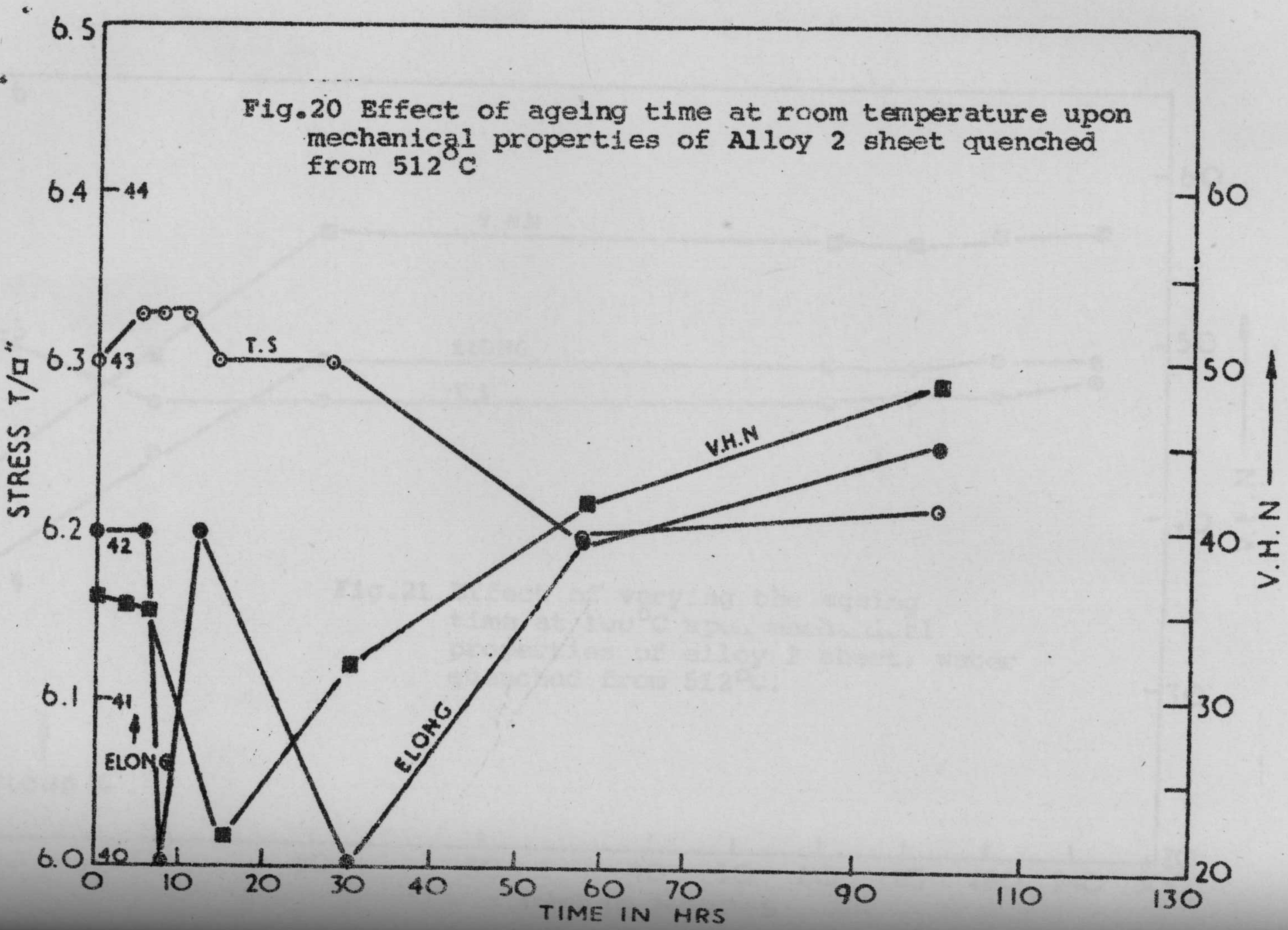


Fig.20 Effect of ageing time at room temperature upon mechanical properties of Alloy 2 sheet quenched from 512°C

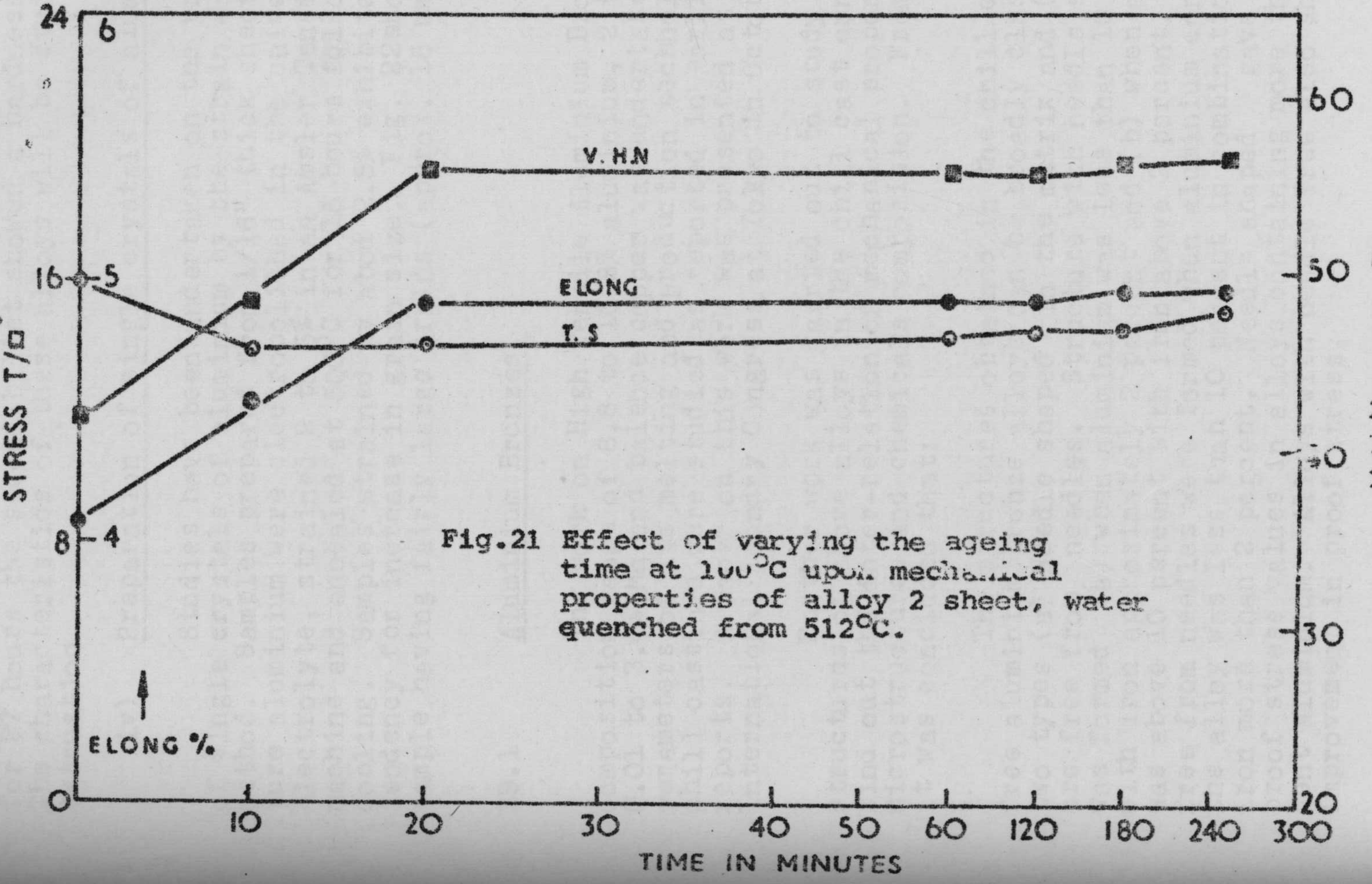


Fig.21 Effect of varying the ageing time at 100°C upon mechanical properties of alloy 2 sheet, water quenched from 512°C.

The billets had an as cast average hardness of I 79 BHN and II 70 BHN, and after homogenisation at 460°C for 27 hours the second billet showed a hardness of 55 BHN. The characteristics of these alloys will be studied after extrustion.

iv) Preparation of single crystals of aluminium:

Studies have been undertaken on the preparation of single crystals of aluminium by the strain - anneal method. Samples prepared from 1/16" thick sheet of super pure aluminium were electropolished in the universal electrolyte, strained 2 to 5% in an Amsler Tensile Testing machine and annealed at 500°C for 15 hours followed by slow cooling. Samples strained by abot 2.5% exhibited marked tendency for increase in grain size. Fig. 22shows a typical sample having fairly large grains (approx. 15 mm x 5 mm).

39.1 Aluminium Bronzes.

The work on High Tensile Aluminium Bronzes in the composition range of 8.8 to 10.8% aluminium, 2 to 6% iron, 0.01 to 3.4% Mn and balance copper was undertaken and the parameters of its melting and production technology for chill castings were studied as reported in earlier annual reports. A paper on this work was presented at the 35th International Foundry Congress at Tokyo in October, 1968.

Further work was carried out to study the microstructures of above alloys in the chill cast condition to find out the inter-relation of mechanical properties, the microstructures and chemicals composition. From the work it was concluded that:

The structures obtained in the chill cast nickel free aluminium bronze alloys can be broadly classified into two types (a) Needle shaped in the matrix and (b) Structure free from needles. Structure with needle shaped was formed (a) when aluminium was less than 10 percent with iron approximately 2 percent and (b) when aluminium was above 10 percent with iron above 2 percent. Structure free from needles were formed when aluminium content of the alloy was less than 10 percent in combination with iron more than 2 percent. Needle shaped gave high proof stress values in alloys containing more than 10 percent aluminium. Alloys with needle free also showed improvement in proof stress.

39.2 Conductivity of Aluminium and its Alloys.

Earlier work had shown that the PM-2 conductors developed in the laboratory has superior electrical, mechanical and corrosion properties than those obtained in the conductors made from electrical grade aluminium (99.5% purity).

Since the semi-commercial scale working of the ingots was done by the conventional rolling and wire drawing, efforts are now being made to study the behaviour of PM-2 under the Properzi method of working.

40.0 Study on the Indigenous Foundry Moulding Materials.

This project was taken up with a view to assess the moulding characteristics of Indian foundry sands, bonding clays and other binding materials, such as dextrine, resin etc. and to determine their suitability for various types of castings.

Investigations were completed on the following:

Sands:

i) Rajmahal sand (Coarse)

The sand received from Rajmahal quartz sand and kaolin Co. Calcutta was investigated. It was coarse to fine grained and had an A.F.S. Fineness No. 20.57 and 45.66 in the 'as received and washed' states respectively along with clay content of 2.48%. It assayed 97.53% silica, its sintering temperature was above 1350°C.

In view of two sieve distribution high permeability, high refractoriness of the sample, this aand can be used for heavy steel casting jobs.

ii) Moulding characteristics of some sands of U.P:

With a view to assist the foundries located in Uttar Pradesh a short review of the moulding sands available in Uttar Pradesh based on the data collected from the investigations carried out at the National Metallurgical Laboratory, has been presented in this paper.

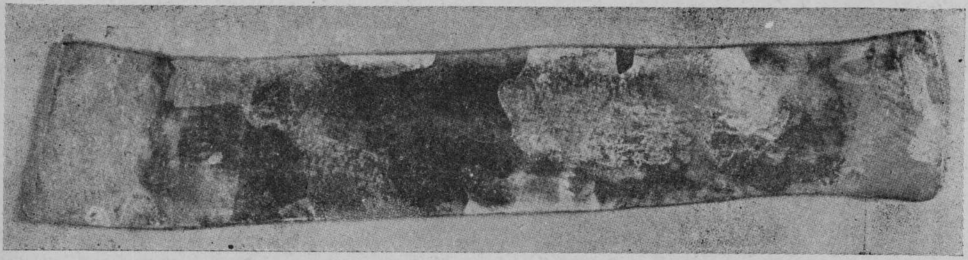


Fig. 22 Photomicrograph showing single crystal grains obtained x 3

Fourteen sand samples were investigated. Based on the data it may be concluded that all the sands reported herein were well graded and the samples were mostly concentrated in three adjacent sieves while a few either in two or four adjacent sieves. The sands are more or less free from A.F.S. Clay. Clay contents were ranging from 1 - 5.16%. The finess contents in these samples were less than 3% for half of the samples and less than 8% for all of them. Except for sand samples number 14 (hardwar River sand) all were highly refractory and as cuh are suitable for making moulds and cores for use in steel foundry. Sample No. 14 would meet the requirement of cast iron and non-ferrous foundries.

It may be added here, that combination of two or more sand sample would be tried to obtain wider range in the moulding properties, specially the strength and flowability. The finer varieties of sand samples may also be used for shell moulding purpose.

iii) Foundry moulding sands in Ratnagiri District of Maharashtra State:

A similar report as in the above case was prepared for the sands available in the Ratnagiri district of the State of Maharashtra. Ten sand samples were investigated. Based on the data it may be concluded that all the sands of Maharashtra except Kanhan I & II may be made use of for moulding jobs in steel foundries for light and medium steel castings. The sands particularly MSS 2 and MSS 3 are non-uniform in grading whereas MSS-6, MSS-7, MSS-8 and Kanhan - I exhibit wide distribution on 5 to 6 sieves. Sands like MSS-4 and 5 are suitable for making cores. Kanhan I and II sands may be used for non-ferrous moulding jobs as well as for some cast iron jobs.

BENTONITES

The following bentonite samples were investigated during this period:

i) Assam Bentonite II:

The sample was received from M/s. Steelsworth Pvt. Ltd., Assam. The sample was of yellow colour and in powder form. The sample had an initial moisture content of 3.36% and pH 8.5. The chemical analysis of the sample showed it to consist of 42% silica and rather a high per-cent of oxides of iron while the total alkali and alkaline earth oxides amounted to 9.0%. Studies on bonding characteristics of the sample showed it to develop maximum green compression strength of 3.2 lbs/sq. inch at 1.98% moisture on addition of 5% of the sample to Rajmahal silica sand. Maximum dry strength obtained was 64 psi at 7.5% moisture. The total base exchange capacity of the sample was 21 miliequivalents for 100 gms. The sintering temperature of the sample was in the range of 900°C - 950°C. Study of the differential thermal curve showed that the sample was not a montmorillonite but an admixture of beidellite with other types of clays.

It may be concluded that the bentonite may be used for bonding purposes in the foundry after mixing with a good quality bentonite.

ii) Kutch bentonite marked 'SC':

The bentonite sample marked 'SC' was received from M/s. Kutch Minerals, Bunder Road, Kutch Mandvi, Gujrat to investigate its suitability as a binding material in the foundries. The sample was in the powdery form having whitish colour and was associated with 9% initial moisture content. It showed gel value, pH and swelling index 12.8, 7.4 and 8.3 respectively. The total base exchange capacity was 75.5 milliequivalents/100 gm. The sodium to calcium ratio was 1.93. The predominant exchangeable ion was sodium. The sample appeared to be containing beidedite. The analysis and D.T.A studies showed the presence of high iron content in the sample. The sintering range of the bentonite sample was 1000°C. The sample contained 46.16% SiO_2, 23.04% Al_2O_3, 10.0 (FeO, Fe_2O_3), 1% TiO_2, 4.45% MgO, 4.15% CaO, Trace K_2O, 1.27% Na_2O, 10.10% L.O.I. The sample mainly consisted of microcrystalline aggregate of clay minerals. Quartz, feldspar and muscovite were presented in small amounts. Bonding characteristics of the bentonite sample was studied by using 5% bentonite with Rajmahal sand of A.F.S. G.F. No. 56.7 as a base sand. Maximum green compression strength value of 12 lb/sq.in. was obtained at a moisture content of 2.02%. Green shear, mould hardness and shatter index values at this moisture content were 3.2 lbs/sq. inch, 80 and 49.12 respectively.

The dry compression and dry shear strength increased with moisture and exhibited maximum value of 95.5 and 29.6 psi respectively at 5.32% moisture. The bentonite can be used for bonding in the iron and steel foundries in the moisture range of 1% to 2.02% when 5% of the sample is used as binder.

iii) Kutch bentonite marked 'MI':

The bentonite sample was received from the above firm. The sample was in the powdery form having yellowish colour. It was associated with 7.6% moisture content which is in agreement with the specification of Steel Founder's Society of America for Western bentonite. It contained 47.8% SiO_2, 21.56% Al_2O_3, 8.8% (Fe_2O_3, FeO), 4.29% MgO, 3.45% CaO, 1.27 Na_2O, 11.13% L.O.I. The ratio of sodium to calcium was 1.06 which showed that the predominent exchangeable ion was sodium. Its pH, gel and swelling index values were found to be 7.7, 32 and 3.1 respectively. The total base exchange capacity was 100.33 milliequivalents/100 gms. The sintering range of the sample was 1100°-1150°C. Petrological examination of the sample revealed that it mainly consisted of microcrystalline aggregate of clay minerals. Feldspar, muscovite, quartz, calcite and chloride were present in small amounts. The bonding characteristics studies by using Rajmahal high silica sand of AFS GF, No. 56.7 bonded with 5% addition of bentonite showed satisfactory green and dry strength properties. It developed a maximum green strength of 13.1 psi at 5.23% moisture. The sample appears to contain essentially the minerals of montmorillonite group. DTA studies showed that the sample contained nontronite and saponite mixed with montmorillonite. The sample also contained oxides of iron in an appreciable amount. It may be concluded that the sample investigated with a fairly good gel value, can be used for bonding in the iron and steel foundries in the moisture range of 1.62% to 2.24% when 5% of the binder is used.

40.1 RESIN.

A sample of solid resin in the powder form was received from M/s. India Cements Ltd., Foundry Division, Madras to carry out tests and find out its suitability in shell moulding.

The tests were carried out as per tentative specification laid down by American Foundrymen's society of America. The tests revealed that the resin was soluble in 80% alcohol. It developed about 500 lbs/sq.in. tensile strength when curved at 500°C for a time of 3 minutes. However, the resin could be cured at lower temperatures also, provided correct curing time is allowed for the development of strength. The resin was quite stable and did not deteriorate on prolonged curing. Its melt point and thus, the curing temperature seem to be on the higher side.

The resin appears to be an useful binder in the shell moulding foundry. The use of accelators, such as, "hexa" etc. may help to cut down its curing time, as well as, increase the strength of the shell.

40.2 Evaluation of Different grades of Dextrines as Foundry Bonding Materials.

Dextrine belongs to a class of cereal binders and is commonly used as a binder in the foundry moulding sands. It is manufactured by the degradation of starch obtained from products like corn, topice, potato etc. in their dry state by the application of heat and chemicals. Most commonly used chemical are the mineral acids as weak aquas solutions sprayed over dried starch. The acidified starch is then subjected to roasting process. Different properties in dextrine is obtained by varying the amount of acid, the roasting temperature and the length of exposure to heat. In this way the dextrine is obtainable with different degrees of water solubility, the highest water solubility being in the neighbourhood of 95%. This type of dextrine imparts high hardness to the outer skin of the core and a soft centre, whereas the dextrine having low water solubility of about 10% gives uniform hardness throughout the core cross-section. Dextrine when properly used, contribute many desirable properties some of which are given below:

i) Maximum surface hardening of core on air or torch drying.

ii) Improves green deformation, green compression, dry shear and tensile properties.

iii) Minimizes drying time of cores and thereby increases the product and the capacity of the oven.

iv) Burns out fast and completely, hence, easy clean out minimum build up of fines and collapsible cores.

v) Due to the high solubility, the dextrine migrates to the outer surface with the moisture of the core and builds up a smooth hard exterior surface, which resist metal penetration and results in cleaner and better castings.

vi) Dextrine gives the ability to cores to retain moisture that are not to be poured immediately.

vii) It combines with other binders, such as, linseed oil, bentonite and resins and thus it is possible to obtain a range of properties to suit the foundry purpose.

A project has been taken up to test the various grades of dextrines with a view to their uses as foundry bonding materials specifying their individual properties. Under this project three samples of dextrines were already tested while work is under progress on two more dextrine samples.

Tests are being conducted as per I.S.I. Draft Specification IS/DOC SMDC 17/(832)F, August 1967 and these comprise of: (i) Fineness (ii) Initial moisture content (iii) Ash content (iv) Water soluble content (v) Air set strength with 2% mixture at 1.5% moisture content.

In addition to the above, gas content of the dextrine samples were also determined and bonding characteristics were studied with dextrine additions ranging from 0.5 to 2 percent at various workable moisture contents. Casting characteristics were also observed by making cores of the same composition, as used for the above tests.

40.3 Castable Sand Project.

Castable sand moulding is yet another addition to the existing various automatic moulding processes. The process uses as binders calcium disilicate and sodium silicate. However, a wetting agent is required which helps to make the sand mixture flow as a liquid of low viscosity with uniformity in density of the sand particles at any point.

Success was achieved to a large extent in formulating the sand mixture having consistency in properties. Another difficulty which was being faced was removal of core from the pattern as the sand mixture tended to stick to them. This difficulty was partly solved by using some emulsified mixtures. However, certain liquid sprays were tried and now it is possible to remove the core very cleanly after 5 minutes. Nevertheless, green strength was found to be very small and removal of core in such a short time as five minutes, although possible, is prohibitory. Work is under progress, therefore, to develop the following:

i) Green strength, so that the cores may be removed out of patterns in much shorter time and allowed to pick-up dry strength in air.

ii) Combination of binders which might produce a much harder outer surface of the core.

iii) Improved method of mixing the various ingredients.

iv) Deformer of better quality.

41.0 Development of Wear and Abrasion Resistant Cast Iron.

The project was taken up at the instance of Tata Locomotive & Engineering Co., Jamshedpur, who had been importing the impeller blade castings for their shot blasting fetlling machines. The object is to develop suitable composition and heat treatment technique for producing wear and abrasion resistant cast iron suitable for such machines. The basic requirements of impeller blades is specified in terms of hardness, and in importing blades it had been found as Rc 66; but hardness alone does not qualify to give satisfactory castings unless a proper service life is obtained.

To improve the service life impeller blade castings were made with different molybdenum and chromium contents in addition to the high chromium variety mentioned in the earlier report. These blades were subjected to various hardening and tempering treatment inclusive of sub-zero treatment. The blades are subjected to service trials. Further work is in progress.

42.0 Appraisal of Raw-materials for Iron Making.

i) Physical and chemical characteristics of iron ore:

The reducibility of ore exerts profound influence on the smelting parameters. In continuation of the investigation, the reducibility of goethite variety and compact laterite variety of Madhya Pradesh iron ores were determined with hydrogen gas as reductant by comparison with the Orissa mineral iron ore. In the case of Orissa mineral iron ore, the influence of reaction temperature on reducibility was studied at 600°C, 700°C, 800°C and 900°C with variation in grain size of the ore -25 +13, -13 +6 and -6 +3 mm while for the Madhya Pradesh

Iron ore and size in mm.	State	At 900°C 800 cc/min gas flow		At 800°C 800 cc/min gas flow		At 700° C 800 cc/min gas flow		At 600°C 800 cc/min gas flow	
		% Reduction.	Time in min.	% Reduction.	Time in min.	% Reduction.	Time in min.	% Reduction.	Time in min.
Orissa mineral iron ore -25 +13	Orissa	90.00	36	83.5	42	85.7	52	84.0	58
Orissa mineral -6 +3	Orissa	91.74	32	87.3	36	85.7	40	85.7	44
Orissa mineral -6 +3	Orissa	92.1	30	89.0	34	86.0	34	85.0	40
Geothite variety Madhya Pradesh -13 +6	Kanhwara M.P.	77.6	50	76.85	65	--	--	--	--
Compact laterite Madhya Pradesh -13 +6	Kanhwara M.P.	77.0	45	75.5	75	--	--	--	--

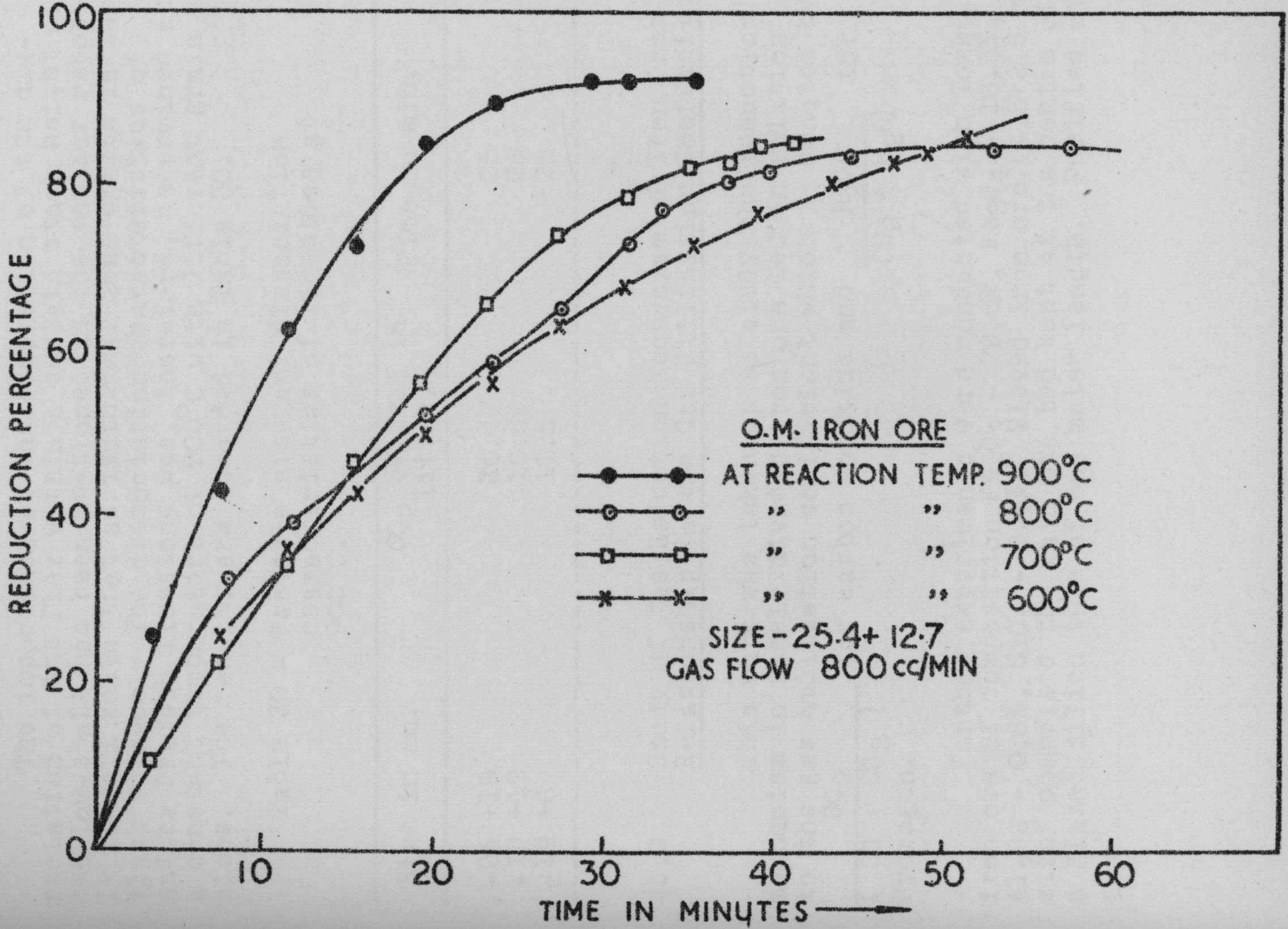


Fig.23 Effect of temperature on reducibility

iron ores, the reduction was conducted at 800°C and 900°C. The gas flow was maintained at 800 cc/min. for all the experiments. The results are recorded in Table 29. It was observed that the extent of reduction generally decreased with decrease in reaction temperature as shown in the Fig. 23, but it is considered that there is an optimum temperature. Reducibility curves for Madhya Pradesh ore are shown in Fig. 24.

ii) Dissociation Characteristics of limestone:

The importance of the completion of the dissociation of the flux within a certain stack height of the low-shaft has been mentioned in the earlier reports. It is well known that as isothermal zone exists in the blast furnace. The dissociation characteristics of Andhra Pradesh limestone was, therefore, determined at a constant temperature of 900°C with different grain sizes. The results are recorded in Table 30.

Table 30 - Particle size and dissociation characteristics of limestone at 900°C.

Size in mm.	CO_2 evolved in litres.	Time in min.
-25 +19	10.47	25
-19 +12	10.64	25
-12 +6	11.5	25

43.0 Study of the Reduction Mechanism of Iron Ores in Relationship to the Gas Utilization Coefficient.

This work was taken up to study the reduction mechanism in finely divided iron ore bed in relation to the gas utilization coefficient which is denoted by $\frac{CO_2}{(CO + CO_2)}$ for carbon monoxide and $\frac{H_2O}{(H_2 + H_2O)}$ for Hydrogen.

Further experiments were conducted with Tomka iron ore of composition Fe_2O_3 - 79.6%, Fe_3O_4 - 18.5%, Al_2O_3 - 0.6%, SiO_2 - 0.7%. Sieved iron ore (4.5+2.5 mm size) comprised the main ore bed kept at the centre of a glazed silica tube of one meter length. Purified and measured volume of carbon monoxide (500 cc/min. at N.T.P.) was passed through the ore bed kept at a temperature of 950° ± 10°C. Series of experiments with Tomka iron ore were carried out to study the effect of the ore weight on carbon monoxide utilization coefficient. Ore weights were varied from 12.5 to 100 gm. Fig. shows the variation of carbon monoxide utilization coefficient with the weight of the ore. It was observed that as the ore weight increases from 12.5 to 100 gm. the carbon monoxide utilization coefficient increases. Further work is in progress with different iron ores following the same procedure.

45.0 Study of the Reduction Mechanism of Iron Oxide by Solid Carbon.

The reduction of iron ore can be obtained by carbon by direct or indirect reactions (The direct reduction means the reduction of iron oxides by carbon and indirect means reduction by carbon monoxide). In a blast furnace both the reactions take place but which should be more predominant over the other could not be dogmatically established as yet. Maintenance of 100% of either modes of reactions, has got its own advantages and disadvantages in blast furnace. A great deal of experimental work has been done and various theories have been advanced on the different aspects of gaseous reduction or indirect reduction but relatively little has been confirmed for direct reduction (solid-solid reduction). It is with this in view the investigation was taken up to study the interactions of carbon and iron oxides occuring by the two known mechanisms and also to compare the reduction mechanism when ore bed is heated with coke/carbon along with small amounts of carbon monoxide.

Experiments were carried out to study the reduction mechanism of iron oxides by using Barajamda and Barbil iron ores with graphite, both crushed to 8 to 10 mesh size. 8.5 gm of iron ore and 5.5 gm of graphite were mixed and kept inside the tube at a temperature of 975° ± 15°C in a horizontal tubular furnace. Purified nitrogen gas was passed down the outer tube and brought back through the charge. The carbon monoxide formed during reduction was measured by separating and then converting it to carbon dioxide. Ratio of iron ore and graphite was varied for the same combination to observe the rate and degree of reduction. The sweeping rate of nitrogen was also varied to observe its effect

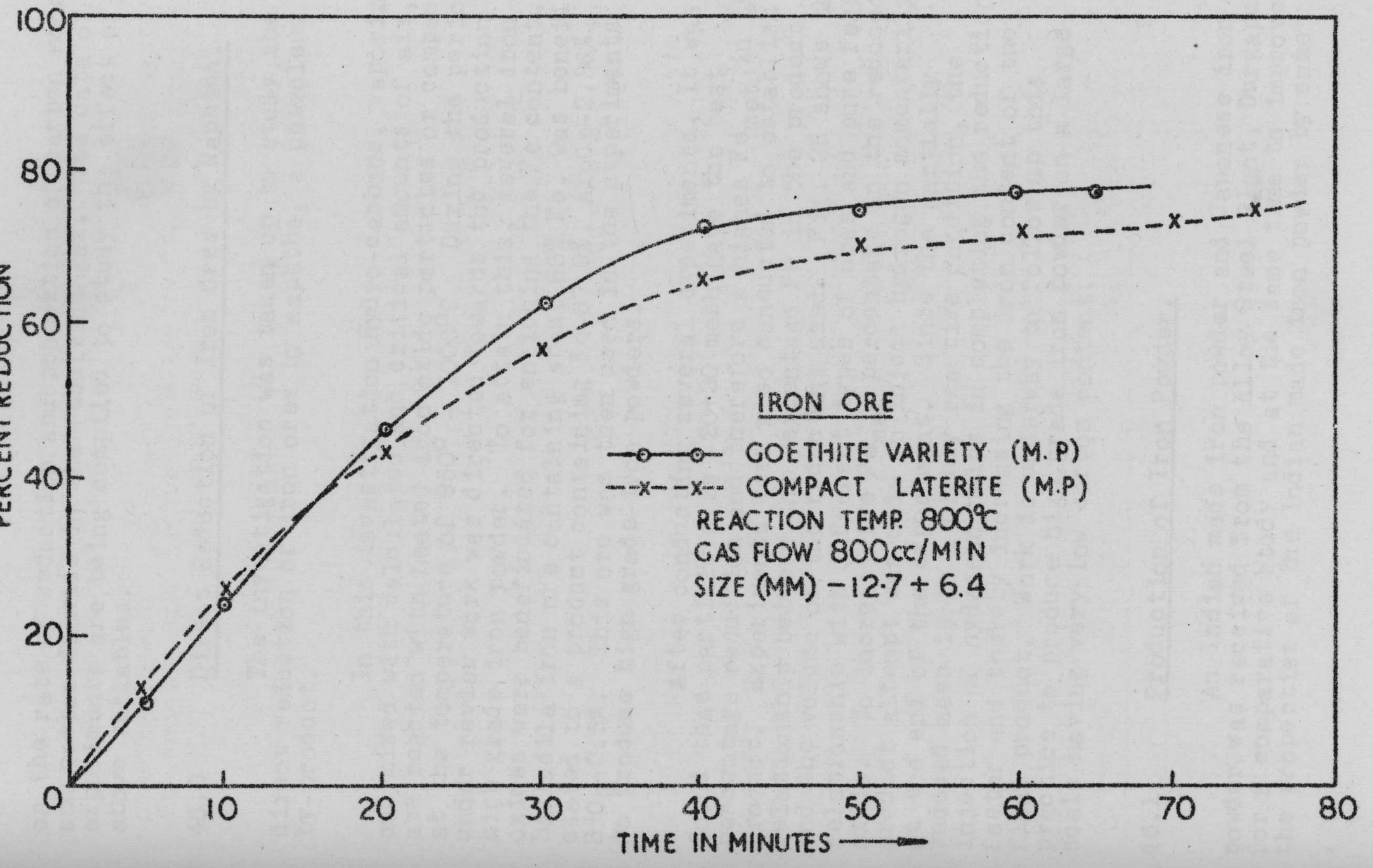


Fig.24 - Reducibility characteristics of two varieties of iron ore from Madhya Pradesh

on the rate of reduction and proportion of carbon monoxide and carbon dioxide in the resultant gas. The data of these experiments are being compiled to study the effect of the above variables.

46.0 Direct Reduction of Iron Ores by Naphtha.

The investigation was taken up to study the direct reduction of iron ores by naphtha, a petroleum by-product.

In this investigation hydro-carbons, vaporized and mixed with calculated and critical amounts of air, are reacted with heated iron oxide particles or compacts at the temperature of $950^o - 1000^oC$. During the period under review work was directed towards the production of high grade iron powder. To attain this, several iron oxides were beneficiated for enriching the Fe content. Bailadila iron ore containing about 66% Fe, was beneficiated to a product containing Fe-67.9%, Al_2O_3-2.36%, SiO_2-0.5%. This ore was then used in the experiments to produce high grade iron powders.

After conducting several experiments, it was found that particle size -20+30 mesh gave the best percentage reduction and therefore a higher Fe_{met} in the product. Experiments were also conducted to establish the relationship between the percentage Fe in the product and the volume of the reductant used. Fig. 25 shows this relationship with different types of ores and pure ferric oxide. To increase the Fe_{met} percentage in the reduced product attempt was made to inject hydrogen momentarily at the end of the experiment. Since the partially reduced mass is in a highly reactive condition, the injection of hydrogen helps in completing the reduction faster and thereby increasing the iron content of the final product. Work is underway to follow up this practice to produce high grade iron powder on a large scale having very low carbon content.

46.1 Production of Iron Powder.

An Indian made iron powder and Japanese iron powder was received from the Alloy Steel Plant, Durgapur for a comparative study and at the same time to improve the properties of the Indian made iron powder by some treatment. It was concluded from the tests that the Indian made iron powder was very inferior compared to the Japanese powder. Hydrogen annealing of the iron powder was carried out with a view to improve its metallic iron content and flowability. Though hydrogen annealing improved its flowability but iron content of the treated powder was found to be very low.

11 kg of iron powder made sent to Alloy Steel Plant, Durgapur for their trials, which were highly successful.

Iron ore containing small percentage of molasses and moisture were pressed into brick form of 4.5" x 2" x 1" at different pressures. These were then reduced at 1100°C in presence of coke breeze. The effect of pressure on the density of the powder produced was not very encouraging. Now it is planned to pre-sinter the pressed iron ore bricks at different temperatures to improve the density of the iron powder.

47.0 Determination of Porosity of Iron Ores and Sinters.

It is well known that porosity of iron ores and sinter plays an important role in their reduction in a blast furnace. Higher the porosity, higher is the rate of reduction due to increased surface area. It is with this in view an apparatus was fabricated for the determination of macro and micro porosities of iron ores and sinters, collected from various ore occurrences in India. With this apparatus, the porosities of 11 more iron ores were determined. Table 31 shows the macro and micro porosity values of various iron ores, crushed to -3+4 mesh size and heated to 125°C to remove moisture prior to porosity measurement. Further work is in progress.

Table 31 - Macro & Micro Porosities of Indian Iron Ores.

Sl. No.	Name of the iron ore.	Density in gm/cc.	Macro Porosity %	Micro Porosity %
1.	Bayaram	4.14	1.1	12.6
2.	Barbil II	4.04	1.3	16.6
3.	Bailadilla	5.01	0.61	6.4
4.	Rajasthan	3.53	0.63	2.23
5.	Barbil III	4.27	0.91	15.55

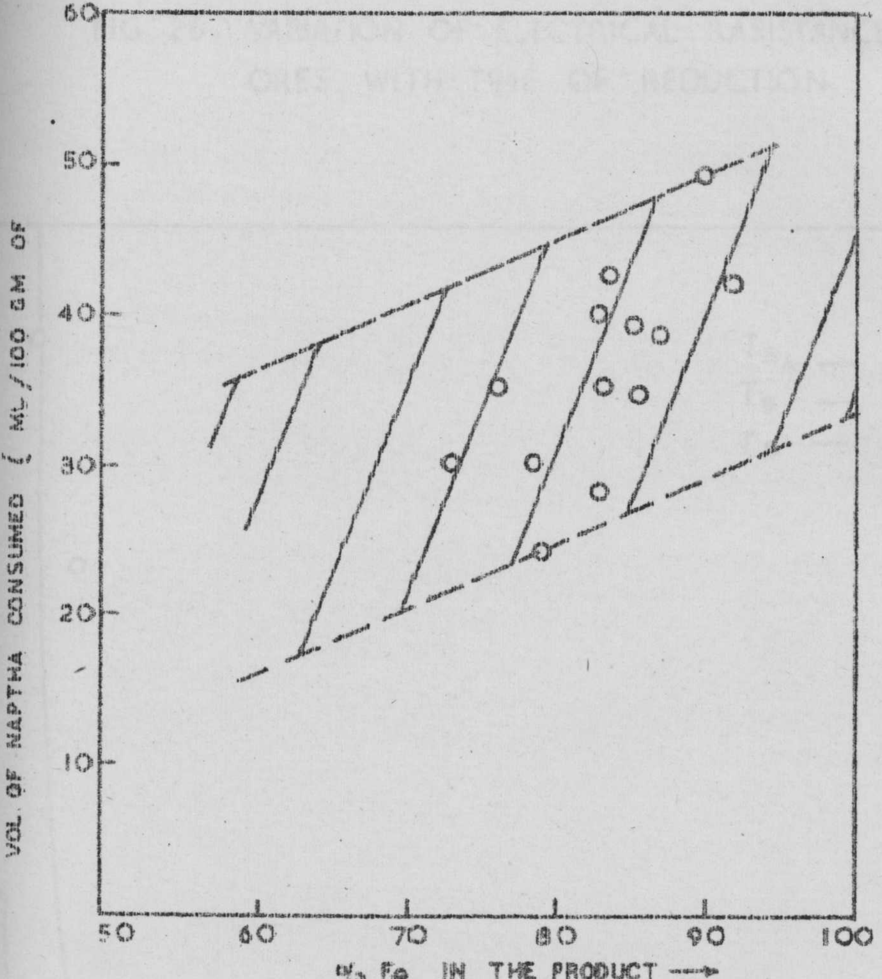
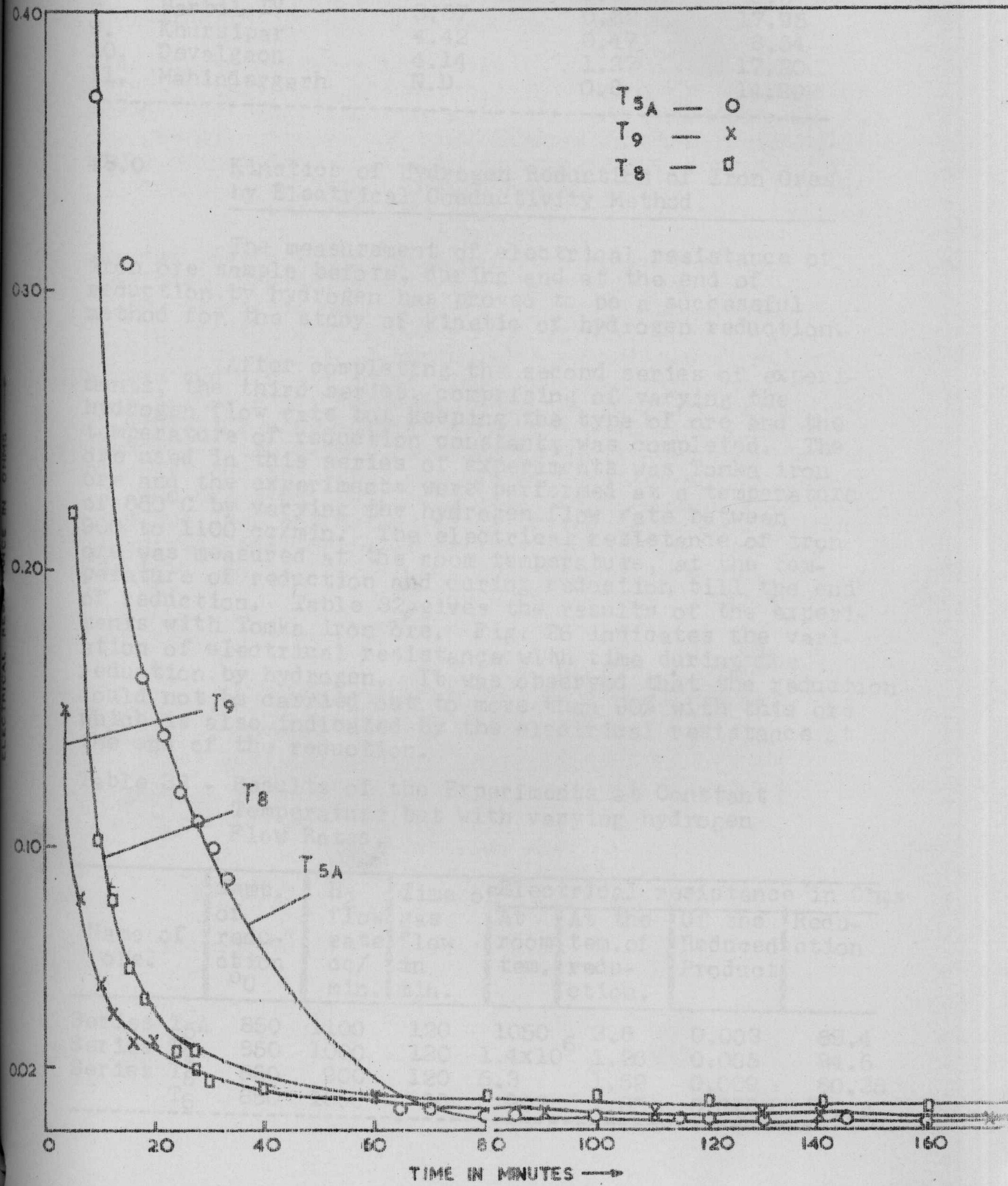


FIG: 25. RELATIONSHIP BETWEEN THE REDUCTANT USED Vs Fe IN THE PRODUCT

FIG: 26. VARIATION OF ELECTRICAL RASISTANCE OF IRON ORES WITH TIME OF REDUCTION.



6.	Kiriburu	4.14	0.92	10.51
7.	Madras	3.61	0.47	6.0
8.	Barbil IV	3.87	0.62	17.95
9.	Khursipar	4.42	0.47	8.54
10.	Devalgaon	4.14	1.27	17.20
11.	Mahindergarh	N.D.	0.8	14.20

48.0 Kinetics of Hydrogen Reduction of Iron Ores by Electrical Conductivity Method.

The measurement of electrical resistance of iron ore sample before, during and at the end of reduction by hydrogen has proved to be a successful method for the study of kinetic of hydrogen reduction.

After completing the second series of experiments, the third series, comprising of varying the hydrogen flow rate but keeping the type of ore and the temperature of reduction constant, was completed. The ore used in this series of experiments was Tomka iron ore and the experiments were performed at a temperature of 850°C by varying the hydrogen flow rate between 900 to 1100 cc/min. The electrical resistance of iron ore was measured at the room temperature, at the temperature of reduction and during reduction till the end of reduction. Table 32 gives the results of the experiments with Tomka iron ore. Fig. 26 indicates the variation of electrical resistance with time during the reduction by hydrogen. It was observed that the reduction could not be carried out to more than 90% with this ore which is also indicated by the electrical resistance at the end of the reduction.

Table 32 - Results of the Experiments at Constant Temperature but with varying hydrogen Flow Rates.

Name of ore.	Temp. of reduction °C	H_2 flow rate cc/min.	Time of gas flow in min.	Electrical resistance in Ohms			
				At room tem.	At the tem. of reduction.	Of the Reduced Product	Reduction
Series T_5A	850	1100	120	1050	3.8	0.003	89.4
Series T_9	850	1000	120	1.4×10^6	1.26	0.005	84.5
Series T_8	850	900	120	5.3	1.52	0.009	80.35
T_6	850	1000	60	902	1.85	0.010	74.8

49.0 Influence of Associated Ingredients on the Mechanism of Gaseous Reduction of Hematite

The kinetics of gas-solid reducing reactions decides the efficiency of iron smelting in blast furnace. It is considered that the minerals associated with hematite may significantly influence the reaction rate. A comprehensive investigation has been undertaken to study the effects of various associated ingredients on the mechanism of gaseous reduction of hematite.

In view of wide use of fluxed sinters, experiments were carried out to study the effects of calcium carbonate on the mechanism of reduction of ferric oxide pellets by reduction at 600° - 900°C in a current of hydrogen by continuous determination of loss in weight. Initially the reduction of pure Fe_2O_3 pellets was studied. The sample being of spherical in shape the reducing gas completely envelopes the outer surface, and therefore the reaction interface advances at a more or less constant rate. At the initial stages, the radius of the unreacted core decreases linearly with time, but there are the deviations from linearity as the unreduced core decreases in diametar. Assuming that the reaction takes place only at the interface of the pellets, the following equation holds good:

$$\frac{dw}{dt} = K.A. \quad \ldots\ldots\ldots\ldots \quad (1)$$

where W = wt. of oxide reacted

t = time (in mins.)

A = Reacting interfacial area (cm^2)

K = Constant, (a function of composition of pellets, temperature and the shape of the pellets)

The fraction of the pellet reduced, f

$$f = \frac{Ro - R}{Ro}$$ --- Where R = rad. of reduced <u>oxide</u> pellets

Ro = initial rad. of oxide pellets.

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K = Constant, (a function of composition of pellets, temperature and the shape of the pellets)

The fraction of the pellet reduced, f

$$f = \frac{Ro - R}{Ro}$$ --- Where R = rad. of reduced <u>oxide</u> pellets

Ro = initial rad. of oxide pellets.

Fig.27 Dependence of rate of reduction on the temperature, time and composition of the pellet

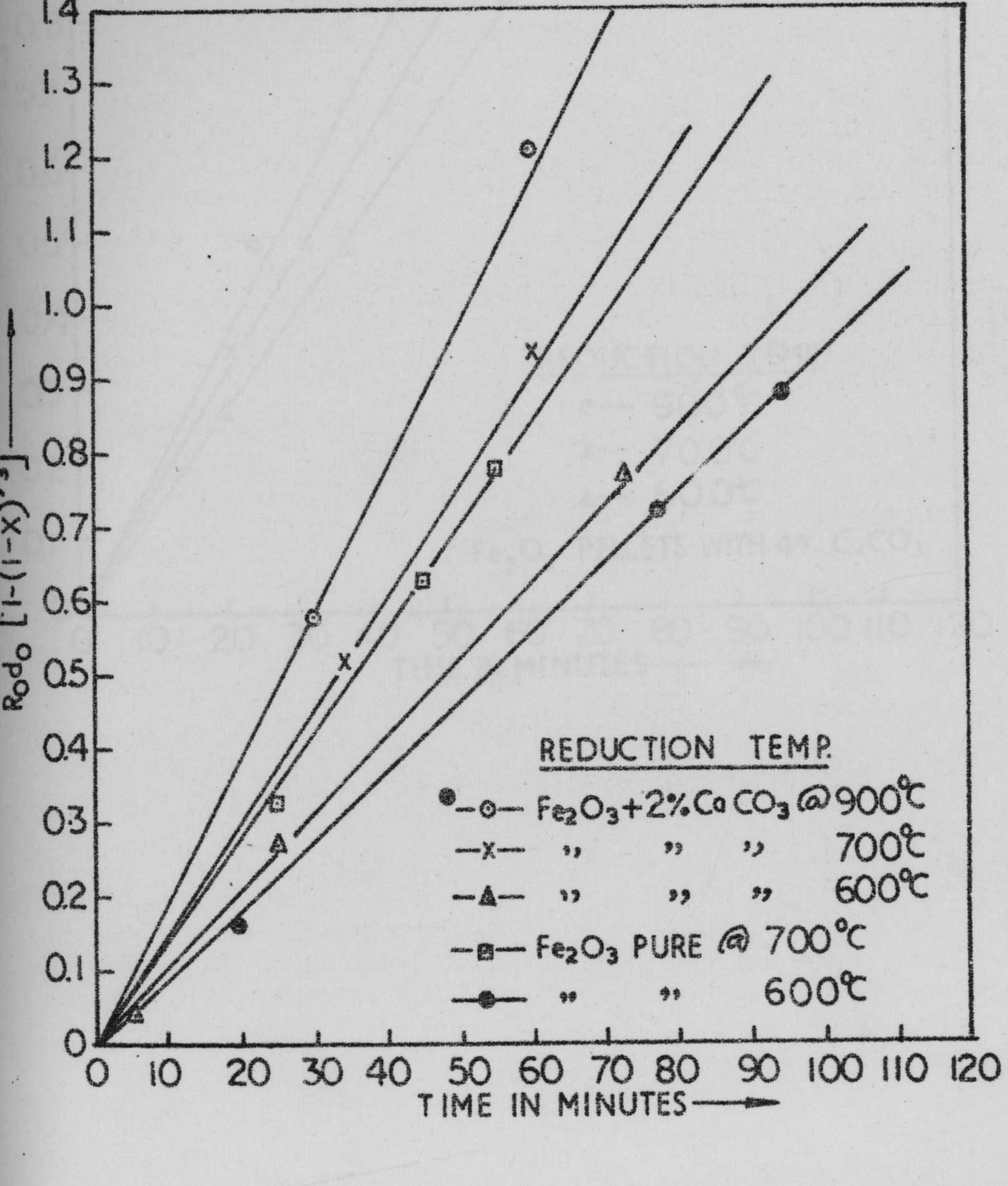
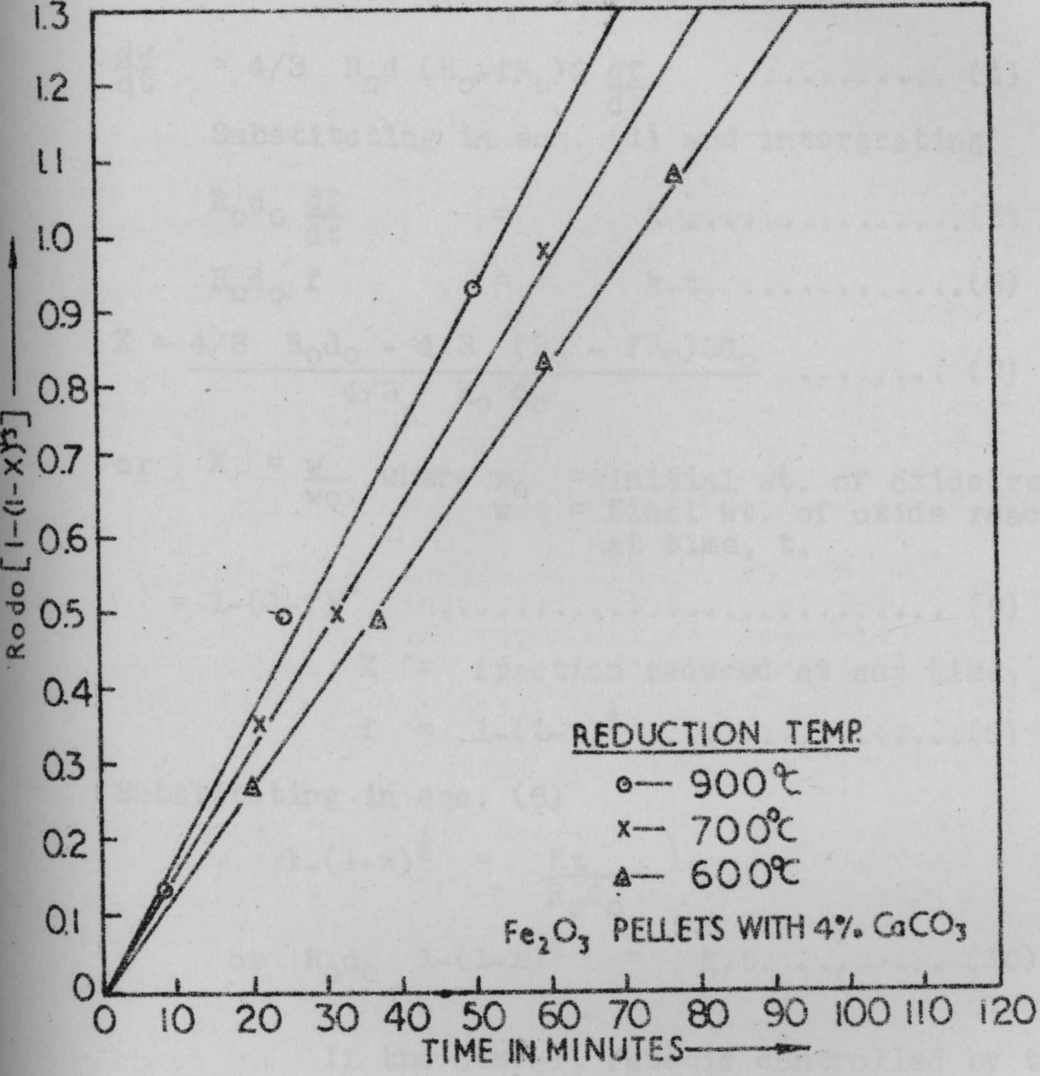


Fig.28 Progress of reduction with time and temperature of Fe_2O_3 pellets containing 4% $CaCO_3$



$$A = 4\ (R_o - fR_o) \quad \ldots\ldots\ldots\ldots (2)$$

Weight oxide reacted $w = 4/3\ R_o{}^3 d_o - 4/3\ (R_o - fR_o)3d_o \ldots(3)$

where do = density of pellet.

$$\frac{dw}{dt} = 4/3\ R_o d\ (R_o - fR_o)2\ \frac{df}{dt} \quad \ldots\ldots\ldots (4)$$

Substituting in eqn. (1) and intergrating

$$R_o d_o \frac{df}{dt} = K \quad \ldots\ldots\ldots\ldots\ldots (5)$$

$$R_o d_o f = k.t. \quad \ldots\ldots\ldots\ldots (6)$$

$$X = \frac{4/3\ R_o d_o - 4/3\ (R_o - fR_o)3d_o}{4/3\ R_o d_o} \quad \ldots\ldots\ldots (7)$$

or $X = \frac{w}{wo}$ where w_o = initial wt. of oxide reacted
w = final wt. of oxide reached at time, t.

$$= 1-(1-f)^3 \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (8)$$

X = fraction reduced at any time, t.

$$f = 1-(1-x)^{\frac{1}{3}} \quad \ldots\ldots\ldots\ldots\ldots\ldots (9)$$

Substituting in eqn. (6)

$$1.(1-x)^{\frac{1}{3}} = \frac{Kt}{R_o d_o}$$

$$\text{or} \quad R_o d_o\ 1-(1-x)^{\frac{1}{3}} = k.t. \quad \ldots\ldots\ldots (10)$$

If the overall rate is controlled by the rate of the surface reaction at the Wustite iron interface, then a plot of $R_o d_o\ 1-(1-x)^{\frac{1}{3}}$ vs 't' should yield a straight line, slope of which is equal to the specific reaction rate constant.

Results of the above plot of pure hematite pellets and with the addition of 2%, 4% and 10% calcium carbonate are shown in the Figs. 27, 28 and 29. It was realised that the dissociation of $CaCO_3$ will increase the porosity of the pellets in comparison with pellets without it. However, eliminating the effect of increase in the porosity, difference in reducibilities of pellets with or without $CaCO_3$ will disclose the effect of CaO on reducibility. It was concluded that the CaO has a measurable effect, mechanism of which is being investigated.

50.0 Reactivity of Solid Fuels Employed for Iron Smelting in Low Shaft Furnace.

Reactivity of the fuel greatly influences the gaseous reduction in the stack of the blast furnace and thereby reflects on the fuel consumed. It is of greater significance in a low-shaft furnace as the limited stack height requires fuel of optimum reactivity.

In continuation of earlier work done further experiments were carried out on the reactivity towards CO_2 of fuels employed in iron smelting in low-shaft furnace. The experimental details and conditions for the test were mentioned in the previous report.

The fuel was soaked in normal NaOH solution for about 24 hours, air dried for about 2 hours and then dried in air oven for about 3 hours at 110°C. The fuel was crushed to standard size of -14 + 22 mesh BSS. The temperature of the fuel bed was raised to 950°C within one hour in a current of pure, dry nitrogen and the temperature was maintained for one hour.

Pure CO_2 stored in a gas holder of 200 ml capacity was passed through the fuel bed by displacement with conc. magnesium chloride solution. The first 150 ml. of CO_2 was used for purging. The gases $CO+CO_2$ resulting from the passage of remaining 50 ml. of CO_2 were collected in a nitrometer over conc. caustic potash solution to absorb the unconverted CO_2. The volume of gases collected, mainly consisting of CO, is directly taken as an index of reactivity of the fuel towards CO_2. The results for experiments on pearl coke and Lodna coke are given in Table 33.

Fig.29 Effect of time and temperature on the reduction rate of Fe_2O_3 pellets containing 10% $CaCO_3$

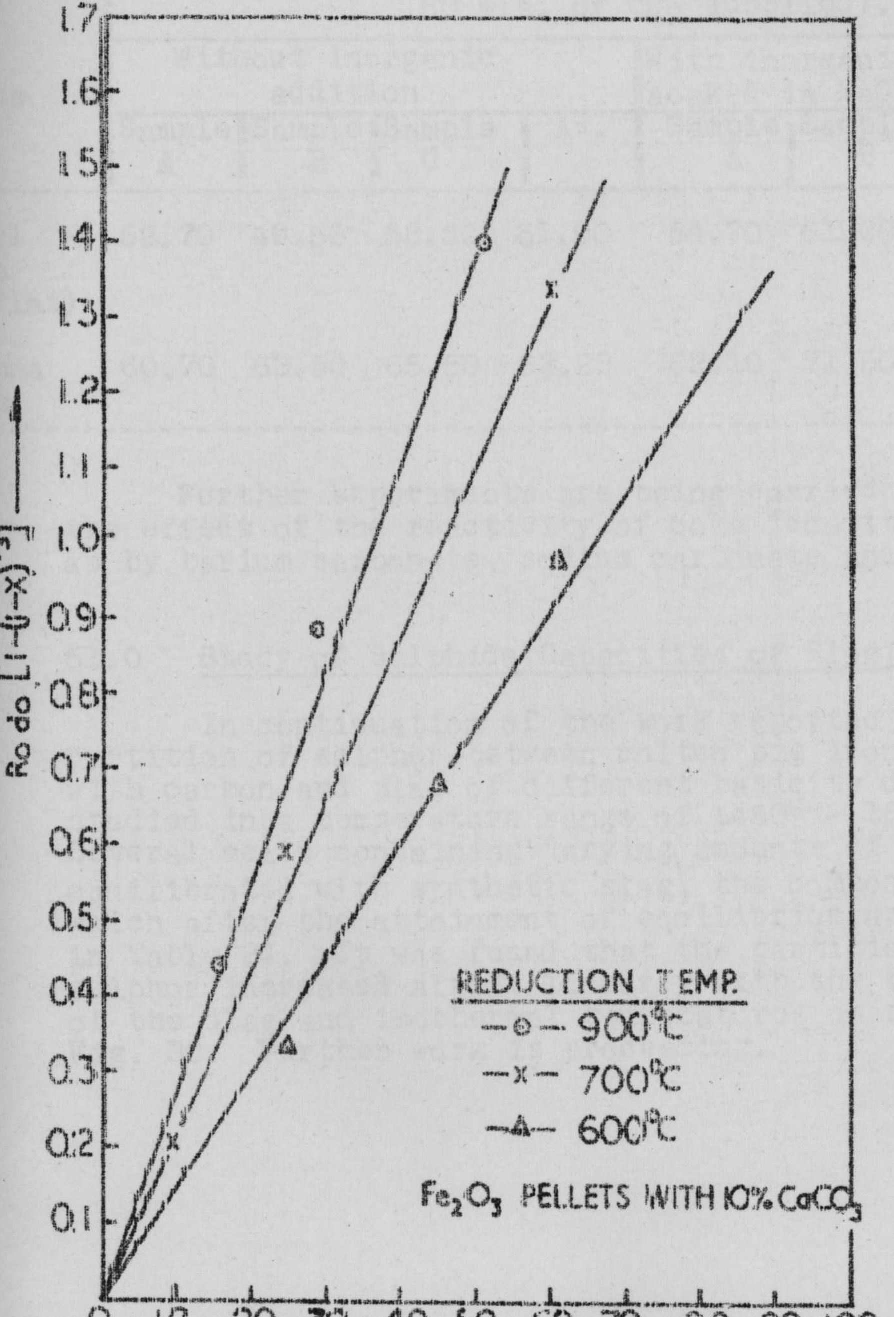


Table 33 - Reactivity of Pearl Coke (Bhilai) and Lodna Coke with Inorganic Addition.

Coke	Reactivity (ml. of CO produced per 50 mls. of CO_2 supplied).							
	Without inorganic addition				With inorganic addition aoaked in NaOH solution			
	Sample A	Sample B	Sample C	Av.	Sample A	Sample B	Sample C	Av.
Pearl coke (Bhilai)	52.70	49.50	53.50	51.90	54.70	61.00	53.50	56.40
Lodhna coke	60.70	63.50	65.50	63.23	62.10	71.50	77.90	70.50

Further experiments are being carried out to study the effect of the reactivity of coke "conditioning" such as by barium carbonate, sodium carbonate and iron oxide.

51.0 Study of Sulphide Capacities of Slag.

In continuation of the work reported earlier, the partition of sulphur between molten pig iron saturated with carbon and slag of different basicity degrees were studied in a temperature range of 1450° - 1500°C. Several melts containing varying amounts of sulphur were equilibrated with synthetic slag, the compositions of which after the attainment of equilibrium are mentioned in Table 34. It was found that the partition of sulphur increased almost linearly with the basicity degree of the slag and isothermal temperatures as shown in Fig. 30. Further work is proceeding.

Table 34 - Equilibrium Sulphur Partition between Carbon Saturated Iron and Slag.

Analysis of iron			Analysis of slag						Basicity degree	Partition coefficient
C %	Si%	S%	CaO%	SiO_2%	MgO%	Al_2O_3%	FeO%	S%	$\frac{CaO+2/3\ MgO}{SiO_2 + Al_2O_3}$	$\frac{(S)}{S}$
3.08	1.80	0.016	39.19	41.63	2.32	16.34	-	0.193	0.703	12.0
3.90	2.09	0.018	31.40	43.10	9.90	9.70	-	0.234	0.720	13.0
4.06	2.08	0.012	48.91	25.60	2.28	13.10	-	0.952	1.3	79.4
4.8	1.27	0.011	46.97	26.96	9.63	13.60	-	0.892	1.32	81.2

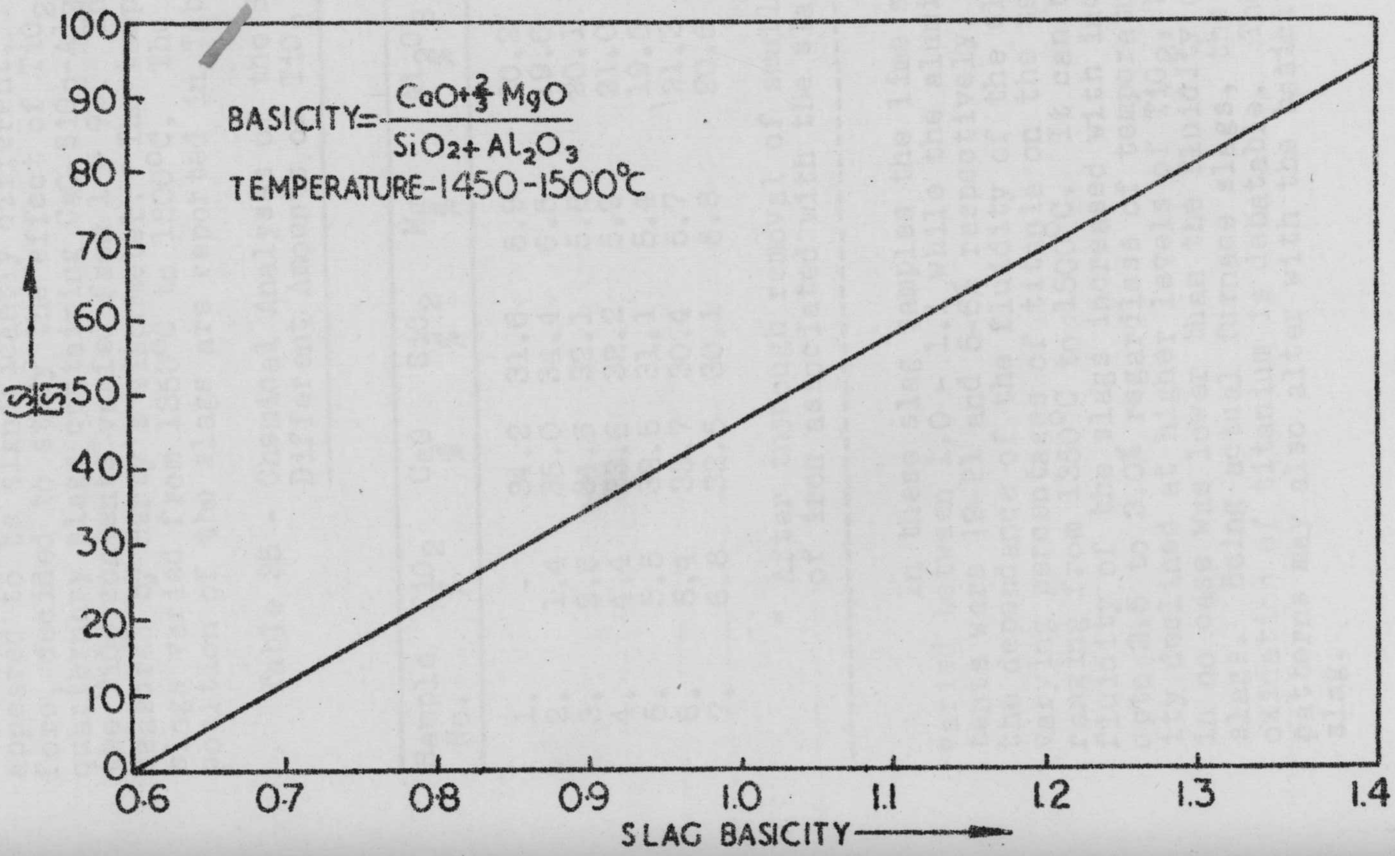


Fig.30 The effect of slag basicity on the effect of sulphur.

52.0 A Comparative Study of the Viscosity of Quarternary Slags Containing Titania.

During smelting of titaniferous ores in low-shaft furnace, the viscosities of slags of different basicity degrees and containing different amounts of TiO_2 therein appeared to be significantly different. It was, therefore, decided to study the effect of TiO_2 on viscosity of quarternary slags containing $CaO-SiO_2-Al_2O_3-MgO$ in which the TiO_2 contents varied from 1 - 7%. The fluidity was measured by Herty's viscometer. The temperature of the slags varied from 1350°C to 1500°C. The chemical composition of the slags are reported in Table 35.

Table 35 - Chemical Analyses of the Slags Containing Different Amounts of TiO_2

Sample No.	TiO_2 %	CaO %	SiO_2 %	MgO %	Al_2O_3 %	FeO* %
1.	-	34.2	31.6	5.9	20.2	1.4
2.	1.4	35.0	34.4	6.5	19.6	1.2
3.	2.6	34.6	32.1	5.8	20.1	0.8
4.	4.4	33.6	32.2	5.9	21.0	0.6
5.	5.5	32.5	31.1	5.4	19.5	0.5
6.	5.4	33.7	30.4	5.7	21.3	0.5
7.	6.8	32.5	30.1	5.8	20.6	0.4

* After thorough removal of small particles of iron associated with the slag.

In these slag samples the lime silica ratio varied between 1.0 - 1.1 while the alumina and MgO contents were 19-21 and 5-6% respectively. Fig. 31 shows the dependance of the fluidity of the slags containing varying percentages of titania on the temperatures ranging from 1350°C to 1500°C. It can be seen that the fluidity of the slags increased with increase in TiO_2 upto 2.5 to 3.0% regardless of temperature. The fluidity declined at higher levels of TiO_2, but the fluidity in no case was lower than the fluidity of titania free slags. Being actual furnace slags, the state of oxidation of titanium is debatable. The fluidity patterns may also alter with the basicity degree of the slag.

53.0 Studies on the Growth and other Properties of Metal Fibres.

It is known that fibre-reinforced composites possess high strength over a wide temperature range. Investigations have been undertaken to develop these new engineering materials. Of all metallic fibres, iron whiskers are known to possess very high strength. It was felt, therefore, that work should be mainly directed on the study of iron whiskers. However, it was felt that other whiskers may also be studied with regard to development of new procedures and techniques and so the work has also been extended to include other types of whiskers.

Studies carried out during the period can be classified under two groups namely (i) growth properties of whiskers, and (ii) mechanical properties of whiskers.

i) The growth properties of whiskers:

Experiments were conducted on the growth properties of copper, iron, silver, cobalt, nickel and platinum whiskers, which were produced by thermal dissociation or chemical decomposition by methods outlined in Table 36.

Table 36 - Preparation of Whiskers by the Thermal Dissociation and Chemical Decomposition of Metal Halides.

Metal	Chemical compound	Method used	Reaction temperature Range.
Platinum	$PtCl_3$	Thermal dissociation	700/900°C
Copper	$CuCl_2$	Chemical reduction	500/850°C
	$CuBr_2$	with H_2/Ar mixtures	500/850°C
	CuI_2		500/850°C
	Cu_2Cl_2		500/850°C
Silver	$AgCl$	"	400/850°C
	AgI	"	400/850°C
	$AgBr$	"	400/850°C
Iron	$FeCl_3$	"	700/800°C
	$FeCl_2$	"	700/800°C
Cobalt	$CoBr_2$	"	600/900°C
Nickel	$NiBr_2$	"	600/900°C

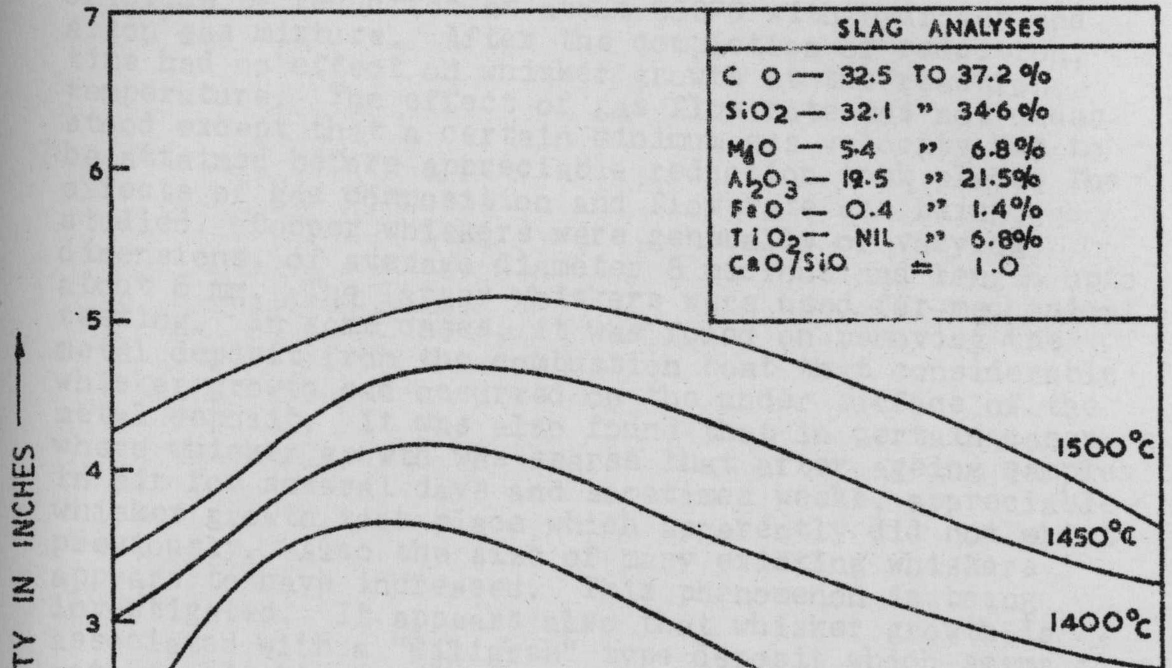


Fig.31 - Effect of TiO_2 on the fluidity of slags at different TiO_2 content.

The optimum temperature for producing platinum whiskers was 750°C. The whiskers were straight and very small, of length about 1 mm, with diameters less than 5 microns. Copper whiskers were produced from cupric chloride by reduction at about 650°C with hydrogen and argon gas mixture. After the completion of reduction, time had no effect on whisker growth at the reaction temperature. The effect of gas flow rate was not understood except that a certain minimum gas velocity had to be attained before appreciable reduction took place. The effects of gas composition and flow rate are being studied. Copper whiskers were generally of varying dimensions, of average diameter 8 microns and length upto about 5 mm. The larger whiskers were used for mechanical testing. In some cases, it was found on removing the metal deposit from the combustion boat that considerable whisker growth had occurred on the under surface of the metal deposit. It was also found that in certain cases where whisker growth was sparse that after ageing samples in air for several days and sometimes weeks, appreciable whisker growth took place which apparently did not exist previously. Also the size of many existing whiskers appears to have increased. This phenomenon is being investigated. It appears also that whisker growth is associated with a "Filigree" type deposit which seems to act as suitable base or nucleation site for whisker growth. At higher temperatures where the 'filigree' type deposit gives way to an "alligator skin" type deposit very little whisker growth took place. The cross section and general morphology of these whiskers will be investigated. Experiments on growth of silver whiskers showed that the optimum temperature of growth of silver whiskers from silver chloride was about 720°C. The whiskers were of comparatively heavy cross sections of the order of about 20 microns diameter and generally of short length. However, there were a few whiskers produced of length of about 3 mm. As in the case of copper whiskers, silver whiskers appear to grow from nucleation sites in silver "Filigree". Ageing of specimens of silver produced by chemical decomposition of silver-chloride did not produce any increase in size of the whiskers grown, nor did new whiskers appear. The chemical decomposition of silver bromide and silver iodide showed very low shisker production. All attempts to grow iron whiskers from ferric chloride under different temperatures have proved futile. Further experiments are being carried out on the thermal decomposition by hydrated and anhydrous ferrous chloride. Experiments on production of cobalt and nickel whiskers by chemical decomposition of coblat and nickel bromide gave no positive results. Further experiments are also to be conducted on the effect of impurities, stresses, catalysts and effect of substrate on the growth properties of whiskers.

ii) Mechanical properties of whiskers:

A small tensile testing apparatus was designed and fabricated for the tensile testing of whiskers. The whisker is mounted between two copper loops which act as grips of a tensile testing machine. The upper loop is held rigid and the lower loop is loaded by means of weights on a small pan attached to it until failure occurs. The diameter of the shisker is measured microscopically. The whisker is attached between the loops by means of shellac which is melted on to the end of the copper loops. The melting characteristics of the shellac are controlled by passing current through the copper loops. Two types of whiskers namely those of copper and silver were tested. Results from copper whiskers were inconclusive. Values of tensile strength of silver whiskers were above the normal tensile strength of cast silver and further tests are being conducted on whiskers of smaller diameter. An instrument suitable for handling of small minute parts, namely a 'micro-manipulator' is being designed and fabricated. Bend tests on whiskers will also be carried out with this instrument. A suitable equipment for accurate measurements of elastic strain is being designed. The future programme of work includes preparation and testing of fibre reinforced composites.

54.0 Structure of Liquid Metals.

i) Aluminium-zinc system:

It was earlier shown that (i) liquid metallic solutions over the entire range of composition in binary eutectic systems may not be homogeneous and stable particularly in presence of characteristic inflection in the liquidus (ii) that liquids outside the composition range of the liquidus inflection on either ends of the binary phase diagrams are stable and consists of colloidal despersion of solute and solvent rich clusters (iii) the essential immiscibility existing in the solid state is extended in

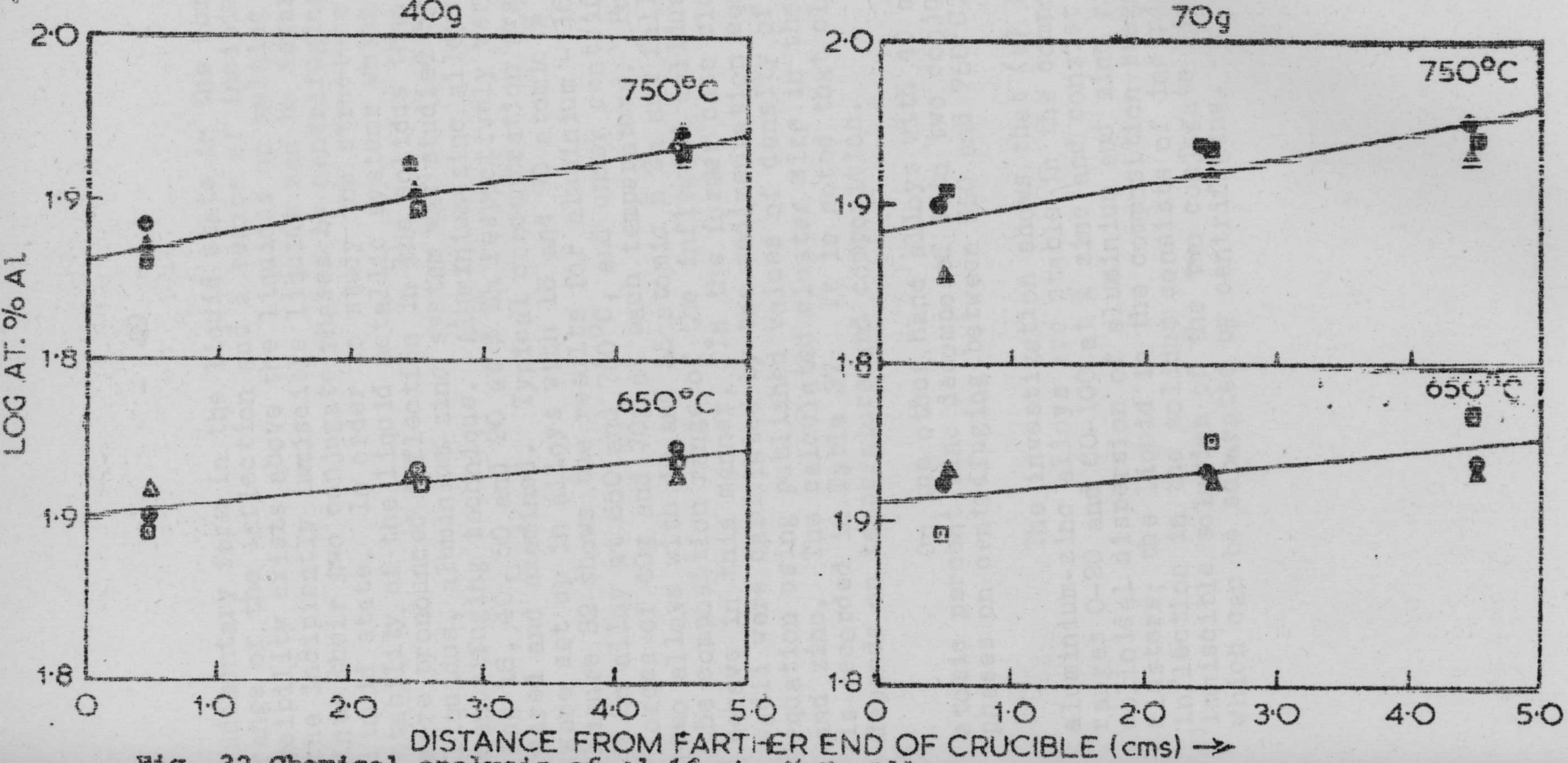


Fig. 32 Chemical analysis of Al-16 at % Zn Alloys

rudimentary form in the liquid state in the composition range of the inflection and a region of incipient immiscibility exists above the liquidus on melting and (iv) the incipiently immiscible liquids can be separated into their two conjugate phases by centrifuging in liquid state. In order to study the structure and stability of the liquid metallic systems which show more pronounced inflection in the solidus than in the liquidus, aluminium-zinc system was studied by the centrifuging technique. Aluminium-zinc alloys containing 16, 40, 50 and 90 at % Zn respectively were prepared and examined. Typical concentration gradients were set up in alloys with 16 and 90 atomic % Zn. Figure 32 shows the results for aluminium - 16 atomic % Zn alloy at 650 and 750°C, and under centrifugal forces of 40g and 70g at each temperature. But the two alloys with 40 and 55 atomic % Zn and falling in the composition range of the inflected solidus did not behave in this manner. In the former case cluster radii were calculated by the sedimentation equilibrium equation using published values of density of aluminium and zinc. The calculated cluster size in these alloys is recorded in Table 37. It is noted that cluster size depends on temperature and composition.

On the other hand alloys with 40 and 50 atomic percent zinc decomposed into two conjugate phases on centrifuging between 550 and 750°C.

The investigation shows that (a) liquid aluminium-zinc alloys are stable in the composition ranges 0-20 and 60-100 at % zinc and consist of colloidal dispersion of aluminium and zinc rich clusters; the liquid in the composition range of the inflection in the solidus consists of incipiently immiscible solution of the two conjugate liquid phases which can be separated by centrifuging.

Table 37 - Cluster size in Al-Zn System

Composition at % Zn	Centrifugal force xg	Temperature °C	Calculated values of cluster (Å) size at mean, minimum and maximum slopes.			
			Al		Zn	
16	40	650	28	22 32	16	12 12
		750	38		21	
	70	650	24	16 29	13	9 16
		750	30	17 37	17	10 20
90	40	450	40	22 47	19	11 23
		550	56	10 70	27	5 34
	70	450	34	30 38	17	15 19
		550	50	27 60	24	14 30

ii) Liquid metals and solidification:

Thin films of aluminium containing 4.8%, 7.4% copper respectively were obtained by the modified splat cooling technique based on centrifuging metals in liquid state. The alloys were drastically cooled from 900°C to -4°C and the films thus obtained were examined metallographically and by transmission and back-reflection x-ray techniques. It was observed that (i) in dendritic crystallization growth occurs along the (III) and (321) directions, (ii) asterism was present but disappeared on annealing at elevated temperatures. During annealing polygonization or sub-grain formation was observed before the on set of general recrystallization. These metallographic observations were also confirmed by x-ray data. Changes in lattice parameter and microhardness of all samples were measured after heat treatment. It was found that the lattice parameter decreased as the annealing temperature was raised initially.

Further work on these alloys as well as on others are in progress.

55.0 Co-efficients of Thermal Expansion of Fe-Al and Fe-Si Alloys.

Co-efficients of thermal expansion of iron-aluminium and iron-silicon alloys were measured in the temperature range of R.T. - 400°C. Their variation with increasing contents of alloying elements (Al-Si) bear close correspondence to the variation of curie temperatures of these alloys, suggesting magnetic interaction to be one of the main factors influencing the thermal expansion co-efficients of these alloys. The results are interpreted in terms of electron theory of transition metals.

56.0 Study of Interaction in Aluminium-Silicon Alloys.

The Al-Si alloys containing 1, 4, 9 and 11 % Si respectively were prepared by casting in metal moulds (2" sq x 10" long). The 1% alloy was contrifuged at 700°C, 500 rpm using graphite covering for different times upto 2 hours. The centrifuging of the 9 % Si alloy is in progress.

57.0 Effect of Titanium in Iron and Steel.

Titanium in steel:

In order to understand the mechanism of precipitation, x-ray analysis was carried out with PM-501 alloy in differently heat treated conditions and the precipitating phases have been identified. Several heats corresponding to the nominal Fe_2Ti and FeTi compositions were also made synthetically in the electron beam melting furnace and x-ray studies were carried out after heat treatment with a view to study the possibility of the decomposition product of $Fe_2Ti = FeTi + Fe$. In view of the high hardness of the heat treatment, attempts are being made to put the steel in service for cutting purposes. Few heats were made for finding the suitability of the alloy as a tool steel for industrial purposes.

58.0 Grain Size Control of Non-ferrous Metals and Alloys.

After completing a basic study of grain size control in magnesium-aluminium alloys, attention was focussed on the mechanism of grain size control in cast aluminium. The solidification behaviour of two Al-Ti alloys containing respectively 0.14 and 0.4% titanium was studied. Heats were made by dissolving appropriate amounts of titanium in molten aluminium in an electric furnace and were cast in metal moulds. Specimens of 35 g each were taken; a set was melted and solidified in refractory and graphite crucibles and another solidified after centrifuging in refractory and graphite crucibles at 700 and 900°C for various times.

The following conclusions were drawn:

i) The structure of liquid aluminium alloys consists of a small fraction of dense clusters suspended in randomized atoms which behave like true liquids.

ii) Technological freezing depends on the presence of either kindred surfaces of the clusters or of the heterogeneous particles.

iii) In Al-4 % Cu alloys, nucleation occurs on the Al-Cu cluster surfaces and experimental observations on the variation of cast grain size in a centrifuged specimen could be explained on the basis of isolation theory of Turubull for the solidification of tiny liquid droplets.

iv) The role of titanium as effective grain refiner was confirmed.

v) Both the primary particles of Al_3Ti and the Al-Ti clusters provide surfaces to nucleate the crystallization of aluminium from the melt.

vi) The marked influence on grain size if the pouring temperature exceeded 750°C in Al-Cu alloys could be explained on the basis of randomization of clusters of Al-Cu.

vii) Presence of TiC in aluminium melts in which titanium was not intentionally added could not be confirmed if the melt was contained in refractory crucibles.

Experiments are in progress for the development of commercial inoculents for aluminium and its alloys.

59.0 Structure and Properties of 9 percent Nickel Steel.

Work on the development of cryogenic steels for use at sub-zero temperature was undertaken with a view to study (i) The mechanism by which the established cryogenic steels (especially nine percent nickel steel) derive their cryogenic properties and (ii) To develop cryogenic steels, based on indigenous alloying elements.

The work on (i) was carried out on nine per cent nickel steel which has gained prominance during recent years as a steel suitable for low-temperature application.

Heats were made in basic lined high frequency induction furnace. The average chemical analysis is shown in Table 38.

Table 38 - Chemical Analysis of Heats.

Carbon %	Silicon %	Manganese %	Nickel %	Phosphorus %	Sulphur %
0.07	0.33	0.55	8.7	0.017	0.03

The specimen blanks were austenitised at 800^{o}C for 1 hour and water quenched. The quenched specimens were tempered at 570^{o}C for 2 to 8 hours. A set of tempered specimens were quenched in liquid air and the rest were water quenched. In the case of austenite micro-hardness for each heat treated condition, average of ten micro-hardness values was taken as the mean micro-hardness value.

Thin foils for electron microscopy were prepared from mechanical ground and polished thin strips which was subsequently chemically thinned. Final thinning was carried out in a modified Bollman's apparatus using perchloric - butyl cello solve electrolyte.

The Fe-Ni-C system with very low concentration of carbon resumbles that of Fe-Ni system. With 9% Ni, the austenite phase is in stable equilibrium with ferrite even at low temperatures. Thus on tempering the quenched 9% Ni-alloy at or above 550^{o}c, considerable proportion of austenite is formed by the reverse martensite transformation. In the ternary system of Fe-Ni-C, the stable carbide is Fe_3C.

Thus with the increase in tempering parameter (tempered for long hours at 570^{o}C upto 25 hours) more austenite was formed and the carbide particles grew leading to gradual decrease in hardness value. One set of tempered specimens were quenched in liquid nitrogen and the other in water. For specimens quenched in liquid nitrogen and the tempering time above 2 hours, the hardness increased with the progress of tempering whereas for the water quenched specimens there was a decrease in hardness with the increase in the time for tempering. The lattice parameter values of the austenite decreased with the increase in tempering time, when tempered at 570^{o}C, (Table 39).

Table 39 - Lattice Parameter of Y and -iron in Heat-treated 9% Ni Steel.

Tempering time at 570°C, in hours.	Lattice parameter of Austenite (kX)	Lattice parameter of Ferrite (kX).
2	3.592	2.8598
4	3.566	2.8559
6	3.550	2.8554
8	3.545	2.8500

The lattice parameter of Y for 2 hours tempering being 3.592 kX decreased to 3.545 kX after 8 hours tempering. If the high lattice parameter value Y for 2 hours tempering at 570°C due solely to carbon enrichment the carbon content of Y at that condition would be approximately 1.15 percent. The high micro-hardness value of 256 HV, also suggested enrichment of Y with interstials. Therefore high micro-hardness and lattice parameter value of the austenite formed by tempering at 570°C for 2 hours confirms the view of Marschall and others that this austenite contains larger amount of interstitials than the austenite produced subsequently. It was also confirmed that the Ms temperature of this initial austenite is below -196°C due to presence of large amount of interstitials. The subsequent increase in tempering time leads to dilution of the austenite with respect to interstitials with a consequent increase in Ms temperature leading to instability of austenite at sub-zero temperatures.

The good ductility of f.c.c. metals at sub-zero temperatures is thought to be due to low stacking-fault energy. The carbon stabilised the f.c.c. phase and would thus be expected to cause an increase in stacking fault energy. Stacking fault energy of Y-iron is 75 erg/cm^2 increased with the carbon content of Y and with 0.8% C the value is 120 $ergs/cm^2$. Addition of Ni to iron caused a decrease in stacking-fault energy and a minimum value of 35 erg/cm^2 at a composition of 36 % Ni was obtained. Thus high nickel seems to be more beneficial than high carbon so far as low temperature properties are concerned. The advantage of high interestials content in gamma is due to correspondingly lower amount of interstitials in the matrix which would lead to a reduction in the friction stress of the matrix.

The structure of martensite obtained on water-quenching was in the form of laths. From the S-curve for 9% Ni steel it appeared that there was some overlap with the bainitic transformation and some part of the structure was having similarity with the bainitic ferrite.

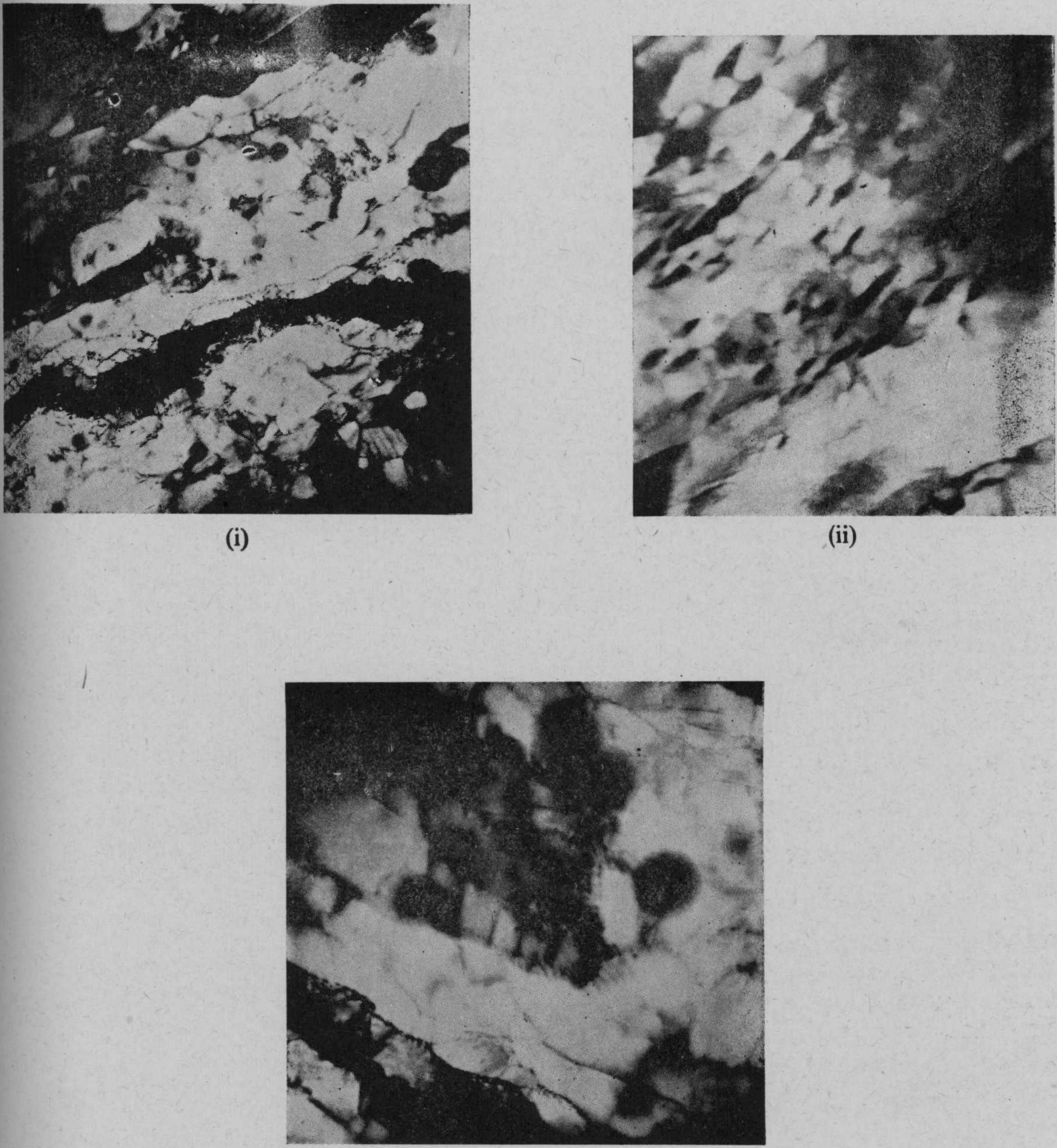
When tempered for 4 hours at 570°C the structure seems to be more akin to upper bainite and this structure persists even after 6 hours of tempering. After 8 hours of tempering a fully tempered structure was formed. In the transmission electron micrograph, (Fig. 33) there was no sign of internal twinning in the martensite areas. There are numerous carbide precipitates, both small and large in the tempered structure.

60.0 Development of High-Temperature Materials.

i) Oxidation study of high temperature metals:

The reactivity of niobium with common gases has been the subject of considerable amount of research during the recent years. Most of the interest in this field has been based on applications that will expose the metal to either oxygen or air at elevated temperatures. The fact that niobium undergoes severe oxidation above 500°C has somewhat limited the scope of its successful use in an oxygen-bearing environment. It is recognised that a clear understanding of the exact nature of these oxides is essential before a method to protect the metal against oxidation can be sought.

Work on the oxidation characteristics of niobium which was reported earlier was mainly concerned with the preliminary investigation on different oxides that formed on oxidation of pure niobium metal powder in air. Further work on similar lines revealed that after the initial dissolution of a small percentage of oxygen into the niobium lattice, a new sub-oxide is formed below 350°C. The structure of this sub-oxide closely resembles to that of the oxygen saturated metal. It is believed that the sub-oxide found in this investigation corresponds to the oxide ascribed as NbO_x by earlier workers. The stability of the sub-oxide was found to decrease with increasing temperature which disappeared completely at 400°C. Interference colours



(i)

(ii)

(iii)

Fig. 33 The electron micrograph of 9% Ni-steel tempered at 570°C for 2 hr, thin foil

(i) × 40 000

(ii) × 60 000

(iii) × 80 000

were observed on several oxidised niobium samples during the initial stages. The interference colours were prevalent under conditions when the formation of the suboxide predominated. This may also be caused by the other known lower oxides of niobium which exist as relatively thin layers and hence insufficient to be detected by x-rays. The x-ray analysis of niobium samples oxidised at various temperatures are summarised in Table 40. The interplaner spacings along with the intensities of the sub-oxide detected at 350°C are given in Table 41.

The oxide formed on heating niobium metal powder in air above 400°C was found to consist of Nb_2O_5. X-ray analysis of Nb_2O_5 obtained at various temperatures showed that two polymorphic forms are exibited by Nb_2O_5. A low temperature α-form which exists upto 850°C rapidly transforms to the high-temperature β-form when heated above this temperature. β-Nb_2O_5 once formed, remains stable at all higher temperatures. On the contrary, α-Nb_2O_5 transforms slowly to β-form even at temperatures within the α-range. α-Nb_2O_5 is therefore considered as metaslable while β-Nb_2O_5 is the true stable form of Nb_2O_5 at all temperatures. The x-ray result on the α to β transformation was further corroborated by dilatometry which indicated that the transformation temperature lies in between 850 **and** 900°C. The transformation is accompanied by a relative expansion and is irreversible.

ii) Phase equilibria in Oxide systems: (Nb_2O_5 - Al_2O_3 system)

The system was investigated by metallographic and x-ray diffraction methods. Priliminary investigation showed that both Nb_2O_5 and Al_2O_3 enter into extensive solid solution with each other. Only one compound was observed having a composition $AlNbO_4$. This phase was found to be in equilibrium with both Nb_2O_5 and Al_2O_3 solid solutions.

Further work should be carried out on oxidation of pure niobium metal sheets and the effect of minor addition of other oxides on the oxidation resistance of the metal. The eventual aim of this type of work would be to find out oxidation resistant niobium metal alloys.

Table 40 - X-ray Identification of Oxide Phase of Niobium.

Temperature ^{o}C	Time min.	Phase Identified
280	30	Nb(O) solid solution
300	30	Nb(O) with further expanded lattice.
330	60	Nb(O) + traces of NbO_x
350	75	Nb(O) + NbO_x
400	45	Nb(O) + Small amount of α-Nb_2O_5.
600	60	Large amount of α-Nb_2O_5
800	60	α-Nb_2O_5 + trace of β-Nb_2O_5.
850	60	Larger amount of β-Nb_2O_5.
900	30	β-Nb_2O_5.
1000	30	β-Nb_2O_5.

Table 41 - Interplaner Spacings of NbO_x

No.	Intobs.	dcalc.
1.	s	2.3864
2.	s	2.3357 Nb.
3.	f	1.6981
4.	f	1.6604 Nb.
5.	f	1.3949
6.	m	1.3747
7.	m	1.3527 Nb.
8.	v.f	1.2028
9.	m	1.1820
10.	v.f	1.0879
11.	v.f	1.0698
12.	f	1.0556
13.	v.f	1.0407 Nb.
14.	v.f	0.9847
15.	v.f	0.9689
16.	v.f	0.9633
17.	v.f	0.9186
18.	v.f	0.9134

N.B:- s = strong; m = medium; f = faint and v.f = very faint.

61.0 Isothermal Transformations in 17% Cr-Mn-N Stainless Steels.

An extensive study of the isothermal transformations in 17% Cr-Mn-N stainless steels was made. It revealed that the addition of 10% Mn at a constant level of ~0.4% C+N makes them entirely austenitic throughout the range of holding temperatures. However, a grain boundary precipitation is observed on prolonged annealing.

Below 10 % Mn, these steels are duplex in nature and at the solution - treatment temperature of 1100°C have a delta + Gamma phase structure. The mode of transformation in these duplex series of steels has been studied in detail employing x-ray diffraction, optical and electron microscopic techniques. It was observed that at temperatures above the austenitic range, the formation of an aggregate of lameller nitrides and carbides takes place from the delta-ferrite. The austenite forms from delta-ferrite at the delta-delta and delta-gamma phase boundaries and has an acicular structure.

At temperature below A_1, the austenite transformed allotropically into alpha-ferrite. This was followed by the formation of a pearlitic aggregate of carbide in alpha-ferrite. A general precipitation of the Widmanstattan type containing $Cr_{23}C_6$ was observed in the range of 800°C and below on prolonged holding.

62.0 Preferred Orientation in Extruded Rods.

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

The coventional X-ray diffraction technique was used in which the axis of the extruded rod was kept horizontal and prependicular to the X-radiation which was incident just on one edge of the specimen. The measurement of the azimuthal positions of the intensity maxima on the semi-Debye-Scherrer rings obtained in the diffraction photographs, enable to deduce the preferred orientations developed in the extruded rods.

During the period under review, the variation of the texture from the surface to the centre of the commercially pure aluminium rods extruded at 400°C with exist speed of 60 ft/min. to rods each of 0.785 in. dia. was studied. The X-ray diffraction photographs taken for 20 positions of the above rod at a pitch of 0.0392 in. (5% of the original diameter of the extruded rods), indicated two zones (i) the surface zone (No. 1 - No. 5) and (ii) the central zone (No. 6 - No. 20).

i) Surface zone:

This zone has preferably a single texture [100] superimposed upon a random orientation. Comparatively the larger grains are oriented in a preferred manner showing the presence of [100] texture and the smaller grains are oriented randomly.

ii) Central zone:

In this zone the grains were oriented randomly without showing any indication of the presence of any fibre texture. The grain orientation did not change appreciably with the position in the radial direction towards the centre.

The textures of three specimens of the above extruded rods (i) front, (ii) middle and (iii) rear portion had been studied. No appreciable change in the development of textures was found in the above three specimens. Further work on duralumin and commercially pure aluminium is in progress.

63.0 Studies on the mechanism of Producing Good Battery Activity in MnO_2.

MnO_2 is an important component in the manufacture of dry batteries. The battery active properties of MnO_2 are however not the same in all naturally occuring and synthetic forms of MnO_2. The characteristics of a good battery active MnO_2 (as found in natural West African Ore) are its high and steady voltage and current on discharge), when used in a battery. It has long been suggested that the difference in the battery active properties of different types of

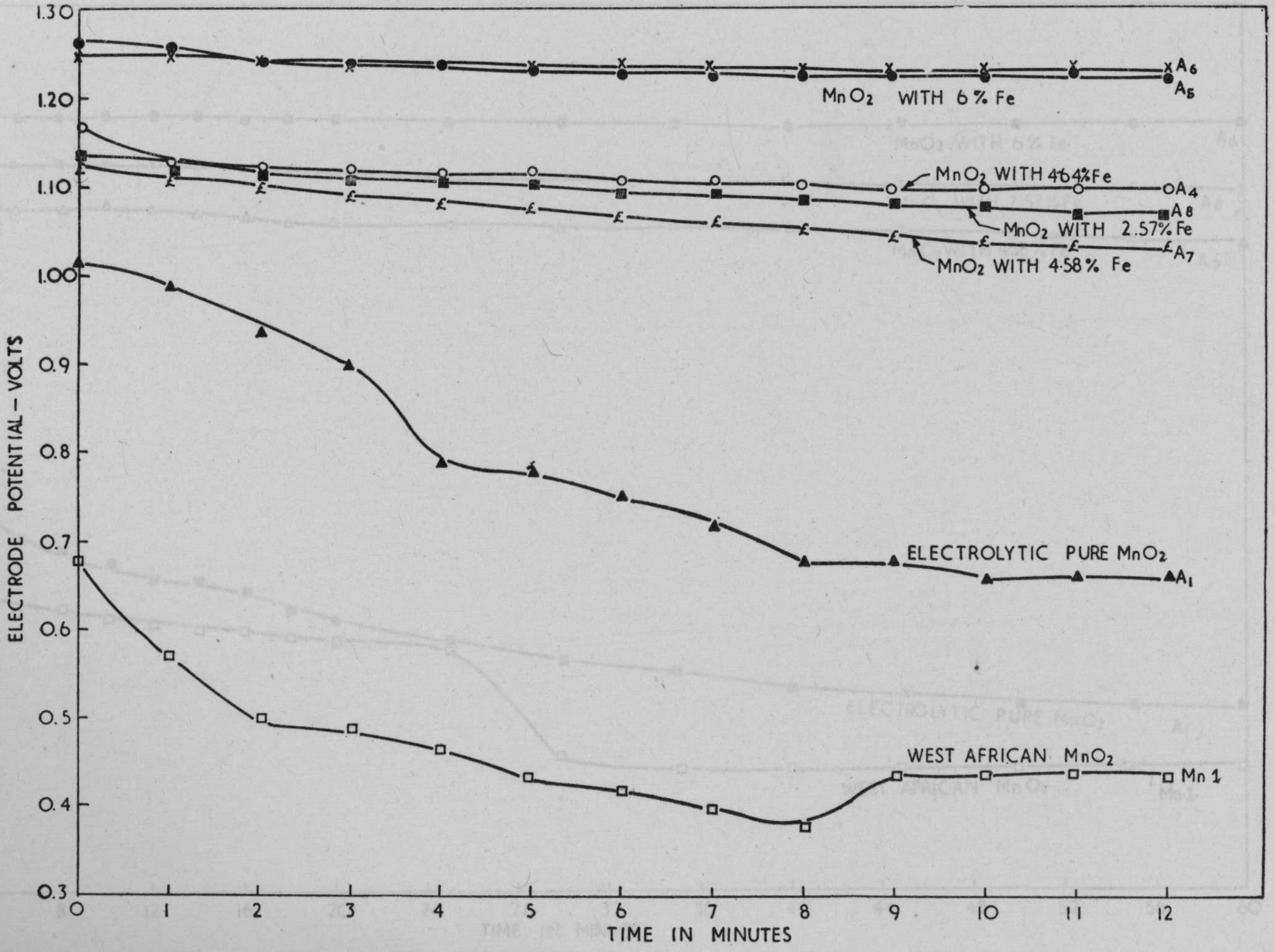


Fig. 34 Discharge of MnO_2 electrodes containing iron (Electrode potential against time)

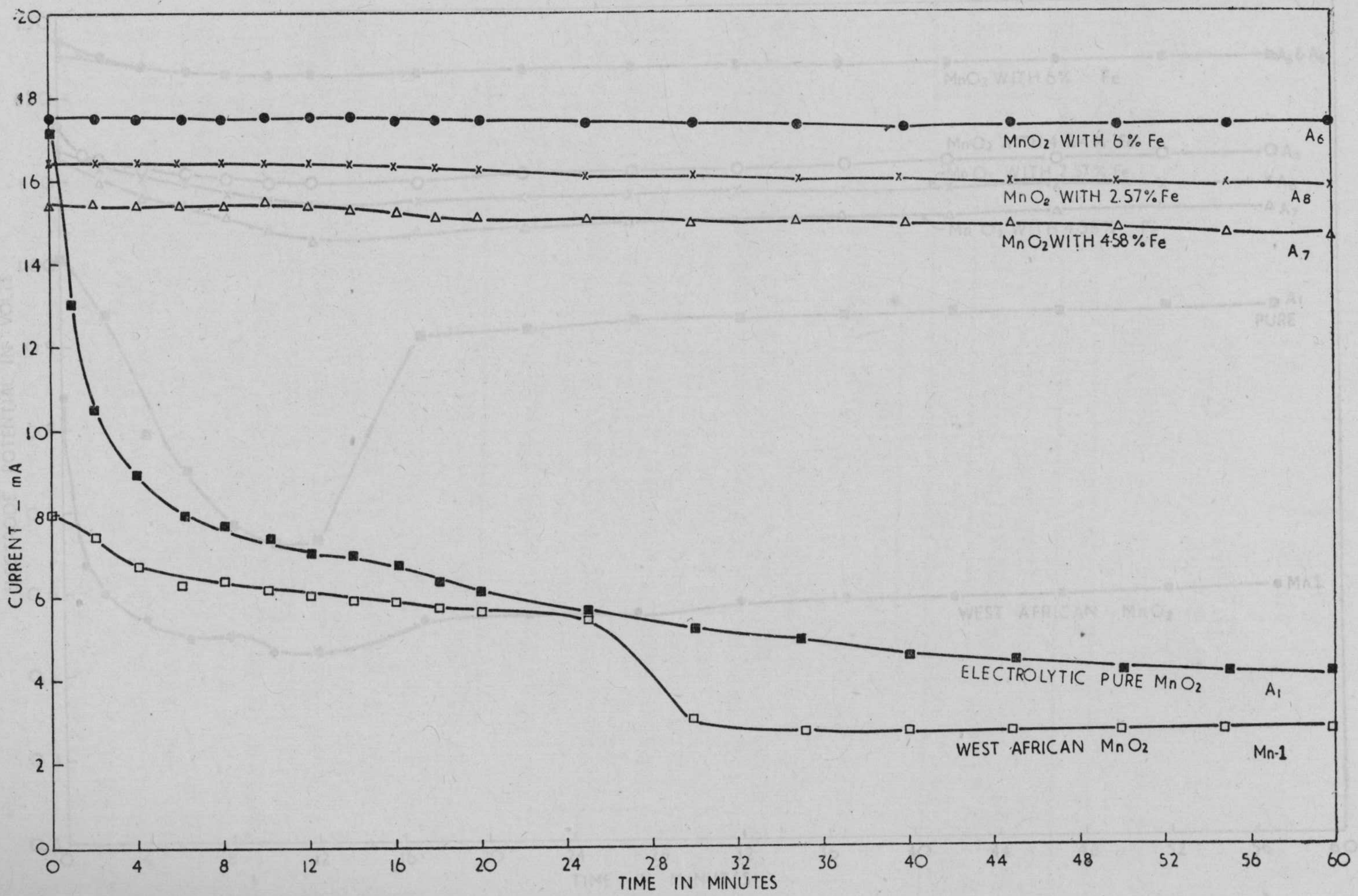


FIG. 35 DISCHARGE OF MnO_2 ELECTRODES CONTAINING IRON (CURRENT AGAINST TIME)

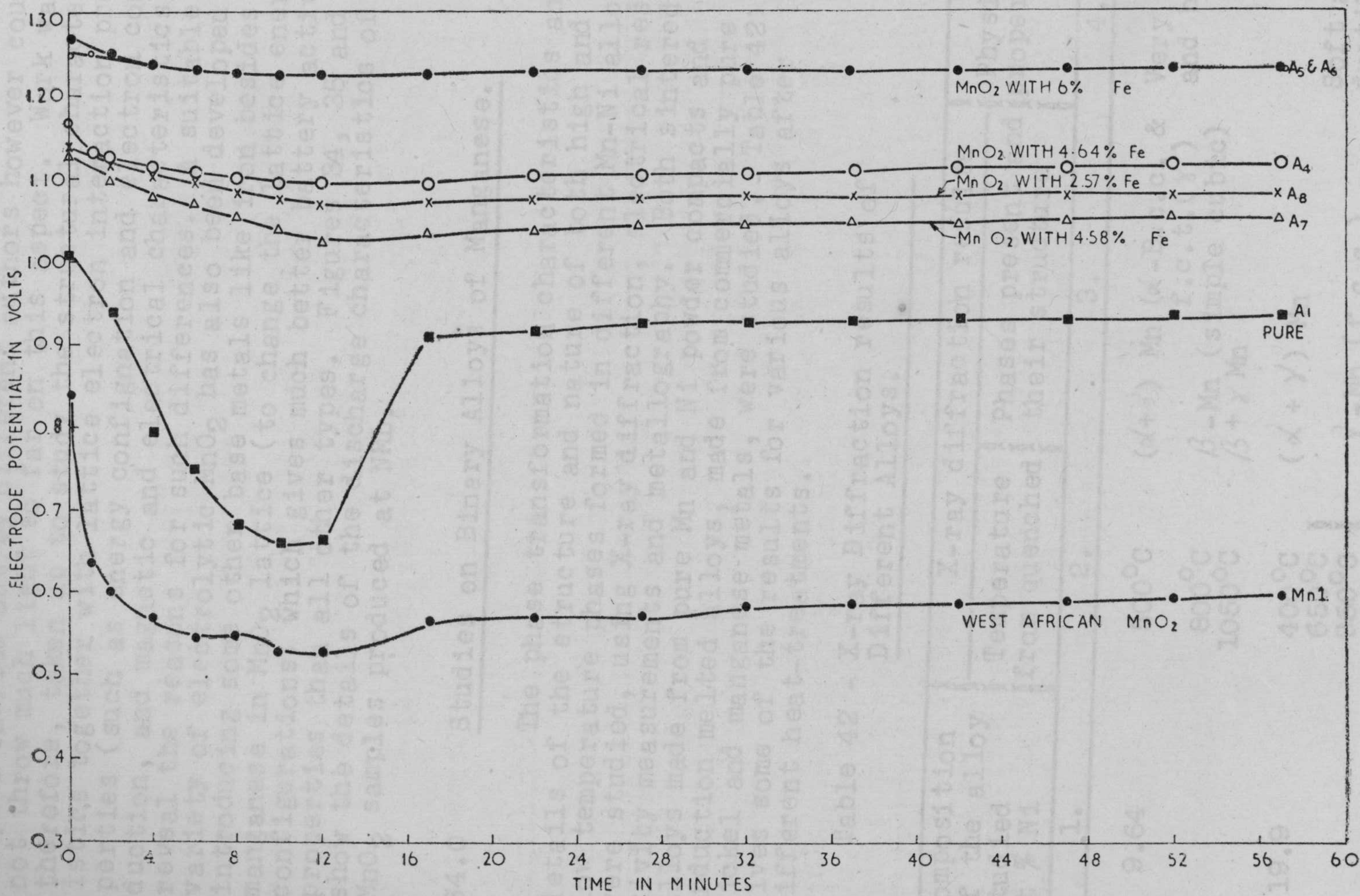


FIG. 36 DISCHARGE & RECOVERY OF MnO_2 ELECTRODES CONTAINING IRON (ELECTRODE POTENTIAL AGAINST TIME)

MnO_2 are due to some difference in their crystal structures. Studies on the crystal structure of different MnO_2 samples carried out by different authors however could not throw much light so far on this aspect. Work was, therefore, taken up to study the structural characteristics together with lattice electron interaction properties (such as energy configuration and electron conduction, and magnetic and electrical characteristics) to reveal the reasons for such differences. A suitable new variety of electrolytic MnO_2 has also been developed by introducing some other base metals like iron besides manganese in MnO_2 lattice (to change the lattice energy configurations) which gives much better battery active properties than all other types. Figures 34, 35 and 36 show the details of the discharge characteristics of the MnO_2 samples produced at NML.

64.0 Studies on Binary Alloys of Manganese.

The phase transformation characteristics and details of the structure and nature of both high and low temperature phases formed in different Mn-Ni alloys were studied, using X-ray diffraction, electrical resistivity measurements and metallography. Both sintered alloys made from pure Mn and Ni powder compacts and induction melted alloys, made from commercially pure nickel and manganese metals, were studied. Table 42 gives some of the results for various alloys after different heat-treatments.

Table 42 - X-ray Diffraction results of Different Alloys.

Composition of the alloy studied wt % Ni	X-ray diffraction results		Physical properties
	Temperature from quenched	Phases present and their structure	
1.	2.	3.	4.
9.64	500°C	$(\alpha+\gamma)$ Mn (α-B.c.c. & f.c.t.(γ)	Very hard and brittle
	800°C	β-Mn (simple cubic)	
	1050°C	$\beta+\gamma$ Mn	
19.9	400°C	$(\alpha+\gamma)$ Mn	
	650°C	γ-Mn (f.c.c.)	Soft and ductile
	950°C		

1.	2.	3.	4.
24.5	500°C γ 800°C γ	γ-Mn (f.c.c.)	Soft and ductile with workable properties.
39.3	500°C 800°C 950°C	γ+ Mn Ni(L) f.c.t. γ+ Mn NI(H) f.c.c. γ- solid solution	Hard and less ductile
51.6	500°C 850°C 950°C	Mn Ni(L) f.c.t. Mn Ni(H) f.c.c. γ-solid solution(f.c.c.)	very hard and brittle
76.00	400°C and slowly cooled 750°C	Ordered $MnNi_3$(f.c.c.) disorded $MnNi_3$	Ductile with workable properties

Crystal structures and transformation characteristics of Mn-Ni alloys as found in this investigation agreed well with those reported by earlier workers. The controversies on high temperature phase in the equiatomic range which was reported as f.c.c. by earlier workers and as b.c.c. by Hume Rothery in his high temperature X-ray work has been clarified. The phase was found to be b.c.c. at high temperature having lattice parameters of 2.961Å and transforms to a f.c.c. structure having 3.74Å lattice parameter values which is practically same as that of γ-Mn solid solution. These aspects were studied both by X-ray diffraction and metallographic observations. An interesting observation on the presence of a phase similar to sigma phase have been made in alloys having 30-60% Ni at higher temperatures with the addition of silicon in small quantities. Silicon in small quantities seems to stabilize this phase. This has not been reported by any of the earlier workers and confirm one of the predictions by Hume Rothery.

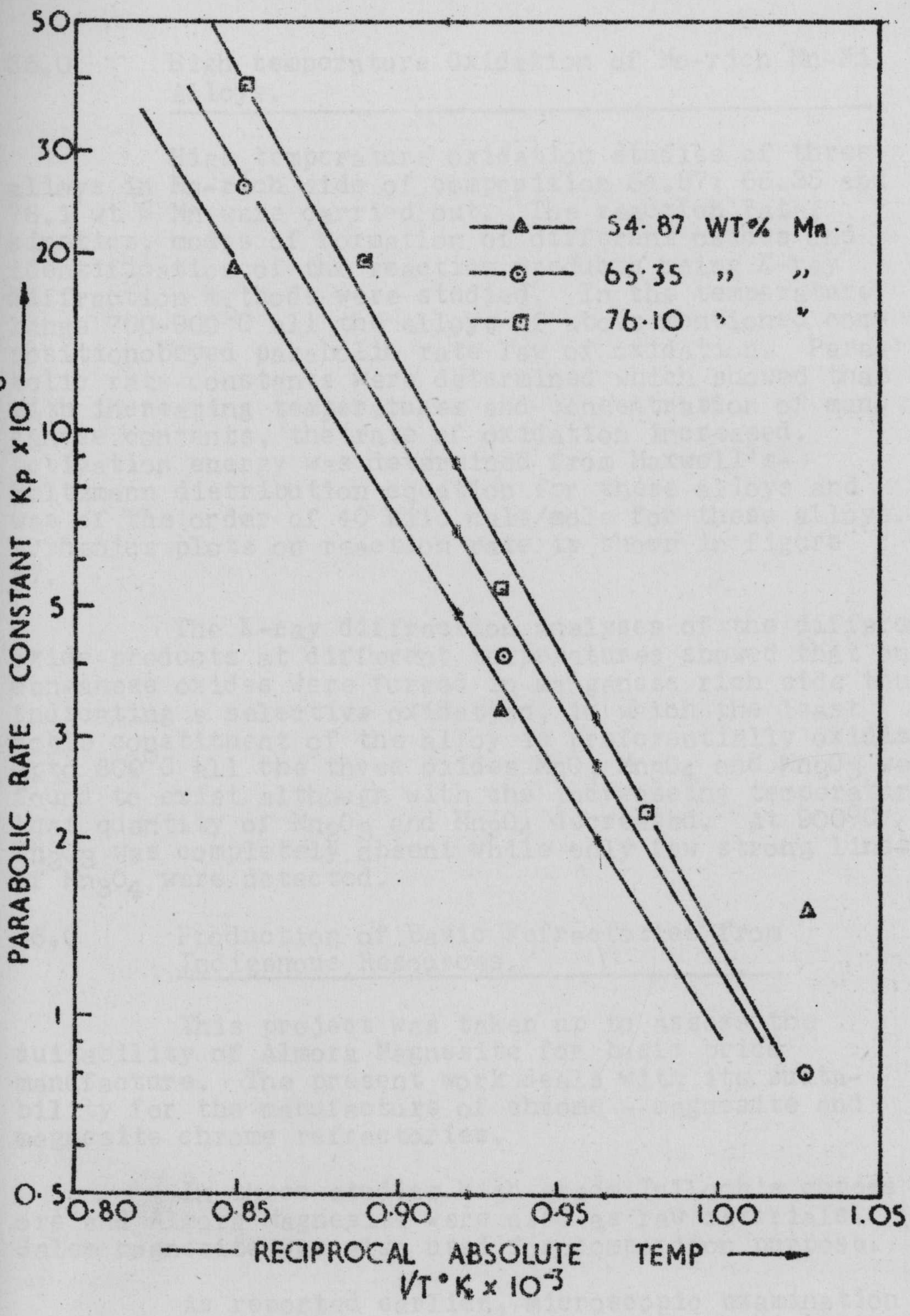


FIG. 37. ARRHENIUS PLOT OF SCALING RATE FOR THE MnNi ALLO AT HIGH TEMP.

65.0 High temperature Oxidation of Mn-rich Mn-Ni Alloys.

High temperature oxidation studies of three alloys in Mn-rich side of composition 54.87; 65.35 and 76.1 wt % Mn were carried out. The reaction rate, kinetics, modes of formation of different oxides and identification of the reaction products using X-ray diffraction methods were studied. In the temperature range 700-900°C all the alloys of above mentioned compositionobeyed parabolic rate law of oxidation. Parabolic rate constants were determined which showed that with increasing temperatures and concentration of manganese contents, the rate of oxidation increased. Activation energy was determined from Maxwell's-Boltzmann distribution equation for these alloys and was of the order of 40 kilo cals/mole for these alloys. Arrhenius plots on reaction rate is shown in figure 37.

The X-ray diffraction analyses of the different oxide products at different temperatures showed that only manganese oxides were formed in manganese rich side thus indicating a selective oxidation, in which the least noble constituent of the alloy is preferentially oxidised. Upto 800°C all the three oxides MnO, Mn_3O_4 and Mn_2O_3 were found to exist although with the increaseing temperatures that quantity of Mn_2O_3 and Mn_3O_4 decreased. At 900°C Mn_2O_3 was completely absent while only few strong lines of Mn_3O_4 were detected.

66.0 Production of Basic Refractories from Indigenous Resources.

This project was taken up to assess the suitability of Almora Magnesite for basic brick manufacture. The present work deals with its suitability for the manufacture of chrome - magnesite and magnesite chrome refractories.

In these studies high grade Tulloch's chrome ore and Almora Magnesite were used as raw materials. Salem magnesite was also used for comparison purpose.

As reported earlier, microscopic examination of the samples in the reflected light revealed cavities round the chrome grains and no direct bonding of the periclase and chrome grains even at the highest firing temperature (1700°C). To study the phenomenon of cavity formation some samples were made with pure chromite and pure pericalse with different additions of iron oxide and in these samples it was observed that samples with 10% and more of iron oxide in the periclase had very good direct bonding.

Keeping the above observations in view some Salem magnesite was sintered with 5 and 10% mill scale additions and samples having composition 70% chromite - 30% magnesite, 60% chromite, 40% magnesite and 85% magnesite, 15% chromite were made using beneficiated chrome ore (-72 mesh size and 1.8% SiO_2). Samples of the same composition were also made with -10, and -18 mesh chrome ore (non benificiated).

Microscopic examination of these samples under reflected light revealed that in samples made from benificiated ore porosity had hindered the direct bonding of the chrome and periclase grains. 70/30 and 60/40 compositions which are highly porous (porosity above 30%) have very little direct bonding but in magnesite/chrome samples in which graded magnesite was used and which had less porosity, direct bonding was quite good.

With -10 and -18 mesh chrome ore samples containing magnesia sintered with 5% mill scale addition, very good direct bonding was not observed, though it was definitely better than the old samples. But with samples containing magnesia sintered with 10% mill scale addition, very good direct bonding observed. Particle size of the chrome ore seems to have little effect on direct bonding.

Some samples were also made wth Dewaldhar magnesite (sintered with 5 and 10% iron oxide additions) and the same chromite (-10, and -18 mesh). The properties of these samples are being studied.

67.0 Studies on Carbon Refractories.

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

i) Development of dense carbon aggregate as substitute for imported anthracite coke:

Work during the period has been concentrated on determining certain data necessary for designing and fabrication of equipment for the carbon pilot plant that is now under installation. Mainly the effect of time and temperature on heat treatment of these aggregates are being assessed in a quantitative manner by determining the dimensional changes, weight losses and electrical resistivity of compacts heat treated at specific temperatures in the range of 400 to 1300°C. Experiments on corrosion by sodium under electrolytic cell conditions are also in progress.

ii) Development of substitute for petroleum coke:

The indigenous supply of uncalcined petroleum coke may fall far short of the actual demand in near future and may not be able to cover even 50% of the consumption. In view of this, experiments are now in progress to produce substitute carbon materials which can take the place of petroleum coke and incidentally serve as raw materials for the dense carbon aggregate process.

A comprehensive programme for the examination of some readily available steel plant pitches is underway. The idea is to determine the physical properties of these pitches particularly viscosity and co-efficient of viscosity, densities and variation in densities with respect to temperature as well as calcining characteristics at various temperatures and under various pressures. Apparatus for these experiments have been rigged up during the period under review. An apparatus for determining the C/H ratio of these materials is also under fabrication. A reactor to treat these pitches at different pressures and different temperatures has been fabricated and is undergoing preliminary trials.

68.0 Study on Clays and Bauxitic Clays from Jammu & Kashmir.

The objective of this study is to find the possibility of utilising the clays from (salal area of Jammu province) for the manufacture of refractories. Earlier studies, made on the physico - chemical properties of these clays indicated that these clays could be used for the manufacture of superduty type fireclay refractories. On the basis of their mineralogical constitution these clays can be classified into two categories. (1) Kaolnitic clays and (2) Bauxitic clays with Kaolinitic and diaspore intimately mixed in varying proportions. Out of these, the four kaolinitic clays and one of the bauxitic clays were further investigated for their suitability for the manufacture of superduty fireclay refractories. Results on physical properties of some of the selected composition made from these clays, which showed promise for their utilisation have already been reported earlier.

Study on the suitability of the remaining three bauxitic clays for the manufacture of superduty refractories was taken up during the period under review. The chemical analysis of these bauxitic clays have already been incorporated in last year's report.

Grogs for these bauxitic clays were prepared by firing them at 1500°C. Test specimens for each of these clays were prepared from mixes containing graded grog (80-95%) and plastic bond clay. C_5 from the same deposit (5-20%). Final firing was carried out at 1300°C and 1500°C. Physical properties of some of the selected compositions which have given good results are given in Table 43.

69.0 Determination of the Properties of Insulation Refractories (I.S. 2042 - 1963) - Laboratory Examination of Indian Standards.

The objective of this work is to study the various physical properties of some commercial Indian Insulation fireclay refractories.

Samples of eleven brands were obtained from various manufacturers. Some contained 50-52% Al_2O_3 some lower than 34%, one of 40-42 Al_2O_3, one, of type 3-3 I.S. 2042 and another of type 2-IS-2042.

Properties studied were:

1. Visual examination; 2. Dimensional variation, 3. Porosity, Bulk density, apparent specific gravity etc. were determined as per I.S.I. standards 4. C.C.Strength of samples were determined on half bricks. 5. Transverse strengths were determined on full bricks given a span of 7". 6. Reversible thermal expansion upto 1000°C was

Table 43 - Physical Properties of some Selected Compositions.

No.	BC4-15	BC4-20	BC4-5	BC4-10	BC4-15	BC4-20	BC9-10
Grog %	85	80	95	90	85	80	90
Clay %	15	20	5	10	15	20	10
Temperature of firing	1350°C	1350°C	1500°C	1500°C	1500°C	1500°C	1500°C
Linear Change %	-0.38	-0.33	-0.38	-0.12	-0.48	-0.60	-2.22
Apparent Porosity %	23.9	23.5	24.4	25.3	24.10	22.4	23.5
Bulk density gm/cc	2.18	2.17	2.18	2.15	2.18	2.20	2.01
CCS lb/sq.in	2380.0	2720.0	3980.0	2810.0	2590.0	2604.0	2990.0
MOR lb/sq.in	553.0	510.0	721.0	806.8	1156.0	1102.0	692.0
Reheat change vol.percent	-0.91	-2.04	-1.23	-0.70	-0.10	-0.18	-3.64
R.U.L.	To be determined						
Spalling	--	--	--	--	--	--	--

The refractories under load of these compositions is yet to be determined. However from the data on the other physical properties given above it is seen that these clays can be successfully used for the manufacture of super duty type fire-clay refractories.

determined by heating the sample at 50°C/mt. 7. Reheat shrinkage was done at temperatures varying as per the Al_2O_3 content in the bricks. Samples used were 9" x 4½" x 3" straights. Tests were done at 1000, 1300, and 1400°C with a soaking period of 5 hour. Reheat liner change was calculated as percent of the original length. 8. Thermal conductivity was done at temperatures of 350°C hot face and 350°C mean temp. on the Blakeley & Cobb apparatus. 9. R.U.L. of some samples were done as per I.S.I. standards using a sample of 2" dia and a load of 10 lbs. per square inch. using a carbon resistance furnace.

70.0 Study on Refractory Plastics and Castables.

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

A plastic refractory is generally a mixture of a graded refractory aggregate and a plastic fire clay whilst a castable refractory contains a hydraulic setting refractory cement and refractory aggregate with or without some chemical binder added if necessary. Not only can the mixture be rammed, moulded or cast, but these may also be gunned in position. These refractory specialistic are manufactured in our country only to a very limited extent because (i) suitable hydraulic cement and binder that can stand high temperature use have hitherto not been developed or manufactured in our country and (ii) a systematic formation of a wide and increasing variety of these mixes suitable for different applications in ferrous and non-ferrous metallurgical as well as other high temperature furnaces necessitates drawing up acceptable specifications. As a first step in this direction it was considered necessary to evaluate the qualities of indigenous plastics and castables. Accordingly the following properties of plastics and castables were determined.

1) Chemical analysis. 2) Sieve analysis. 3) Moisture content 4) Workability index 5) Drying and firing shrinkages at 1600°C, 1650°C and 1700°C for plastics and 1200°C, 1300°C and 1400°C for castables 6) Modulus of rupture at 1100, 1200, 1300, 1400, 1500 and 1650°C 7) Bulk densities and porosities at all those temperatures.

During the year under review the testing programme was completed and the results are given in tables 44 and 45.

The plastics tested show all have different particle sizes and workability index. Three of them have very poor workability index. Measure should be adopted to impart optimum workability index, adequate intermediate strength and volume stability.

In the case of castable the results show that all have good dry strength as well as fired strength at 1100°C. Only castable No. 1 with 100 lb/sq. inch ruptural strength at 1100°C might be for its low plasticity at room temperatme and high refractoriness. The service temperature of these refractories is low. It can be improved by improving the hydraulic cement used and using high temperature refractory aggregate.

Table 44 - Physical Properties of Plastics.

Chemical analysis in %	I	II	III	IV	V	VI
L.O.I.	2.30	3.80	3.66	1.56	1.88	4.20
SiO_2	31.40	36.56	38.80	39.12	57.64	49.88
Al_2O_3	55.90	51.20	52.28	53.37	34.76	39.58
Fe_2O_3	4.40	5.36	3.36	0.28	1.91	4.34
TiO_2	1.14	2.40	1.20	1.25	1.99	1.38
CaO	4.67	Tr	Tr	0.98	0.88	Tr
MgO	Tr	Tr	Tr	Tr	0.43	Tr
Alk	Tr	Tr	Tr	1.10	Tr	Tr
Sieve analysis in %						
Sieve Nos.						
B.S.S.+5	0.80	3.85	0.0	0.0	2.67	1.00
-5 + 25	40.00	55.36	27.90	63.30	52.33	11.50
-25+100	19.60	25.12	37.50	22.80	20.33	5.50
-100	39.60	16.11	34.50	13.90	25.34	82.00

	I	II	III	IV	V	VI
Moisture content	0.36%	0.42%	0.12%	0.21%	0.28%	20.17%
Workability index	.40%	15.7%	16.3%	16.00%	40%	40%
Permanent liner change at						
1100°C	nil	-0.52%	-0.51%	-0.51%	nil	-0.15%
1600°C	-0.56	+3.70	-0.20	-2.60	-1.62	-3.85
1650°C	-1.47	+2.49	-1.86	-2.90	+.86	-4.70
1700°C	-4.55	fused	-3.14	-3.36	fused	-5.05
Modulus of rupture at						
110°C	100	105	109	73	nil	83
1100°C	188	788	338	300	300	300
1200°C	263	750	405	473	240	938
1300°C	580	750	700	450	375	1725
1400°C	1035	1260	900	570	450	2460
1500°C	1890	1255	960	630	1050	2483
1650°C	2060	1312	1998	1380	1875	2600
Apparent porosity						
1100°C	--	--	--	--	--	--
1200°C	35.4	20.1	36.5	28.2	28.0	36.70
1300°C	33.5	20.3	35.3	27.6	24.5	35.50
1400°C	30.3	21.8	32.8	27.2	22.9	31.90
1500°C	26.2	18.6	32.2	27.0	18.3	28.80
1650°C	22.4	16.5	26.6	17.2	12.0	17.3

Table 45 - Physical Properties of Plastics.

	I	II	III	IV	V	VI	VII	VIII	IX	X
Chemical analysis in %										
L.O.I.(%)	2.96	0.96	1.28	3.38	0.32	0.26	1.02	1.11	0.44	3.54
SiO_2 (%)	9.60	28.80	17.00	52.68	56.48	30.48	39.97	37.24	48.76	42.08
Al_2O_3(%)	53.94	47.40	69.45	33.93	38.48	61.96	41.42	42.64	38.23	46.08
Fe_2O_3(%)	7.16	4.60	1.80	1.92	1.57	2.64	4.40	4.00	3.52	3.60
TiO_2 (%)	2.25	2.50	3.75	1.25	2.00	2.00	2.70	2.80	3.25	2.25
CaO (%)	23.10	14.42	7.10	6.44	2.90	2.68	11.70	10.11	5.20	2.28
MgO (%)	1.55	0.40	0.50	0.66	0.52	0.25	Tr	Tr	Tr	Tr
AlK (%)	Tr	Tr	Tr	Tr	Tr	Tr	1.21	1.67	0.97	1.34
Sieve analysis in %										
Sieve Nos.										
B.S.S +5	0.0	0.0	0.0	0.0	2.55	1.77	0.67	0.67	0.67	0.0
-5 +25	40.0	52.50	52.50	47.66	51.70	53.90	22.34	61.85	43.72	10.00
-25+100	20.0	12.10	5.40	13.05	10.33	20.55	17.11	6.44	15.43	14.43
-100	40.0	35.50	41.40	39.27	35.40	23.66	59.91	31.52	40.19	75.57
Workability index	10	11	12	14	12	10	22	13	12	13

Permanent Linear change at(%)										
1200°C	+0.001	nil	nil	-0.001	-0.003	nil	0.330	-0.610	-1.080	-0.002
1300°C	+0.021	-0.006	-0.003	fused	-0.815	+0.016	-0.110	-0.140	-0.250	-0.024
1400°C	+0.028	-0.004	-0.607	fused	-0.013	0.020	-0.460	-0.485	-1.380	-0.226
Modulus ofrupture at temp.										
110°C	300	383	975	278	240	263	750	440	660	450
1100°C	75	260	315	323	195	200	653	250	320	338
1200°C	375	278	390	803	473	263	685	256	431	675
1300°C	400	675	473	1710	720	338	985	480	750	1140
1400°C	425	1125	960	--	1515	450	1931	1050	1875	2025
1500°C	825	1890	-	--	1740	1800	--	--	1500	2625
Apparent porosity(%) at temp.										
1100°C	41.6	36.3	35.3	29.2	30.5	32.5	46.3	34.5	33.3	35.6
1200°C	40.7	35.0	33.7	29.7	29.8	34.3	41.3	40.2	35.8	35.1
1300°C	38.5	31.5	33.6	7.7	28.0	24.6	37.0	31.4	30.1	35.2
1400°C	42.2	29.1	31.7	--	17.4	19.4	24.8	26.1	17.4	28.6
1500°C	39.7	16.2	30.0	--	12.0	17.2	--	--	17.0	24.2

Table 46 - Chemical analysis of burnt basic bricks obtained from Steel plants and refractory manufacturer.

%	ROURKELA			DURGAPUR			IISCO			TISCO			BELPAHAR		
	A	B	C	A	B	C	A	B	C	A	B	C	A	B	C
SiO_2	8.82	7.50	6.60	6.00	3.28	5.68	7.24	8.26	6.36	7.08	17.52	6.36	5.72	6.52	5.40
Al_2O_3	14.61	4.19	1.56	8.88	10.37	0.80	10.99	2.12	3.26	6.80	Tr	0.12	13.52	10.83	1.30
CaO	1.90	2.75	2.91	0.57	1.85	2.00	2.58	2.55	3.00	0.94	1.21	0.12	0.97	1.16	1.29
MgO	33.32	72.66	85.00	50.86	62.86	88.64	48.42	58.06	83.50	42.87	51.00	89.28	55.97	62.62	88.03
Cr_2O_3	23.63	4.57	Tr	23.65	13.62	Tr	18.03	17.54	Tr	30.03	15.95	Tr	15.34	12.10	Tr
Fe_2O_3	16.00	6.40	2.00	9.32	9.17	2.640	11.00	10.00	2.00	12.00	13.40	3.12	7.84	5.61	3.52
LOI	0.50	0.28	0.80	0.29	--	0.43	0.75	0.28	0.56	0.30	0.70	0.60	0.41	0.98	0.60
K_2O	Tr	Trace	Trace	Trace	Trace	Trace	Trace	Trace	Trace	-	-	-	-	-	-
Na_2O	0.93	0.93	0.93	Trace	Trace	Trace	0.53	0.50	0.60	-	-	-	-	-	-

A - Chrome-magnesite
B - Magnesite-chrome
C - Magnesite

71.0 A Comparative Study of the Constitution, Physical and Mechanical Properties of Magnesite and Chrome Magnesite Bricks Used in the Indian Steel Plants.

This project was undertaken to evaluate the quality of basic bricks used in different steel plants of India. Some of the steel plants are using both Indian and foreign made bricks. It is envisaged that this investigation would give a comparative idea of the properties of the indigenous bricks with imported bricks. It was also envisaged that any improvements, if necessary, could be suggested to the Indian manufacturers for making better type of bricks using indigenous raw materials.

During period under review some more samples of magnesite and chrome magnesite include chemically bonded and metal clad bricks (indigenous and foreign made) were received from Tisco and Bhilai Steel Plants. Some more thin sections were prepared for petrological studies. Preparation of polished sections are also under progress. Several basic bricks were crushed and ground and sent for chemical analysis. Some of the representative analyses of magnesite, magnesite chrome and chrome magnesite bricks are shown in Table 46.

72.0 Development of Ramming Mixes for Low Frequency Induction Furnace.

Objective of this investigation is to develop suitable refractory ramming mix compositions from indigenous raw materials, for lining low and high frequency induction furnaces melting ferrous and non-ferrous metals and alloys.

Studies carried out earlier resulted in development of high alumina ramming mix composition based on natural corundum available in the country with appropriate additions of refractory bond clays etc. Laboratory trials in an experimental high frequency induction furnace have shown encouraging results. As it was felt that the dry strength and strength at low temperature (in the range 200° - 800°C) of this mix needed further improvement, the influence of certain organic binders such as 'Viscoles' on the strength of the ramming mix was further examined and it was found that desired strength could be obtained by their use in the mix in proper proportions. To try the performance of this mix on an industrial furnace necessary raw materials for one ton batch of this ramming mix have been prepared and the mix is expected to be ready soon for trials.

73.0 Development of High Alumina Cements.

The objective of this investigation is to develop suitable process based on indigenous raw materials for manufacture of high alumina cements, which are widely used in the production of refractory castable and the like for use as monolithic linings in high temperature furnaces used in metal and non-metal industries.

Studies carried out earlier resulted in the development of successful compositions for making high alumina cement, from various bauxities and limestones available in the country by the melting process. It was found that high alumina cements can also be made by sintering process and some results of work carried out in this direction were reported earlier.

During the year under review the possibility of making these cements by the sintering process has been further examined on two more bauxite samples. The chemical analysis of these bauxites are given in table 46.

Table 46 - Chemical Analysis of Bauxites.

%	Bauxite No. 1.	Bauxite No. 2.
L.O.I.	25.27	32.46
SiO_2	14.24	1.88
Al_2O_3	51.12	59.38
Fe_2O_3	7.80	4.60
TiO_2	0.50	1.68
CaO	0.94	
MgO	9.42	
Alk	--	

Bauxite No. 1 is from Shevroy and No. 2 from Mysore. The Shevory bauxite was of the red variety. Properties of cements obtained with these bauxites in combination with Madras limestone are given in table 47.

Table 47 - Physical Properties of Cements.

Bulk Density	1.748 gm/cc
Cold compression strength according to ASTM	6180 lb/sq.inch
Setting times	
Initial:	90 minutes
Final	260 minutes
Soundness	Good

It was noted that Shevroy red bauxites on preliminary crushing through jaw and roll crushers yield two distinct fractions with different chemical analysis, the + 10 mesh fraction being more ferruginous than - 10 mesh fraction.

Table 48 shows the chemical analysis of these fractions.

Table 48 - Chemical Analysis of Bauxites.

	+ 10 mesh	- 10 mesh
L.O.I. %	19.40	29.10
SiO_2 %	22.60	8.20
Al_2O_3 %	45.10	60.00
Fe_2O_3 %	7.36	1.90
TiO_2 %	0.50	1.90
CaO %	2.00	0.60
MgO %	2.00	1.80
Alk %	--	--

Cements with an Al_2O_3/CaO ratio of 1.2 were prepared using the two fractions separately with Madras limestone and sintering at 1450°C. Properties of these cements are given in table 49.

Table 49 - Physical Properties of Cements made With -10 mesh (A) +10 mesh Fraction (B).

		A	B
Bulk density		2.66 gm/cc	2.44 gm/cc
Cold compression strength according to ASTM		Very low	10,000 lbs/sq.in.
Setting times:			
	Initial:	142 minutes	45 minutes
	Fina:	330 minutes	152 minutes
Soundness		Good	Good

Table 49 from which is seen that use of the +10 mesh fraction of crushed Shevroy bauxite gave good cement.

Further work on the preparation of these cements on large scale are in progress.

74.0 Development of Sub-merged Arc-welding Fluxes.

During the year under review, further work was carried out on the development and testing of submerged arc welding fluxes. A few compositions containing high percentage of MnO were melted and their quenching properties and chemical compositions after melting were studied. The effect of heat on the phase changes in the fluxes was studied by D.T.A. Two compositions of high MnO content were tested at Indian Sugar & General Engineering Corporation, Yamunanagar and at Bharat Heavy Electricals, Trichy. The properties of the all-weld deposites obtained by using high MnO fluxes are given in Table 50.

Table 50 - Properties of Weld Deposits using High MnO Fluxes made at N.M.L.

	Tested at ISGEC Ltd., Yamunanagar	Tested at Bharat Heavy Electricals, Trichy.
Yield Strength kg/mm^2	31.1	36.4
U.T.S. kg/mm^2	48.7	51.6
% Elongation on 5 d.	33	29
180° bend	O.K.	N.D.
Impact kgm/cm^2	8.4; 7.6	10.6
Tensile joint, U.T.S. kg/mm^2	47.4 (broken outside joint)	N.D.
Microscopical examination	Satisfactory	N.D.
X-ray result	Satisfactory	Satisfactory
Name of the wire used	Bostrand No. 2 wire (Mn about 1.8%)	Union S3 (Mn about 1.5%).
Composition weld deposits	N.D.	C - 0-08 Mn - 0.88 Si - 0.20 S - 0.014 P - 0.037

The physical properties given in Table 50 are considered satisfactory. By using raw materials containing low phosphorus it is intended to test more fluxes with a view to get lower phosphorus values in the weld deposits.

The welding fluxes produced by our licencees also were tested at M/s. Indian Sugar & General Engineering Corporation, Yamunanagar as well as M/s. ACC Vickers Babcock Ltd., Durgapur in our presence. At both these places, it was found that the indigenously manufactured flux as per NML know-how performed excellent by for welding pressure vessels. A copy of the test results received from M/s. A.V.B. Ltd., Durgapur is given below:

Flux used: No. 12 B flux manufactured by M/s. Tapadia Engineers & Traders Pvt. Ltd., as per NML technical know-how.

Wire used: Modi No. 2 manufactured by Modi Steels, Modinagar
Diameter - 5/32".

Plate thickness: 2 inches.

Plate length: 24 inches.

Plate specification and plate used: BS 1633 Gr c
Min Tensile strength 28 T/sq.in
Min elongations: 23% on 5.65 A

Chemical composition: C, 0.28 max., Si 0.1 to 0.30; Mn. 0.4 to 0.9; S 0.05 max., P 0.05 max. Actual tensile strength of plate used 28.8 T/sq. inch. Actual chemical analysis: C, 0.26; Si 0.21 Mn 0.72 S 0.028 P 0.024.

Weld Operation: 10 degree level, backed by a backing strip 1/4" thick and $1\frac{3}{4}$" wide. Bottom gap $\frac{3}{4}$".

No. of runs: 24

Speed of welding: 8" to 12"/min.

Voltage: 28 to 34 V

Amerage: 430 to 600 Amps.

Properties of the welded test piece

1. Tensile test for joint:

Section	50.3 x 37.7
Area sq.mm.	1900 sq.mm.
Yield point	25.9 kg/sq.mm 16.4 T/sq.inch.
Max. stress	46.6 kg/sq.mm 29.6 T/sq.inch.
Remarks	Broken outside the weld.

2. All weld tensile test:

Section	14.30 mm dia.
Area	161 sq.mm
Distance between gauge points	2 inches
Yield point	36.0 kg/sq.mm; 22.3 T/sq.inch
Max stress	50.3 kg/sq.mm; 32 T/sq.inch
Elongation	37%
Section after break	8.2
Reduction area:	67%

3. Bend test inner surface	180° satisfactory
Bend test outer surface	180° satisfactory

4. Izod impact V notch at room temperature:

Inner surface	M.kg. 9.00 (64.8 ft. lbs)
Outer surface	M.kg.10.8 (77.7 ft. lbs)

5. Charpy V-notch test at minus 40°C

i) On weld metal (top)	4.4 M.kg.(31.8 ft lbs)
ii) -do-	5.0 M.kg.(36.2 ft lbs)
iii) -do-	4.6 M.kg.(33.3 ft lbs)
iv) On weld metal (bottom)	7.0 M.kg.(50.6 ft lbs)
v) -do-	4.0 M.kg.(28.9 ft lbs)
vi) -do-	6.4 M.kg.(46.3 ft lbs)

6. Microhardness test:

Parent metal	135 HV
HAZ	149 HV
Weld	155 HV

7. X-ray examination	Satisfactory

8. Analysis of weld deposits:

	Top	Bottom
Carbon	0.10	0.11
Silicon	0.42	0.44
Manganese	1.40	1.42
Sulphur	0.024	0.026
Phos.	0.026	0.028

---

9. Macro etching: Satisfactory

10. Microscopic examination: Satisfactory

11. The test reuslts are satisfactory and as good as those of imported Gr 80 flux in combination with B.C.C. wire No. 2.

75.0 Development of Resistor Elements for High Temperature.

It is common experience that for electrical furnaces, running at temperatures of about 1300°C elements have to be imported from foreign countries. This need is being felt all the more, with a fairly large number of such furnaces being built in the country both for metallurgical as well as for other industries requiring heat treatment equipment. A number of laboratories also need, these elements and at least one high tension insulator plant has a tunnel kiln running with such resistor elements through out. In view of this need a project has been under taken in NML to examine the possibility of these elements in the country. During the year under review certain necessary equipment for processing the materials at very high temperatures of the order of 2000°C have been designed and they are being fabricated.

Polishing techniques necessary for preparation of specimens for examination under microscope are also being perfected. Certain compositions of silicon carbide have been under preparation and are waiting thermal treatment prior to study of their properties.

76.0 Development of Tar Bonded Basic Refractories For L.D. Furnaces.

Tar bonded basic refractories for L.D. furnace lining has a vast commercial potential in the steel industries in India as a consequence of the use of L.D. process of steel making. At present Rourkela Steel Plant, HSL (which is the only steel plant using L.D. process in India) manufacture tar bonded dolomite blocks for their own use. The fifth steel plant under public undertaking at Bokaro is also expected to use pneumatic process of steel making (L.D.). This is expected to create need for data on indigenous raw materials as well as systematic study of lining that can be developed from them. With this end in view a project for the development of tar bonded basic refractories has been taken up under the following heads:

1. Literature review of the most recent developments in this field.
2. Study of the indigenous raw materials, dolomites, magnesites, tars and pitch etc.
3. Development of suitable compositions of the refractory tar bonded basic bricks.
4. To make large pilot scale studies.

The first part of the project, i.e. making a literature review is under progress and it is hoped that a review report will be ready very soon.

77.0 An Apparatus for Measuring the Thermal Conductivity of Refractory Bricks.

Thermal conductivity determinations are of importance both in the manufacture of as well as the utilisation of insulating refractories. In the former, it is a necessary step in quality control and in the latter it is essential for designing effective and at the same time economic linings.

While the data are so important, their determination is time consuming as well as complicated. Further, in India, it is difficult to instal and keep in operation many of the standard equipments designed in other countries for the purpose, as they involve imported parts. The Indian Standards Institution has therefore decided to try out the efficiency of some simple equipment that can be easily fabricated in this country and can be expected to be reasonably acurate. Blakely and Cobb appears to answer both this needs.

So at the I.S.I's request Blakely & Cobb apparatus was prepared and several commercial samples were tested.

78.0 Preliminary Investigation on Hot Top.

This investigation was sponsored by M/s.Khannas Caplos Co. In the preliminary investigation attempts were made to find out the chemical composition of the hot top as well as some of its physical properties. The investigation indicated that submitted sample of hot top is made of quartz sand, clay material and some fibrous organic material. Physical properties tested were porosity, bulk density, modulus of rupture and thermal conductivity. A report embodying the recommendations has been submitted to the party.

79.0 Mineralogical Constitution of Indian Steel Plant Slags.

During the period under review some more blast furnace slags from different Steel Plants were melted in platinum crucibles for liquidus studies. A small coke oven gas oxygen furnace was used for this purpose. After melting the slags were quenched in a water bath to get homogenous glasses. Liquidus studies of these slags are in progress.

Further studies on the mineralogical constitution of Indian Open Hearth and L.D. Slags showed that all the Indian open hearth slags are high in CaO. Slags produced at IISCO had the lime content of 49.72 to 51.95%. IISCO slag had also the highest P_2O_5 content of 4.52 to 8.01%.

The texture of the open hearth slags varied with cooling history and phase constitution. Beta dicalcium silicate and wustite were predominant phases. Nagelschmidtite occured in slags where P_2O_5 was comparatively high. Tricalcium silicate was also present in many of the slags. The chemical and mineralogical compositions of open hearth and L.D. slags were more or less similar but free lime was present only in L.D. slags, which indicated incomplete reaction. L.D. slags were also predominantly composed of Beta dicalcium silicate and wustite Dicalcium ferrite was present in amount in Bhadravati L.D. slags. Other minerals found in small amounts were monticellite, brown millerite, ilmenite and pyrophanite. Two photomicrographs, one of open hearth slags and other of L.D. slags, are given in Figs. 38 and 39. A summarised table showing constitution of these slags is shown in Tables 51 and 52. A paper on the constitution and mineralogy of Indian open Hearth and L.D.Slags was presented at 33rd Annual Convention of India Ceramic Society at Delhi.

Table 51 - Mineralogical Composition of Indian Open Hearth Slags.

Minerals	Source of Slag.					
	TISCO	ROURKELA	DURGAPUR	BHILAI	IISCO	BHADRAVATI
Beta Dicalcium silicate	MX	MX	MX	MX	MX	MX
Wustite	MX	MX	MX	MX	MX	M
Nagelschmidtite	MX	M	X	--	M	M
Tricalcium silicate	X	--	X	X	X	--
Dicalcium ferrite	--	X	--	--	X	--
Monticellite	--	X	--	X	--	--
Brown millerite	X	X	--	X	X	--
Illmenite	X	X	--	--	X	X
Pyrophanite	X	X	--	--	X	X

Table 52 - Mineralogical Composition of L.D. Slags.

MINERALS	ROURKELA	BHADRAVATI
Beta dicalcium	MX	MX
Wustite	MX	X
Nagelschmidtite	M	MX
Dicalcium ferrite	X	X
Monticellite	X	--
Tricalcium silicate	X	X
Brown millerite	X	X
Eree lime	M	M

M - observed microscopically

X - determined by X-ray.

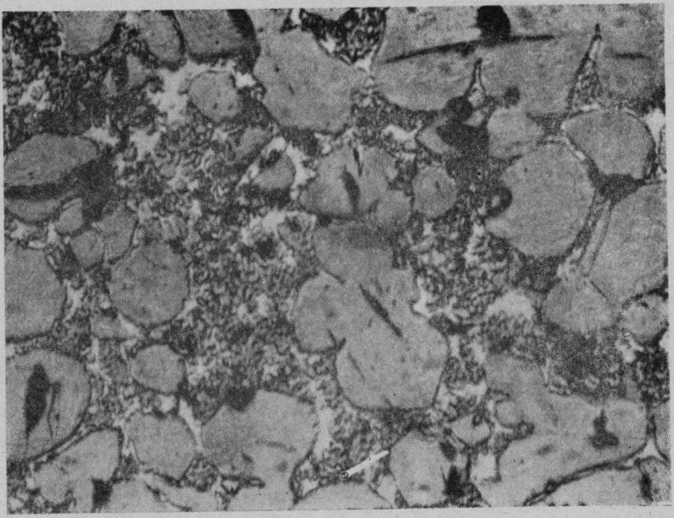


Fig. 38 Photomicrograph of open hearth slag from IISCO showing silicate and oxide phase

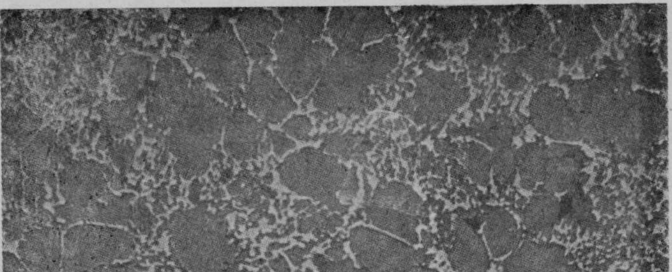


Fig. 39 Photomicrograph of L.D. slag from Rourkela showing silicate and oxide phases

80.0 Development of Al-base Sacrificial Anode for Cathodic Protection.

The use of sacrificial anodes for cathodic protection of sea going vessels and underground pipelines is receiving increased importance. Generally Mg and Zinc or thin alloys are mostly used for this purpose but both these have their own limitations. Recently in foreign countries the possibilities of using aluminium alloys as sacrificial anode have been examined. Work on similar lines has been started in NML to successfully develop aluminium alloys suitable for sacrificial anode for cathodic protection.

Various compositions based binary and ternary aluminium have been developed. Methods of melting and casting, effect of impurity content on efficiency, etc. have been studied. Both laboratory tests and field trials have shown excellent performance of the alloy developed having a driving potential for steel in salt water of 500-800 mV; which is in between that of Zn and Mg. Several Port Commissioners and anode manufacturers have recently indicated their interest in this alloy. An application for patenting the alloy has been filed. Further work on the production technique is being continued.

81.0 High Temperature Oxidation of Alloys in Presence of Fuel Ash.

Residual oil and low grade coal are used for many power generating units mainly on economical ground. One of the disadvantage of using low grade fuel is the accelerated oxidation of super-heater tubes, turbines, etc. due to residual ash. For economic operation such attack is to be prevented. The project has been taken with an objective to develop suitable material of construction from indigenous sources and also to develop protective coatings or additives to oil to prevent high temperature oxidation in presence of fuel ash.

Studies on oxidation of nickel free stainless steel, developed in NML were carried out in contact with the artificial fuel ash mixture ($V_2O_5Na_2SO_4$) under accelerated conditions at high temperature of 500^oC-900^oC and the performance of the steel was compared with that of Std. 18:8 Cr-Ni stainless steel. The rate of oxidation of Ni-free stainless steel was comparatively low in comparison to 18:8 stainless steel because of the nature of the oxide growth which was non-porosu thus hindered the rapid diffusion of oxygen to the oxide/metal interface. Protective property of aluminium coating against vanadium attack has been evaluated and found to be quite satisfactory. Additives like CuO, MgO, etc. were found to markedly reduce the attack. The results obtained so far are encouraging.

82.0 Oxidation of Copper and Copper-Alloys.

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

Oxidation of Cu-Mn (5-40% Mn) alloys has been studied in oxygen upto 900°C. Addition of manganese was found to first increase the attack slightly and then decrease it markedly beyond 20% Mn. The nature of oxidation products, internal oxidations, etc. were studied. Thesis based on the work has been submitted for Ph.D. by a Research Fellow.

83.0 Failure of Stainless Steel Valves in the Antibiotics Plant of the Indian Drugs & Pharmaceuticals Ltd. at Rishikesh.

The services of the National Metallurgical Laboratory were sought for an "on-the-spot" survey of the various corrosion problems being experienced at the Antibiotics Plant of the Indian Drugs and Pharmaceutical Ltd. at Rishikesh. The corrosion of stainless steel valve in the sodium and procaine section of the Recovery & Purification block was found to be most serious and was therefore referred to NML for investigation on payment basis.

Different parameters of localised corrosion of stainless steel were studied and the results indicated that the localised corrosion of the stainless steel is due to the presence of scale on the surface and abnormal inclusions in the alloy. As remedial measure better grades of steel, e.g., AlSl No. 316L has been suggested as the material construction from the view point of the service requirement of the pharmaceutical production where even minutest quantity of corrosion of the handling equipment is not tolerable.

84.0 Feasibility of Applying of Cathodic Protection to the Underground Steel Pipe Lines for Transportation of Filtered Water at Calcutta.

Bare steel and cast iron pipelines without any kind of protection from corrosion are under use for the transportation of filtered water in the Calcutta Metropolitan area. Occasional failures due to corrosion have been experienced since long. Recently these troubles have increased to a greater extent causing grave concern to Calcutta Metropolitan Water & Sanitation Authority. It has been decided to apply cathodic protection to these lines and a preliminary survey work for the zinc covered by 35 kilometers net work of water mains was made by M/s. Indian Cathodic Protection Co. and National Metallurgical Laboratory acted as a Consultant to scrutinise the data and prepare a feasibility report on the request of CMW & SA.

85.0 Gases in Metals.

Samples of ferrous and non ferrous metals and alloys were received from different research projects in the Laboratory for the determination of gases in them by vacuum fusion method. Samples were also received from different Laboratories and Institutes for the determination of gases in metals and alloys as a regular routine work. The new electro-chemical method for the determination of nitrogen in solid solution in steel developed in NML is being used to determine solid soluble nitrogen in creep testing samples.

86.0 Pickling of Ferrous Materials with Hydrochloric Acid.

A comparative study was made on the pickling of ferrous materials by hydrochloric acid and sulphuric acid. Hot rolled mild steel sheet of 1/16" thickness having a black coating of scale was cut into panels of ½" x 1" which were used as experimental specimens. The specimens were degreased by benzene and acetone before immersing them in pickling solution. It has been established that pickling of ferrous metal with hydrochloric acid shortens time, reduces attack on base metal and furnishes a pit free pickling surface with less absorbed hydrogen even without inhibitor. In continuation of the pickling work it has been decided to study the effect of addition of sulphuric, nitric and phosphoric acid in hydrochloric acid pickling bath. It was observed that addition of other acids in hydrochloric acid did not improve the pickling time, hydrogen pick-up, or dissolution of the base metal. Results are furnished in Tables 52, 53 & 54.

Table 52 - Dissolution and H_2 pick-up during Pickling in mixture of HCl and H_2SO_4.

Sl. No.	Composition of pickling bath	Pickling temp.	Pickling time	Dissolution in gm/dm^2	H_2 pick-up in cc/dm^2
1.	4 NHCl	28°C	17 min	1.44	2.12
2.	4 NHCl+1% H_2SO_4	28°C	24 min	1.58	2.75
3.	4 NHCl+2% H_2SO_4	28°C	25 min	1.60	3.34
4.	4 NHCl+5% H_2SO_4	28°C	26 min	2.05	5.10

Table 53 - Dissolution and H_2 pick-up during pickling in mixture of HCl and HNO_3.

Sl. No.	Composition of pickling bath	Pickling temp.	Pickling time	Dissolution in gm/dm^2	H_2 pick-up in cc/dm^2
1.	4 NHCl	31°C	14 min	1.43	2.05
2.	4 NHCl+1% HNO_3	31°C	15 min	1.55	2.10
3.	4 NHCl+2% HNO_3	31°C	15 min	1.98	2.12
4.	4 NHCl+5% HNO_3	31°C	14.5 min	2.34	2.16

Table 54 - Pickling time, dissolution and H_2 pick-up in mixture of HCl and H_3PO_4.

Sl. No.	Composition of pickling bath	Pickl ing temp.	Pickl-ing time	Dissolu-tion in gm/dm^2	H_2 pick-up in cc/dm^2
1.	4 NHCl	31°C	14 min	1.45	2.15
2.	4 NHCl+1% H_3PO_4	31°C	14.5 min	1.47	2.18
3.	4 NHCl+2% H_3PO_4	31°C	14.5 min	1.46	2.16
4.	4 NHCl+5% H_3PO_4	31°C	15 min	1.48	2.19

From the experimental data it is evident that pure hydrochloric acid has got the best performance as a pickling agent. Addition of increasing amounts of sulphuric acid appreciably undermined the pickling property. Similarly the addition of phophoric acid and nitric acid failed to improve the pickling property. They also adversely affected pickling but not to the extent of sulphuric acid.

87. Electroplating.

i) Bright nickel plating process:

A process for bright nickel plating has been developed and the process is under negotiation for lease to an industrial firm.

Work on further improvement of the "N.M.L." Bright Nickel Plating process was taken up by using indigenously available organic brightners and levellers.

ii) Stripping composition for the removal of nickel from rejected nickel plated goods:

A process for stripping nickel plated coatings by simple immersion in an alkaline solution of m-nitrobenzoic acid has been worked out and standardised in this laboratory. A reputed firm in India is utilising the process on industrial scale. Efforts are being made to develop an alternative non-poisonous stripping composition.

iii) Gold and gold-alloys platings:

Investigations were taken up to find suitable plating of gold or its alloys on watch cases and watch straps for a decorative and protective purpose. The colour, composition and physical properties of the coatings are important factors. It was possible to obtain bright guinea gold finishes of pure gold from solutions containing organic acid and gold salts. Various shades of gold alloy plates containing upto 20% of alloying metal were prepared. Copper and silver were used as alloying metal.

88.0 Production of Electrolytic Iron Powder.

For production of high purity electrolytic iron powder an investigation was undertaken to standardise the composition of bath, its pH, cell characteristics, and cell design. Experiments were carried out with mild steel anode and stainless steel cathode using solutions of ferrous sulphate and ferrous chloride together with some auxilliary chemicals as electrolyte. It was found that by changing the composition of the bath and using organic compounds as addition agents particle sizes of the powder can be controlled. The powders obtained so far are free from contaminations. Oxygen content of the deposit is around 0.5%.

89.0 Preparation of Metal Catalysts for use in the Organic Reaction Processes.

i) Nickel catalysts:

For the hydrogeneration of fats and oils nickel is practically the only metal used as catalyst. Many nickel alloys, such as those with Al, Si, Zn, Mg and other elements and various nickel compounds like Ni (ous) oxide, Ni-sulphide, nickel-boride, Ni-formate etc. also have catalytic properties.

Ni is prepared in and catalytically highly dispersed active form by three principal methods: The first involves precipitating nickel carbonate or nickel hydroxide from solution on Kiesselguhr or other inert and unractive carrier, filtering, washing, drying and reducing the resulting powder at a temperature of 370°C to 540°C with hydrogen. The material after reduction is highly pyrophoric and must be suspended in oil before it is permitted access to the air. In the second method of catalyst preparation, generally termed "wet reduction" a finely ground heat labile nickel salt is suspended in oil, which is heated to a temperature high enough to bring about thermal decomposition of the salt. During the heating period, H_2 is passed under pressure. The temperature usually does not exceed about 240°C to avoid extensive thermal decomposition of the oil. Nickel forate is generally used as the starting material for wet reduction. The third method of Ni-catalyst preparation is the well known process of leaching out aluminium from Raney's Nickel + Aluminium alloy. This last named process has been adopted in NML for the study and preparation of Ni-catalyst.

Method followed was as given below:

The Ni and Al alloy powder was added in small amounts to caustic soda solution under stirring. The addition was completed in about 1½ hour and the solution and reacting slurry were stirred for another 1 hour. The resulting sludge was washed with distilled water and ethyl alcohol mixture by decantation. The sludge was then separated out by centrifugation.

The sludge material was dense, greyish black powder and was very pyrophoric even in wet condition, when exposed to the atmosphere. It ignited in the air. It was found to contain nickel upto 90%.

Studies are being continued to see the effect of temperature and time of leaching and also the concentration of NaOH in the leaching solution on the activity of the product.

ii) <u>Granular silver catalyst:</u>

This noble metal catalyst is used in the oxidation and dehydrogenation reaction for producing formaldehyde and ethelene oxide from methyl alcohol and ethelene respectively. There are at present only two units producing formaldehyde. The requirements for the silver catalyst are mostly met by import. Highly pure silver powder is used as the starting material for the manufacture of the catalyst. The silver powder is manufactured by various methods, viz. electrolysis, galvanic reduction, chemical reduction, cathodic reduction of silver oxide etc. The product is finally activated for use in the oxidation reaction to produce formaldehyde.

Electrolytic method was adopted for preparing fine crystals of pure silver with a view to obtain suitable material for silver catalyst. Conditions of electrolysis were as detailed below:

Electrodes	--	Platinum
C.D.	--	12-13 Amp/dm^2
Cell voltage	--	3 V.
Temperature	--	30°C

The product obtained was subjected to treatment at high temperature and powdered to -36+75 mesh. The work is being continued.

90.0 A Study on Techniques of Electrolytic Deposition of Metal-Ceramic Composite Materials and their Properties.

The electro deposited composite materials are of interest for better resistance to wear, abrasion, corrosion, oxidation and high temperature. Hard metal-ceramic composite coating can be applied on suitable substrate for protecting base metals by electro-deposition technique. Sheets also can be made by electro-deposition. For the present investigation nickel and copper have been taken as metal matrix and fine particles of alumina, silicon carbide used as second phase ceramic particles.

For codeposition of Ni-Al_2O_3, Ni-SiC and Cu-Al_2O_3, Cu-SiC, different sets of experiments were done to study the influence of various parameters such as current density, pH of the electrolyte and bath agitation etc. to get the best composite and find out the possible manner of migration of ceramic particle into the deposit.

From experimental results it appeared that the pH of the electrolyte played an important part for the migration and amount of nonmetallic inclusion in the deposit. A comprehensive report about the electrodeposited technique of metal ceramic has been prepared.

Specimens for physical and mechanical testing of the composites were prepared. For these purposes many samples of pure nickel, Ni-SiC and Ni Al_2O_3 have been prepared by electrolytic deposition and some specimen from these deposited samples have been prepared according to design for testing tensile strength and wear. It was found that the hardness of composite materials are in general greater than the pure metal. Further work for testing thermal and electrical conductivity and salt spray corrosion test has been taken in hand.

91.0 Rapid Methods for Analyses of Silico-Chrome Metal and its Slag.

i) Metal:

0.5 g of Si-Cr metal was treated with HNO_3 and HF in a beaker and then with sufficient perchloric acid. It was fumed for 15 to 17 minutes. Water was added and volume was made upto 250 ml. From 100 ml solution Cr was determined after reduction with standard ferrous ammonium sulphate solution as usual. From another 100 ml solution, Fe was precipitated with NH_4OH, filtered, washed and dissolved in hot dil Hcl. Then Fe was estimated by the dichromate method after reduction with $SnCl_2$ solution as usual. Carbon was determined volumetrically. Si was obtained by difference of Cr, Fe and C from 100. As the Si-Cr sample contained high Si and less than 1% Al, the percentage of Si obtained by difference was accurate enough to guide the operator of the furnace. Some results of analyses by this method are given below:

Radicals	Samples		
	1	2	3
Si %	45.3	37.06	40.7
Cr %	35.1	43.00	40.87
Fe %	18.55	18.74	17.33
C %	0.05	0.20	0.10

Further work on direct estimation of silicon is under progress.

ii) Slag:

0.25 g of powdered slag was fused in used clean silica crucible with sodium peroxide. The cooled mass was dissolved in water and acidified with hydrochloric acid. It was boiled for 12-15 minutes. SiO_2 was filtered and washed with acidified water. R_2O_3 was determined from the filtrate by double precipitation with NH_4Cl and NH_4OH. The ignited R_2O_3 was fused with potassium bisulphate and made upto a volume. From an aliquot Fe_2O_3 was determined. Al_2O_3 was obtained by difference from R_2O_3.

The filtrate from R_2O_3 was made upto a volume. From an aliquot MgO + CaO was determined with EDTA using Erichrome Black-T as indicator. As the charge was magnesite and the CaO% was less than 1%, the EDTA value was calculated only for MgO. Cr_2O_3 and SiO_2 were determined on a separate sample after fusion with sodium peroxide in a nickel crucible and analysed by usual methods. As the Si-Cr slags are insoluble in acids, the complete analysis is a difficult problem and requires two to three days for completion. By this method four samples of slags can be analysed within 6 hours. The results obtained by this method were sufficiently accurate for the purpose of controlling the furnace.

Some results obtained by this method are given below:

Radicals	Samples 1.	2.	3.
SiO_2 %	39.0	41.5	41.0
Cr_2O_3%	8.5	1.5	1.8
Al_2O_3%	22.0	27.0	30.1
MgO %	18.2	23.4	22.6
FeO %	11.7	5.4	3.8
C %	0.6	1.0	0.5

92.0 Investigations on Molybdenum Concentrates Obtained from Rakha Mines.

While Mo concentrates are easily and completely decomposed by nitric acid sulphuric acids, the one obtained from low grade copper ore of Rakha Mines left an insoluble black residue by the above treatment. Moreover, the total of the constituents generally present in the concentrates was only 76.1%. Further, the total sulphur present was much in excess of the amount necessary to be present in combination with Mo, Cu, Fe. To ascertain the reasons for the above peculiar behaviour of the Mo concentrate, systematic investigation by chemical, spectrographic and X-ray diffraction methods was undertaken in our Laboratory.

Presence of graphite and free sulphur besides molybdenite and chalcopyrite in the concentrate and graphite in the acid insoluble residue respectively have been revealed by X-ray diffraction. Shortage in the total was due to the presence of organic matter and graphite in the concentrate and 11.8% free sulphur accounted for high total sulphur content of the sample.

The composition of the concentrate can be given as follows from the results of this investigation:

MOS_2	--	51.70%
$Cu\text{-}Fe\text{-}S_2$	--	5.20%
FeS	--	1.93%
Al_2O_3	--	1.10%
CaO	--	0.40%
MgO	--	0.30%
Graphite	--	11.50%
Free S	--	11.80%
SiO_2	--	2.00%
Organic matter	--	Balance

93.0 Analysis of Metals, Alloys, Minerals etc.

i) Chemical Analysis:

3428 samples were chemically analysed during the year for different Divisions of the laboratory and in connection with investigations sponsored by outside parties.

ii) Petrological and ore-microscopic studies of low grade ores and mineral samples:

Comprehensive petrological and ore-microscopic studies of the various low grade ores received for beneficiation studies were undertaken. The scope of investigations included the physical, mineralogical character of the ores, textural relationship, inter-locking and modal analysis of the mineralogical constituents. In addition to this, a continuous support was provided to the ore dressing investigators by microscopic examination of the test products of the beneficiation processes so that the effectiveness of a process could be adequately judged. Detailed petrological study of the following ore samples was conducted:

1. Iron ore from Bailadila iron ore project (M.P.) of N.M.D.C.
2. Pyrite ore samples from Amjhore, Bihar for Pyrites, Phosphates&Chemicals Ltd.
3. Graphite samples from Andhra Pradesh.
4. Cu-Ni-Mo bearing complex Uranium ore from Jaduguda mines received from Dept. of Atomic Energy.
5. Graphite sample from NEFA received from G.S.I.
6. Nickeliferous ore from Orissa.
7. Lead ore from Agnigundala area, Andhra Pradesh received from G.S.I., Hyderabad.
8. Iron ore samples from Kiriburu Mines, Orissa of N.M.D.C.

9. Phosphate rock samples from Rajasthan, received from Govt. of Rajasthan.

10. Limestone sample from Uttar Pradesh.

11. Silica sand from Maharashtra.

12. Emery powders from Shevaroy Bauxite Products Co., Yercaud, Salem Dist., Madras.

Thus, in the year under review, nearly 25 samples were subjected to detailed petrological examination.

iii) X-ray fluorescence analysis of ores, minerals and metals:

The non-destructive, highly sensitive, selective and very rapid method of X-ray fluorescence analysis is in use since past five years to expedite analysis of routine test and processing products of various divisions of the National Metallurgical Laboratory as well as for specialised analytical problems.

The results of analysis obtained were highly satisfactory in their degree of accuracy as well as to the rapidity of the method, as compared to the other conventional time consuming analytical techniques. The testing method comprises of comparing the unknown sample with a standard sample of known chemical analysis having a similar elemental environment as that of unknown. For greater accuracy of results where the number of samples in any concentration range to be analysed run in hundreds, calibration curves are prepared using a few analysed samples which are within that concentration range. Synthetic standards are also prepared for use where analysed samples of similar nature as the unknown are not available. The range of concentration of the elements of interest that could be tested varied from trace amount to 100%

Following is the list of products and elements or radicals for which X-ray fluorescence analyses were performed:

1. Phosphate rock flotation products from Kanpur block, Rajasthan for P_2O_5.
2. Limestone flotation products of Maharashtra for CaO.
3. Kyanite-sillimanite products of Maharashtra for Al_2O_3.
4. Rakha copper ore products for Cu, Ni and Mo.
5. Diamondiferous tuff beneficiation products for TiO_2.
6. Phosphate rock flotation products from Mussoorie for P_2O_5.
7. Cu-Ni-Mo bearing uranium ore products from Jaduguda mines for Cu, Ni and Mo.
8. Pyrite beneficiation products of Amjhore for S.
9. Iron ore beneficiation products of Kiriburu mines for Fe.
10. Phosphate rock beneficiation products of Kataria hill, Rajasthan, for P_2O_5.
11. Iron ore beneficiation products of Donimalai iron ore project for Fe, SiO_2 and Al_2O_3.
12. Nickeliferous ore beneficiation products for Ni.
13. Fly ash and flue dust samples for Ge.
14. Slimes and residues of selenium project of Ext. Met. Division for Te, Ag, Se, Cu & Ni.
15. Aluminium bronze alloys for Cu, Fe, Mn and Al.
16. Iron ore samples from Iron and Steel Division for Fe.

During the year under review, about 4,800 elemental or radical analyses were performed on about 2,900 samples.

iv) X-ray diffraction analysis:

X-ray diffraction analysis was done for determining the different phases and crystal structure of ten mineral samples from Geology Deptt. of Andhra University.

Similar such help was provided to outside industries such as Hindustan Aluminium Corpn. Ltd., Renukoot, Mirzapur (X-ray diffraction analyses of mud samples), Hindustan Photo Films Manufacturing Co. Ltd., Indunagar Utakmand, (X-ray diffractometric analyses of certain compounds synthesised in their organic Laboratory), The Madras Aluminium Co. Ltd., Mettur Dam, RS Salem Dist., Madras, (X-ray diffractometric analyses of bauxite and other aluminium ore samples). Besides the outside investigations analyses results of large number of samples from different Divisions of the Laboratory were supplied.

v) Spectrographic analysis:

Three hundred samples were qualitatively analysed. A draft on 'Standard Method of Spectrographic Analysis of Aluminium' was prepared in connection with I.S.I. - SMDC 2:2 "Spectrographic Analysis of Metals & Minerals Sub-Committee".

94.0 Treatment of Zinc Waste.

During galvanizing, considerable quantities of wastes are produced and are obtained in the form of zinc dross, ashes, skimmings and blowings which do not directly find any application. Generally, in galvanizing of tubes about 25% zinc is lost as zinc dross. The investigation aims at the recovery of metallic zinc from zinc dross for reuse in the galvanizing plant.

In the earlier experiments a recovery of 85-90% zinc could be obtained from the zinc dross samples. While carrying out distillation experiments with a sample of zinc dross received from the Post & Telegraph Department, Calcutta higher zinc recoveries between 95 and 97% was recorded as against other zinc dross samples tested so far. On analysis the dross was found to contain small amounts of aluminium that was not found in the samples of zinc-dross received from other sources. A few experiments were carried out to study the effect of aluminium in the dross on the distillation rate.

The dross was melted with different amounts of aluminium in a refractory crucible and thereafter charged inside a 2 feet dia. M.S. Vacuum Pot designed and fabricated at the National Metallurgical Laboratory.

Table 55 - Results of treatment of zinc waste.

Exp. No.	Period of distillation (in hrs.)	Pressure at which distillation was carried out. (in mm of Hg)	% Zn recovery	Al% in the dross
45	19	0.3 - 0.4	46.44	5.0
46	12	1.0 - 1.7	38.19	0.5
47	12	0.2 - 1.5	60.89	0.3

Aluminium addition had no appreciable effect on the rate of zinc distillation. Further experiments are in progress after incorporation of a few modification in the pot design, based on the studies so far carried out.

94.1 Treatment of Zinc Base Die Casting Scrap Alloys.

Zinc base die casting alloys of zamak group contain extremely small quantities of impurities like Fe, Pb, Cu, Sn and Cd. Scraps produced during melting and casting are collected together for reclamation. The re-melted products generally do not conform to the rigid specifications and need refining. A few samples of such remelted scraps were received from the Defence organization for refining to the specified limits for re-use in die casting alloys. In the samples, received, all the impurities except Sn and Pb were above the specified limits. Tin and lead contents were in the region of 0.0024 - 0.0097% and 0.015 - 0.01% respectively.

The samples were crushed in a jaw crusher followed by ball milling to liberate the metallic particles from the fluxes. The crushed product was seived through 10 mesh seive. Most of the metallic aluminium was in the +10 mesh fraction and the seived product was melted with suitable fluxes. The results are given in Table 57.

Table 57 - Results of experiments on recovery of aluminium from aluminium dross.

Sl. No.	% metals recovered.	% metallic aluminium lost in the -10 mesh fraction.
1.	61	3.5
2.	62.5	4.0
3.	57.0	4.0
4.	56.5	6.5
5.	48.0	5.8
6.	59.0	-
7.	53.0	-
8.	53.0	-

The recovery varied from sample to sample i.e. from 48% to 62%, depending on the metallic content and the proportions of gangue materials associated with each sample.

94.3 Recovery of Metals from Leaded Brass.

Unserviceable leaded brass scrap containing Cu 60%, Zn 38%, Pb 2% with traces of iron was received from Ordnance Factory, Katni, Madhya Pradesh to investigate the possibility of recovery of electrolytic copper.

Experiments were carried out for electrolytic recovery of Cu after partial removal of zinc from the leaded brass. The residue left after partial removal of zinc contains 2.6% Zn, 0.6 Pb and rest copper.

Electrolysis was carried out at 6 volts and a current density of 10 amps/sq.ft. Pure electrolytic copper was obtained with a current efficiency of 87%. The purification of $ZnSo_4$ left after the electrolysis is under progress.

95.0 Tailing Sands from Kolar Gold Field.

(Utilisation of the tailing sand from Kolar Gold Field).

The Kolar Gold Mining Undertaking of the Government of India is producing about 1200 tons of the tailing sands per day. The undertaking wishes to find profitable outlet for this waste material. So this investigation was taken up on their request. The scope of this project is to study their raw material and to suggest a commercial outlet for the same.

As the tailing sands were low fusible with a very short vitrification range, some bodes were made with clay additions to see their suitability as a raw material for stone ware pipes fired between 900-1100°C and subsequently salt-glazed. Report on this investigation has already been submitted to the sponsorers.

PILOT PLANTS

96.00 Low Shaft Furnace Project:

During the period under review, extensive investigations on the smelting of iron ore fines, fluxes and low-temperature carbonized coke made from wholly non-coking coals from different collieries were undertaken with additional alterations in the operational parameters. It has been reported earlier that despite the short height of the stack, the temperature of the top gas can be lowered to reasonable limit by the suitable adjustment of the particle size of the ore and flux, particularly the ore. It has been conclusively proved that the oxygen enrichment of the blast with consequent reduction of the gas volume was not essential for iron smelting in a low-shaft furnace. However, during the smelting trials with blast additives, such as fuel oil and naphtha with simultaneous enrichment of the blast with oxygen, considerable improvement in the smelting efficiency, as reckoned by the decrease in coke rate and the increase in productivity, was noticed. It was, therefore, considered that the effect of different degrees of oxygen enrichment of the blast, in the absence of any auxiliary fuel injection on the smelting parameter will yield useful data for comparison and investigations were conducted with different burden schedules and oxygen enrichment of the blast by 0.5, 1.0, 2.0 and 3.0% percent in different stages.

Besides, the extensive trials on ironmaking to evaluate the possibilities of exploitation hitherto unused ores, coal and fluxes in the low-shaft furnace pilot plant, substantial progress has been made on the diverse industrially oriented investigations concerned with iron and steel making, which were undertaken with the object of development of commercially acceptable efficient techniques, based on the economic utilization of available raw-materials and metallurgical wastes. However, fundamental ferrous metallurgical problems were not totally divorced and a few of them were undertaken to elucidate the phenomenon observed in the pilot plant trials, or from the future developmental purposes. Apart from the investigations, maintenance and modifications of the various components comprising a heavy pilot plants were simultaneously done.

Programme of Research and Development work in the Low-Shaft Furnace Pilot Plant.

In order to conduct comprehensive smelting trial for the development of a suitable alternative process for ironmaking with raw-materials available in India, an exhaustive programme was drawn up as given in Phases I to VI.

Phase I

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

ii) Iron ore from Chanda district and non-coking coals from Ballarpur, Kamptee and Wardha Valley, Maharashtra State.

iii) Iron ores from Chapra, Antribeharipur in Mohindergarh Punjab, with nut coke or non-coking coals.

iv) Iron ores from Anantpur, Warrangal etc. with non-coking coals (and low-temperature carbonized coke made thereof) from Kothagudem, Yellandu etc. in Andhra Pradesh.

v) Iron ore from Katni in Madhya Pradesh with non-coking coals from adjacent coalfields (Kanhan and Pench Valleys).

vi) Iron ores from Nathara-ki-Pal near Udaipur and Morinja (Chomu-Samod) near Jaipur, Rajasthan, initially with nut coke and then with high temperature carbonized Palana lignite (as and when it becomes available) vide Phase III (i).

Phase II

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

Phase III

i) Utilization of lignites after its high temperature carbonization for iron smelting.

ii) Utilization of Salem magnetite after its beneficiation and agglomeration by pelletizing or sintering in conjunction with carbonized lignite briquettes.

Phase IV

Research and development work on oxygen-enrichment of air blast, direct injection of naphtha and furnace oil, thorough auxiliary tuyeres with simultaneous enrichment of air blast with oxygen and study of overall economics of production of such operations.

Phase V

Utilization of agglomerated fine grained soft iron ores, flue dust in the form of sinter, self-fluxed sinter, pelletised or briquetted ore burden 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.

It may be mentioned here that investigational work on Phase 1(V), Phase III (i) and (II) and Phase V could not yet be undertaken for non-availability of the requisite raw-materials. Apart from the main objective of iron smelting with non-coking coals, it has been used as an experimental blast furnace to study the smelting characteristics of hitherto unused ores and fluxes, study of the effect of variation of the operational conditions on the smelting parameter and for studying the feasibility of injecting auxiliary fuels. Several Feasibility Reports incorporating economic viability of the smelting process utilizing regional raw-materials have been issued.

The investigations on iron-making undertaken during the period under review can be broadly classified in the following campaigns:-

FORTYFIRST CAMPAIGN

Smelting trials were conducted with two different varieties of iron ores (Orissa Minerals) and mixed ores with subsize coke, limestone (Maihar, M.P) and dolomite (Assam) with the object of assessing the effect of oxygen enrichment of the blast on the smelting characteristics.

FORTYSECOND CAMPAIGN

Smelting trials were conducted with two different varieties of iron ores (Bolani) and mixed ores with nut-coke, low-temperature carbonized coke made from totally non-coking coal(Singareni), limestone (Satna) and dolomite(Assam) to find out the compatibility of these ore fines with a non-metallurgical fuel, and also the effect of different degrees of oxygenation of the blast on the smelting characteristics.

DETAILS OF FURNACE CAMPAIGNS

The burden comprised of iron ore fines (Orissa Minerals), 91% below -12 mm, or mixed iron ore 66% below -12 mm, subsize coke of particle size range of 60-75% below -25 mm and limestone. A certain amount of dolomite was also added to the burden. The chemical and physical characteristics of these raw-materials are given in Tables 58 and 59 respectively.

Table 58 - Chemical Analysis of Raw-materials.

1. Chemical Analysis of Iron Cre:

	Fe%	SiO_2%	Al_2O_3%	S%	P%
Iron ore (Crissa Mineral)	64.14	3.28	4.57	0.01-0.03	0.02-0.04
Mixed ore (M.B.P.P)	66.00	1.48	3.20	Trace	0.04

2. Analysis of Fluxes

	CaO%	SiO_2%	Al_2O_3%	MgO%	Fe_2O_3%
Limestone (Dutta Chowdhury)	52.0	2.30	1.85	1.16	0.55
Dolomite (Assam)	31.30	0.63	0.40	20.70	-

3. Analysis of Fuel

	V.M.%	F.C.% (by diff.)	Ash%	S%	Moisture%
Nut coke (Durgapur)	2.3	70.7	24.7	0.5	2.3
Nut coke (TISCO)	1.5	74.4	22.9	0.5	1.2

4. Ash Analysis of Fuel:

	CaO%	SiO_2%	MgO%	Al_2O_3%	Fe_2O_3%	P%
Nut coke (Durgapur)	3.1	50.9	1.9	25.6	13.9	0.27
Nut coke (TISCO)	3.8	52.08	2.12	33.0	-	0.69

Table 59 - Screen Analyses of Raw-materials Employed:

Raw-materials	+50 mm %	-50 mm +25 mm %	-25 mm +12 mm %	-12mm +6 mm %	-6 mm %	-3mm %
Nut coke (Durgapur)	-	18.83	59.00	6.41	2.58	13.18
Nut coke (TISCO)	-	18.20	74.00	4.50	0.60	2.70
Iron ore (Crissa Mineral)	-	-	8.80	41.60	30.33	13.27
Mixed ore (MBPP)	-	1.10	31.40	26.50	14.60	26.40
Limestone (Dutta Chowdhury)	-	36.20	42.20	10.45	4.36	6.79
Dolomite (Assam)	-	-	83.00	2.50	8.80	5.70

The mixed iron ore has slightly higher iron content and little lower silica. The reducibility characteristics on particle size of -13 + 6 mm of both the ores were almost identical. Therefore, it was considered that replacement of one ore with the other did not seriously affect the operational variables in assessing the behaviour of oxygen enrichment of the blast on the smelting efficiency.

As is normally practised, the burden schedule was drawn up on the calculated basicity degree of the slag CaO/SiO_2 of 1.2 to 1.3 and enough dolomite was added to yield 6-8% MgO in the slag. The furnace was operated under the following conditions:

Tuyere diameter, mm	75
Hot blast temperature °C	500 - 550
Hot blast pressure mmWG	1400 - 1600
Hot blast volume Nm^3/hr	2250 - 2500

The variation of the hot blast temperature, pressure and volume were kept within the limits indicated above. The operation with a definite oxygen enrichment of the blast was continued for a sufficiently long duration for attaining stable smelting condition, which assured correctness of the data and the conclusions derived thereof. The operational characteristics are recorded in Table 60.

Compared with base period operation without oxygen enrichment of the blast, 0.5% oxygen enrichment did not significantly alter the fuel rate, slag rate and productivity. With 1.0% oxygen enrichment, however, a distinct improvement in the regularlity of the discent of the burden coupled with lowering of the fuel rate and increase in productivity were noticed. However, in order to fully assess the effects of oxygen enrichment on the smelting efficacy, another comprehensive trial was considered necessary.

FORTYSECOND CAMPAIGN

In this campaign, iron ore fines (Bolani), 87% below -12 mm, mixed iron ore 57% below -12 mm, low temperature carbonized coke made from Singareni Colliery, limestone (Satna) and dolomite were employed for smelting with the object to determine the effect of oxygen enrichment of 1.0, 2.0 and 3.0% respectively on the smelting parameter. The chemical analyses and the particle size classification of the raw-materials are recorded in Tables 61 and 62 respectively. The Bolani iron ore was somewhat inferior to the mixed variety.

Table 60 - Operational Characteristics with 0.5 and 1.0% Oxygen Enrichment of Blast.

Data on	Oxygen enrichment	
	0.5%	1.0%
Fuel Rate F.C. tonnes/ tonne of pig iron.	1.87 - 2.0	1.86
Slag volume tonnes/tonne of pig iron	1.14 - 1.17	1.10
Av. top gas temperature °C	500	450
Average flue dust %	4.5	5.0
Pig Iron analysis, %		
C	2.5 - 2.75	2.5 - 2.7
Si	3.5 - 4.5	3.5 - 4.2
S	0.04 - 0.08	0.03-0.08
P	0.23	0.24
Mn*	0.24	0.25
Slag Analyses, %		
CaO	35 - 37	37 - 38
SiO_2	34 - 35	33 - 34
FeO	0.5- 1.2	0.5- 1.5
Al_2O_3	22 - 23	22 - 23
MgO	5 - 6.5	5 - 7
Average Top Gas Analysis, %		
CO	25 - 26	25.5
CO_2	3.65	3.6
CH_4	3.5	3.55
H_2	Nil	Nil

* Manganese Ore was not added in the burden.

Table 61 - Chemical Analyses of Raw-materials Employed

1. Chemical analysis of iron ore.

	SiO_2%	Fe%	Al_2O_3%	S%	P%
Mixed Variety (from MBPP)	1.48	66.00	3.20	Trace	0.04
Iron ore (Bolani)	5.0	57.0	6.5	0.03	0.05

Analysis of Fluxes

	CaO%	SiO_2%	Al_2O_3%	MgO%	Fe_2O_3%
Limestone (Satna)	48.3	3.5	0.9	3.0	0.34
Dolomite (Assam)	31.30	0.63	0.40	20.70	-

Analysis of Fuel.

	V.M.%	F.C.%	Ash%	S%	Moisture%
Nut coke (TISCO)	1.5	74.4	22.9	0.5	1.2
Kolsit	9.2	69.1	19.3	-	2.4

Ash Analysis of Fuel.

	CaO%	SiO_2%	Mg.O%	Al_2O_3%	Fe_2O_3%	P%
Nut coke (TISCO)	3.8	52.08	2.12	33.03	-	0.68
Kolsit	0.7	66.3	2.2	23.3	-	0.023

Table 62 - Screen Analyses of Raw-materials Emelted.

Raw-materials	-50mm	-50mm +25mm	-25mm +12mm	-12mm +6 mm	-6mm	-3mm
1. Nut coke (TISCO)	5.91	46.55	45.60	1.88	0;03	0.03
2. Kolsit	11.61	57.12	24.51	3.82	1.36	1.58
3. Iron ore(Mixed)	-	1.74	41.46	28.56	12.11	16.13
4. Iron ore (Bolani)	-	-	12.60	35.06	18.84	33.50
5. Limestone (Satna)	-	45.22	43.90	6.44	2.93	1.61
6. Dolomite (Assam)	-	1.1	92.0	6.0	0.9	-

As in the previous trials, the burden was made up for a calculated slag basicity degree CaO/SiO_2 of 1.2 to 1.3 and enough dolomite was added to yield 6-8% MgO in the slag. The furnace was blown under the following operational conditions:-

Tuyere diameter, mm	75
Hot Blast temperature °C	500 - 600
Hot Blast Pressure, mmWG	1400 - 1800
Hot Blast Volume Nm^3/hr.	2200 - 2550

At the initial stage, the smelting operation was stabilized with mixed iron ore, employing nut coke as the fuel and reductant. Subsequently Bolani iron ore was employed in which nut coke was progressively replaced in stages of 25%, 50%,75% by the 'char'. The movement of the burden with 75% char in the fuel burden was smooth and there was no operational fluctuations. In fact the fuel rate was reduced by the replacement of the nut coke with the char. Subsequently the enrichment of the air blast with 1.0%, 2.0% and 3.0% oxygen on smelting behaviour was studied in three distinct stages with a fuel burden comprising 75% char and 25% nut coke. The operational results are summarised in Table 63. The suitability of low-temperature carbonized coke made from the non-coking coal of Singareni Colliery for iron smelting in a low-shaft furnace has been indicated earlier. In this trial it has been further confirmed that it can be used with other varieties of fine grained iron ores.

It may be observed that the oxygen enrichment of the blast reduced the top gas temperature slightly and there was a minor reduction in the fuel consumption. However, the only benefit of the increase in output may not justify the additional cost of oxygen enrichment of the air blast. In fact oxygen enrichment in the absence of auxiliary fuel injection is not recommended.

Scrap Oxygen Steelmaking in Basic-Oxygen Converter.

It has been mentioned earlier that extensive investigations are being done to evolve a suitable technique for the conversion of solid iron and steel scrap generated in foundries to molten steel of desired composition by a modified basic top blown oxygen steel making technique. Comprehensive data based on the previous trials have indicated the technical feasibility and commercial adaptability of the process, but further trials were undertaken.

It is understandable that the size of the scrap added to the normal L-D steelmaking vessel depends on the heat size. Although the experimental basic-lined converter was small (diameter at mouth 150 mm, diameter of the vessel 300 mm for treating 100 Kg hot metal), it is considered that the

Table 63 - Consolidated Operational Data with Progressive Increase of 'Char' in Fuel Burden and Different Degrees of Oxygen Enrichment.

Operational data	Kolsit in Burden			Kolsit in burden 75%, Oxygen Enrichment %		
	25%	50%	75%	1.0	2.0	3.0
Fuel Rate F.C. t/ tonne of pig iron	2.00	1.35	1.55	1.45	1.42	1.4
Slag volume tonnes/ tonne of pig iron	1.6	1.55	1.4	1.31	1.31	1.22
Average Top Gas temperature °C	520	500	470	450	420	400
Average flue dust%	2.5	2.8	3.4	4.5	4.75	4.75
Average Pig Iron analysis, %						
C	2.85	2.35	2.9	2.75	2.9	2.5
Si	3.7	4.9	4.7	4.2	4.1	4.4
S	0.02	0.03	0.03	0.02	0.01	0.06
P	0.22	0.23	0.22	0.24	0.22	0.23
*Mn	0.24	0.25	0.24	0.25	0.23	0.24
Average Slag analysis, % CaO	36.3	36.5	36.6	35.2	37.7	35.2
SiO_2	29.9	33.0	31.1	32.0	36.2	34.6
FeO	1.0	1.0	0.2	0.7	0.5	0.3
Al_2O_3	22-24	23-24	22-23	22-25	22-24	23-25
MgO	5-6	6-7	5-7	6-8	5-7	6-8
Average Top Gas Analysis, %						
CO	25.1	25.9	25.2	27.4	27.2	27.2
CO_2	3.4	3.2	3.5	3.8	4.5	4.2
CH_4	3.0	3.5	4.0	3.6	3.6	3.4
H_2	0.1	0.2	0.3	0.3	0.3	0.3

* Manganese Ore was not added in the burden

acceptability of different sizes of pig iron scrap should be investigated particularly in relation to the time required for melting influencing the tap to tap time and oxygen consumption. The effective size of the vessel limited the charging of a maximum size of scrap of 10 mm x 5 mm. The larger size of the scrap, higher will be the void volume inside the vessel and, therefore, for melting the same quantity, frequent charge addition will be necessary. The chemical analyses of the scrap and the additions of limestone etc. were kept identical in all the experiments. The operational results are given in Table 64.

Table 64 - Dependance of melting time and oxygen consumption on the scrap size.

Size of the Scrap	O_2 Consumption in Cu meter	Time in minute.
2.5 cm x 2.5 cm	16	25
3.75cm x 2.5 cm	16	25
5 cm x 2.5 cm	18	29
8 cm x 2.5 cm	19	32
10 cm x 5 cm	22	40

From the experimental results, it was observed that the oxygen consumption and the time of conversion of the scrap to molten steel analysing on average 0.11% C, 0.10% Si, 0.02% S, 0.01% P increased with the size of the scrap. It was also noticed that the increase in oxygen requirement and time were related to initial melting of the pig iron scrap, while the refining time was identical irrespective of the size of scrap. It conclusively proved that scrap of assorted size generated in a foundry can be absorbed in this process provided the size of the scrap was suitable for charging in the vessel. Further experiments with other types of fuels like the low-temperature carbonized coke, non-coking coal, calcium carbide are under progress.

Experimental Baby Blast Furnace:

It has been mentioned earlier that the objective of iron smelting in the baby blast furnace is to comprehensively evaluate the effect of burden preparation, oxygen and fuel injection on the fuel rate and productivity. A similar work in a commercial blast furnace will not only involve heavy expenditure, but also will be impracticable, it is believed that the data obtained can be reasonably extrapolated to estimate the benefit of burden preparation on blast furnace operation.

Employing iron ore fines, limestone and low-temperature carbonized coke, the effect of oxygen enrichment of blast upto 3% in three different stages was evaluated under identical conditions for comparison with the data obtained that of nut coke as fuel. The chemical analyses and the particle size classification of the raw-materials used were given in the previous report. The operational conditions and the experimental results are given in Table 65.

Table 65 - Smelting Behaviour with Different Percentages of Cxygen Enrichment using Low-temperature Carbonized Coke.

Data on	Cxygen enrichment of air blast			
	Nil	1% O_2	2% O_2	3% O_2
Fuel rate/tonne of pig iron (fixed carbon)	1.45	1.35	1.30	1.01
Rate of smelting ore fed/Hr. (in Kg.)	20	22	26	36
Slag volume t/tonne of pig iron	1.10	1.00	1.00	0.8
Average Hot Blast Temp. °C	280	280	280	280
Average Hot Blast Pressure mmHg.	30	40	30	30
Average Pig Iron analysis %				
C	2.0	2.19	2.0	2.2
Si	1.20	0.80	1.1	1.3
S	0.26	0.27	0.26	0.19
P	0.40	0.50	0.48	0.45
Average slag analysis, %				
CaO	37.62	32.30	32.80	33.12
SiO_2	31.04	32.60	32.00	31.48
FeO	2.10	3.00	2.00	2.70
Al_2O_3	20.90	18.30	19.00	19.08
MgO	9.20	8.30	9.00	8.65

Despite the small quantities involved, the separation of the slag and metal was satisfactory. From the experimental results it was found that with the progressive increase in oxygen enrichment, the rate of smelting increased and the fuel rate decreased, which was related to low blast temperature.

Further experiments on oxygen enrichment upto 7% with the same type of raw-materials, employment of sinter, pellets etc. are under progress.

97.0 Investigations on Cupola Melting Practise:

Oxygen Enrichment of the Air-Blast:

In view of the difficulties of iron smelting in cupola with high ash Indian coke, systematic investigations were conducted with the object of studying the effects of oxygen enrichment of the air blast on carbon saturation in the metallic charge, containing optimum amount of steel scrap for obtaining hot metal of desired chemical composition for making special castings. Incidentally the effect of oxygen on melting rate and other associated factors were studied.

The investigations were conducted under identical conditions with a coke to metal weight ratio of 1:3 and 50% steel scrap in the charge, both in the acid and in basic-lined cupolas. In the acid lined cupola, the basicity degree CaO/SiO_2 of the slag was maintained at four different levels of 0.6, 0.8, 0.9 and 1.0 by adjusting the limestone addition to the charge. The blast was enriched by 1% and 2% respectively in two stages. It was observed that carbon contents of the metal increased with the increasing slag basicity ratio and also with higher oxygen enrichment. The temperature of the hot metal was raised by 25 and 50°C at 1% and 2% enrichment respectively. It was, however, noticed that the acid refractory lining was attacked severaly and consequently the further trials were conducted with the basic-lined cupola and the basicity ratio of the slag was maintained at 1.0, 1.1, 1.2 and 1.3 in different trials. The oxygen enriched at three different levels of 1%, 2% and 3% respectively. With 3% oxygen enrichment, the rise in temperature of hot metal was 70°C. It was observed that the same carbon content of 2.61 at a slag basicity of 1.3 with a normal air blast can be obtained with 1%, 2% and 3% enrichment of the blast and slag basicities of 1.2, 1.12 and 1.08% respectively. The saturation of carbon was, therefore, dependant on the basicity degree of the slag as well as oxygen enrichment of the air blast as shown in Fig.40 With oxygen enrichment to the extent of 3%, 100% steel scrap can be successfully melted to produce hot metal of desired chemical composition as shown in Fig. 41. Although almost linear relationship of the rise in metal temperature with oxygen enrichment was initially observed, higher enrichment did not show proportional rise in temperature. The oxygen enrichment lead to absorption of larger amount of steel scrap and consequently lowering of phosphorus in hot metal, thereby making it suitable for making special castings.

Heading Arrangement at the Top:

Bearing in mind the advantages of cupola iron melting with preheated air blast, various types of simple preheating devices are being designed and tried. In a cupola provided with preheating facilities at the top, further experiments were conducted with coke: metal weight ratios of 1: 9 and 1:11 with variation in the hot blast temperature between 100°C - 400°C and the results are shown in Fig. 42. The rise in temperature of the hot metal justifies the incorporation of the facility for heating the blast. Further investigations are being done.

Utilization of Heat lost by Radiation:

A simple ingenious process for preheating the air blast without the consumption of additional fuel has been designed and patented (No. 112574). It can be easily incorporated in the existing cupola with minor modification. The temperature of the air blast can be raised to 350°C and substantial amount of coke can be saved. Alternatively, the metallic charge can comprise of pig iron and steel scrap, which leads to production of low-phosphorus hot metal suitable for making malleable and other special castings:

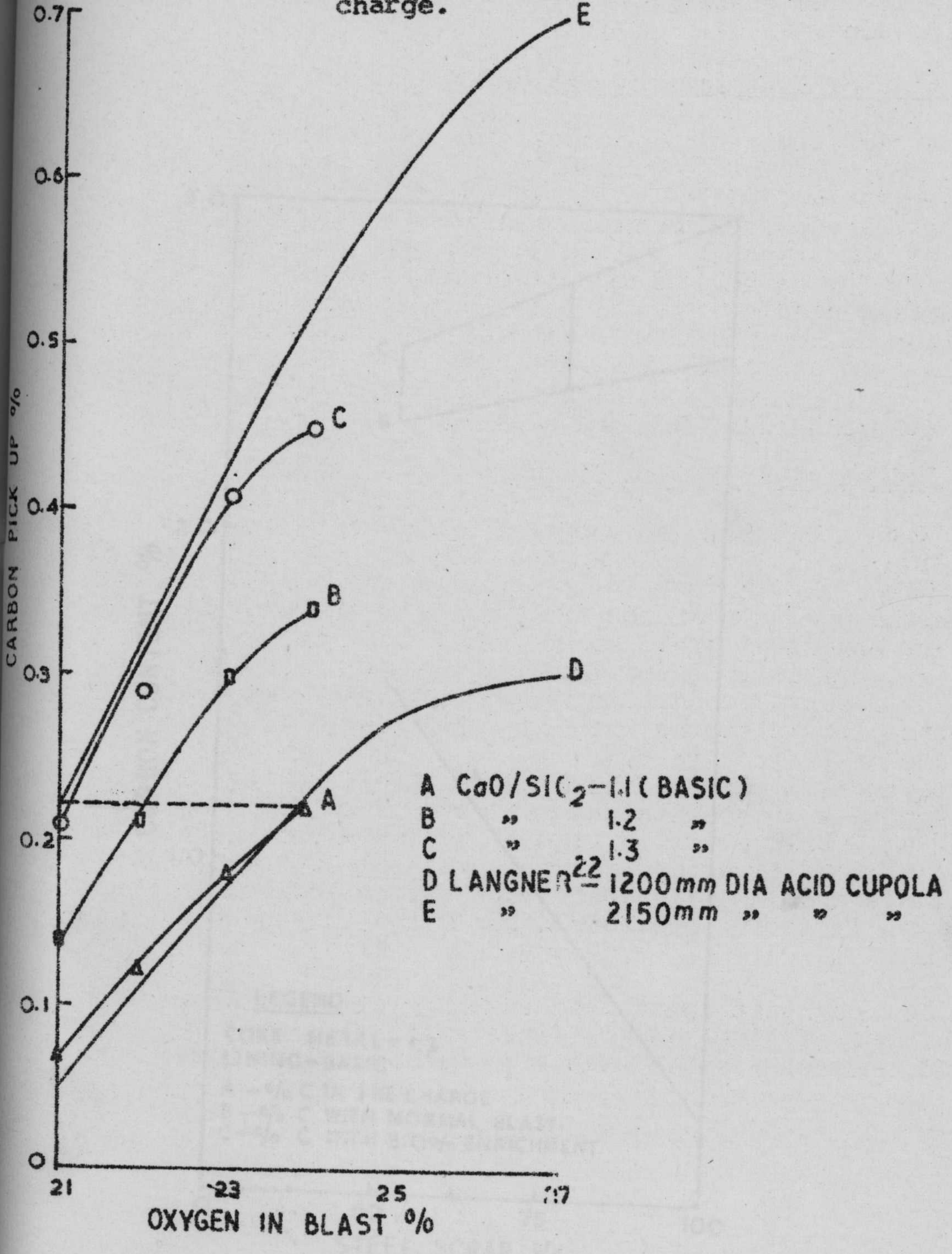
98.0 Investigation on Agglomeration:

(i) Briquetting of Lime Sludge:

With a view to utilize lime sludge containing 56% CaO, 1.32% SiO_2 and 1.56% Al_2O_3 in the form of briquettes for steel-making purposes, further investigations were conducted to improve the strength of the briquettes by the addition of limestone having of grain sizes (-1.00+0.50 mm) and (-0.50 + 0.252 mm). Apart from the variation in particle size, the amount of addition was also varied. Lime sludge briquettes were prepared with the addition of 10 and 20% limestone of grain size (-1.0 + 0.50 mm), employing 6% molasses and 10% moisture as binders. The shatter index of the briquettes in green condition were 15 to 20% respectively on + 12 mm. Briquettes of lime sludge were made with 10 and 20% addition of limestone of grain sizes (-0.50 + 0.252 mm), 6% molasses and 10% moisture. The shatter indices of these briquettes in green condition were 18 and 22 respectively on + 12 mm.

Although the shatter index was slightly improved by the addition of limestone of finer grain size, yet the physical characteristics were somewhat unsatisfactory.

Fig.40 Carbon content of the melt with different amount of steel scraps in the charge.



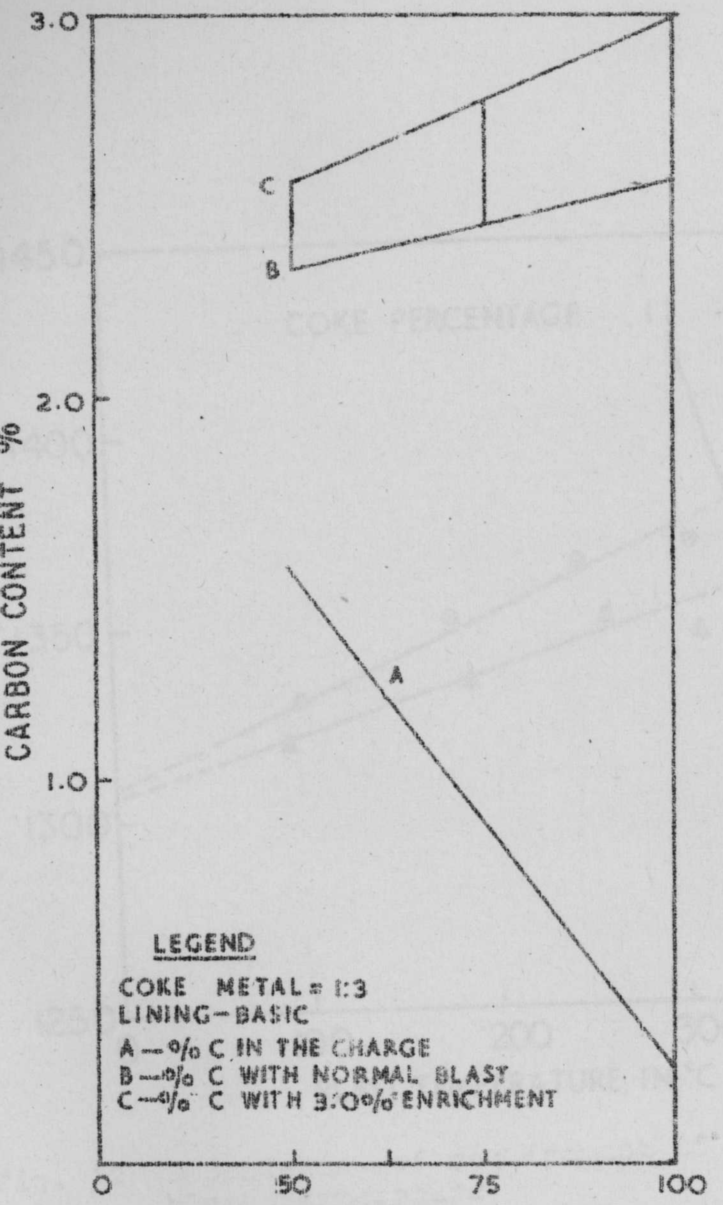


Fig.41 Effect of increasing O_2 enrichment on the carbon saturation at increasing basicity degree of slag.

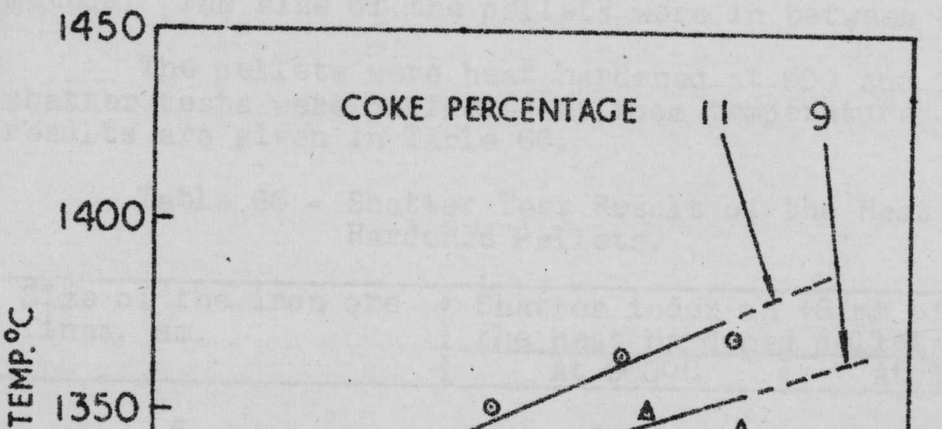


Fig. 12 Dependance of requirement of coke on blast temperature

It was felt that the physical strength of the lime-sludge briquettes might be improved on heat hardening after the incorporation of small quantity of fine grained mill-scale. Accordingly lime sludge briquettes were prepared with the addition of 5, 10, 15 and 20% mill scale and the briquettes were heat hardened at 200, 400 and 600°C but the physical strength did not improve.

(ii) Pre-reduced Self-fluxed Pellets:

a) Size Requirement:

In order to incorporate the optimum amount of flue dust sludge from the wet gas cleaning plant etc. investigations on the pelletization of iron ore fines were conducted. Besides it was desired to develop self-hardening pellets and a technique of making self fluxed prereduced pellets.

In order to determine the optimum size of iron ore fines, iron ore was crushed to different particle sizes of -6, -3 and -1 mm.

Pellets were prepared from the iron ore fines with the addition of 0.5% bentonite and 15% moisture by manual balling method. The size of the pellets were in between (-12 + 6 mm).

The pellets were heat hardened at 900 and 1000°C and shatter tests were performed at room temperature. The results are given in Table 66.

Table 66 - Shatter Test Result of the Heat Hardened Pellets.

Size of the iron ore fines, mm.	Shatter index on +6 mm of the heat hardened pellets.	
	At 900°C	At 1000°C
-6	69	80
-3	80	87
-1	82	89

b) In order to study the progress of reduction during the process of heat-hardening, iron ore fines (-3 mm) and 10% coke breeze (-3 mm) were pelletized with addition of 0.5% bentonite and 15% moisture. The heat-hardening and prereduction were performed in an inert atmosphere. However, the physical strength of the pellets was not satisfactory.

Attempts are made to incorporate flue dust in the pellets. The chemical and sieve analysis of the flue dust are given in Tables 67 and 68.

Table 67 - Chemical analysis of the flue dust.

L.C.I. %	CaO %	SiO_2 %	FeO %	MgO %	Al_2O_3 %
43.19	6.29	25.34	12.00	2.98	9.70

Table 68 - Sieve analysis of Low-shaft Furnace Flue Dust Sample in mm pct.

-2.41 +1.00	-1.00 +0.50	-0.50 +0.252	-0.252 +0.152	-0.152 +0.076	-0.076
0.4	3.6	26.7	14.2	29.2	25.9

As the pellets made from flue dust exclusively with binder addition and 20% moisture did not develop adequate strength, further investigations will be done on pellets containing optimum amount of flue dust. Attempts are also made to develop a technique of making self-hardening pellets.

99.0 Malleablization Characteristics of Low-Shaft Furnace Pig Iron.

It has been mentioned earlier that low-phosphorus pig iron made with low-temperature carbonized coke from fully non-coking coals, leads to the production of low-phosphorus pig iron which is more suitable for malleabilization. It is also known that certain elements like copper, boron, aluminium etc. affect the malleabilization cycle. With a view to reduce the malleabilization cycle, the effect of addition of varying amounts of boron is being studied dialatometrically. The composition of the heats are given in Table 69.

Table 69 - Chemical Analyses of Casts Cotaning

Heat No:	Chemical Composition, %						
	C	Si	P	S	Al	B	Mn
1	2.25	0.96	0.165	0.06	0.10	0.011	0.45
2	2.25	0.96	0.165	0.06	0.10	0.023	0.45
3	2.25	0.96	0.165	0.06	0.10	0.031	0.45
4	2.25	0.96	0.165	0.06	0.10	0.043	0.45

A suitable salt bath has been developed for holding the specimen at different malleabilization temperature schedules.

The effect of various other elements are being studied:

100.0 Production of Pig Iron from Run-of-mine Iron Ore, Limestone fines and coke or coal breeze in a Rotary Furnace.

Investigations on iron smelting with fine grained raw-materials with an acid lined furnace have been reported previously. Smelting trials were conducted with iron ore fines (Orissa Mineral), limestone chips (Bisra) and coke breeze in a basic (magnesite) lined rotary furnace primarily to study desulphurization and for controlling the phosphorus contents in the pig iron produced. The chemical analyses of the raw-materials employed and their screen analyses are reported in Table 70 and 71.

Table 70 - Analysis of Ore, Fuel and Flux.

1. Analysis of iron ore:

Fe%	SiO_2%	Al_2O_3%	CaO%	MgO%
64.14	3.28	4.57	Trace	Trace

2. Analysis of fuel:

	H_2O%	Ash%	V.M.%	F.C.%	S%
Coke breeze	2.50	26.0	1.51	70.10	0.50

3. Analysis of fuel ash:

SiO_2%	CaO%	Al_2O_3%	MgO%	Fe%	P%
50.90	3.10	25.50	1.80	4.80	0.27

4. Analysis of limestone:

CaO%	SiO_2%	Al_2O_3%	MgO%
44.88	6.96	1.60	3.27

Table 71 - Screen analyses of Raw-materials Employed(%)

Name of the material	-50.8 mm	-50.8 +25.4mm	-24.4 +12.7mm	-12.7 +6.35mm	-6.35 +3.17mm	-3.17mm & below
Iron ore (Crissa Mineral)	-	-	3.80	16.28	26.07	53.85
Limestone (BISRA)	-	4.65	50.72	26.87	10.70	7.06

Particle Size of Coke Breeze %

+7 mesh Sieve	-7 mesh sieve +16 mesh sieve	-16 mesh sieve +30 mesh sieve	-30 mesh sieve +60 mesh sieve	-60 mesh sieve +100 mesh sieve	-100 mesh sieve & below
9.07	21.78	26.90	15.68	12.77	13.80

The mode of conducting the iron smelting has been previously reported. It was found that after initial heating of the vessel by oil burner followed by the burning of CO to CO_2 inside the vessel by O_2 injection could not maintain the smelting temperature and, therefore, the furnace had to be heated by burning oil periodically. The pig iron analysed 3.4% C, 0.30% Si, 0.49% P and 0.07% S. Although the basicity of the slag CaO/SiO_2 was maintained at 1.2, and it contained 4-5% FeO, the dephosphorization was not pronounced, while the partition of sulphur also suffered due to high FeO contents in slag. Further investigations are under way.

101.0 Pilot Plant for Hot-dip Aluminizing:

Aluminising of Heat Resistance Wire:

Experiments were conducted for aluminising the heat resistance Fe-Cr-Al alloy wire. Specimens from 1/8" Die-wire and ¼" sq. bar of the alloy were aluminised with variation in the dipping time and temperature.

18 and 20 gauge wires were aluminised. Experiments are under progress to get the optimum condition for aluminising the wire.

Refractory lined pots:

Experiments are under progress to study the suitability of the refractory lined pots for aluminising.

Petrol Tanks:

Petrol tanks sent by Chief Inspector of Vehicles, Ministry of Defence, Ahmedabad were aluminised.

Coating wt. of Wires:

Tests were conducted to determine the coating weight of aluminised wires sent by M/s. Venkata Durga Aluminising Works, Nandigama, Vizayawada.

New Licenses:

New license was issued to M/s. Engineering Enterprises, Vijayawada.

102.0 100 Lb Electrolytic Manganese Metal Pilot Plant and 100 Lb Electrolytic Manganese Dioxide Pilot Plant.

During the year the following investigations were conducted.

(a) To examine the suitability of manganese ore received from Manganese Ore (India) Ltd., Nagpur, M.P. for production of electrolytic manganese metal.

(b) To collect operational data from the 100 Lb pilot plant for the design of an industrial electrolytic manganese plant.

(c) Studies on the reduction of manganese ore with solid fuels, viz. charcoal and coke.

(d) To design a 6 tons/day rotary kiln for reduction of manganese ore on the basis of the present 35 Lbs/hour rotary kiln.

(e) Effect of sulphur dioxide content in the electrolyte on sulphur content of deposited manganese metal.

(f) To examine the possibility of upgrading the manganese dioxide obtained as a byproduct from the production of saccharin to a product similar to chem. grade.

(a) Investigation work on the suitability of the 14 samples of manganese ore, supplied by Manganese Ore (India) Ltd., Nagpur for the production of electrolytic manganese metal, was completed and a report incorporating the results was prepared. The 14 samples received from the firm were from the following mines namely(1) Beldongri Mine I (2) East and West Tirodi Mine I(3) Sita Saongi Mine I (4) Sita Patori Mine

(5) Sita Saongi Mine II (6) Sample without identity (7) East-West Tirodi Mine II (8) Kandri Mine I (9) Ukwa Mine I (10) Kandri Mine II (11) Ukwa Mine II (12) Ukwa Mine III (13) Beldongri Mine II and (14) Munsar Mine Main Hill. The samples were crushed and ground to -35 + 100 mesh through jaw crusher, Roll crusher and pulverizer. The ground ore was reduced with coke oven gas at 600°C and cooled. The reduced ore was then leached with a solution having the sulphuric acid concentration of a spent liquor from electrolytic manganese metal cell. The results of analysis of the 14 samples are given in Table 72.

The percentage recovery of manganese, and manganese that can be recovered by treating 100 tons of ore, along with the manganese contents of each ore have been presented in Table 73. The conclusions arrived at are given below:

(i) Ore samples 12, 11 and 9 from Ukwa Mines, and 7 and 2 from Tirodi Mines have good percentage of MnO_2 of the available manganese, and have good percentage recovery of manganese. All these five samples have given more than 30 tons of recovered manganese by treating 100 tons ore. The gangue materials in the ore are not intimately mixed with manganese but only loosely in these samples, and hence after leaching, settles down quickly as fine free silica without much time loss. Ukwa Mines and Tirodi Mines samples have been found to be the very best of the 14 samples supplied and can be safely recommended for production of electrolytic manganese. Amounts of alkaline earth metal content in these ores are also fairly low excepting in sample No. 11.

(ii) The sample Nos: 3,5,6,10 and 8 as a whole have a high percentage of MnO of the available manganese, though the MnO_2 content is low.

(iii) The sample Nos. 14, 1,13 and 4 give less than 23 tons of manganese by treating 100 tons of ore and do not recommend so highly as the other groups (1) and (2) though electrolytic manganese can profitably be produced from these ores also.

(b) Pilot Plant work on production of electrolytic manganese metal.

During the period under review, the pilot plant was run to collect data for the design of an industrial unit. About 700 Kg of metal was collected with a rectifier capacity of 1800 amp. The flake metal was supplied to outside parties and was also consumed inside the laboratory for studies on manganese bearing alloys. The metal was used for making heats to prepare special steel, low alloy steels, stainless steel and low alloy impact resisting steel and preparation of pure manganese nitrate etc.

TABLE-72 ANALYSIS OF 14 SAMPLES OF MANGANESE ORE

%	1	2	3	4	5	6	7	8	9	10	11	12	13	14
Total Mn	35.67	39.33	37.24	43.30	30.45	35.03	34.38	28.1	44.17	34.88	35.41	36.86	35.25	33.63
MnO	15.06	41.0	24.50	21.66	18.28	31.50	44.84	14.76	45.60	24.16	40.96	39.45	18.27	27.31
Total Iron as Fe_2O_3	6.95	17.38	12.42	13.43	11.86	8.8	13.91	9.61	8.16	9.60	10.40	9.20	8.80	12.80
Al_2O_3	2.40	3.75	1.16	3.11	7.1	2.96	18.05	6.79	4.96	9.30	4.60	5.20	4.80	4.85
SiO_2	33.24	13.01	26.30	13.30	30.02	29.24	12.80	34.72	22.52	23.50	26.08	25.56	28.46	29.14
CaO	2.38	0.97	1.24	1.67	0.55	3.01	9.08	0.44	0.22	3.42	2.74	1.64	3.97	3.00
BaO	0.85	1.66	1.80	1.44	1.49	1.60	1.99	1.02	0.99	0.45	0.72	0.60	1.02	0.22
MgO	1.57	1.00	1.12	1.35	1.82	0.96	0.87	1.31	0.07	3.52	2.56	2.74	3.17	1.37

TABLE - 73 RECOVERY OF MANGANESE FROM THE ORES

Sample nos.	1	2	3	4	5	6	7	8	9	10	11	12	13	14
% Total manganese in raw ore	35.67	39.33	37.24	43.30	30.45	35.03	34.38	28.1	44.17	34.88	35.41	36.86	35.25	33.63
% Manganese as MnO_2 in raw ore	15.60	41.0	24.50	21.66	18.28	31.5	44.84	14.76	45.6	24.16	40.96	39.49	13.27	27.31
% Manganese MnO in raw ore					24.34	19.82		24.19	19.57					
Percentage recovery of Manganese in solution	61.43	77.67	72.3	36.63	86.70	72.86	89.98	82.67	71.05	67.4	87.0	86.1	54.3	67.50
Recovered Mn in tons per 100 tons of ore treated	21.91	30.55	26.92	15.86	26.49	26.29	30.87	23.23	31.48	23.48	30.80	32.73	19.14	22.70
% of total iron as Fe_2O_3 in raw ore	6.95	17.38	12.42	13.43	11.86	8.8	13.91	9.61	8.16	9.60	10.40	9.20	8.80	12.80
% Al_2O_3 in	2.40	3.75	1.16	3.11	7.1	2.96	18.05	6.79	4.96	9.30	4.60	5.20	4.80	4.85
% SiO_2 in raw ore	33.24	13.01	26.30	13.30	30.02	29.24	12.8	34.72	22.52	23.50	26.08	25.56	28.46	29.14

A part of the flake metal was successfully powdered in the ball mill to -200 mesh and supplied to an outside party. The gas analysis, complete chemical analysis and the sieve analysis of the powdered metal have been done.

(c) Reduction of manganese ore with solid fuels viz. charcoal and coke:

Studies on the reduction of high grade and low grade manganese ore by charcoal was completed. Work on reduction with coke is under progress. The reduction of low grade manganese ore with powdered coke alone in the rotary kiln did not give good results mostly due to low temperature inside the kiln as the present rotary kiln can attain a maximum of 600-650°C temperature only. Work is being carried out to raise the inside temperature of the kiln.

(d) Design of 6 tons/day rotary kiln:

The design of a 6 tons/day rotary kiln for reduction of manganese ore with coke oven gas on the basis of the present 35 Lb/hr rotary kiln was completed after collecting all the necessary data, such as, inclination of the rotary kiln, consumption of coke oven gas per pound of ore for reduction, revolutions per minute, system of the feeding spiral, composition of the coke oven gas etc.

(e) Effect of sulphur dioxide content in the electrolyte on the sulphur content of deposited manganese:

It has been seen that if the SO_2 gas content becomes less than 0.1 gm/litre, manganese hydroxide formation increases and the cell condition deteriorates having the metal with innumerable holes. On the other hand, it is found that sulphur content of the metal can be controlled by retaining the cathodes for more than 32 hours in cell, keeping the SO_2 content in the solution at 0.1 gm/litre. Further work is under progress.

(f) Investigation on the utilization of manganese dioxide obtained as a by product from the production of saccharin.

The manganese dioxide was received from a Calcutta firm to examine the possibility of upgrading to suitable chemical grade. Spectroscopic analysis showed that the sample contained appreciable quantities of sodium and potassium but no trace of iron and lead was found.

Chemical analysis showed:

Mn	...	39.8%
MnO_2	...	60.0%
MnO	...	2.42%
Moisture (dried at 120°)	...	17.64%
Foreign matter (by balance)	....	19.39%

It was found that the sample could be upgraded to chemical grade by suitable chemical and physical methods.

103.0 Pilot Plant for Production of Refractories:

During the period under review, work was taken up to prepare complete plans and layout for the production of dense carbon aggregate on a pilot plant scale. Work was also undertaken to produce special refractories needed in putting up kilns needed for the above production.

Fig. 43 is a lay out plan of the dense carbon pilot plant which is nearing completion. All the equipments in this plant including the electric tunnel kiln have been designed in NML and/or fabricated under the direction of NML indigenously This is probably the first pilot plant of this nature in which the design, layout construction as well as fabrication of major equipments have been handled by NML personnel.

Pilot Plant is designed for a production of 1/2 ton of dense carbon aggregate per day. In the initial stage it will utilise raw petroleum coke as base material but as soon as the laboratory experiments are completed it will employ processed Bhilai pitch and Bituminous coal as base materials. The aggregate produced is expected to replace anthracite coke and imported low ash metallurgical coke that are now being employed by the Aluminium Industry in its cathode linings. Incidentally it will also provide strong raw material for structural carbon industry, which can be expected to come into being in this country shortly.

The Pilot Plant as designed is capable not only of producing dense carbon aggregate but also of producing carbon structurals etc. at a later stage. In fact there is every hope that it will supply the need for a well equipped pilot plant to study the suitability of various raw materials and process methods on a pilot scale before the same are utilized on a industrial scale.

All the machines have been ordered for and some of them have already arrived. The installation is expected to be completed in near future. Figures 44 to 46 show the main working shed and down draft kilns under construction as well as - main working shed.

The refractory plant was busy in making special refractory shapes necessary for the construction of tunnel kiln and D.D. kiln in the dense carbon site. In the initial stage it was apparent that high alumina refractories necessary for the purpose would not only cost too much but would also need a considerable time for deliveries to start and therefore the programming of the pilot plant construction would become very difficult and indefinite. Therefore work was undertaken in the pilot plant to (a) produce high alumina cement

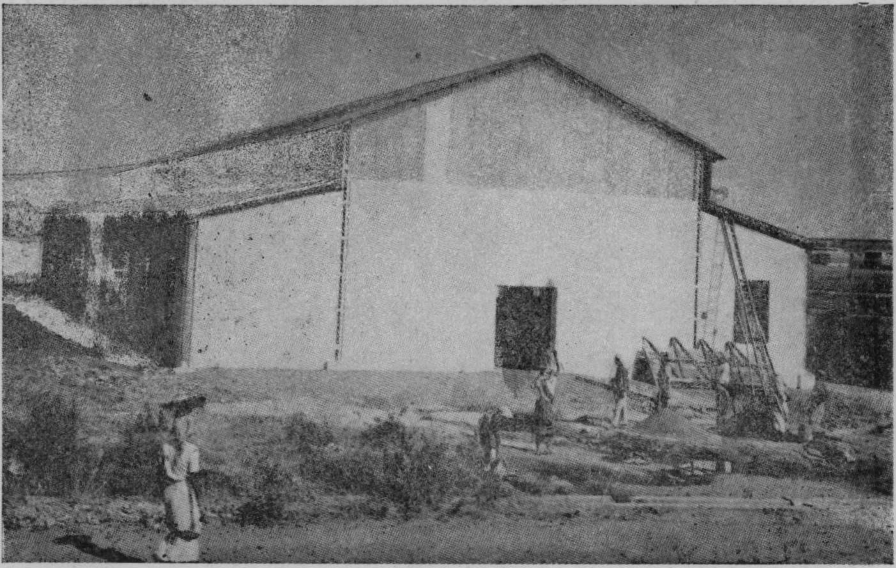


Fig. 44 Main bay of dense carbon aggregate pilot plant extension

Fig. 45 Interior of down draft Kiln under construction

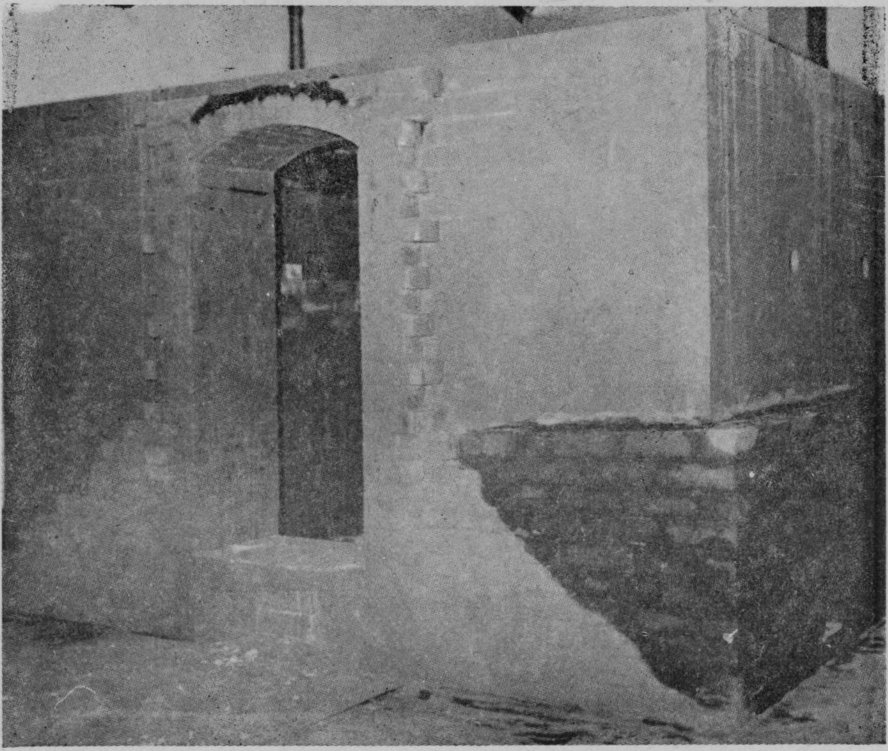


Fig. 46 Exterior of down draft Kiln under construction

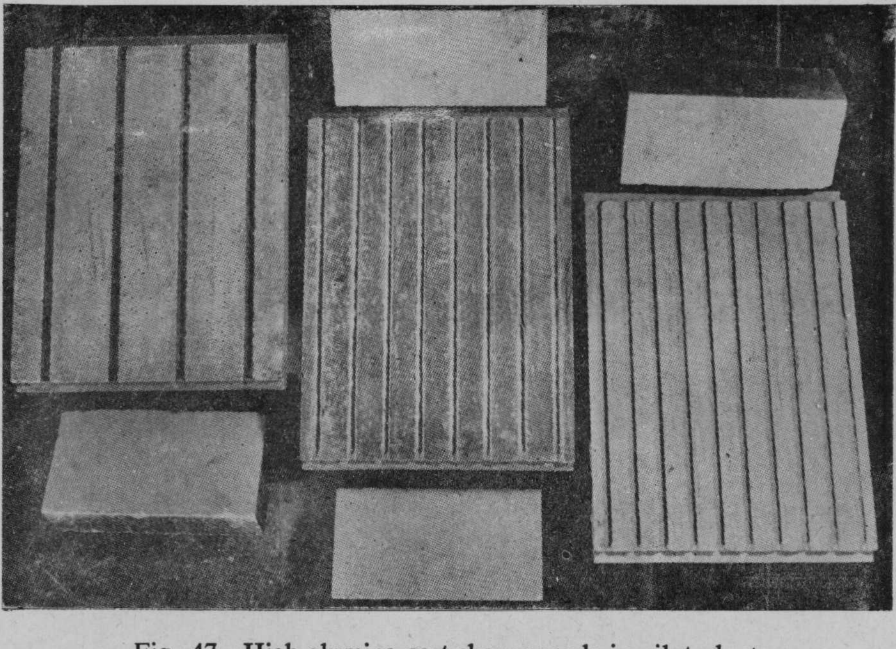


Fig. 47 High alumina cast-shapes made in pilot plant

(b) calcining of Kyanite and crushing etc. (c) formulate certain castable and rammable compositions from the above two materials and (d) fabrication of the shapes necessary for the dense carbon plant. Fig. 47 show all the shapes produced. Upto date 250 pallets of 18" x 12" were fabricated and nearly 30 pallets of 30" x 18" were made ready. High alumina bricks of 9" x 4½" x 3" are now being produced at the rate of 100 bricks per day. The savings thus effected in the production of special shapes and refractories was considerable.

104.0 Pilot Plant for Production of Synthetic Cryolite:

A pilot plant for production of 50 kg per day synthetic cryolite is under installation. Most of the heavy equipment and machinery has been obtained and being installed. The reactor vessels were damaged during transit. The damaged parts have been sent to firm for repair and awaiting receival. The drawing of steam line has been approved.

RESEARCH GROUP

HIGH TEMPERATURE CREEP RESEARCH GROUP

Development of Substitute Creep Resisting Austenitic Steels:

Previous work on the precipitation and creep characteristics of Cr-Mn-N-C system of austenitic steels had shown that these alloys can have creep strength with sufficient rupture ductility around 650°C provided the composition specially with respect to C & N and the heat treatment is properly controlled. For further improvement in the creep properties of these alloys addition of strong carbide and nitride forming elements viz. Ti, Al and W was made to the basic alloy and with the aim of also precipitation hardening through intermetallic compounds.

A number of 10 kg heats were made in high frequency induction melting furnace. The ingots of 3" square size were hot forged to bars of 1½" square size between 1200°C - 900°C. The hot-forged bars were then solution-treated at various temperatures covering a range of 1050 to 1250°C and with soaking times of 1 and 4 hr. This was done with a view to determine the correct solution temperature and time so as to obtain complete dissolution of the precipitates without excessive grain growth. This study indicated that 1200°C/4 hr. is the most suitable condition for solution treatment. These alloys after the above alloying additions were found to have considerable resistance to grain coarsening during the solution treatment at higher temperatures.

X-ray examination of the electrolytically extracted residues of the samples solution treated at 1200°C/4 hr. and aged at 700°C and 750°C for 1000 hrs. were made. The predominant precipitates were $Cr_{23}C_6$ and Cr_7C_3 along with possibly Fe_2W and the nitrides of titanium and tungsten in the respective alloys. These are being further studied.

Creep tests on the above alloys are in progress at 700°C. The results of the alloys C_6, C_7 and C_8 have been given in Table 74.

Since the creep fracture characteristic of the Cr-Mn-N-C alloys specially those with lower C and higher N are predominantly influenced by the precipitate structure obtained during aging under stress, it was thought to conduct experiment with the alloy under initially solution treated (alloy L_{2-3}) and solution treated and aged condition (alloy L_{2-4}). It can be seen from the results shown

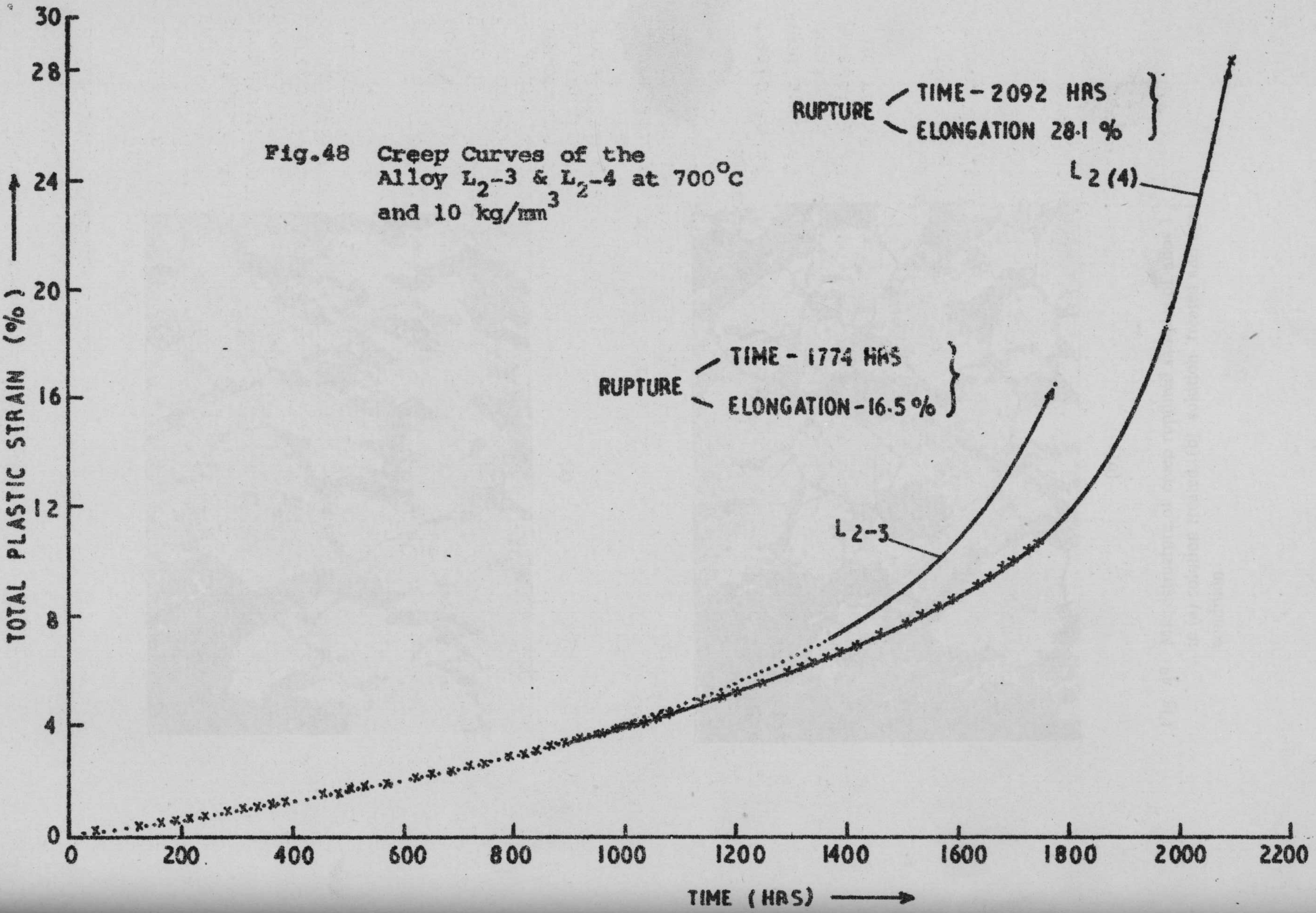
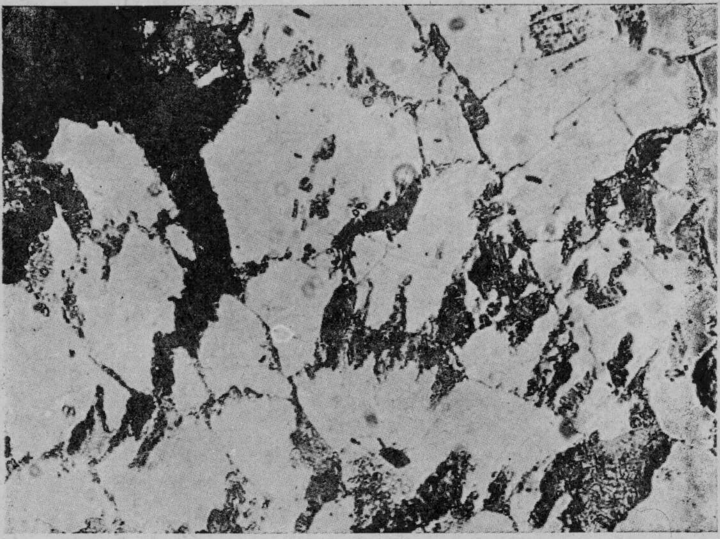
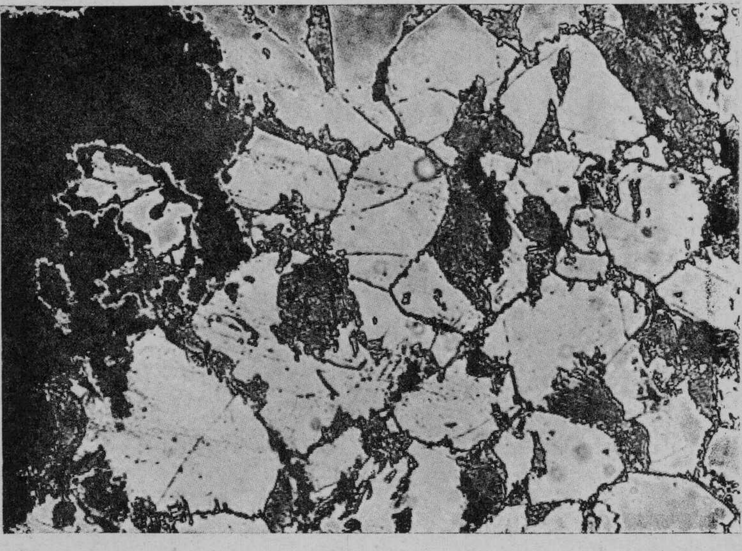


Fig.48 Creep Curves of the Alloy L_2-3 & L_2-4 at 700°C and 10 kg/mm^3



(a)



(b)

Fig. 49 Microstructure of creep ruptured samples of alloy L_2 in (a) solution treated (b) solution treated and aged condition in figure 48, that on prior aging treatment the creep-rupture life and rupture-elongation of the alloy were improved without any deterioration of the min. creep rate values. This improvement is also seen from the microstructure of the fractured specimens (Fig. 49) which shows that the fracture in the aged alloy was not so distinctly grain boundry type and the precipitate orientation not so predominantly stress - induced as in the case of the alloy given only solution treatment.

Establishment of High Temperature Creep Laboratory <u>for Long-time Creep Testing of Indigenous Alloys</u>:

With the rapid developments of the basic heavy engineering industries in the country to manufacture high temperature boilers, pressure vessels, steam turbines for power plants and gas-turbines for jet engines etc., the demand for heat and creep resisting steels and other alloys for use in these high temperature applications has immensely increased in recent years.

At present, all these alloys are imported resulting in considerable drainage of scarce foreign exchange. Serious attempts as now being made to produce these alloys indigenously. But to make the indigenous steels and alloys acceptable to the designers it is essential to determine the high temperature properties viz. the long-time creep and stress-rupture and elevated temperature proof-stress or hot-yield values which are the basic parameters used as the design criteria of high temperature equipments.

According to the agreed international standards it is essential to obtain creep test data for periods extending to over 10,000 hours for the purpose of extrapolating the data to obtain stress to produce rupture or specific creep deformation in 100,000 hours, which is normally the design life of some components. Creep tests lasting for periods over 30,000 hours is quite common practice and in a few cases even 100,000 hrs. tests have been carried out in some of the advanced countries.

To obtain these long-time data, a large number of creep test-points are required and efforts are now being made to provide the requisite testing facilities in the country.

As the creep tests are quite time-consuming and expensive, and since creep is a highly structure sensitive property, the metallurgical variables playing significant role in the long-time high temperature creep behaviour of the complex alloys, it has been considered most advisable to undertake the creep testing work at the NML in close collaboration with the manufacturers, designers, and users of the high temperature alloys in the country. This is also the practice in other advanced countries.

Even though the individual organizations may have their own creep testing laboratory for shorter-time tests on their day-to-day production materials, the long-time creep tests for design data are conducted in centralized creep laboratories, working in close collaboration with the various organizations concerned. This collaboration is sought to be established through committees as the one on boiler quality steels set-up by the Ministry of Steel, Mines and Metals and the other on high temperature bolting and other steels for use in heavy electrical plants by the Research & Development Organisation for electrical Industries.

With this end in view the existing creep testing laboratory with about 26 creep/stress-rupture test points is being suitably expanded to 150 test points in the initial stages with provision to further raise the capacity in future with the growing need for creep tests on indigenous alloys in the country.

The Creep laboratory has been planned on the pattern of modern creep testing laboratories in U.K. and other advanced countries incorporating sarfegards against power failure/shut down, mains voltage stabilization, ambient temperature regulation etc.

The creep laboratory will consist of high sensitivity single specimen double lever creep testing machines to cover the materials and test conditions with extremely low creep rates; medium sensitivity single-lever creep testing machines for materials and test condition encountering high creep rates and larger rupture-elongation; multispecimen-creep testing machines for stress-rupture tests of intermittent type to economise the tests. Besides, there will be provision for stress relaxation test on high temperature bolting materials and also special creep tests under controlled atmosphere or under vacuum for reactive metals and to study the effect of environment on creep behaviour of special materials.

High Temperature Tensile Test:

For determining elevated temperature proof stress (0.1, 0.2 or 5% off-set) or hot-yield point of the various categories of creep resisting steels and other high temperature alloys, a test set-up has been assembled in conjunction with a 50-ton Avery Universal Testing machine available in the laboratory. In this test set-up shown in Fig. 50, necessary provisions have been made for the multizone wound creep furnace, using similar temperature controlling panels as for other creep furnaces so as to allow temperature controls within ± 2°C and temperature gradient within 2°C. At present nercury relay type proportional temperature is being used but it is to be subsequently

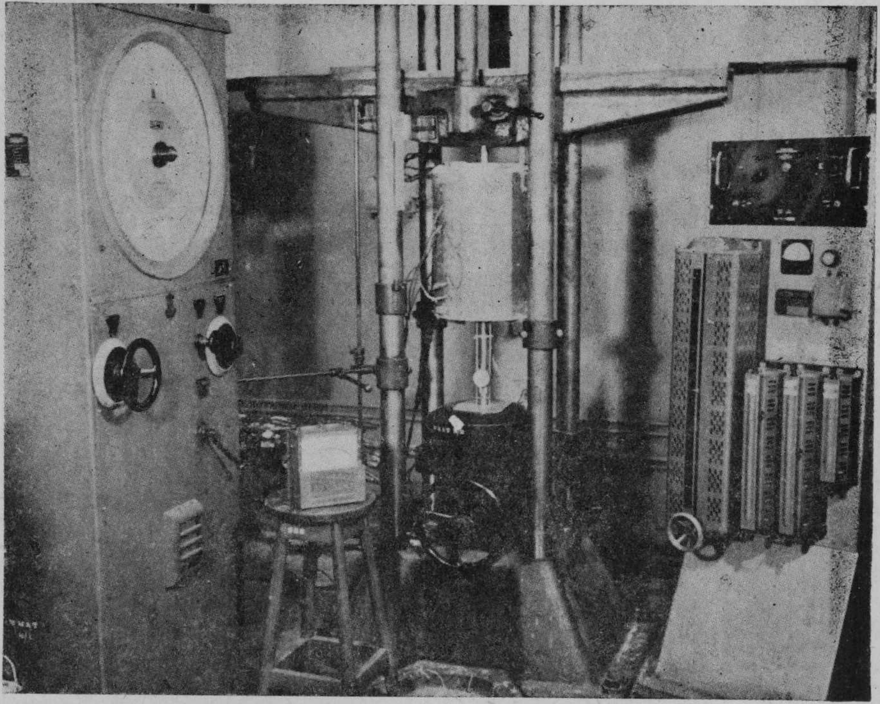


Fig. 50 Elevated temperature tensile testing set up

replaced by the saturable reactor type temperature controller. Since elevated temperature yield or proof stress is sensitive to strain rate variation, the latter is to be controlled within prescribed range of the various standards. The strain rate is controlled at present manually within reasonable limits for which the particular strain-rate settings of the machine have been pre set by initial experiments. However, a tensile testing machine specially designed for high temperature proof stress determination accompanied with strain-pacer device for precise control of the actual strain rate developing across the specimen's gauge length with at least three sets of furnaces, extensometers etc. to allow a number of tests to be completed in a day has been provided for in the scheme for expansion of the creep laboratory.

Table 74 - Creep Properties of alloy C_6, C_7 & C_8 at temperature 700°C,& stress 10 kg/mm^2

Test mark	Minimum creep rate per hr.	Rupture time hr.	Rupture elongation %
C_6	60.3×10^{-5}	392	23.2
C_7	24.1×10^{-5}	425	36.8
C_8	29.8×10^{-5}	459	30.5

RADIO-ISOTOPE RESEARCH GROUP

A radio-isotope research laboratory is under installation in the National Metallurgical Laboratory with a view to use radio-active isotopes as tools in metallurgical research problems such as grain boundary and surface segregation of metals in alloy systems, friction, lubrication and wear studies, zone melting of metals, corrosion of metals, electrodeposition, mineral ore dressing, radio-activation analysis for determination of the minute quantity of elements (10^{-8} to 10^{-12}g), etc. Radio-active isotopes will also be used for industrial studies like study of processes in blast furnaces and ancillary metallurgical equipment, studies on the refining and casting of steel, measurement of wear of blast furnace linings, production control using thickness gauges, etc. Necessary specimens for radio-tracer studies in the Radioisotope Laboratory would be irradiated in the atomic reactor piles available with the Bhabha Atomic Research Centre, Trombay.

With the above objectives in view; the setting up of the Radio-isotope Laboratory made considerable progress. Following electronic instruments and accessories were set up for the Radioisotope Laboratory.

Electronic Instruments:

1. Pulse generator - type -PG 700
2. Radiation survey meter - type - MR 120
3. Contamination monitor, type - CM 167
4. High voltage unit - type - 204
5. Non-overload amplifier - type - PA 520 P
6. Toshniwal cathode ray oscilloscope (double beam), Cat. No. EE 60. Band width: DC to 15 MC/sec.

Accessories:

1. Stainless steel sample pans, type - P 1851
2. Aluminium absorbers type A 1750
3. Set of gamma reference sources - Type R 2300 A
4. Mount for endwindow gas flow detector - Type M 1905
5. G.M. Detector (liquid sample) - Type I 1200
6. Polythene reagent bottles
7. Wire wound potentiometers
8. Stainless steel trays
9. Flexible transparent PVC tubing
10. Polythene carboys with screw cap for storage of liquid active wastes.
11. Mica moulded condensers
12. Air oven (thermostatic). Temperature range - A few degrees above room temperature to 350°C.
13. Mahendra temperature controller -type 154/T Range 0°-1200°C.
14. Soldering iron 25 watt, pencil bit, 220/250 V. AC/DC
15. Wolf type SD 4 C - General duty electric drill 220/250V, AC/DC
16. MPE's microscopic specimen polishing machine
17. MPE's metallographic specimen mounting bakelite press with hydraulic ram 1¼" dia.
18. Carbon film resistors

The installation of the necessary ventilation plant in the Radio-isotope Laboratory has been completed in consultation with Bhabha Atomic Research Centre, Trombay. The distributions of the supply and the exhaust air of the ventilation plant at various rooms of the Radioisotope Laboratory (considering the minimum air velocity in the fume hood with one foot opening and in the beta-gamma boxes with full opening, being 100 linear feet per minute) are given in Table 75.

Table 75 - Distribution of Supply and exhaust air at the Radio-Isotope Laboratory.

Space	Size	Total volume c. ft.	Supply Air		Exhaust Air	
			Qty. C.F.M.	Change per hr	Qty. C.F.M.	Change per hr
Hall	41'3"x14'11" x 12'	7400	2100	17	2300	18.6
Active store	9'5"x9'3"x12'	1050	350	20	400	22.8
Decontamination room	9'4"x9'9"x12'	1080	450	25	550	30.5
Auto-radiography room	9'4"x9'9"x12'	1080	350	19.4	350	19.4
Change room	15'x9'2"x12'	1640	350	12.8	400	14.6
			3600		4000	

The air of the active area of the radioisotope laboratory is exhausted through the fume hoods led through proper ducting to a small stack reaching ten feet above the topmost point of the Main Laboratory Building. The inlet air and the outlet air of the ventilation plant are filtered by a bank of very highly efficient filters to make the active area of the laboratory completely free from dust. Uniform circulation of the air throughout the Laboratory is ensured by installing the grills at proper locations inside the various rooms and by adjusting the various air dampers.

The floor of the active area of the Radioisotope Laboratory has been covered with linoleum which can easily be decontaminated. Necessary lead chambers for the counting of the radioactive solid and liquid samples by Geiger counting setup, were designed and fabricated.

The percentage of U_3O_8 equivalent present in Jadugudah active ore samples received from the ore Dressing Division of the Laboratory, was determined using the standard samples from Bhabha Atomic Research Centre, Trombay with the Geiger Counting set up.

The installation of the glove boxes for the tracer studies in metallurgical research problems and the necessary ejector systems for maintaining a negative pressure of 1" to 2" W.G. inside the glove boxes by compressed air with necessary equipments (water trap, air regulator, pressure gauge, polythene tube with proper clamps, square type suitable glove box filters etc) are in progress.

SURFACE COATING DEVELOPMENT GROUP

Plastic Coating on
~~Phosphating of~~ Steel:

A series of phosphate bath compositions were tried to obtain a unform zinc phosphate coating of about 500 mg to 1000 mg per sq. foot on cold rolled steel. Bath compositions without an accelerator did not yield unform coating weights. Moreover after working the bath for some time, baths without accelerator yielded coatings which are associated with iron phosphates also as evidenced from colour of the coating.

Bath compositions with nitrite accelerator and polyphosphate additions for grain refining yielded fine dendritic coatings but the coating weights were not very uniform as indicated in the Table 76.

Table 76 - Coating weight using nitrite accelerator and polyphosphate.

Temperature =80^{o}C

Duration of Phosphating= 4 mts.

Sl. No.	Acid Ratio	Coating weight gms/sq.ft.	Remarks
1.	5.4	1.439	Even though the deposit consists of fine dendritic crystals, the coating weight was not uniform.
2.	5.4	1.538	
3.	5.4	1.345	
4.	5.4	1.376	
5.	5.4	1.433	
6.	5.0	2.135	
7.	5.0	1.995	
8.	5.0	1.952	
9.	4.8	0.4956	
10.	4.8	0.4261	
11.	4.8	0.3754	
12.	4.8	0.5316	

Results given in Table 76 are representative of quite a few experiments carried out under optimum conditions.

Baths were tried using nitrite accelerator, polyphosphate and a surface wetting agent and the coating deposits are indicated in the table 77.

Table 77 - Coating weight using nitrite accelerator, polyphosphate & surface wetting agent.

Temperature = 80°C Duration of phosphating= 4 mts.

Sl. No.	Acid Ratio	Coating weight gms/sq.ft.	Remarks
1.	5.2	1.999	Deposit is fine dendratic crystals. Coating weight is rather high per sq. foot of the base material even though the deposit is fairly uniform.
2.	5.2	2.033	
3.	5.2	2.026	
4.	5.2	1.954	
5.	5.2	1.755	
6.	5.2	1.382	
7.	5.2	1.109	
8.	5.2	1.129	
9.	5.2	0.9802	
10.	5.2	0.9380	
11.	5.2	1.165	
12.	5.2	1.295	
13.	5.2	1.241	
14.	5.2	1.432	
15.	5.2	1.475	
16.	5.2	1.414	

Subsequently zinc phosphate baths with only nitrite accelerator were tried using a surface active agent and the coating deposits and coating weights were found to be extremely uniform. The results are represented in Table 78.

Table 78 - Coating with using nitrite accelerator and surface active agent.

Temperature= 80°C Duration of phosphating=4 minutes

Sl. No.	Acid ratio	Coating weight gms/sq. ft.	Remarks
1.	5.6	1.389	All the samples are quite uniform The deposit consists of fine dendratic crystals. The samples are quite uniform.
2.	5.6	1.346	
3.	5.6	1.198	
4.	5.6	1.054	
5.	5.6	0.8447	
6.	5.6	0.7686	
7.	5.6	0.6615	
8.	5.6	0.7180	
9.	5.6	0.9441	
10.	5.6	0.8316	
11.	5.6	0.6141	
12.	5.6	0.5852	
13.	5.6	0.6933	
14.	5.6	0.6713	
15.	5.6	0.6012	

1	2	3	4
16.	5.6	0.6814	
17.	5.6	0.8153	
18.	5.6	0.6853	
19.	5.6	0.5685	
20.	5.6	0.5339	
21.	5.6	0.5114	
22.	5.6	0.5222	
23.	5.6	0.6049	
24.	4.8	0.5676	
25.	4.8	0.5588	
26.	4.8	0.6474	
27.	4.8	0.6287	
28.	4.8	0.6688	
29.	4.8	0.5988	
30.	4.8	0.6343	

Table 76 indicates the non-uniformity in coating weight. Table 77 shows that even though the coating weight is high, the deposit is quite uniform. Table 78 shows that the coating weights with the exception of a few initial samples varied between 500 mg and 1000 mg per sq. foot. Photographs of a few typical phosphate coated samples are given in Figs. 51 to 53. Figs 51 and 52 indicate the nature of deposit with nitrite accelerated bath containing polyphosphate and Fig. 53 indicates the deposit with nitrite accelerated bath. The crystal size of the deposits seen in the photographs are in accordance with the coating weights represented in Tables 76 to 78. It was found that nitrite accelerated bath containing a surface active agent will give a coating weight in the range 500 mg to 1000 mg per sq. ft. under the conditions studied.

It is contemplated to use treated bentonite as a stabilizer for PVC and hence the c.e.c. of Kutch bentonite was determined and the rate of exchange of cations from a cation exchange resin to bentonite was studied using an ion exchange resin column. The c.e.c. of bentonite was found to be 106 me/100 gms and the total cations as Na^+ were found to be 89 me/100 gm. The extent of hydrogen exchange from Amberlite IR-120 to Kutch bentonite was found to be 78 me/100 gm at a flow rate of 5 cc per minute. Rate and extent of exchange by column technique with other cations is under study.

As iron and aluminium chlorides act as catalysts in the decomposition of PVC, it is felt that bentonite should be made free from these cations as much as possible without considerable destruction of the clay structure. With this in view, acid dissolution of bentonite and the X-ray analysis of acid treated material is being investigated along with the c.e.c. of acid treated samples.

Fig. 51 Deposit with nitrite accelerated bath containing polyphosphate

Fig. 52 Deposit with nitrite accelerated bath containing polyphosphate

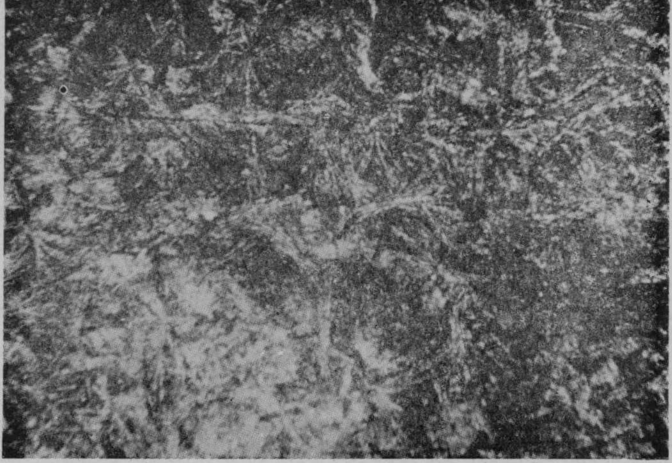


Fig. 53 Deposit with nitrite accelerated bath

Exudation of plasticizer from PVC films was investigated by casting films with different proportions of commercial plasticizers and the film apparance was studied for exudation after ageing for a week. The results are indicated in Table 79.

Table 79 - Exudation of Plasticizer

Sl. No.	Plasticizer	Resin Plasticizer ratio	Touch	Exudation of plasticizer	Remarks
1.	Di-iso-octyl phthalate	1 : 1	Hard	Nil	
2.	Di-iso-octyl phthalate	1 : 2	Medium	Nil	
3.	Di-iso-octyl phthalate	1 : 3	Soft	Nil	
4.	Di-octyl phthalate	1 : 1	Hard	Nil	
5.	Di-octyl phthalate	1 : 2	Soft	Nil	
6.	Di-octyl phthalate	1 : 3	Soft	Nil	
7.	Dibutyl phthalate	1 : 1	Hard	Nil	
8.	Dibutyl phthalate	1 : 2	Medium	Nil	
9.	Dibutyl phthalate	1 : 3	Soft	Nil	

As seen from the above table, there was no exudation of plasticizer from the film at different resin plasticizer ratio. Viscosity measurements of the thinner compositions and the resin-plasticizer mixes has been undertaken and in this connection, calibration of Rheo-viscometer is being carried out.

STANDARD METALS DEVELOPMENT GROUP

The standard samples prepared earlier such as manganese ore, Iron-ore and 0.6% C steel ore ready for sale.

32 Kg of Bright steel sample, were collected from a bar of 4" dia; the millings were obtained after removing ¼" all round the surface. The seiving crushing were carried out (-12 + 20) mesh fraction was choosen to be the proper size. The sample was homogenised.

The analysis of this sample was carried out in this Laboratory and found to be as follows:

C% - 0.25
Si% - 0.10
P % - 0.025
Mn% -- 0.80

This sample has to be sent to the other organisations for chemical analysis.

18 Kg millings Cast Iron sample was prepared from a casting made in the Laboratory. To study the homogeneity of the above, drillings were collected from five different sections and the analysis carried. The homogeneity was observed from the analysis report given in Table 80. After proper mixing and sampling the sample was sent to the following organisations for chemical analysis.

National Test House, Alipore
Italab Private Ltd., Bombay
Director of Inspection, Tatanagar
Inspection Metallurgy, Burnpur
Alloy Steel Project, Durgapur and
C.M.E.R.I., Durgapur.

Table 80 - Analysis of the cast iron sample.

Different sections	Si%	C%	Mn%	S%	P%	C.C.
No.1 (3"-2½")	1.93	3.48	0.68	.05	0.266	
No.2 (2½"-2")	1.91	3.50	0.67	.05	0.263	
No.3 (2"/1½")	1.97	3.17	0.68	.052	0.263	
No.4 (1½"-1")	1.93	3.40	0.69	.048	0.254	
No.5 (1"/¾")	1.99	3.15	0.67	.052	0.25	0.96

24 Kg of millings of stainless steel sample has been collected, from an ingot casted in our Laboratory. It was forged into bars. The millings were obtained after removing ¼" all round the surface. The seiving and crushing were carried out (-12 +30) mesh fraction was taken to be the proper size. The analysis of this sample is in progress in the Laboratory. For finalising and establishing the certificate of the sample it will be sent to different collaborating laboratories for chemical analysis.

Standard samples prepared by the Laboratory have been supplied to a number of firms at their request on payment basis.

SUMMARY OF RESEARCH THESIS SUBMITTED FOR THE AWARD OF DOCTORATE DEGREE BASED ON THE WORK UNDERTAKEN AT THE NATIONAL METALLURGICAL LABORATORY.

Structure of Electro-deposited Manganese:

Manganese has been electro-deposited in two allotropic modifications, viz alpha and gamma, from aqueous solutions of its salts. Interestingly, from pure solutions of manganese and ammonium sulphate, gamma manganese, ordinarily stable between 1095-1134°C, is obtained, while with addition of 0.1 gm per litre of sulphur dioxide to the same electrolyte, alpha manganese stable at room temperature is obtained. The electro-deposited gamma manganese is in a metastable state and completely transforms to alpha manganese in about 10 days at a room temperature of 32°C.

The effect of sulphur dioxide in the deposition of alpha manganese has been explained by various authors but the views expressed have been generally not concordant. A survey of the literature shows that there is yet no general agreement on the nature and growth of electro-deposits of manganese. The thesis is a systematic study on the various factors involved, in an attempt to evolve an explanation on the deposition of manganese in alpha modification in presence of reducible sulphur or selenium compounds and in gamma modification in their absence.

The standard electrode potential of pure alpha manganese (containing no sulphur or selenium) is found to be less negative than that of gamma manganese, and the values for the two phases differ only very slightly as would be expected from the thermo-chemical data of the two phases. But the standard electrode potential values of alpha manganese containing sulphur or selenium are found to be more negative than those of pure alpha manganese (containing no sulphur or selenium) and gamma manganese. The cathode potentials in the deposition of these phases are of the same order of sequence as the standard electrode potentials, viz:

i) Gamma manganese
ii) Alpha manganese containing sulphur
iii) Alpha manganese containing selenium

It has been found in the course of this investigation that a flash layer of armorphous manganese (vide Fig.I) is deposited from pure solutions fitting in or resembling that of alpha manganese as is to be expected from the standard potential values, over which gamma manganese begins to build up in absence of reducible sulphur or selenium compounds. In presence of sulphur dioxide, the amorphous nature continues to build up to an increasing thickness over which crystalline pattern of alpha manganese is obtained. The difficulties in the deposition of pure alpha manganese to an increasing thickness, in absence of reducible sulphur or selenium compounds in the electrolyte, have been attributed to its low hydrogen overvoltage.

It has been conclusively established in this work that the substrate has a directing influence only in the first few atomic layers of the deposit and beyond that, with all other conditions of deposition like current density, pH, temperature, flow of electrolyte and even current efficiency remaining the same, on the same and similarly prepared substrates, gamma and alpha manganese can be prepared with an addition of 0.1 gm per litre of sulphur dioxide or selenious acid for alpha deposition and their absence for gamma deposition. Even gamma manganese can be deposited on alpha manganese and vice versa depending upon the absence or presence of reducible sulphur or selenium compounds in the electrolyte (vide Fig. II and III).

The presence of reducible sulphur or selenium compounds in the electrolyte have been found to increase the cathodic polarization with a resultant increase in the hydrogen content of the alpha deposits obtained. Both these properties have been attributed to the negative catalytic effect of sulphur and selenium on the recombination of hydrogen atoms to form molecules.

Organic inhibitors which also increase the cathodic polarization do not yield the deposition of alpha manganese. Alpha manganese is obtained when not only the cathodic polarization is increased but also the hydrogen content of the deposit is increased. Other than Group VIB elements, these effects are brought about by the use of very high current densities when hard brittle manganese is obtained.

The alpha manganese deposits obtained in presence of sulphur or selenium and the amorphous brittle hard deposits obtained at high current densities from pure solutions contain high hydrogen. Deposits containing over 500 cc of hydrogen per 100 gms were generally amorphous. Heating this amorphous deposit at 125°C for an hour when part of the hydrogen is released gives crystalline pattern of alpha manganese. Similarly the amorphous deposit obtained initially in presence of sulphur dioxide gives a pattern of crystalline alpha manganese after annealing for a period of over four years at room temperature.

The manganese initially obtained in presence of sulphur di-oxide in the electrolyte and the deposit obtained at very high current densities bear a close resemblance in that both give an X-ray powder pattern of broad halo at low angle and no back reflection lines indicating the amorphous nature of the deposit. But the hardness of both deposits is found to be similar to that of alpha manganese. The appearance of the amorphous layer below the crystalline pattern of alpha manganese in the micro-photographs (vide Fig. II) is likely to lead to the impression that manganese is initially deposited in the gamma form which subsequently transforms to fine grained alpha and further deposition takes place as macroscopic crystals of alpha manganese. No evidence for initial deposition of gamma manganese was found from X-ray studies of deposit obtained in presence of sulphur dioxide, though in its absence gamma manganese lines were distinguishingly clear.

Based on the above experimental evidences, a new theory on the mechanism of deposition of manganese in alpha and gamma modifications has been suggested.

ENGINEERING DIVISION AND SECTION

DESIGN AND MECHANICAL ENGINEERING

A. Major development projects underway during the period under review:

i) Strip Aluminizing Prototype Plant:

Work is underway on the coating furnace unit, the lining of which is now to be taken up. Installation of the pre-treatment line is almost complete. There is a good possibility of commissioning the plant during 1969-1970.

ii) Vanadium Pentoxide Plant

A feasibility report for the setting up of a 100 tons/year Vanadium Pentoxide Plant was well under way. Some experimental data was required for finalizing the optimum process route. Steps were taken to obtain this data.

iii) Continuous pickling with HCl

A project was planned for determining optimum conditions for the continuous pickling of steel strip with hydrochloric acid as a substitute for sulfuric acid. This will be directly utilized in the Strip Aluminizing Plant under installation. It also has a wide scope for application in Industry (Galvanizing lines, Tube plants etc.)

The regeneration of spent liquor and the possible production of by product iron powder will also be investigated.

B. Design and Fabrication Projects completed or underway during the period under review:

i) Baby Cupola

The fabrication of this 500 kg/hr unit was completed.

ii) Jib Crane

The design and fabrication of a ten tons jib crane with a 5 meter swivel boom was completed.

iii) Martinson Coater

The design of a Martinson type of Coating unit was completed.

iv) Rotary Kiln

The design of a prototype rotary kiln having a capacity of 6 tonnes/day for roasting Manganese ores for the production of Manganese Dioxide was completed.

v) Magnesium Plant

Work was taken up on the rectification and finalization of the layout drawings of the 250 tons Magnesium Plant Project.

vi) Zinc Distillation Unit

The design of an advanced type of retort for the vacuum distillation of zinc was completed.

vii) Liquid Metals Centrifuge

A high speed centrifuge for liquid metals with in built melting furnace was designed.

viii) Abrasion Apparatus

An apparatus for conducting of abrasion tests on coatings was designed.

ix) Extrusion Rheometer

A rheometer for determining extrusion characteristics was designed.

x) Stormer Viscosimeter

A viscosimeter of the Stormer type was designed, fabricated, and handed over to the Refractories Division.

xi) Truck Cabin

A Cabin for Driver and passengers was designed and fabrication taken up.

xii) Boris Furnace

The detailed design of a Boris type of Research Furnace was completed.

xiii) Electrodes Actuating Mechanism

Two sets of equipment for the controlled movement of carbon electrodes were fabricated.

xiv) Corrosion of Moving Bodies

An apparatus for determining corrosion of moving bodies is being designed.

ELECTRONICS ENGINEERING

A. Development Work:

1. High Current Potentiostat

Work on the development of high current potentiostat (upto 10 amps) for corrosion studies under controlled potential/current conditions with special reference to electrode reactions, was continued. Different methods for amplifying error signal and control of cell currents were studied. A high gain differential amplifier was selected. This D.C. amplifier is connected to the cell circuit. The potentiometer is set to the desired potential of the working electrode. The difference between this set potential and the actual electrode potential is amplified by the high gain D.C. amplifier. The amplifier is coupled to the control winding of the saturable core reactor, which controls the current through the electrolytic cell.

2. Transistorised automatic thermogravimetric balance

Design of a suitable transistor D.C. Amplifier was undertaken. Several types of amplifier circuits were studied and designed for amplifying signals from cadmium sulphide cells.

3. Instrumentation of carbon plant:

Design of an electric tunnel kiln and associated instrumentation and control systems were undertaken jointly with Refractories Division. Various zones of the tunnel kiln will be separately controlled and temperatures recorded by suitable temperature recorders.

B. Maintenance, installation and Calibration work:

Among the major maintenance installation and calibration work the following may be mentioned:

1. Installation of Ferrotester for hysteresis loop.

2. Testing of various types of electronic instruments for radio-isotopes laboratory such as Pulse Generator, Dosemeter, Counting rate meter, single channel analyser, Gamma ray Spectrometer etc.

3. Testing of Brentford Voltage regulator for radioisotopes Laboratory.

4. Repair, calibration and overhauling of various types of electronic and process control instruments such as temperature controllers and recorders, spectrophotometer, oscilloscopes, electron beam melting furnace, ultrasonic flaw detector, Autrometer, X-ray diffractometer, Derivatograph etc.

ELECTRICAL ENGINEERING

i) Power Distribution in Dense Carbon Pilot Plant:

The peak load of Dense Carbon Pilot Plant was estimated as 400 K.V.A. In view of the future expansions, capacity of 500 K.V.A.power is being arranged. This power will be drawn from the sub station of Mineral Beneficiation Pilot Plant as the provision of 500 K.V.A. spare power was made at the time of the installation of this sub-station in the year 1963-64. The bulk power at 410 volts will be transmitted through two parallel underground cables to the plant. The cables will be terminated to a Main distribution Board. From Main distribution board, power will be distributed to a Central panel for 250 KVA Electric Tunnel Kiln and to several sub-distribution boards which will supply power to individual motor control panels.

Scheme of industrial and street lighting was designed. Lay out for above schemes were prepared. Detailed specification of materials were prepared. Quotations were scrutinized and final selection of materials was made.

ii) Installation of new equipment

Installation and commissioning of several equipment were planned and executed. Amongst those, the following were the major jobs:-

a) Installation and commissioning of 50 KW Bengal lyne high temperature furnace.

b) Installation and commissioning of 10 T capacity chilling plant.

c) Installation of temporary power for Dense Carbon Pilot Plant.

d) Installation and commissioning of Press Machine, Milling machine, Lathe and 5 T air conditioning unit.

iii) Electrical Resistance Furnaces:

Several electric resistance furnaces of low, medium and high temperatures were designed and fabricated to meet the demands of various research and development work in National Metallurgical Laboratory.

iv) Development of electro-slag refining equipment:

The equipment was designed. Detailed specifications of various components were prepared and enquiries have been floated to know the potentiality of procuring the materials indigenously.

v) Development of electrical resistance furnace for creep testing:

From the trials of two furnaces, designed and fabricated earlier, the following conclusions were drawn.

(i) the refractory material developed in National Metallurgical Laboratory will stand the long service.

(ii) The zone of constant temperature of these furnaces is too narrow.

Another modified design of the furnace is being evolved.

CIVIL ENGINEERING

Various Civil engineering jobs relating to maintainence and erection of plants and equipment, building, extension of building, etc. were conducted during the period under review.

INDUSTRIAL LIAISON, INFORMATION AND EXTENSION SERVICES

Liaison, Information and Extension services of the National Metallurgical Laboratory played a significant role in maintaining regular flow of contact with industrial and other organizations. Industrial problems were undertaken on behalf of many industries and other organization and technical consultancy was provided in many cases. The different techniques and 'know-how' which are developed by the Laboratory for commercial exploitation were brought to the notice of the industrial organization resulting in fruitful discussions and negotiations for releasing the processes for commercial production. The Laboratory was actively consulted by the Technical Development Establishment in the matter of granting license to firms for foreign collaboration. The laboratory has scrutinised the cases critically and have discouraged foreign collaboration wherein indigenous 'know-how' or indigenous talent is available.

Collaboration has been maintained with other organizations in the matter of various research and development projects of the Laboratory. Enquiries from various industries; large, medium and small; were attended to and suggestions furnished in respective cases. Collection of industrial, economic and market data were continued during the period.

A brief account of the activities is furnished below:

CONSULTANCY WORK

During the period under review, metallurgical industries both in public and private sector continued to call on heavily the assistance of the National Metallurgical Laboratory in diversified subjects of metallurgical research pertaining to production, process control etc. Consultancy assistance provided to industry comprised of beneficiation of limestone samples from Yeotmal District of Maharashtra and from Dungri Limestone Quarry, Sambalpur Dist. for making it suitable for use in cement making, upgrading of silica sand sample from Ratnagiri Dist. of Maharashtra for making it suitable for use in glass, foundry and silica bricks manufacture, beneficiation of pyrite samples from Amjhore supplied by Pyrites, Phosphates and Chemicals Ltd. for manufacture of sulphuric acid, beneficiation of poor grade kayanite from Amda Lapso mines, Bihar, graphite from Gangavaram mines, Errametla mines of Andhra Pradesh, phosphate rock from Masrana block, Mussoorie area, U.P., low grade phosphate from Rajasthan, pelletization and calcination of lime shells from Kerala, crushing and wet screening tests with iron ore sample from Deposit No. 5 of Bailadila Iron Cre mines of NMDC Ltd. and crushability and reducibility of iron ore samples from Surajagarh deposit of Chanda Dist., Maharashtra.

Batch as well as pilot plant beneficiation studies were completed on Ambadongar fluorspar sample for drawing an optimum beneficiation flow sheet and working out equipment and machinary details for submitting a proposal for establishment of 500 tonnes per day fluorspar beneficiation plant by M/s. G.M.D.C. and report on the studies had been submitted.

Besides, batch as well as pilot plant beneficiation studies for the recovery of copper, nickel and molybdenum from low grade copper ore from Rakha mines and detailed proposals for setting up of a 1000 tonnes per day beneficiation plant were completed and report on the studies had been submitted.

Among the work relating to ore preparation referred to NML during the current period mention may be made of (i) the pilot plant beneficiation studies of a low grade phosphate sample from Maton Block, Udaipur District, Rajasthan taken up on behalf of Hindustan Zinc Ltd., Udaipur District, Rajasthan taken up on behalf of Hindustan Zinc Ltd., Udaipur, (ii) the pilot plant beneficiation studies on a low grade limestone sample from Tal, Garhwal Dist., U.P. and proposals for setting up of a 1200 t.p.d. beneficiation plant taken up on behalf of the Director of Geology & Mining, Govt. of U.P., Lucknow and (iii) pilot plant studies on washing and pelletisation of Donimalai Iron Cres taken up on behalf of M/s. N.M.D.C. Ltd., New Delhi.

Smelting trials were conducted in submerged arc smelting furnace for production of silico chrome referred to by M/s. Ferro Alloys Corporation Ltd., Shreeramnagar and 'know-how' was furnished to the firms.

COLLABCRATIVE RESEARCH AND DEVELCPMENT WCRK

The National Metallurgical Laboratory is collaborating with the Commonwealth Committee on Mineral Processing as well as International Working Group on Bonding Clays. Besides, NML is collaborating with Indian Standards Institution in the formation and rationalization of standards, and have conducted and undertaken a number of investigations apart from comments furnished on various draft specifications. Close collaboration is maintained with the defence establishments to meet their needs for various special products as per their rigid specifications. With the Research Design and Standard Organization of Ministry of Railways, the National Metallurgical Laboratory is in close touch regarding the development of technical 'know-how' relating to production of some specific materials based on indigenous materials.

INDUSTRIAL SURVEYS AND COLLECTION OF STATISTICAL, ECONOMIC AND MARKET DATA

Statistics relating to latest production and consumption figures of different types of raw materials, ferrous and non-ferrous metals and alloys of Indian and other countries of the world were collected. Scanning of technical news of the industry and research development in home and overseas press was done. Data thus collected were classified and catalogued and made available to research workers of the Laboratory as well as made use of in connection with enquiries from outside organizations.

TECHNICAL AID TO INDUSTRIES

Over two hundred enquiries on different subjects pertaining to process details, raw materials, quality, production etc. were attended to during the period under review. The majority of the enquiries originated from privat industries as well as individuals. Detailed technical notes were furnished whenever necessary. About fifty short-term investigations and testings were conducted out of which about 41 percent related to government and semi-government organisations and 59 percent from private industries and individuals.

TRAINING

Instructional and training facilities were extended to nineteen students of Metallurgy from the I.I.T., Madras and special lectures were arranged on (i) Ore dressing technique for Indian iron and manganese ore (ii) Modern physico-chemical methods of metallurgical analysis and (iii) Manufacture of iron by Low Shaft Furnace.

Besides, many technical personnel from Government and Semi-Government organisations, engineering colleges, universities, CSIR sister laboratories and students from technical institutes, universities and engineering colleges were given training in heat treatment, induction melting, mechanical testing, refractories, ore dressing techniques, spectrographic analysis, creep testing etc.

NML FOUNDRY STATIONS AND MARINE CORROSION RESEARCH STATION

N.M.L. FOUNDRY STATIONS

The NML Foundry Station, Batala; was actively engaged in rendering technical service facilities to a large number of foundries in and around Batala as well as to foundries situated at Jullunder, Ludhiana, Amritsar etc. In collaboration with the Department of Industries of the Govt. of Punjab, a beginning has been made in introducing modern scientific methods of foundry practice and production of quality castings in a few selected foundries. It is planned to extend such development work to other foundries in the region in a phased manner.

Extensive investigation work aimed at exploring the resources in the Northern region with regard to foundry raw materials is underway at the NML Foundry Station, Batala. As a first step, the sand deposits in the area are being examined in collaboration with the Department of Industries of the Govt. of Punjab. So far nearly 40 sand samples were/investigation /collected and was completed on twenty five sand samples. Work on further fifteen samples is under various stages of investigation. In addition to the above, the Foundry Station at Batala is also providing facilities for the analysis of metals and alloys, testing of sand samples etc. During the period under review 117 samples were analysed chemically, and about 200 technical enquiries were attended to by way of personal inspection or through correspondance. Testing facilities like hardness testing etc. has been provided at the Foundry Station for the benefit of the foundry industry in the region. It is proposed to equip the Foundry Station with other testing facilities like universal testing machine, Impact testing machine etc.

The NML Foundry Station, Howrah, has a well equipped sand testing laboratory and has facilities for analysis of both ferrous and non-ferrous metals and alloys. The Foundry Station has been recently equipped with testing equipments for tensile, impact, hardness tests etc. which facilities are extensively used by the local foundries and other small scale industrialists.

Regional survey and investigation of foundry raw materials was taken up in collaboration with the Geological Survey of India, Investigation has so for been completed on nineteen sand samples and work on further eleven samples is under various stages of investigation.

During the year the NML Foundry Station, Howrah, analysed chemically as many as 130 samples. Twenty five foundries were visited for the purpose of giving technical guidance and 110 technical enquiries were attended to.

The NML Foundry Station, Madras has a well equipped sand testing laboratory and a chemical analysis section for the analysis of metals and alloys. Some Mechanical testing equipments have also been provided. Action is under way to procure other mechanical testing equipments.

Investigation of foundry raw materials available in the southern region with a view to find out their suitability for foundry purposes has already been taken up. As a first step, investigation of foundry moulding sands available in the region was taken in collaboration with Geological Survey of India. Eleven sand samples were investigated. A large number of samples of foundry sand, metals, alloys etc. from foundries were analysed. During the period under review, technical guidance was rendered to a number of foundries.

The Chemical analytic laboratory of NML Foundry Station, Ahmedabad, was set up and work on testing and analysis of samples of metals and foundry raw materials has started. The setting up of the sand testing laboratory is almost complete. Action is underway to equip the laboratory with mechanical testing equipments. In order to be able to take up regional survey of foundry raw materials, arrangements has been made with the Geological Survey of India for location in the first instance of foundry sand deposits in the region and collection and supply of samples for extensive investigation.

Expansion of the service facilities offered by the NML Foundry Stations to include metallographic examination and problems related to metal melting and casting will be taken up. Each NML Foundry Station will be provided with a small Library which will be gradually expanded to provide facilities for technical reference on diverse fields of foundry technology.

MARINE CORROSION RESEARCH STATION

The Marine Corrosion Research Station at Digha on the coast of ~~Bengal~~ Bay of Bengal in W.Bengal has initiated a number of research projects relating to the corrosion and protection of metal and alloys under marine atmosphere which are of interest to ship building industry. transport industry, structural and other engineering industries. Samples of different metals and alloys were exposed to atmosphere to study their rates of corrosion. Corrosion of metals and alloys under actual immension condition in sea water and other related studies are underway.

INTERNATIONAL SYMPOSIUM

A Symposium on 'Recent Developments in Non-Ferrous Metals Technology' was organized by the National Metallurgical Laboratory from 4th to 7th December 1968. The Symposium was held with a view to focus attention on the various aspects of non-ferrous technology with particular reference to the utilization of the indigenous raw materials and indigenous technology and the scope of research and development work in these directions. The Symposium which was attended by more than 200 delegates from home and abroad provided an international forum to discuss the various aspects of non-ferrous technology, exchange of technical 'know-how' and examination of inter-related problems which could facilitate further research and development work on the subject.

Sixty two technical papers covering various facets of non-ferrous technology were presented and discussed in seven technical sessions.

The Symposium was inaugurated by Dr. Atma Ram, Director-General, Scientific & Industrial Research; Sir Jehangir Ghandy, Director, Tata Sons Ltd., presided over the function.

Dr. T. Banerjee, Scientist-in-Charge, National Metallurgical Laboratory, in welcoming the delegates from India and abroad stressed the need for development of non-ferrous metal industry in India from indigenous resources as far as practicable with indigenous talents. He assured the help and service of the National Metallurgical Laboratory to non-ferrous metals industries in solving of their difficult problems connected with raw materials, extraction techniques, alloy development etc. Continuing Dr. Banerjee stated that role of National Metallurgical Laboratory in the development of substitute alloys, study of raw materials, production of non-ferrous metals, and recovery of metallic values from waste products etc., many of which are either now under commercial scale production or being implemented by industries and organizations, both in public and private sectors.

Sir Jehangir Ghandy, Director, Tata Sons Ltd., stated that inter-change of ideas through technical papers and discussions would go a long way towards solving many problems which plague the non-ferrous metal industry for its proper development. He expressed his concern over the staggering amount of foreign exchange that would be needed to import the non-ferrous metals if nothing is done immediately about the development of indigenous resources of non-ferrous metals. He stated that this problem can be solved by making all out efforts to utilise existing resources fully, to make intensive search for new deposits, to substitute imported metals by indigenous ones as far as practicable and to generate requisite resources for purchasing such metals as cannot be substituted.

Continuing, Sir Jehangir stressed the need for further development of the aluminium industry in view of the large reserves which could support an annual out put of 500,000 tonnes for over a century. He welcomed the proposed establishment of an 'Aluminium Cell' at the National Metallurgical Laboratory and a 'Central Design & Research Organization for metallurgical industries' which would cover the existing gap in research and design facilities in the production of non-ferrous metals and alloys.

Regarding copper, zinc and lead, Sir Jehangir commented that even though there is expansion of the present smelting units and new units are coming up, the gap between the production and demand will continue to be met by import if concerted efforts are not made to locate fresh deposits and plan their exploitation. He expressed his satisfaction on the effort of Geological Survey of India to tackle this problem on top priority basis and also the initiation of the air borne survey named as 'Operation Hard Rock' to find deposits of these base metals.

Sir Jehangir did not rule out the desirability of borrowing new technology but he deplored the idea of drawing all the time from the common pool of technology without contributing to it. The necessity for setting up of Co-operative Research Association by non-ferrous metals industries was also stressed by him.

The task of putting non-ferrous metals industries of India on a sound footing is very complex, Sir Jehangir Commented and in this connection he referred to the laudable efforts of the National Metallurgical Laboratory to tackle various problems in the field of non-ferrous metals technology. He complimented Dr. T. Banerjee, Scientist-in-Charge and his colleagues for the fruitful research and development work conducted by them, some of which have already paid rich dividends.

Dr. Atma Ram, Director-General, Scientific & Industrial Research while inaugurating the Symposium said that the need to apply science and technology to economic development was now a well-accepted principle but the question particularly facing the developing countries was how to accelerate this. It was therefore essential that scientists industrialists should appreciate each other's role in economic development.

Dr. Atma Ram urged the industries to be fully aware of the importance of research and development as a tool for increasing and diversifying productivity. Regarding the role of research laboratories of the country, Dr. Atma Ram said, one of the tasks of the laboratories is to assist in the expenditions and efficient development of natural resources so as to provide the material needs of the people.

Some laboratories under Council of Scientific & Industrial Research, including National Metallurgical Laboratory have gained valuable knowledge in regard to the processing and beneficiation of raw materials, which have been provided to the needy organisations. Dr. Atma Ram pointed out that the development of 'know-how' for new products and processes has to undergo a whole sequence of operations from bench research to large scale operation in the production plant. Research work, basic and applied, is to be followed by pilot plant and development work; engineering, fabrication of plants, erection; production operations, sales, etc. The research laboratories, he said, can only deal with the first one or two links of the chain. In the whole sequence of processes and product development, subsequent aspects such as engineering, design, fabrication and erection of the plant; production, mangagement and modern marketing techniques, which are very important to ensure that a research idea becomes fruitful, are mainly the responsibility of industry which need institutional and organisational support. The CSIR, he told, has pioneered in organising objective basic research in several fields and the laboratories have established pilot plants for studying the commercial feasibility of a number of items. Inspite of some failures, valuable data have been provided to the industries from these pilot plant investigations with a view to their subsequent commercial scale production.

Continuing, Dr. Atma Ram said, non-ferrous metals occupy a key position in industrialization. World trends are indicative of the increasing role of non-ferrous metals for the scientist, and the field is becoming more and more fascinating. He urged upon the fruitful utilization of the abundant resources of aluminium, magnesium and titanium in India. Import substitution and efficient utilization of raw materials are vital for development of this industry in India.

He commended the work of the National Metallurgical Laboratory carried out by Dr. T. Banerjee, Scientist-in-Charge and his able colleagues in the multitude fields of study on utilization of raw materials and development of substitute products conducted at the National Metallurgical Laboratory. He stated that National Metallurgical Laboratory has not confined only to laboratory scale experiments and pilot plant studies but is also assisting the industry.

In this connection he referred to the responsibility undertaken by National Metallurgical Laboratory in setting up a 500 tons/day plant for treatment of low grade fluorspar of Baroda on the basis of investigation carried out at National Metallurgical Laboratory. He had no doubt that the Symposium would throw up new ideas and articulate interaction of views which would stimulate further research and development work to the advantage of non-ferrous metals industries of the country.

Shri P.I.A. Narayanan, Officer-in-Charge(Ore Dressing) National Metallurgical Laboratory, while proposing a vote of thanks to Dr. Atma Ram, Sir Jehangir Ghandy and the delegates present, stated that it is very heartening and encouraging to see that the Symposia organised by the National Metallurgical Laboratory are very well received and appreciated both at home and abroad. Contributions in this Symposium cover a wide range of subjects with considerable technical and scientific value to the subject of non-ferrous metals technology.

LIBRARY, DOCUMENTATION, TRANSLATION AND REPROGRAPHIC SERVICES:

LIBRARY & DOCUMENTATION SERVICE

The Library continued to render its service to the staff of the laboratory. The quantum of classification of the abstracts and their processing has increased considerably.

TRANSLATION SERVICE

French, German & Russian articles of direct interest to research projects of the Laboratory were translated into english. Oral translations were also rendered for the benefit of the research workers. Forty technical papers were translated into english.

REPROGRAPHIC SERVICE

Photostat, reflex prints and micro-films were made from scientific papers of interest to research staff. Microphotographs, X-ray photographs, photographs of apparatus and equipment etc. of various projects of the laboratory were also taken.

PUBLICATIONS

NML TECHNICAL JOURNAL

NML Technical Journal has entered into eleventh year of its publication. The journal is continuing to be received with much interest by scientists, technologists and industrialists both in the country as well as abroad. Exchange agreement has been arrived with a large number of Indian and Foreign journals. The leading abstracting services of the world are regularly publishing the abstracts of the papers of NML Technical Journal.

PROCEEDINGS OF THE SYMPOSIA:

The Proceedings of the Symposium on "Metallurgy of Substitute Ferrous & Non-ferrous Alloys" was published during the period. The Proceedings contain papers and their discussions.

MONOGRAPH

A monograph on "Structure of Electro-deposited Manganese Metal" based on the research work done in the laboratory is under publication.

SPECIAL REPORTS

Many special types of reports to meet the need of CSIR, Planning Commission, Parliament etc. were prepared and sent.

PRESS RELEASES

Periodic Press-releases were issued to appraise the general public about the activities and contribution of the National Metallurgical Laboratory.

HAND OUTS & NOTES

Hand outs and notes on the processes of the Laboratory developed for commercial exploitation were prepared and circulated to interested industrial organizations.

GENERAL

Dr. T. Banerjee, Scientist-in-Charge, retired towards the end of February 1969.

Shri P.I.A. Narayanan, Officer-in-Charge (Ore-dressing) took over as Scientist-in-Charge on the retirement of Dr. T. Banerjee.

The following Staff of the Laboratory were nominated to various Committees as indicated:

Dr. T. Banerjee, Scientist-in-Charge, till February 1969 (i) Membership - Advisory Committee - Metallurgical Engineering, I.I.T., Kharagpur. (ii) C.S.I.R. representative on the reconstituted Committee, of 'Chemistry & Metallurgy' of the Department of Atomic Energy, Govt. of India. (iii) Convenor of the Committee on Inhibitor and Cathodic Protection of the Corrosion Sub-Committee constituted by the Metals Committee.

Shri P.I.A. Narayanan, Scientist-in-Charge from February, 1969 (i) NML representative on Sub-Group II on Iron Ore, under the Planning Group on minerals other than coal and oil constituted by the Ministry of Steel, Mines and Metals, Govt. of India. (ii) Nominated to act as Director on the Board of Directors of Pyrites & Chemicals Development Company Ltd., New Delhi (iii) Member of Panels of experts for different sectors of Trade and Industry for consultation and advise to the customs on problems of assessment, classification and valuation in cases of dispute for mining.

Dr. A.B. Chatterjea, Scientist (i) NML representative on the Coal Advisory Council constituted by the Ministry of Steel, Mines & Metals, Govt. of India. (ii) Member of the Panels of experts for different sectors of Trade and Industry for consultation and adviser the customs on problems of assessment, classification and valuation in cases of dispute in the field of ferrous

Shri P.P. Bhatnagar, Head (Mg. Project)- Member of the Panels of experts for different sectors of Trade and Industry for consultation and advise to the customs on problems of assessment, classification and valuation in case of dispute in the field of Non-ferrous.

Shri P.K. Gupte, Scientist - Member of the Panels of experts for different sectors of Trade and Industry for consultation and advise to the customs on problems of assessment, classification and valuation in case of dispute in the field of Ferrous and Non-ferrous.

Shri B.N. Das, Scientist - Member of Panels of experts for different sectors of Trade and Industry for consultation and advise to the customs on problems of assessment, classification and valuation in cases of dispute for metallurgical equipment.

Shri H.P.S. Murthy, Scientist - Member of the Panel on Graphite Crucible Industry constituted by the Ministry of Industrial Development and Company Affairs, Govt. of India.

Dr. A.K. Lahiri, Scientist - Member of the Committee on Fundamentals of Corrosion studies constituted by the Metal Committee.

Shri G.S. Ramakrishna Rao, Scientist - NML representative on Sub-Group III on Manganese, Phosphates, Pyrites, Diamonds and Bauxite, under the Planning Group on Minerals other than coal and oil constituted by the Ministry of Steel, Mines and Metals, Govt. of India.

Shri R. Choubey, Scientist - NML representative on the composite Committee set up to examine the recommendations made by the Ad-hoc Panel for conducting investigation to determine mechanical properties of boiler steel under the Ministry of Steel, Mines & Metals.

Shri K.P. Mukherjee, Scientist, Member of the Committee on Corrosion in the Chemical Industry and industrial corrosion and service failures and on Inorganic surface coatings constituted by the Metals Committee.

Shri S. Rao Addanki, Scientist - Member of the Committee on Atmospheric Corrosion.

Shri D.K. Khan, Alternate Member to the above committee.

Shri H.R. Thilakan, S.S.A. - Member of the Committee on underground and sub-soil corrosion.

Shri A.N. Mukherjee, S.S.A. - Member of the Committee on ~~Marine Corrosion.~~

Shri D.K. Khan, J.T.A., Alternate Member to the above Committee.

Besides the above, the staff of the Laboratory worked in various Committees of Indian Standards Institution.

Shri S.K. Banerjee, Scientist, went on deputation to U.K. under Colombo Plan for training in the field of "Explosive metal working process".

The following staff were awarded Doctorate Degree(Ph.D) during the period under review:

1. Shri A.K. Nayak, Scientist
2. Shri N. Dhananjayan, Scientist
3. Shri Inder Singh, Senior Research Fellow.

The following staff received awards from Invention Promotion Board based on their work done in the Laboratory.

Shri P.P. Bhatnagar,	Head (Magnesium Project)
Shri V.S. Sampath,	Scientist
Shri G. Bysak,	Senior Scientific Assistant

Purchase and Store Section kept up their activities for procurement of Capital equipment, raw materials, consumable stores. Administration and Accounts Section handled the general administration and budgetary accounts with speed and efficiency.

The NML Staff Co-operative Credit Society Ltd. of the National Metallurgical Laboratory continued its good work and handled transactions amounting to nearly Rs. 2 lakhs during the year. Co-operative Stores of the National Metallurgical Laboratory is supplying ration, food-stuffs, stationary articles to their staff members. The Canteen is supplying meals and different types of snacks at reasonable rate.

The National Metallurgical Laboratory participated at the various games of the Shanti Swrup Bhatnagar Memorial Tournament.

Celebration of Silver Jubilee of CSIR.

The National Metallurgical Laboratory celebrated the Silver Jubilee of CSIR. Shri Nityananda Kanungo, Governor of Bihar, inaugurated the function which was presided by Sir J.J. Ghandy. Director, Tata Sons Ltd., The Laboratory was kept open to the public and a large number of visitors including school and college students visited. The Laboratory was illuminated at night during the celebration period. A Silver Jubilee Number of NML Technical Journal was brought out.

Safety First & First Aid Section:

Periodic inspections for safety measures were carried out during the period. Safety materials and garments were supplied to workers who are exposed to various hazards. There was no major casualty during the period.

PATENTS AND PROCESSES

Patents	Ind.Patent No:	Title
Applied for:	116520	An improved and modified process for the manufacture of ferrite magnets.
	118916	A Process for stabilisation of ferro-silicon.
	118902	A Process for the production of electrolytically deposited manganese dioxide containing iron.
	119958	Improvement in or relating to the production of aluminium alloy anodes for cathodic production.
Accepted	106906	An improved method for removal of iron from ferruginous chrome and manganese ores.
	107982	Stable castable suspensions of non-plastic alumino-silicate materials and methods of making the same.
	108081	Refractory ramming, plastering and patching mixes.
	108583	Chemically bonded forsterite refractories and methods of their preparation.
	110834	Improvements in or relating to the production of fluoboric acid.
	112574	A simple process for preheating the air blast in cupola.
Sealed:	105895	A pneumatic process for the conversion of phosphoric pig irons to steels.
	106906	An improved method for removal of iron from ferruginous chrome and manganese ores.
	107982	Stable castable suspensions of non-plastic alumino-silicate materials and methods of making the same.
	108081	Refractory ramming, plastering and patching mixes.
	108583	Chemical bonded forsterite refractories and methods of their preparation.

PROCESSES RELEASED:

The following processes were released during the period to the firms as mentioned:

Process	Firms
1. Hot-dip aluminising of ferrous materials	M/s.K. Rama Krishna Vijayawada
2. Production of electrolytic manganese metal.	M/s. R. Sen & Co., Calcutta

APPENDIX - I

SCIENTIFIC PAPERS PUBLISHED AND PRESENTED

1. Distribution characteristics of burden in low shaft furnace - K.N. Gupta and A.B. Chatterjea - NML Tech. Jr., X(2), 7-14, 1968.

2. Studies on extraction of nickel from nickeliferous laterites of Badampahar area, Orissa - Mis.S.Samanta, R.N. Misra and P.P. Bhatnagar - NML Tech. Jr., X(2), 15-18, 1968.

3. Some observations on bauxitic hardening of 18-4-1 high speed steel - R.K. Dubey, P.S. Nag and P.K. Gupte - NML Tech. Jr., X(2), 19-23, 1968.

4. A centrifuging technique for studying structure of liquid metals - Rajendra Kumar and Manjit Singh, NML Tech. Jr., X(2), 24-26, 1968.

5. Pickling of ferrous materials Part I - with hydrochloric acid - J. Konar, N.G. Banerjee, H.K.Chakravarty and T. Banerjee - NML Tech. Jr., X(2), 27-30, 1968.

6. Thermal expansion characteristics of some Fe-Cr-Mn-N alloys - Y.N. Trehan, NML Tech. Jr., X(2), 31-34, 1968.

7. Research and development work on non-ferrous minerals, metal and alloys at the National Metallurgical Laboratory - T. Banerjee, NML Tech. Jr., X(4), 68-90, 1968.

8. Theory on mechanism of phase-transformation in electrolytic alloys - T.Banerjee, NML Tech. Jr., XI(1), 4-5, 1969.

9. Studies on the resistance of alloy steels against oil-ash corrosion - Part II - H.R. Thilakan, A.K. Lahiri and T. Banerjee - NML Tech. Jr., XI(1), 12-16, 1969.

10. Studies on extraction of vanadium by fluo-solid roasting - A.K. Saha, R.N. Misra and P.P. Bhatnagar- NML Tech. Jr., XI(1), 6-11, 1969.

11. Standardisation and design criteria relating to high temperature tensile and creep properties - R. Choubey and T. Banerjee, NML Tech. Jr., XI(1), 17-22, 1969.

12. Investigation into cause of failure of pinion shaft - a case study - B.N. Das and B.N. Ghose - NML Tech. Jr., XI(1), 23-27, 1969.

13. Studies on nickel-manganese maraging steels - J.K. Mukherjee and T. Banerjee, NML Tech. Jr., XI(1), 24-28, 1969.

14. On the variation of saturation induction and expansion co-efficients of iron with temperature - L.J. Balasundaram - NML Tech. Jr., XI(1), 36,1969.

15. Stress corrosion cracking of alpha prase in $CuSO_4$ $(NH_4)_2$ SO_4 solution - A.K. Lahiri - British Corrosion Jr. 1968, Vol. 3, November.

16. Electrochemical factors in the stress corrosion cracking of Cu alloys containing Zn and Mn - A.K. Lahiri and T. Banerjee - Corrosion Science, 1968, Vol. 8, pp. 895-905.

17. On the structure of beta Al.Mg - Y.N. Trehan - Indian Jr. of Pure & Applied Physics , 1969, 7(3), 209-210.

18. Clusters in liquid metals - Rajendra Kumar - Contemporary Physics, 1969, 10(1), 49-58.

19. A correlation of chemical composition, mineralogical constitution and physical properties of magnesite refractories - Mr. Ramakrishna Rao and N.V. Naidu - Trans. Ind. Ceramic Soc., Vol XXVIII, No. 2, 1969.

20. Some aspects of cation exchange of raolinite - P. Prabhakaram - Trans. Ind. Ceramic Soc., Vol. XXVII, No. 3, 1968.

21. Laboratory study of Indian and foreign nozzles for pouring steel - A.V. Subramanyam and H.P.S.Murthy- Trans. Ind. Ceramic Soc., Vol. XXVII, No. 5,1968.

22. A study of phase relations along six vertical sections in the ternary system CA_2 gehlenite-spinel and a presentation of six isothermal sections in the ternary systems CA_2 gehlenite - spinel and CA-gehlenite - spinel - M. Ramakrishna Rao - Trans. Ind. Ceramic Soc., Vol. XXVII, No. 6, 1968.

23. Beneficiation of Indian iron ores and allied problems - P.I.A. Narayanan and G.S.Ramakrishna Rao - Trans. Ind. Inst. of Metals, Vol. 21, No.2, 1968.

24. Study on the recovery of nickel from low.grade siliceous ores - Miss. S. Samanta, R.N. Misra and P.P. Bhatnagar - Trans. Ind. Inst. of Metals, Vol. 21, No. 3, 1968.

25. A model on the study of slags and their utility as slag cement with the help of O.R. technique - C.P. Saksena - Trans. Ind. Inst. of Metals, Vol. 21, No.3, 1968.

26. Engel Brewer theory and crystal structures of metals and alloys - L.J. Balasundaram - Trans. Ind. Inst. of Metals, Vol. 21, No. 4, 1968.

27. A study of the precipitation and creep characteristics of Cr-Mn-N-C austenitic steels - R. Choubey, K. Prasad and T. Banerjee - Trans. Ind. Inst. of Metals, Vol. 21, No. 4, 1968.

28. Thermal analysis of Mg-Sn alloys by calorimetics measurements for the determination of the liquidus curve (Pt. I) - A.K. Nayak and W. Oelsen - Trans. Ind. Inst. of Metals, Vol. 21, No. 4, 1968.

29. Foundry industry in Japan - R.M. Krishnan - Indian Foundry Jr., Vol. 14, No.10, 1969, (Annual Convention Number).

30. Beneficiation of low-grade ores of Maharashtra region - T. Banerjee - Mineral Research, Vol. I, No. 1, 1968.

31. Non-ferrous metals in India - T. Banerjee and C. Sharma - Mineral Wealth.

The following papers (Sl. Nos. 31-35) were presented at the Annual Convention of Institute of Indian Foundrymen, held at Madras from 8-11th March, 1969.

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32. Studies on moulding sands from West Bengal, Pt. I, Dist. Purlia - Amitava Das and Ashimesh Dutt.

33. A study of the foundry sands of the Southern region for foundry moulding purposes - K.S. Vijayanarayanan and N.V. Naidu..

34. Some observations on cupola iron melting with oxygen enriched blast - J. Goswami and A.B. Chatterjea.

35. Grain size control in cast aluminium alloys - Rajendra Kumar.

The following papers (Sl. Nos. 36-40) were presented at the 32nd Annual Technical Meeting of the Indian Ceramic Society held at Madras in July, 1968.

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36. Effect of composition and firing temperature on the physico-mechanical properties of basic refractories Pt. I - mineralogical constitution and microstructure - T.V. Prasad.

37. Effect of composition and firing temperature on pysico-mechanical properties of basic refractories - Pt. II - Physical properties - T.V. Prasad.

38. Effect of composition and firing temperature of the physico-mechanical properties of basic refractories, Pt. III - high temperatures deformation in torsion - T.V. Prasad.

39. Mineralogical constitution of Indian blast furnace slags - R.V. Hargave and M.Ramakrishna Rao.

40. A correlation of chemical composition, mineralogical constitution and physical properties of magnesite refractories - M. Ramakrishna Rao and N.V. Naidu.

The following papers (Sl. Nos. 41-50) were presented at the 33rd Annual Technical Meeting of the Indian Ceramic Society held at Delhi in March 1969.

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41. Studies on niobium oxides and polymorphism of niobium pentoxide - J.P. Guha.

42. Calcination characteristics of Indian petroleum coke - study by polarised microscopy - R.V.Hargave and H.P.S. Murthy.

43. Role of forming pressure on the properties of basic refractories - T.V. Prasad.

44. Properties of some Indian refractory plastics and castables - A.K. Bose and T.V. Prasad.

45. Constitution and mineralogy of Indian open hearth and L.D. slags - R.V. Hargave and M. Ramakrishna Rao.

46. High temperature mechanical properties of some French castable refractories - M.C. Kundra.

47. Castables in the fabrication of special shapes for an experimental tunnel kiln-M.C. Kundra and H.P.S. Murthy.

48. Studies on the castable refractories using white calcium-aluminate cements - B.M. Dutta, S. Pramanik and M. Ramakrishna Rao.

49. Refractory castables and ramming developed at National Metallurgical Laboratory - T.V.Prasad.

50. High alumina clays from Jammu & Kashmir State - potential raw material for the refractory industry - T.V. Prasad and N.S. Sahota (the Souvener published in connection with 33rd Annual General Meeting of Indian Ceramic Society).

The following papers (Sl. Nos. 51-62) were presented at the 22nd Annual Technical Meeting of Indian Institute of Metals held at Calcutta from 13th to 15th January, 1969.

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51. Preliminary studies on carburizing characteristics of Nb treated steels - R.K. Sinha, M.R. Kulkarni and R. Chattopadhaya.

52. The structure of properties of 9% nickel steel - R. Chattopadhaya, J.P. Tewari and S.S.Bhatnagar.

53. Some observations on the production of ferro-coke - B.K. Paul, S.R. Jyoti and A.N. Kapoor.

54. Production of sponge iron by reduction with petroleum naphthe - S.R. Jyoti, K.N. Gupta, A.N. Kapoor and T. Banerjee.

55. Technology of production of phospher-bronze springs - B.N. Das and S.K. Banerjee.

56. Thermal beneficiation of ilmenite for the removal of iron - Miss. S. Samanta, R.N.Misra & P.P. Bhatnagar.

57. Quantitative thermal analysis of Mg-Sn alloy by calorimetric measurement for the determination of solid and liquid curves - A.K. Naik and W. Oelsen.

58. Co-efficient of thermal expansion of iron-aluminium and iron-silicon alloys - A.N.Sinha & L.J. Balasundaram.

59. Solidification of aluminium - Rajendra Kumar.

60. Embrittlement of maraging steels - Rajendra Kumar and Sivaramakrishnan.

61. Grain size control of magnesium - Rajendra Kumar and R.K. Mahanty.

62. Smelting of high titania ores with non-metallurgical fuels - A.B. Chatterjea, J.Goswami, S.K. Biswas, R. Santokh Singh and J.S. Padan.

APPENDIX II

SCIENTIFIC INVESTIGATIONS COMPLETED AND REPORTS PREPARED

1. Moulding characteristics of sand samples received from M/s. Steelsworth Pvt. Ltd., Assam - Savinder Singh, V.S. Bhandary and P.K. Gupte (I.R. 456/68).

2. Studies on the recovery of zinc from zinc dross produced at the Workshop, Post & Telegraph, Calcutta - G. Basak, V.S. Sampath and P.P. Bhatnagar (I.R. 457/68).

3. Beneficiation of phosphate rock sample from 'Maton Block' of Udaipur Dist., Rajasthan - K.N. Rakshit, P.V. Raman and P.I.A. Narayanan (I.R. 458/68).

4. Investigation on moulding sand from Parbatipur, Dist. Purulia, West Bengal - Amitava Das and Ashimesh Dutt (IR 459/68).

5. Investigation on sand sample from Mejia Dist. Bankura, West Bengal - Amitava Das and Ashimesh Dutt (I.R. 460/68).

6. Beneficiation studies on an Argillaceous limestone sample from Dungri limestone quarry of Orissa Mining Corpn. Ltd. - S.K. Sengupta, P.V. Raman and P.I.A. Narayanan (I.R. 461/68).

7. Production of sized sand for the Glass industry from a sandstone sample from Gujarat - N. Chakravarty, G.P. Mathur and P.I.A.Narayanan (I.R. 462/68).

8. Beneficiation of poor grade kyanite from Amda Lapso mines, Bihar - S.K.Dhar and P.I.A.Narayanan (I.R. 463/68).

9. Settling and filtration tests on the acid grade conc. from a mixed fluorspar sample from Dingarpur, Rajasthan - K.N. Rakshit, S.K. Dhar and P.I.A. Narayanan (I.R. 464/68).

10. Recovery of nickel and apatite from the copper ore of Indian Copper Corpn., Ghatsila - C. Satyanarayana, G.S. Ramakrishna Rao and P.I.A. Narayanan (I.R. 465/68).

11. Recovery of germanium and gallium from coal, fly ash, flue dust - B.P. Verma (I.R. 466/68).

12. Pelletization and calcination of lime shells from Kerala - B.L. Sengupta and P.I.A.Narayanan (I.R. 467/68).

13. Crushing and wet screening tests with an iron ore sample from Deposit No. 5 of Bailadila Iron Ore mines of N.M.D.C. - Prasad Rao, G.P. Mathur and P.I.A. Narayanan (I.R. 468/68).

14. Heavy media separation tests with the pyrite sample from Amjhore, Shahabad Dist., Bihar - M.V. Ranganathan and P.I.A. Narayanan (I.R. 469/68).

15. Reducibility of lumpy iron ores from Dalli mines of Bhilai Steel Plant - S.B. Mathur, M. Subramaniam and P.P. Bhatnagar (I.R.470/68).

16. Batch as well as pilot plant beneficiation studies with a low grade fluorspar from Ambadongar Dist., Baroda and detailed proposals for setting up of a 500 tonnes per day beneficiation plant for the Gujarat Mineral Development Corpn. Ltd. - M.V. Ranganathan, P.V. Raman, S.B. Dasgupta, G.R. Mathur and P.I.A.Narayanan (I.R. 471/68).

17. Recovery of Diamond and concentration of TiO_2 from -20 mesh fraction of Diamondiferous Tuff from N.M.D.C. - P.K. Sinha and P.I.A. Narayanan (I.R. 472/68).

18. Beneficiation of a high silica limestone (sample No. CGI) from Yeotmal Dist., Maharashtra - P.V. Raman, Sachidanand Prasad and P.I.A. Narayanan (I.R. 473/68).

19. Beneficiation of limestone sample (KAG-1) from Yeotmal Dist., Maharashtra - P.V.Raman, S. Prasad and P.I.A. Narayanan (I.R. 474/68).

20. Pelletisation of chrome ore concentrates from Orissa - N. Chakrabarty and P.I.A. Narayanan (I.R. 475/68).

21. Sintering of chrome ore fines from Orissa - B.L. Sengupta and P.I.A. Narayanan (I.R. 476/68).

22. Foundry moulding sands in Ratnagiri Dist. of Maharashtra State - P.K. Gupte, H.P. Singh and V.S. Bhandary (I.R. 477/68).

23. Moulding characteristics of sand sample received from M/s. Supertech Casting Ltd., Madras - K.S. Vijayanarayanan, N.V. Naidu and R.N.P.Gupta (I.R. 478/68).

24. Investigation on Ichhanagari moulding sand received from M/s. Howrah Builders Corpn., Belgachia, Howrah, West Bengal - Amitava Das and Ashimesh Dutt (I.R. 479/68).

25. Investigation on moulding characteristics of Mundeswari river sand received from M/s. Howrah Builders Corpn., Belgachia, Howrah - Amitava Das and Ashimesh Dutt (I.R. 480/68).

26. Moulding characteristics of natural moulding sand received from M/s. Model Foundry, Madras 32 - K.S. Vijayanarayanan, R.N.P. Gupta and N.V. Naidu (I.R. 481/68).

27. Moulding characteristics of BTL-7 sand received from Punjab State Industries Deptt., Chandigarh - M.N.P. Verma and R.C. Arora (I.R. 482/68).

28. Moulding characteristics of sand sample No.KS-4, received from the Deptt. of Industries, Govt. of Punjab - R.C. Arora and M.N.P. Verma (I.R.483/68).

29. Moulding characteristics of sand sample No.LS-2, received from the Deptt. of Industries, Govt. of Punjab - R.C. Arora and M.N.P. Verma (I.R.484/68).

30. Moulding characteristics of sand sample No.GSP-4, received from the Deptt. of Industries, Govt. of Punjab - R.C. Arora and M.N.P. Verma (I.R.485/68).

31. Moulding characteristics of sand sample No. GSP-4, received from the Deptt. of Industries, Govt. of Punjab - R.C. Arora and M.N.P. Verma (I.R. 486/68).

32. Moulding characteristics of sand sample No. GSP-6, received from the Deptt. of Industries, Govt. of Punjab - R.C. Arora and M.N.P. Verma (I.R. 487/68).

33. Flotation studies for the recovery of molybdenum copper and nickel minerals from uranium ore samples from Jaduguda mines - R. Ganesh, G.S. Ramakrishna Rao and P.I.A. Narayanan (I.R. 488/68).

34. Beneficiation of graphite from Gangavaram mines, Mahbubabad, Andhra Pradesh - M.V. Ranganathan and P.I.A. Narayanan (I.R. 489/68).

35. Beneficiation of low grade graphite from Nangrim Hill Lohit Dist. Nefa, Assam - S.K. Sengupta, S.B. Das Gupta and P.I.A. Narayanan (I.R. 490/68).

36. Upgrading of silica sand sample from Ratnagiri Dist., Maharashtra - S.K. Dhar, P.V. Raman and P.I.A. Narayanan(I.R. 491/68).

37. Studies on mineralogy, crushability and reducibility of three iron ore samples from Surjugarh Deposit of Chanda Dist., Maharashtra - Joga Singh, G.S. Ramakrishna Rao and P.I.A. Narayanan (I.R. 492/68).

38. Beneficiation of phosphate rock from Masrana block, Mussoorie area, U.P. - C. Satyanarayana, S.K. Banerjee and P.I.A. Narayanan (I.R. 493/68).

39. Beneficiation of low grade graphite from Errametta mines, A.P. - C. Radhakrishnan, S.K. Banerjee and P.I.A. Narayanan (I.R. 494/68).

40. Investigation on the Kutch bentonite marked SC - R.R. Dash, V.S. Bhandary and P.K. Gupte (I.R.495/68).

41. Investigation on the Kutch bentonite marked MI - R.R. Dash, V.S. Bhandary and P.K. Gupte (I.R.496/68).

42. Investigation on the Assam Bentonite II - H.P.Singh, V.S. Bhandary and P.K. Gupte (I.R. 497/68).

43. Batch as well as pilot plant beneficiation studies for the recovery of Cu, Ni and Mo from low grade copper ore from Rakha mines and detailed proposals for setting up of a 1000 tpd. beneficiation plant- K. Vijayaraghavan, S.K. Dhar, P.D. Prasad Rao, P.K. Sinha, G.S. Ramakrishna Rao and P.I.A. Narayanan (I.R. 498/69).

44. Investigation report on the suitability of the 14 samples of manganese ores supplied by M/s. Manganese Ore (India) Ltd., Nagpur, for production of electrolytic manganese - A.M. Pande, P.L. Sengupta, N. Dhananjayan and H.K. Chakravorty (I.R. 499/69)

45. Beneficiation of three pyrite samples from Amjhore, Bihar - S.B. Das Gupta and P.I.A. Narayanan (I.R. 500/69).

46. Investigation on molybdenum concentrates obtained from Rakha mines - H.P. Bhattacharya, A. Ghosh & J.K. Mukherjee (I.R. 501/69).

47. Some observation on cupola iron melting with oxygen enriched blast - J. Goswami and Dr. A.B. Chatterjea (I.R. 502/69).

48. Grain size control in cast aluminium alloys - Dr. Rajendra Kumar (I.R. 503/69).

49. Studies on moulding sands from West Bengal, Part I - Amitava Das and Ashimesh Dutt (I.R.504/69).

50. A study of the foundry sands of Southern Region for foundry moulding purposes - K.S.Vijayanarayanan and N.V. Naidu (I.R. 505/69).

51. Pelletisation of metallurgical grade of fluorspar concentrate from Ambadongar, Gujarat - N. Chakravorty, G.P. Mathur and P.I.A.Narayanan (I.R. 506/69).

52. Grinding characteristics and work index of ferro-silicon and calcined dolomite samples - A.K.K., G.S.R.K. Rao and P.I.A. Narayanan (I.R. 507/69).

53. Moulding characteristics of sand sample No. LS-1, received from Punjab State Industries Deptt., Chandigarh -R.C. Arora, and M.N.P.Verma (I.R.508/69).

54. Moulding characteristics of sand sample No. GSP-7 received from Punjab State Industries Deptt., Chandigarh - R.C. Arora and M.N.P. Verma (IR 509/69)

55. Moulding characteristics of sand sample No. GSP-22 received from Punjab State Industries Deptt., Chandigarh - R.C. Arora & M.N.P. Verma (IR 510/69).

56. Recovery of vanadium from Bayer process liquors - Part I - utilisation of sodium complex salt - M.S. Mahanty, T.C. Dey, S.R. Srinivasan and P.P. Bhatnagar (RR 253/68).

57. Development of an ICE calorimeter for high temperature calorimetry - Gangotri Misra and Rajendra Kumar (RR 254/68).

58. Effect of rolling scale on corrosion of mild steel - H.R. Thilakan, A.K. Lahiri and T. Banerjee (RR 255/68).

59. A new approach to thermal beneficiation of low grade ferruginous ores - P.P. Bhatnagar, R.N. Mishra, S.R. Srinivasan and P.V. Viswanathan (RR 256/68).

60. Studies on desilication of siliceous bauxite from Kashmir - S.S. Bhoray, V.S. Sampath and P.P. Bhatnagar (RR 257/68).

61. On the structure of β Al-Mg - Y.N. Trehan (RR 258/68).

62. Clusters in liquid alloys-Rajendra Kumar (RR 256/68).

63. Studies on the thermal beneficiation of ilmenite for the removal of iron - S.Samonta, R.N. Misra and P.P. Bhatnagar (RR 260/68).

64. Co-efficients of thermal expansion of iron-aluminium and iron-silicon alloys - A.N. Sinha and L.J. Balasundaram (RR 261/68).

65. Quantitative thermal analysis of Mg-Sn alloys by calorimetric measurements for the determination of solidus and liquiduc curres - A.K. Nayak and W. Oelsen (RR 262/68).

66. High speed chromium plating - D.S. Tandon and B.B. Basu (RR 263/69).

APPENDIX III

MAJOR SPONSORED PROJECTS AT THE NATIONAL METALLURGICAL LABORATORY

Sl.No.	Title.	Sponsor.
1.	Beneficiation of limestone samples from Yeatmal Dist. of Maharashtra.	Director of Geology and Mining, Govt. of Maharashtra Nagpur.
2.	Batch as well as pilot plant beneficiation studies with a low grade fluorspar from Ambadongar Dist. Baroda and detailed proposals for setting up of a 500 tonnes per day beneficiation plant for the Gujarat Mineral Development Corpn. Ltd.	Mg. Director Gujarat Mineral Development Corpn. Ltd. Ahmedabad.
3.	Pelletisation of metallurgical grade of fluorspar concentrate from Ambadongar, Gujarat.	-do-
4.	Pelletisation and calcination of lime shells from Kerala.	M/s.Travancore Electro-Chemical Industries Ltd. Chingavanam Dist. Kottayam.
5.	Crushing and wet screening tests with an iron ore sample from Deposit No. 5 of Bailadila Iron Ore Mines of NMDC Ltd.	National Mineral Development Corpn. Ltd. Faridabad.
6.	Suitability of the 14 samples of manganese ores supplied by M/s. Manganese Ore (I) Ltd., Nagpur for production of electrolytic manganese.	M/s. Manganese Ore (I) Ltd. Nagpur.
7.	Studies on mineralogy, crushability and reducibility of three iron ore samples from Surjagarh Deposit of Chanda Dist. Maharashtra.	Director of Geology and Mining, Govt. of Maharashtra Nagpur.

Sl.No.	Title.	Sponsor.
8.	Heavy media separation tests with the pyrite sample from Amjhore, Shahabad Dist., Bihar.	Mg. Director, Pyrites, Phosphates & Chemicals Ltd. Dehri-on-Sone Dist. Shahabad.
9.	Beneficiation of three pyrite samples from Amjhore, Bihar.	-do-
10.	Beneficiation studies on an argillaceous limestone sample from Dungri limestone quarry of Orissa Mining Corpn. Ltd.	M/s. Orissa Mining Corpn. Ltd., Dungri.
11.	Beneficiation of low grade graphite from Nangrim Hill, Lohit Dist. of NEFA, Assam.	Govt. of Assam, Shillong.
12.	Beneficiation of graphite from Gangavaram Mines, Mahbubabad, A.P.	M/s. S. Lal & Co. Pvt. Ltd. Calcutta.
13.	Beneficiation of low grade graphite from Errametla mines, A.P.	-do-
14.	Upgrading of silica sand sample from Ratnagiri Dist.,Maharashtra.	Director of Geology and Mining, Govt. of Maharashtra Nagpur.
15.	Beneficiation of poor grade kyanite from Amda Lapsa Mines, Bihar.	General Manager, Indian Copper Corpn. Ltd. Ghatsila.
