

The National Metallurgical Laboratory in the development of Mineral Beneficiation and Extractive Metallurgical Techniques

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In the context of India's mineral resources, the author discusses researches on mineral beneficiation and developments in extractive metallurgical techniques, and outlines the role of the National Metallurgical Laboratory in this respect. General particulars are furnished of the work done on beneficiation of manganese, chromite, graphite, pyrites, etc., and of the extractive metallurgical themes under development at the Laboratory, including pilot plant investigations on electrolytic manganese and manganese dioxide. A review of the Laboratory's programme of work during the second Plan, which includes the setting up of pilot plant projects on thermal beneficiation of low-grade manganese and chromite ores and the treatment of low-grade manganese, chrome and other ores by ore-dressing methods, is also given.

METALLIC ores have been known to exist from ancient times in India. India as a whole cannot be said to be poor in mineral wealth. Most of our mineral resources, however, have still to be scientifically and fully exploited for home use and export. Under the powerful stimulus of the Five-Year Plans, the process of assessment, systematic examination, and utilization of our mineral resources is being expedited so as to exploit those in which nature has been generous to us and to make most scientific and effective use of others in which we have been less fortunate.

The production of minerals in India have been steadily on the increase. The total pit-mouth value of minerals excavated, exclusive of the value of petroleum, rose from Rs. 64 crores in 1947 to Rs. 105 crores in 1951, Rs. 108 crores in 1952 and Rs. 113 crores in 1953. The large increase in total value in this period was mainly due to mica, manganese ore, gold, iron ore, and copper ore, of which mica and manganese ore commanded a good export market. Of the 1953 figures Rs. 29.4 crores was accounted for by manganese ores, Rs. 25 lakhs by chromite and Rs. 28 lakhs by bauxite. The total value of metals obtained from ores in India increased from Rs. 45.7 crores in 1950 to Rs. 50.4 crores in 1951 and 59.3 crores in 1952, but slightly decreased to Rs. 55.25 crores in 1953. For 1954, mineral production trends are depicted in Table I in relation to figures for previous years.

The general position of our heavy surpluses and acute deficiencies are depicted below :—

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Surplus reserves which can be utilized for export in return for our deficiencies :

Aluminium, Magnesium, Rare Metals, Cerium Group, Strontium, Iron, Manganese, Titanium, Thorium, and Zirconium.

Fair reserves :

Beryllium, Columbium, Hafnium, Lithium, Chromium, Tantalum, Vanadium, and Uranium.

Insufficient for our requirements :

Zinc, Copper and Lead.

Acute deficiencies :

Antimony, Cadmium, Germanium, Molybdenum, Platinum, Tin, Bismuth, Cobalt, Mercury, Nickel, Selenium, and Tungsten.

SECOND PLAN

The broad outlines of the second Five Year Plan of India provide for an investment of about Rs. 7,100 crores, that is, roughly three times the investment under the first Five Year Plan—the share of the private sector being about Rs. 2,300 crores and that of the public sector about Rs. 4,800 crores. For the development of various industries and minerals Rs. 891 crores have been allotted. As regards industrialization on which great emphasis is laid in the second Plan, Rs. 700 crores have been allotted for basic industries alone in the public sector. The three steel plants at Rourkela, Bhilai and Durgapur are estimated to cost Rs. 350 to 400 crores. Coalmining in the public sector has been allotted Rs. 40 crores, and the shipyard at Visakhapatnam and the proposed heavy electrical plant Rs. 20 crores each. In the private sector Rs. 550

TABLE I—PRODUCTION FIGURES (QUANTITY AND VALUE) OF THE PRINCIPAL MINERALS PRODUCED IN THE INDIAN UNION DURING YEARS 1939, 1947 & 1954.

	Units	1939		1947		1954	
		Quantity	Value (Rs.)	Quantity	Value (Rs.)	Quantity	Value (Rs.)
Coal	Tons	27,769,112	9,87,23,916	30,144,505	43,77,20,245	36,880,810	53,90,92,850
Manganese Ore	"	844,663	(a) 13,69,779	451,034	(b) 2,07,47,564	1,413,847	(b) 19,54,17,452
Mica (Exports)	Cwts.	198,368	(c) 1,53,82,082	192,671	(c) 4,65,89,163	334,853	(c) 6,57,29,609
Gold	Ozs.	314,515	3,24,34,364	171,705	4,89,54,639	239,168	5,62,06,814
Building Materials	Tons.	(d)	1,04,57,262	(d)	3,58,14,385	(d)	(f) 4,00,00,000
Iron Ore	"	3,166,074	52,20,600	2,498,459	80,67,805	4,308,273	2,89,36,086
Copper Ore	"	360,624	47,89,340	323,085	60,35,118	342,750	1,87,22,629
Kyanite	"	13,961	2,89,526	15,965	6,71,249	42,330	87,80,362
Ilmenite	"	13,964	2,89,526	260,955	31,59,271	240,513	79,80,210
Chromite	"	49,136	6,35,511	34,717	9,97,639	45,507	13,59,607
Gypsum	"	71,212	1,67,752	50,569	4,66,283	612,120	41,59,388
China Clay	"	(e)	—	66,561	14,62,994	146,144	25,20,084
Lead Metal	"	—	—	321	4,23,720	1,790	23,08,243
Magnesite	"	33,568	2,11,750	51,536	6,86,956	70,507	14,52,731
Zinc (concentrates)	"	—	—	—	—	3,974	10,80,996
Bauxite	"	8,977	17,021	19,906	1,58,493	74,747	8,27,785
Steatite	"	22,259	2,03,841	20,652	10,02,819	42,326	13,28,191
Asbestos	"	262	41,948	160	15,394	389	2,28,734
Diamond	Carats.	1,604	58,317	1,284	1,72,652	1,955	4,74,236
Silimanite	Tons.	—	—	236	20,299	3,066	1,50,937
Graphite	"	936	32,622	1,235	1,55,317	1,479	1,36,561
Emerald	Carats.	—	—	—	—	509,180	6,70,406
Barytes	Tons.	9,255	60,842	24,310	3,29,557	18,792	2,61,379
Silver	Ozs.	22,746	33,361	12,422	54,725	161,185	2,67,680
Salt	Tons.	1,498,988	83,59,505	1,540,353	2,46,89,794 (e)	2,516,725	4,25,64,000
Total Value of all minerals produced		(Rs.) 21,55,58,541		(Rs.) 60,04,63,699		(Rs.) 1,02,10,56,920	

(a) F. ob. value at Indian Ports. (b) Estimated on the basis of export value per ton. (c) Value of exports.
 (d) Only value figures shown. (e) Separate figures are not available. (f) Estimated.

crores have been allotted for industrial expansion and mineral development and Rs. 200 crores for replacement and modernization of the machinery in existing industries. So far as the targets of production in the second Five Year Plan are concerned, steel is number one in the list of items of industrial and mineral development, the target being an annual production of 6 million tons of ingot steel. The fertilizer target assumes the equivalent of 'three more Sindris'. Considerable increase in the production of aluminium is also planned; its proposed output of 40,000 tons per annum at the end of the second Five Year Plan represents a doubling of the target of the first Plan. The production of coal will be stepped up to 60 million tons by 1960-61, iron ore to 13 million tons, mica to 200 thousand cwt. and manganese ore to 3.5 million tons. Efforts will have to be made to set up industrial units for

the manufacture of heavy plant, forgings and machinery to be supplied to existing and future iron and steel and other metallurgical industries in the country instead of importing bulk plant and machinery.

In the field of a number of minerals, particularly those of non-ferrous metals, we have suffered from severe and chronic shortages although their potential resources within the country have been fair—such as of lead, zinc, aluminium, manganese. Our growing internal demands in these directions have solely been met by imports. Geological Survey of India and Indian Bureau of Mines have undertaken detailed prospecting and proving of Khetri copper belt in Alwar (Rajasthan), deposits of copper ore in Almora and Tehri Garhwal (U.P.), Sikkim, Singbhum (Bihar), Jalpaiguri and Darjeeling districts of West Bengal, and in Andhra (near

TABLE II.—PRODUCTION AND EXPORT OF IMPORTANT INDIAN MINERALS

Year	Coal		Iron Ore		Manganese Ore		Total of all
	Qty.	Value	Qty.	Value	Qty.	Value	Value
Production							
1952	36,304	5,362	3,926	268	1,462	2,245	10,804
1953	35,980	5,276	3,855	281	1,902	2,948	11,278
1954	36,880	5,390	4,308	289	1,414	1,954	10,252
1955	38,230	5,603	4,640	324	1,570	2,188	10,616
Exports							
1952	2,488	876	669	299	1,409	2,155	3,560
1953	1,271	453	1,093	501	1,659	2,570	3,838
1954	1,225	394	1,056	463	1,086	1,520	2,647
1955	724	215	1,299	567	766	892	2,195

Gani). Smaller steps are being taken in respect of lead-zinc ores at Zawar, Bolangir (Mayurbhanj, Orissa) and Wasiri Rupi (Kulu). Investigations on deposits of ilmenite, ores of tin, tungsten, chromium, gold, asbestos, sillimanite, pyrites, diamond, graphite, corundum are also actively under way. Particular attention has also been given to the detailed examination of manganese deposits in Madhya Pradesh; gold and sulphide ores in Mysore; re-survey of coalfields and location of oil-bearing structures in East Punjab, Himachal Pradesh and Rajasthan.

Deposits of lead in Almora district, iron in Keonjhar district, Orissa, and coal near the Karanpura coalfields in Bihar, have been discovered by the Geological Survey of India during its field investigations in 1954-55. The copper mines in Dewal Dhar, Almora district (U.P.), have been examined in detail. Surveys in Almora district led to the discovery of a new promising deposit of lead at Chhanapanni with a traceable continuity over a distance of more than a mile with fairly rich concentrations at places. Structural mapping of the Kolar goldfields to study the pattern and structural control of the gold-bearing lodes, and to apply the knowledge obtained to trace the extension of the existing lodes outside the present mine area and also to prospect for new lodes, has yielded useful results.

The basis of the new coal policy is the estimated demand of 60 million tons of coal by the end of the second Five Year Plan period. The present assessed demand is of the order of 39 million tons, whereas total despatches amount only to 32 million tons. The result is that current demand is never met in full. The estimated demand in tons, of various consumers of coal by 1960-61 is as follows: Bunker and export 2.88 million tons; railways 14 million; iron and steel 12 million; electricity 4 million; small consumers 3.5 million; sugar 0.5 million; paper 1 million; refractories 0.47 million;

glass 0.5 million; potteries 0.18 million; limestone 0.34 million; inland steamer service 0.3 million; ordnance and defence 0.2 million; jute mills 0.74 million; engineering and foundries 0.58 million; tobacco 0.23 million; chemicals 0.43 million; oil mills 0.53 million; food 0.20 million; tea 0.1 million; re-rolling mills 0.1 million; others 2.88 million, and the cost equivalent of the coke deficiency estimated by the Coke Oven Committee 0.6 million. This totals up to 56.5 million tons. The balance is expected to be consumed at the collieries to make the total of 60 million tons which represents an increase of 23 million tons over the 1954 level. For purposes of planning, demand is divided into the requirements of the iron and steel industry and other industries which use coking coals for their operations and the requirements of other consumers. It is estimated that a production of 16.5 million tons of metallurgical coal will be necessary to meet the needs of essential consumers. Present production is 13.67 million tons, against a pegged limit of 14 million tons. It is now common ground between the Government and the collieries that all coking coal, other than grade III should generally be washed.

There is a growing realization that the use of coking coal needs to be substituted by non-coking coal, particularly by the railways. The new locomotives are being designed to use lower grades of coal. Electrification of railways should further reduce the demand for superior types of coal. The railways which use annually about 4.7 million tons of coking coal have agreed to change their locomotive boilers to enable them to use non-coking coal in place of coking coal which will be released for metallurgical use as follows:—

	million tons
By April 1, 1957	... 1.7
By April 1, 1958	... 1.5
By April 1, 1959	... 1.5

Another question engaging the attention of the Government relates to developing alternative supplies in regions other than the Raniganj and Jharia coalfields. Development of zonal production is being re-emphasized. This can be brought about to some extent by an increase in the production of the coalfields in Assam, Hyderabad, Vindhya Pradesh, Madhya Pradesh, and of the Talcher, Korba, and North and South Karanpura coalfields and by the development of lignite deposits in the South.

A team of British experts from Powerill Duffryn, made available under the Colombo Plan, surveyed the lignite deposits at Neiveli, Madras State, and reported to the Government of India on its findings. It has now been decided to go ahead with a project expected to cost Rs. 58 crores. The scheme visualizes development of mining so as to produce 3,250,000 tons of lignite a year, development of 200,000 kW of power and construction of factories to produce annually 700,000 tons of lignite briquettes, 350,000 tons of carbonized briquettes and 20,000 tons of ammonium sulphate. The scheme has been so framed that as it progresses some of the features of alternative schemes, such as producing coal tar and synthetic petrol, could be dovetailed into it. The ultimate objective is to make the whole of South India self-sufficient in industrial coal. There is brown coal at Bikaner also and measures are being taken to develop those mines. The Geological Survey recently announced the discovery of lignite at Umarsar in Kutch where a reserve of 10 million tons is estimated.

It might be worthwhile reviewing the latest Indian and world production figures of important non-ferrous metals.

Copper.—World output of copper 2,700,000 tons. India's production of 7,192 tons per year, satisfies about 28% of the total demand for copper in the country. In 1954-55, India imported 27,000 tons.

Zinc.—World output of zinc is 2,803,000 tons. There is no production on commercial scale in India. In 1954-55 India imported 44,000 tons.

Lead.—World output of lead is 2,932,000 tons. India's output of about 2,370 tons satisfies about 14% of the country's requirement. In 1954-55 India imported 10,000 tons.

Tin.—World output of tin is 167,000 long tons. India's production is negligible. In 1954-55, India imported 4,000 tons.

The targets for 20 key industries to be attained at the end of the third Five-Year Plan (1966) have been quoted at : iron and steel, 15,000,000 tons; pig-iron, 2,000,000 tons; aluminium, 70,000 tons; cement, 32,000,000 tons; ferro-manganese 300,000 tons; refractories 3,000,000 tons; nitrogenous fertilizers, 1,000,000 tons; phosphatic fertilizers 350,000 tons; sulphuric acid 1,000,000 tons; caustic soda, 300,000 tons; soda ash, 700,000 tons; petroleum,

7,500,000 tons. To attain these targets, in the background of Indian raw material conditions metallurgical research in general and the National Metallurgical Laboratory in particular will have to play very important roles.

The world's consumption of metals and minerals is rising at a significant rate. For example, during the past 100 years the demand for iron has increased more than a hundredfold, for mineral fuels seventy-five times, and for copper and other metals sixty-five times. Table III illustrates the increased demand for some important minerals between 1938 and 1953. This heavy consumption of metals and minerals is creating serious problems of supply which is being tackled by different countries in different fashions. One of the purposes of the present expedition to Antarctica is to discover workable deposits of minerals beneath the ice cap. Even if virgin fields are discovered, it can hardly be more than a few centuries before all the main deposits of minerals in the earth's crust will have been seriously depleted.

TABLE III—WORLD PRODUCTION
(in millions of metric tons)
(Year Book. A. Bul. Metal Statis.
Miners—Year Book)

Mineral	1938	1945	1948	1951	1953
Coal (all grades)	1469	—	1719	1915	—
Iron	162	160	217	293	—
Copper	2.19	2.40	2.56	2.90	3.07
Lead	1.80	1.24	1.55	1.81	1.98
Zinc	1.56	1.75	1.98	2.46	2.72
Aluminium	...	0.95	1.35	1.98	2.64
Tin	...	0.10	0.17	0.19	0.20

With this background of general data concerning our mineral wealth, let us now examine the question—how to utilize our mineral reserves to the best national advantage and industrial output? Until recently most of Indian non-ferrous mineral wealth was mostly won for export but this policy is now being re-oriented towards conservation of resources and early development of home industries, without at the same time, wholly closing the door to the export of minerals which are of potential value to our national exchequer, such as, manganese. The successful and economic exploitation of ferrous and non-ferrous minerals is made possible only by the evolution of highly efficient methods for separating the gangue and various minerals from one another and by developing extractive metallurgical processes to suit individual ores—processing and converting them into useful engineering metals and alloys. The problem that is put to a laboratory like ours can be formulated simply. How can we win

minerals and metals from relatively low grade ores and also from ancient dumps of tailings and slag heaps which were once worked upon by our ancestors, such as, the old slag dumps containing lead and zinc at Zawar mines in Rajasthan? These requirements place a heavy duty on metallurgical research and development: a responsibility which receives further emphasis because of the following reasons:—

1. Much of India's mineral wealth is relatively poor grade (particularly in relation to impurity content) and limited reserves in any one area, which militates against the metal contents being utilized efficiently and economically along lines of processes which are 'standard' elsewhere in the world. New extraction and processing techniques therefore must be developed.

2. Some of the coals which must be used as reducing agents in metal winning share with the ores the deficiencies mentioned above; and then there has so far been little experience in electro-metallurgical techniques on industrial scale for metallurgical smelting and refining work.

3. The metals indigenous to India are relatively few, whatever their potential reserves may be, and do not include several regarded as essential by the engineers of other countries. Thus, at an early stage of her industrialization, India needs to develop substitute alloys. As an instance, the large occurrence in India of the main ore of titanium, ilmenite, encourages special interest in it. Similarly, full use of rare earth group of elements in alloy steel metallurgy assumes importance considering India's richness in these.

Both the public and the private sectors have rightfully taken heavy responsibilities in the future development of iron and steel industry, the utilization of iron, manganese, chrome, gypsum and sulphur ores and processing of other non-ferrous ores. These objectives open up new horizons before us for evolving mineral beneficiation processes and extractive metallurgical techniques for the purpose of winning and exploiting India's mineral resources to the maximum national advantage. The slag dumps and tailing heaps of today may well be the mines of tomorrow, as the ores become leaner requiring new techniques in mineral dressing and extractive metallurgy. Improved technology alleviates the situation when it gets critical. Copper mine at Utah in 1911 contained 1½% copper with a 70% processing recovery. The same mine today has ore of only nine-tenth of 1% copper even though recovery is 93 per cent.

NATIONAL METALLURGICAL LABORATORY

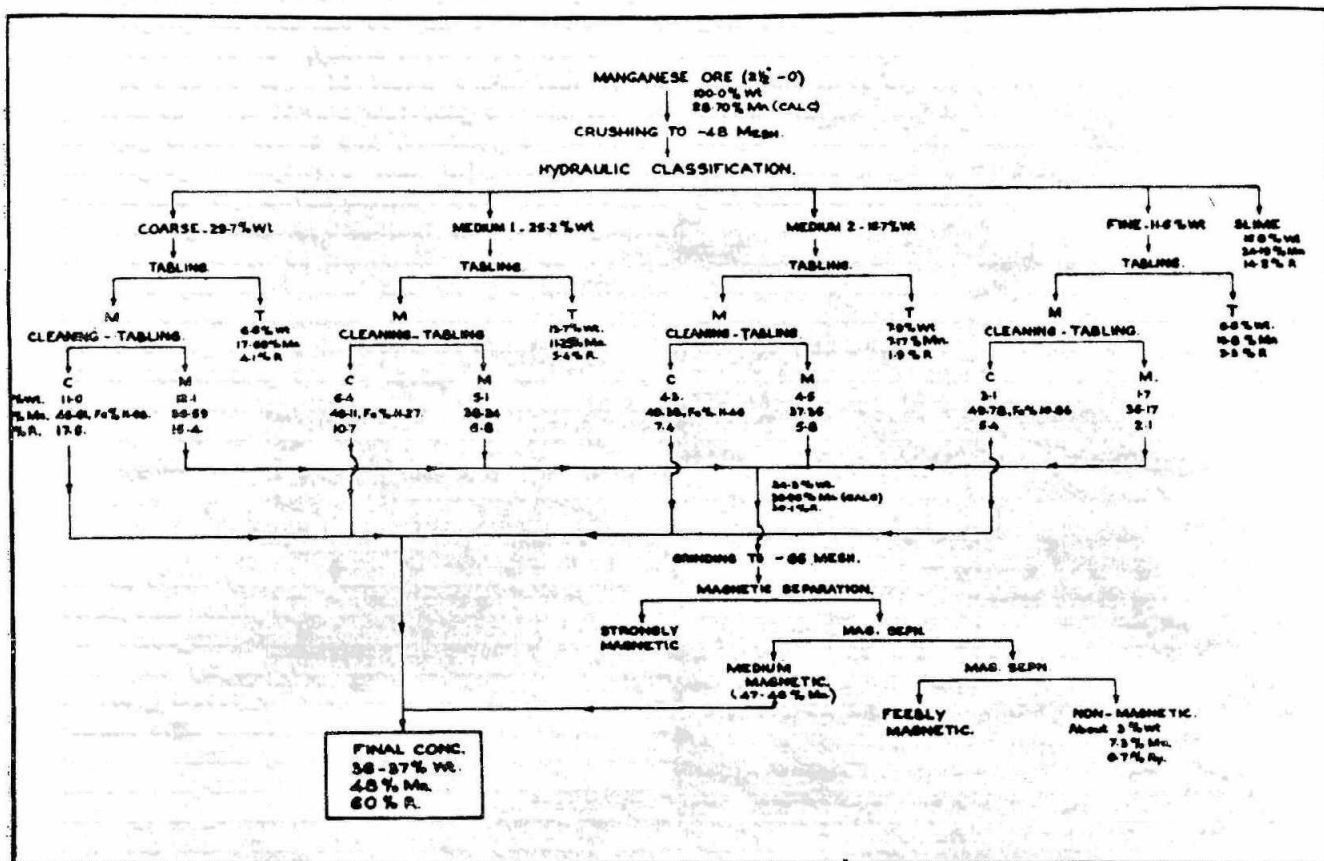
An attempt will now be made to outline the progress of work on mineral beneficiation and extractive metallurgical themes conducted at the National Metallurgical Laboratory. The Labora-

tory is just over 6 years old and, though no spectacular discoveries may be claimed, the progress in diverse fields has been steady. There is also little doubt that in the midst of mass of data on the subject and the problems that confront us needing allotment of priorities, one comes across gaps in our metal research and development programme tuned to maximum national advantage. But if conflicting views were combined 'omnium gatherum'—an unmanageable collection of items will be the result, far beyond the means of any research organisation to tackle. However, efforts at the Laboratory have followed the old adage that limited objectives be chosen in the present context of our development. With these general remarks let us now see what has been done at the National Metallurgical Laboratory on problems covered by the present symposium.

MANGANESE

Excluding Russia, India probably ranks as the world's largest supplier of high-grade manganese ore. The incessant demands for manganese ore made by overseas markets have largely been met by exports from India of raw un-processed ore of high grade. The sub-standard grades have hitherto been chiefly neglected or wholly discarded. High-grade manganese ore is generally hand-picked from the manganese ore burden after blasting in open-cast mining leaving the low-grade manganese ore with no prospects at present for economic utilization. It is roughly computed that for every ton of high-grade manganese ore mined, there remains discarded at the mine site over 1-2 tons of low-grade ore. It has been reported that one single large producer-exporter of manganese ore in Madhya Pradesh has accumulated over six million tons of such low-grade ores at the mine face over the past decades lying as waste. Large dumps of low-grade manganese ore have likewise been accumulated in close proximity to other mines. This material which so far has been dumped aimlessly has got to be turned into a national asset by all possible scientific methods at our disposal. Whilst the estimates of high-grade manganese ores have been reckoned at about 60 million tons, the reserves of sub-standard grades are much greater. Deposits of high-grade manganese ores are not however, inexhaustible. Over 40 million tons of high-grade manganese ores have been exported to world markets during the past few decades. Whilst the indigenous manufacture of exportable grades of standard ferro-manganese in place of large shipments of raw high-grade manganese ore has been recognized as of utmost national importance, mineral beneficiation and upgrading of low-grade manganese ores have not received due attention.

The National Metallurgical Laboratory right from its inception recognized the importance of the



*Flow-sheet for a 48% grade of concentrate
(-46 mesh tabling/magnetic separation)*

subject and initiated survey of manganese ore resources on an all-India basis and undertook investigations into upgrading of low-grade manganese ores.

Concurrently researches have also been undertaken on thermal beneficiation of low-grade manganese ores by a two-stage process and on electrolytic production of manganese and manganese dioxide. The results of a large number of investigations on low-grade manganese ores already completed at the Metallurgical Laboratory have shown that concentration of manganese ores, to render them suitable for standard grades ferromanganese production, is not an easy problem in view of the fact that different manganese ores require varied types of upgrading treatment depending upon the gangue minerals present. Indian manganese ores can be broadly classified under the following categories :—

1. Siliceous ores containing quartz and other light gangue minerals like apatite etc. as the principal gangue.
2. Ores containing ferruginous minerals as the principal gangue.
3. Ores containing appreciable amount of garnet.

4. Complex ores containing gangue minerals falling under two or more of the above categories.

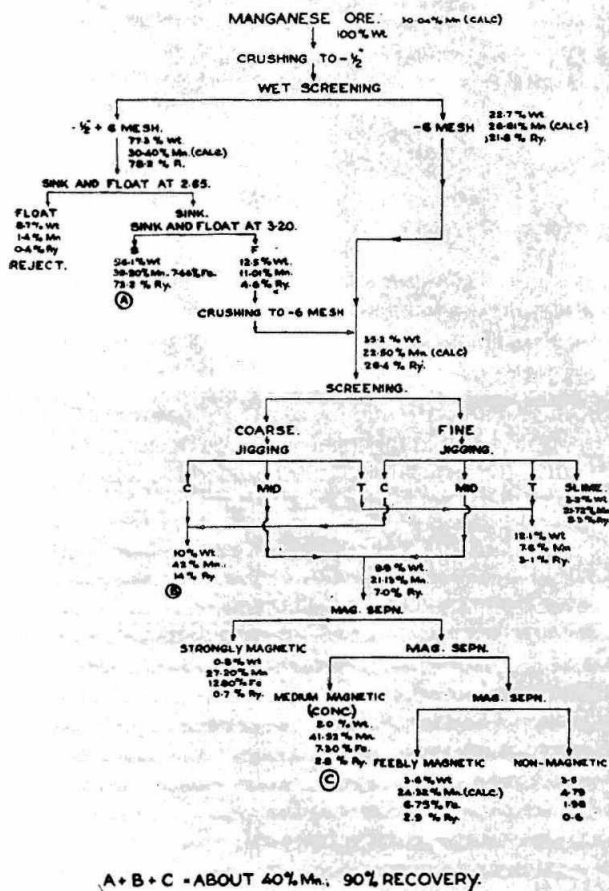
These four types of ores have been comprehensively investigated. Low-grade manganese ores from Mansar Mines, Kachidhana Mines and Balaghat (M.P.), Kodur Mines (Andhra), Shivrajpur Syndicate (Bombay) and Barajamda (Orissa) fall under the first category and require gravity concentration methods such as heavy-media separation, jigging, tabling or straight magnetic separation.

Ores of the second type can be effectively treated by a method (developed at the National Metallurgical Laboratory and patented by the Council of Scientific and Industrial Research) consisting of straight magnetic separation after desliming based on higher magnetic susceptibilities of hematite in relation to manganese minerals, or by a process of magnetizing reduction roast to convert the ferruginous minerals to magnetite followed by low intensity magnetic separation. The method is also applicable to ores containing non-magnetic gangue in addition to ferruginous minerals wherein low intensity magnetic separation following reduction roast treatment will eliminate the magnetite, high intensity magnetic separation will remove the

feebly magnetic manganese constituents and the non-magnetic tailing will comprise siliceous and other non-magnetic gangue. Such treatments can be applied to the following Indian ferruginous manganese ores investigated at the Metallurgical Laboratory :—

1. Nagri-Joida, North Kanara, Bombay ;
2. Chipurupalli, Andhra ;
3. Salur, Srikakulam District, Andhra ;
4. Kodur Mines (Red ore), Andhra ;
5. Sandur, Mysore ;
6. Miragpur Mines, Balaghat, M.P. ;
7. Kamji Mines, Banswara District, Rajasthan ;
8. Sagur, Orissa ; and
9. Keonjhar, Orissa.

In category 3 fall manganese ores from Salur, (Andhra) Banswara District, (Rajasthan) etc. Garnets possess almost the same specific gravity and magnetic susceptibility as manganese minerals and as such, gravity concentration and magnetic separation methods cannot effectively be applied thereto. Electrostatic separation has been found to be the most efficient for separation of garnet from manganese minerals, though flotation in some cases has proved satisfactory.



Flow-sheet for a concentrate of grade 40% Mn. (Heavy-media separation, jigging & magnetic separation)

Under category 4 fall complex ores that contain gangue minerals falling under two or more of the above categories requiring thereby three or even more steps for the elimination of the gangue and production of high grade concentrate. In many cases of this type of ores, garnet, hematite, quartz, manganese silicates, feldspar, apatite, hydrous iron oxides and clay are prominent gangue minerals sometimes present in intimate inter-growth with the ore minerals.

For most Indian manganese ores upgrading flow-sheets have been drawn up based on our investigations and which are currently being made use of by those in the manganese mining and export trade.

The remarks made above, however, should not be taken as meaning that all the problems of the manganese miners have been solved once and for all. It will be noted that the matter has been discussed simply in terms of concentration of manganese. This is not always the whole story. Many manganese ores contain phosphorus in undesirable proportions (i.e. above 0.15%). Most of the phosphorus is intimately associated with the metallic minerals and not with gangue. The result is that gravity and similar methods of concentration tend to raise the phosphorus content of the concentrate. The reduction roast and magnetic separation method results in a partition of the phosphorus between iron and manganese, but the partition is almost equal so that there is no diminution in the phosphorus content from ore to concentrate. This would constitute a serious drawback, except for the providential fact that ferruginous manganese ores are generally not high in phosphorus. For manganese ores in general, the problems cannot be claimed to have been solved, however, until some more positive step can be taken about phosphorus. It is known from work at National Metallurgical Laboratory that flotation, following cheaper methods of concentration, offers some promise.

Another complication remaining is that of garnet. Garnet has a similar density to manganese minerals and is also slightly magnetic. It is accordingly difficult to lay down on the basis of present knowledge an effective schedule for treating garnet-containing manganese ores, with recourse to flotation or electrostatic methods which are usually regarded as over-expensive.

Side by side with the ore-dressing work, three other investigations on manganese have gone forward. The first has been concerned with thermal beneficiation, that is to say concentrating the manganese from a ferruginous ore into a slag, while reducing out the iron. The slag can then be smelted for manganese. One attraction of such a process is that the phosphorus passes largely to the iron. The second investigation concerns electro-

lytic manganese. This, when made at a low enough cost, is fully competitive with ferro-manganese and will in fact serve for purposes for which this is unsuitable e.g. making high-manganese low-carbon steels. Deposition is readily carried out from a sulphate bath according to a process defined at the Laboratory. The more difficult side of the process lies in finding economic methods of producing the sulphate bath especially from low-grade ores. The first problem is that the state of oxidation usually needs to be lowered before the manganese mineral becomes soluble in dilute acid. Reduction with gaseous and other fuels is almost prohibitively expensive. It has, however, been found at National Metallurgical Laboratory that digestion with dilute sulphuric acid, or spent electrolyte, in the presence of saw-dust suffices to reduce the ore and take the manganese into solution. The third investigation has dealt with the electrolytic production of manganese dioxide, to take the place of expensive imported ores for dry battery manufacture. The objective has been achieved.

CHROMITE

Chromite is the only commercial ore of chromium. Theoretical composition is $\text{FeO} \cdot \text{Cr}_2\text{O}_3$, but in nature it occurs as $(\text{Fe}, \text{Mg})\text{O} \cdot (\text{Cr}, \text{Al}, \text{Fe})_2\text{O}_3$.

Chromite is usually associated with ultra-basic rocks such as dunite, peridotites, pyroxenites and serpentines occurring as lenses or tabular masses. Chromite occurring in granular form is usually associated with serpentine, magnesite, talc, pyroxene, olivine, asbestos, dolomite etc.

Jigging, tabling and treatment on Humphrey's spiral are the usual methods. Magnetic separators are also sometimes used depending on the ore. Cr/Fe ratio could be improved (by reduction roast) if at least a part of iron content of the ore is present in the form of another mineral apart from chromite. But if the chromite mineral itself is poor in $\text{Cr}^{2+}\text{O}^{3-}$ content or Cr/Fe ratio, no mechanical process can produce a suitable concentrate. In such cases, chemical methods of beneficiation may have to be adopted.

Specifications

1. Metallurgical—48% Cr_2O_3 , Cr/Fe = not less than 2.8 to 1.

S	—	not to exceed 0.5%
P	—	not to exceed 0.2%
MgO plus Al_2O_3		not to exceed 25%
2. Refractories—Low silica is the main consideration. Chromite should be evenly distributed through the ore and should not occur as coarse grains with aggregations of silica. Cr_2O_3 over 40%.

Chromic oxide plus alumina	63%
iron oxide	... max. 10%
SiO_2	... max. 5%
3. Chemical—

Cr_2O_3	... 44% min.
SiO_2	... 5% max.

Indian occurrences

Keonjhar, Cuttack, Singhbhum district, Mysore State, Salem, Ratangiri.

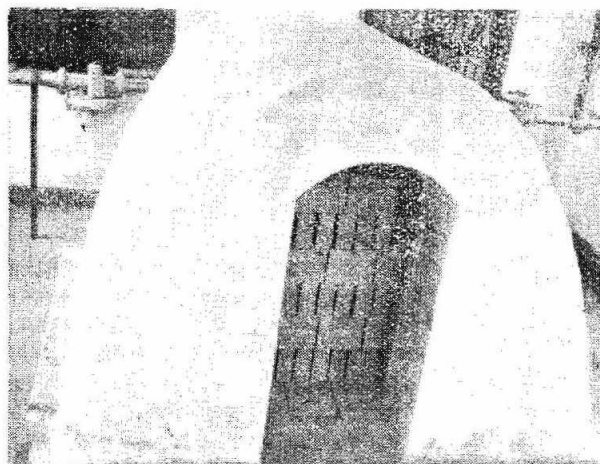
Estimated so far—1,320,000 tons.

Indian chrome ores in general may be characterized by their high iron content which, being present in chemical combination with mineral chromite, cannot be reduced by mechanical processes.

Ten low-grade chromite samples from different mines situated in the states of Mysore, Bihar, and Orissa, have been investigated at the laboratory to study their amenability to the various beneficiation processes. The Indian chrome ores can be broadly classified into the following categories:—

1. Simple ores containing serpentine, talc, chlorite, magnesite, calcite and dolomite as principal gangue minerals.
2. Ferruginous ores containing hydrated oxides of iron as the principal gangue.
3. Chrome-spinel.

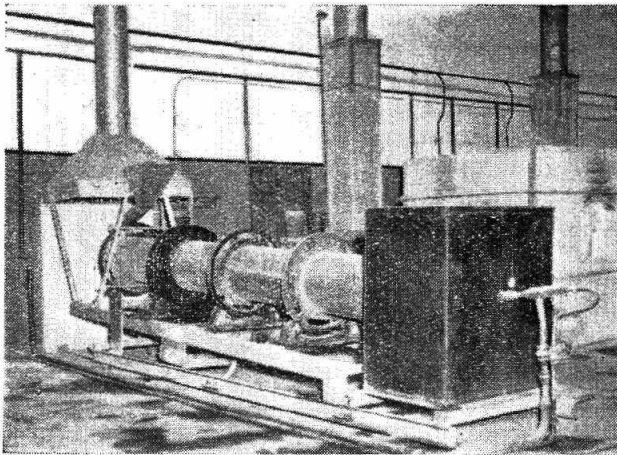
Simple ores, like samples from Nausahi in Orissa and Kittubara in Bihar, could be easily concen-



Catenary arch kiln designed and constructed at N. M. L. for firing 500 bricks to a temperature of 1750 deg. C. Such pilot plant trials are needed for the different projects under way in the refractories division.

trated by some of the well-known ore-dressing methods. The treatment practised in most cases, consists of fine grinding and elimination of admixed impurities by the use of jigs, concentrating tables or Humphrey's spirals, as best suited to the specific nature of the ore. In spite of obtaining high-grade concentrates, the Cr. to Fe ratio in most cases is low. The concentrates could be used for chemical and refractory industries but not for production of standard ferro-chrome.

Ferruginous chrome ore from Gujang in Orissa, contained limonitic ochre as the principal gangue. The sample was subjected to reduction roast followed by magnetic separation of the reduced ore after



Rotary kiln designed and fabricated at N. M. L. for calcining refractory raw materials at a maximum temperature of 1600 deg. C.

crushing to yield a high grade of chromite concentrate having a Cr. to Fe ratio of 3 : 1. The concentrate could be used for production of standard grade of ferro-chrome.

The chrome-spinels like those from Mysore cannot be beneficiated to any appreciable extent by ore dressing methods.

GRAPHITE

Six graphite samples from Betulganj, Madhya Pradesh; Kolar, Mysore; Titilagarh, Orissa; and Attipra, Trivandrum were investigated. It was found that good grade of graphite concentrates could not be obtained from samples from Madhya Pradesh and Mysore due to very intimate association of mica and quartz with graphite. But graphite in the samples from Orissa and Trivandrum, was found to be liberated from gangue minerals at fairly coarse sizes. High grade of flotation concentrates assaying over 90% carbon, obtained from these samples could be used for crucible manufacture.

PYRITE

Shortage of sulphur in the country has necessitated a thorough search for minerals containing sulphur. A number of low-grade pyrite samples from Amjor, Bihar; Chitaldrug, Mysore; and other localities, as well as the coal washery rejects from Nowrozabad which are high in sulphur, were tested at the Laboratory. High grade of pyrite concentrates with high sulphur recoveries, could be obtained from all the samples by employing gravity and flotation methods. The concentrates were found to be suitable for manufacture of sulphuric acid.

In the case of the coal washery waste a two ton sample was treated in pilot plant tests and yielded a concentrate assaying 44.0% S (82% pyrite) and 5.6% C, with a recovery of 50.0% S. Such a pyrite

concentrate should be completely suitable for burning in sulphuric acid manufacture. The coal washeries, together with the known ore reserves, should be capable of providing well over 10,000 tons pyrite per annum.

URANIUM ORES

Nine low-grade uranium samples including the uranium-bearing copper flotation tailing from Indian Copper Corporation Limited, Ghatsila, have been investigated and methods have been determined for concentrating some of the samples employing gravity, flotation and magnetic methods. The results have been reported to the Atomic Energy Commission.

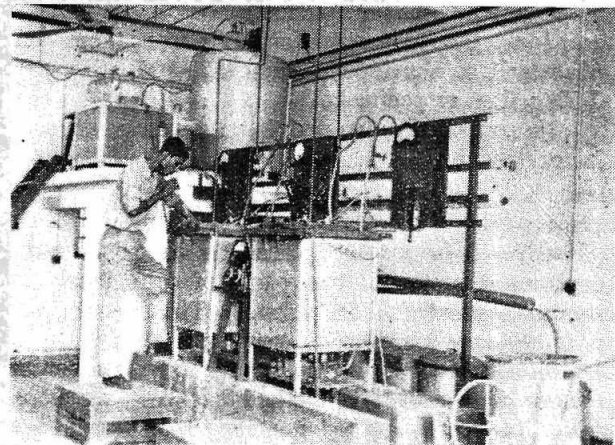
MISCELLANEOUS

Successful methods have been found for concentrating quartz-sand for steel foundry purposes and vermiculite samples from West Bengal and Mysore States.

Raw materials for Bhilai Steel Plant were investigated and methods were developed for washing and concentration of their manganese and iron ores. Optimum conditions have been determined for producing a good iron blast-furnace sinter from their iron ore fines, derived from ores of Rajharapahar, Madhya Pradesh, and other related raw materials. The effects of variables such as coke and moisture contents in sinter mix, basicity and raw material proportions, on the sintering time and their effects on the quality of sinter produced, have been studied with a sintering plant designed and fabricated in our Laboratory.

ELECTROLYTIC MANGANESE

A method for production of electrolytic manganese has been developed at the National Metallurgical Laboratory which can deal effectively with all kinds of low-grade manganese ores and can produce 99.95% pure manganese metal. The low-grade ores



Electrolytic manganese pilot plant designed and set up at N. M. L.

are first powdered and digested with a liquor containing sulphuric acid, usually the spent liquor from electrolytic cells, at about 90-100°C, with saw-dust as the reducing agent. The supernatant liquid is treated in a second stage with a small quantity of dioxide manganese ore when organic matter, iron, arsenic, phosphorus and silica are removed. The filtrate from this stage is purified by treatment with ammonium sulphide which removes all traces of heavy metals. The pH, and concentrations of manganese sulphate, ammonium sulphate and sulphur dioxide of this liquor are then adjusted to suit as a feed liquor for electrolytic cells. Alternatively, the manganese ores can be reduced with coke-oven gas and dissolved in the spent liquor from the electrolytic cells. The pregnant liquor from this stage is then further processed for removal of iron, arsenic, phosphorus, heavy metals and silica and adjusted as stated before for being used as a feed liquor for manganese cells. Recovery of manganese from ores is over 90%.

The purified liquor is then electrolyzed in lead lined diaphragm cells between silver lead alloy anodes and 18 : 8 titanium stabilized stainless steel cathodes. 99.95% pure manganese metal is obtained in the form of flakes at a C.E., of 60-65%. About 3.2 to 3.5 kWh are consumed for one pound of manganese metal. This process has been thoroughly examined on a laboratory scale and is now under operation on pilot plant scale with a capacity of 16-32 lbs. of metal per day.

Studies have also been successfully carried out for producing electrolytic manganese dioxide. It is hoped that India's complete requirement of 5000 tons of electrolytic manganese dioxide for dry cell manufacture can be met from indigenous sources and foreign exchange to the extent of Rs. 7.5 million saved.

The electrolysis for the production of electrolytic manganese dioxide is carried out at 70-90°C, with anodic current density of 0.75 to 1.25 amp/dm², the concentration of manganese sulphate and sulphuric acid being 150-300 g/litre and 0-50 g/litre respectively.

The products obtained have given very satisfactory results.

ZIRCONIUM

India is favourably placed as regards the zircon and it occurs to the extent of 4 to 6 per cent in the Travancore beach sand. The metal is important for use in atomic reactors.

As a first step for the production of zirconium dioxide an electrolytic method has been developed which consists in fusing the zircon sand with potassium silico-fluoride. The sintered product is leached and electrolyzed. The sediment of the hydroxide is filtered, washed and ignited and a

pure zirconium dioxide obtained. The product could thereafter be used for chlorination.

BERYLLIUM

This brittle metal is difficult to extract economically—once called the 'World's Number-One Metallurgical Headache' because of its high melting point coupled with its high vapour pressure at temperatures little above the melting point and its apparent toxicity—seemed to have reached its limit of application.

Then sixteen years ago beryllium in the atomic energy field became the new directive for research into industrial beryllium. The metal was seen to be of great value in reactors, the most effective practical element of low atomic weight which could serve as moderator capable of 'slowing down' high-velocity neutrons. Moreover, beryllium metal is also superior to other materials as a reflector of neutrons.

The principal source of beryllium is the mineral beryl which occurs fairly widespread in Rajasthan, Bihar, Andhra. The metal finds wide use as moderator in nuclear reactors and as an alloying agent for the copper and nickel to produce precipitation hardening alloys.

As a first step towards the production of beryllium metal, investigations were taken up to produce beryllia. The electrolytic method developed consists in electrolyzing a solution of sodium beryllium fluoride obtained by leaching with water a sinter of beryl and sodium silico fluoride. The leach liquor containing the complex salts of beryllium forms the catholyte in the outer vessel and a 10 per cent sodium chloride solution contained in a porous pot placed at the centre of the vessel is the anolyte. On electrolysis of the solution a slurry containing the beryllium hydroxide is obtained in the cathode compartment which on washing, drying and ignition gives the oxide.

The electrolytic process offers advantages over the conventional chemical process in that the product is purer and the process is simpler and much cheaper with a cost estimate of As. 0-14-0 per lb. instead of Rs. 6-8-0 per lb. by the chemical process.

TITANIUM

Titanium is a metal of great interest to India in view of the large reserves of ilmenite and occurrence of rutile in India. As a first step in establishing the possibility of making titanium metal here, chlorination experiments were made on ilmenite. A process for the preferential chlorination of iron from ilmenite has been worked out. The process consists in the removal of almost the entire amount of iron by selective chlorination leaving a product that could subsequently be chlorinated with chlorine and a reducing agent to

produce titanium tetra-chloride. It is hoped that the process offers great scope for commercial exploitation and the reduction in the cost of titanium tetrachloride. An apparatus was then constructed for reducing titanium tetrachloride to study the best conditions for production of titanium sponge by Kroll's process on a scale of 1-lb. per batch. The effect of the rate of addition of titanium tetrachloride to the magnesium used as a reducing agent and the influence of using magnesium in more than the theoretical amount were studied. In order to stop even slight hydrolysis of $TiCl_4$ and also to minimize the risks in transferring large bulk of $TiCl_4$ from the distillation assembly to the reduction set-up, the distillation assembly was made a part of main reduction assembly. The apparatus worked quite satisfactorily.

ELECTROLYTIC METHODS

Amongst these is the electrolysis of titanium trichloride. It was not possible to buy anhydrous titanium trichloride for use in the electrolytic cell. Even the reduction of titanium tetrachloride with the help of St. Claire Deville 'Hot-cold' tube gave difficulty. Attempts were then made to produce titanium trichloride by reduction of titanium tetrachloride with hydrogen at a heated tungsten rod, but so far without much success. Another possibility lay in the thermal decomposition of titanium oxalate. In attempting to follow this up, it was observed that, on addition of absolute alcohol to a solution containing oxalic acid and ortho-titanic acid, a precipitate is thrown down. The precipitate is highly soluble in water and oxalate-titanium dioxide ratio is 1.07 to 1.1. Various factors like temperature of solution, volume of solution, acidity, etc. leading to precipitation are being studied. Unfortunately the precipitate has proved to be of titanyl oxalate, which on heating decomposes to a sub-oxide of titanium and not to titanium metal.

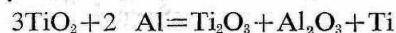
Attention is being directed now to the decomposition of titanium-aluminium alloy (50%) which can readily be produced by the alumino-thermic process.

PREPARING TITANIUM SALTS

Preparation of titanium salts that may be of any use in metal winning was taken up and a new method for producing titanium tetra-iodide was developed. The method consists in heating aluminium powder, iodine and titanium dioxide in a sealed evacuated glass bomb. The process gives a titanium tetra-iodide and after subsequent purification the salt could be used for making pure metal by Van Arkel's process. The process would be a definite improvement on Van Arkel's process as a much cheaper raw material, titanium dioxide, is used instead of crude sponge.

ALUMINO-THERMIC REDUCTION

Modified alumino-thermic has been applied to the preparation of aluminium-titanium alloys by the reduction of titanium dioxide. To understand the nature of the reaction, theoretical aspects of the reduction were considered, according to the equation :



Theoretical calculations indicated that a temperature of 1600°C. could be attained but would not be high enough to melt the reactants and products. Consequently preheating was adopted.

It was observed that intimate mixture of titanium dioxide powder and aluminium powder reacted at about 580°C. but without any metallic separation. A comprehensive study of various factors like addition of cryolite, cryolite and fluorspar, holding time, preheating temperature, and method of mixing the reactants has been made. Cryolite helped in getting better titanium content in the alloy. Addition of calcium fluoride to cryolite as a flux did not show any improvement. It was noticed that the reaction and the separation were favoured by a definite time given for the reaction. Higher temperature gave higher titanium contents in the alloy, but different mixing techniques did not have any pronounced effect.

Titanium-aluminium alloys were prepared by modified alumino-thermic reduction with the following conditions :—

1. The charges was preheated to near about 1000°C. and higher temperatures were found favourable for increasing titanium contents in the alloy.
2. Cryolite equivalent to approximately 20% alumina solubility was necessary for the reaction and separation of the alloy.
3. Fluorspar additions to cryolite did not serve any useful purpose.
4. Holding time of 25 minutes was found to be the optimum for the preparation of the alloy.
5. Preheated titanium dioxide additions to molten aluminium instead of addition of molten aluminium to suspended TiO_2 in cryolite did not show any difference in the titanium contents of the final alloys.

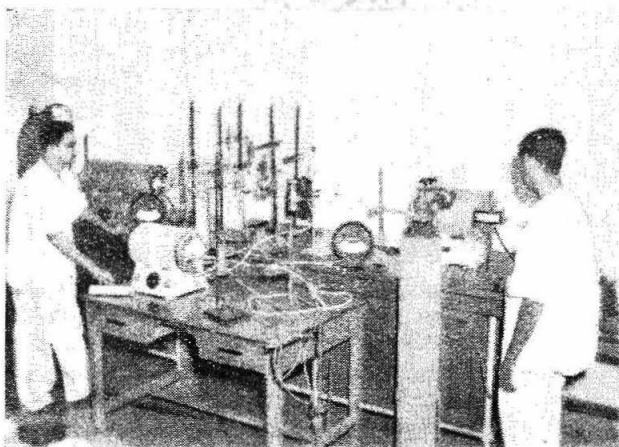
It was concluded that by preheating the charge near about 1100°C. using cryolite for 20% alumina solubility and giving 25 minutes of holding time, a titanium-aluminium alloy containing about 19% titanium could be made by the modified alumino-thermic reduction, but higher amounts of titanium could not be obtained by this technique.

An attempt to produce alloys of higher titanium content using energizers, such as, BaO_2 , $KClO_3$, Na_2O_2 etc. was made. Good results were obtained with $KClO_3$ and BaO_2 . Ultimately alloys of 65% titanium contents using BaO_2 as energizer and 58%

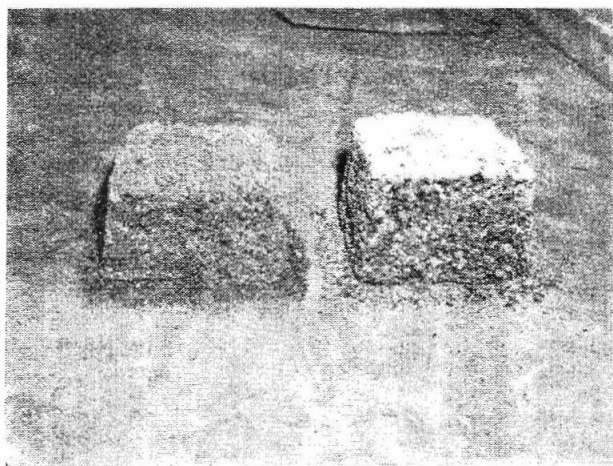
titanium with $KClO_3$ as energizer were successfully obtained. The aim of this project is to utilize these master alloys in the development of creep-resistant alloys and to recover titanium by preferential removal of aluminium by methods yet to be explored.

FERRO-CHROME PRODUCTION

India has got sufficient reserves of chromite—the only source of metal chromium, but the Cr : Fe ratios being mostly very low, it cannot be used as such for manufacture of high-grade ferro-chrome, suitable for alloy steel making. Most of the Indian ores cannot be enriched to obtain the required Cr : Fe ratio by ordinary ore-dressing methods as the excess iron present in the ore is not in the form of free gangue but in chemical combination with the ore mineral. Only method of enriching this type of ore is to attack the spinel structure chemically and eliminate as much iron as possible.



An experimental set-up to study reduction of iron-oxides.



Samples of good and bad 'iron sinter' obtained at N.M.L.

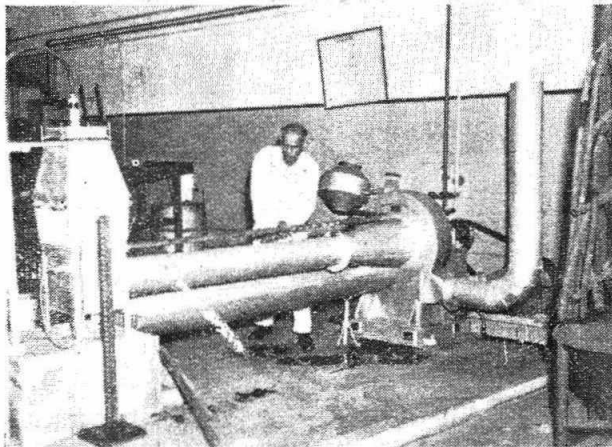
Work on two ores were taken up and it has been possible to enrich both of them to meet the specifications. The process consists of reduction of crushed ore (-7 mesh) with coke oven gas between $900-1000^{\circ}C$, followed by leaching the crushed calcine with dil H_2SO_4 at about $105^{\circ}C$. The leached solution which is high in $FeSO_4$ content may be utilized in the ferro-vanadium project for precipitating iron-vanadate. One ore from Nausahi, Orissa analysing $50\% Cr_2O_3$ (Cr/Fe—2.92) gave a concentrate assaying $53.15\% Cr_2O_3$ having Cr : Fe ratio of 8.12 : 1, with a recovery of 80.6% . Another ore, analysing $49.11\% Cr_2O_3$ (Cr : Fe ratio of 1.8 : 1) from Mysore, gave a concentrate assaying $55.46\% Cr_2O_3$ having Cr : Fe ratio of 4.08 : 1 with a recovery of 90.6% on reduction at $950^{\circ}C$. Best concentrate from this ore was obtained by leaching the ore (pre-oxidized at $850^{\circ}C$.) reduced at $1000^{\circ}C$. Assaying 53.15% having Cr : Fe ratio of 7.30 : 1 but with a lower recovery of only 63.4% . Further work is under progress to find out the optimum conditions of leaching for best grade and recovery. Concentrates made by this process can be utilized for the manufacture of very high-grade ferrochrome by another process developed in this Laboratory.

This process consisting in the production of low-carbon ferro-chrome by the reduction of chromite ores having Cr/Fe ratio of 2.63 to 4.03 with ferro-silicon containing $73-79\% Si$ in an indirect arc furnace has resulted in a ferro-alloy containing $58\% Cr$, $0.8\% C$ and $5\% Si$ instead of theoretically calculated amount of $66-68\% Cr$. The particle size of the charge has practically no influence on the process. As a reductant Si is not 100% efficient as in spite of its presence in the alloy the slag contained $15\% Cr_2O_3$ and only $64\% Cr$ is recovered. Increase in the basicity of the slag has slightly lowered its Cr_2O_3 content. It appeared that an equilibrium is established between the slag containing $15\% Cr_2O_3$ and the metal containing $5\% Si$ and therefore one stage reduction is not possible. Slag basicity index of 1-1.2 has resulted in a clean separation of the alloy. The addition of aluminium towards the end has lowered the amount of Cr_2O_3 in the slag and substantially increased the recovery of chromium. It has been found that with the grades of chrome ore and ferro-silicon used ferro-chrome containing $58\% Cr$, about $0.8\% C$ and $5\% Si$ is obtained instead of theoretically calculated chromium content of $66-68\%$, when the ratio of the amount of Cr_2O_3 in the charge to that of Si in ferro-silicon employed for the reduction, is in the neighbourhood of 3. Particle size of the ore and ferro-silicon did not appear to affect the process. As a reductant Si is not 100% efficient as in spite of its presence in the alloy the slag contained $15\% Cr_2O_3$ and only $64\% Cr$ is recovered. Increase in the basicity of the slag has slightly lowered its

Cr₂O₃ content. It appears that an equilibrium is established between the slag containing 15% Cr₂O₃ and the metal containing 5% Si and therefore complete reduction of all the Cr₂O₃ present in the chromite ore by ferro-silicon in one stage operation is not possible. Slag basicity index of 1-1.2 has resulted in a clean separation of the alloy. The addition of aluminium towards the end although lowered the Cr₂O₃ content in the slag and raised the recovery of chromium, caused undesirable rise in silicon content of the ferro-alloy. Further work is required to explore the possibility of lowering the silicon content in ferro-chrome produced by the aluminosilico-thermal reduction method.

PRODUCING STEEL DIRECTLY

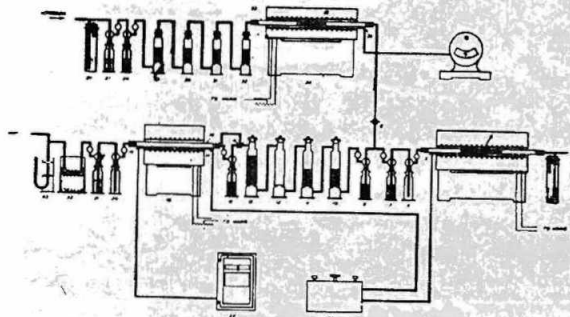
A process of producing steel directly from



Sintering plant designed and constructed at N.M.L.

Indian iron ores has been investigated at the National Metallurgical Laboratory with a view to its application on a cottage industry scale.

In the process developed, crushed iron ore, dried and ground preferably finer than-70 mesh, is packed in a paper tube and embedded in solid charcoal in a sealed steel canister. The purpose of the paper tube or mould is to hold the loose fine ore in the desired shape until the charcoal has been packed round it in metal canister. The paper

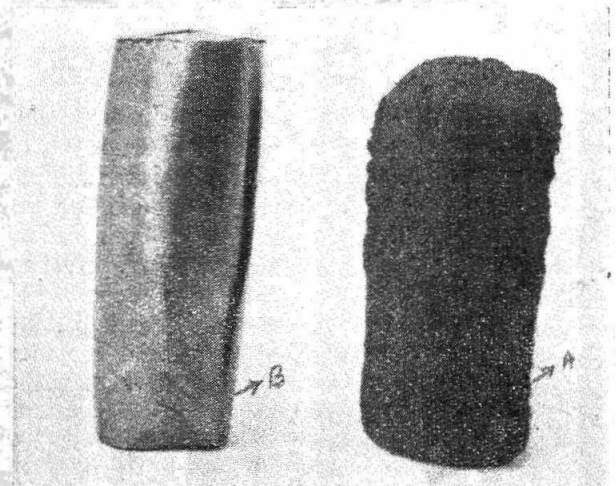


Arrangement for the reduction of CO₂ to generate pure CO gas for iron-ore reduction.

burns off in the subsequent operation leaving a compact mass of the ore. The canister is heated to the reduction temperature and then raised to a sintering temperature of the order of 1100°C. for several hours depending on the carbon content required in the steel. A fairly compact steel bar is obtained after reduction which lends itself readily to hot working. Optimum conditions for obtaining full reduction and the desired density in the compact were determined initially by passing carbon monoxide gas over pellets made from ground iron ore of different mesh sizes. The carbon monoxide was obtained from CO by means of a reducing train. Iron ores from different Indian localities were examined. It was observed that each ore possessed different characteristics in respect of shrinkage, subsequent swelling, percentage loss in weight and reducibility.

When the reduction was carried out with charcoal, and coarse and fine mesh iron ore sizes were mixed together, the resultant density of the reduced product was higher than that obtained when only coarse or fine mesh ore size were individually treated; in the former case, the product showed freedom from surface cracking. Variations in sintering conditions had pronounced effects on the density of the product obtained, for all ores. Reduction at 900°C. and subsequent sintering carried out at 1100°C. gave a denser steel product than when the reduction was done at 750°C. High sintering temperatures such as 1200°C. gave low density products. Addition of mill scale to the charge increased the density of the steel, particularly in the case of ores low in iron contents.

The density of the sintered product obtained



Reduced compacts as obtained before (A) and after grinding (B).

varied from 4 to 7. The compact could be hot worked by forging or swaging, with increase in density. The resultant material was amenable to heat treatment.

It is believed that the process has useful potential applications on the cottage industry scale in this country for the fabrication of small agricultural and other tools in rural areas where high grade iron ore is available.

Iron ores occur in most of the States of the Indian Union but several of the deposits are not rich enough or large enough to be considered as economically workable at the present day. Relatively rich deposits are to be found in Bihar, Madhya Pradesh, Orissa, Bombay, Mysore and Madras. The ancillary materials required for steel making in the normal way are coking coal, flux and refractories. So far, known coking coal is



Reduced compact of 2 lb. in weight after grinding.

available only from the Jharia, Raniganj and Bokaro coalfields in Bihar and to a small extent in the Kanhan valley field in Madhya Pradesh. Ores will have to be crushed and ground for the direct reduction which will cost a considerable amount of outlay for machinery, like, jaw crusher, grinding mill etc. Fairly extensive deposits of powdery ore occur as pockets and lenses passing into one of the other types. The material crumbles to powder easily. Sieve analysis shows that the bulk of the ore will pass through 10 mesh and as much as 40 to 50 per cent may be as fine as 200 mesh. The powdery ore consists largely of hematite with some quantity of martite. This naturally occurring powdery ore is ideally suited for the process. As regards fuel any carbonaceous material such as coal dust, coke, charcoal, can be used. Electric furnace was used in the Laboratory experiments, but for cottage industry a kiln similar to that used for brick making is more suitable.

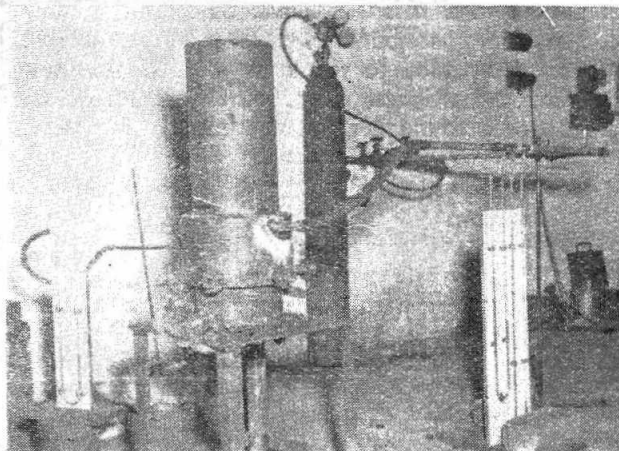
As a matter of fact the industry can be started in conjunction with the brick making industry where the brick kiln will be available and also the labour can be trained easily for making moulds and pots. It may be mentioned here that instead of steel canisters, ceramic pots can be used for holding the paper moulds. These pots can be made out of fireclay. Instead of paper moulds, porous sand moulds can also be used for holding the powdered iron ore.

From the above description it will be seen that the process is very simple and does not require any expensive equipment. The investigation in the Laboratory was carried out on experimental basis with small paper moulds and as such it is difficult to give any definite figures on the economics of the process at this stage of the investigation.

ALUMINIUM-SILICON ALLOYS

Alloys of aluminium-silicon have today attained considerable commercial importance in their wide use for multifarious applications and as much as 80% of the world's production of light alloy castings has been estimated to belong to the family of these alloys.

Comprehensive investigation was undertaken at the National Metallurgical Laboratory on the preparation of aluminium-silicon alloys by the aluminothermic reduction of quartz. Based on thermodynamical and experimental data obtained, the reduction of quartz has been observed to depend upon a number of factors, the chief of which are: particle size of quartz, amount of cryolite in the charge, temperature of the reactants and holding time. Physical properties of the alloys experimentally made by this method have been determined and compared with the physical properties of corresponding alloys produced experimentally here by direct alloying of the two metals. This comparison yielded identical results in the



A small 5" dia. Cupola employing oxygen enrichment of air-blast designed and fabricated at N.M.L.

two cases. Reference to the general economics of this process has also been made. It has finally been stressed that this method can be usefully exploited for commercial applications in countries dependent on silicon imports and rich in bauxite and quartz such as India. The process is simple and can be easily worked without any complicated prior treatment of raw materials. The temperature necessary for aluminio-thermic reduction is easily attainable and therefore does not need any costly equipment. The reduction and alloying of silicon can be completed at one stage. It is, therefore, recommended that this process can be adopted for commercial exploitation in countries dependent on silicon import and rich in bauxite and quartz.

Mention may also be made of secondary metal recovery work done at National Metallurgical Laboratory such as recovering of zinc from dross, copper from scrap etc.

FURTHER PROGRAMME

The future programme of work relating to extractive metallurgical process has been kept quite fluid. Some important research themes along which work is proposed to be directed are given below.

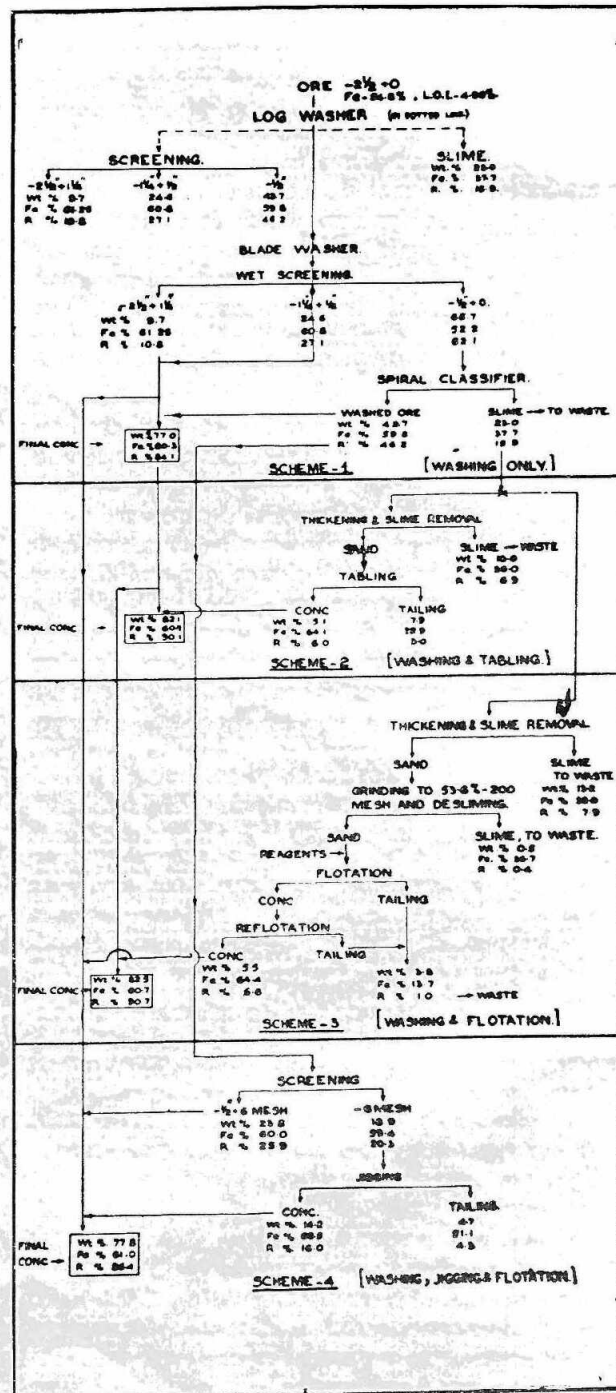
It will be worthwhile to investigate the possibilities of alternative methods of aluminium production. It is known that 'Catalytic Distillation of Aluminium' offers interesting possibilities not only for the production of aluminium from the ore but also from scraps. With the setting up of unit-process equipment at the National Metallurgical Laboratory catalytic distillation of aluminium from titanium-alloys obtained through aluminio-thermic reduction will be attempted.

Development of extractive technique based on fundamental thermo-dynamic theory to apply to different Indian non-ferrous ores will be pursued—attention being focussed on two themes viz., (i) study of gaseous oxidation and reduction of mixtures and (ii) metallic reduction processes. Development work is proposed on pressure leaching of ores to replace conventional modes of extracting metals—such as, pressure leaching of copper ore with the object of making electrolytic purity copper which may replace conventional costly modes of thermal refining followed by electrolysis.

This new technique of pressure leaching of ores followed by selected reduction by hydrogen can be used to produce high purity nickel, cobalt, and copper at a very low cost. The oxidation of sulphide to sulphate is exothermic and supplies the heat required to raise the solution to the leaching temperature. This new technique is receiving much attention in the U.S.A. and Canada. The process depends upon the solution of copper from copper concentrate on being treated with ammonia under pressure of air. Copper is subsequently precipitated as powder by reduction with hydrogen under

pressure when ammonia is regenerated and can be used in first step. The process can be adopted with the same equipment for aqueous oxidation of pyrites in caustic soda medium.

In collaboration with Tata Iron & Steel Co., Limited, use will be made of the pilot low shaft



Flow-sheet for laminated iron-ore.

furnace of 15 tons a day capacity (i) for utilising non-coking coal for iron smelting with or without oxygen injection (ii) for the experimental produc-

tion of ferro-manganese and other ferro-alloys with or without oxygen injection.

On the electro-metallurgical side attention will be given to aqueous electrolysis i.e. electrolytic production of Mn, MnO_2 , Cr, Cu from copper scrap, Sn from tin scrap, Zn, etc.

Attention will also be given to the production of purified metallic chlorides, oxides and other half-products in metal extraction and also carbides, nitrides, etc. which can be put to direct application. A scheme has been submitted to the Atomic Energy Commission for the preparation of small quantities of separated metals such as cerium, lanthanum and didymium, which is important not only from the applied fields relating to beneficial utilisation of the separated metals in many branches of ferrous and non-ferrous metallurgy but also from the fundamental aspect of developing extractive metallurgical technique for their separation.

The work on the preparation of rare earths is proposed to be taken up in these stages detailed below :—

1. Chemical separation of the rare earths.
2. Reduction of rare earth compounds to metals through (a) electrolytic and (b) metallo-thermic reduction.
3. The applications of rare earths to different ferrous and non-ferrous alloys and development of rare-earths-metal alloys as such.

GENERAL

A related field in foundry melting in which encouraging results have been achieved is the setting up of a baby cupola. An improved acid and basic lined cupola has been designed and fabricated which gives much higher temperature than the conventional iron cupolas and enables large steel scrap charges to be melted without any oxygen injection.

Pilot plant schemes related to mineral beneficiation and extraction metallurgy have been submitted on the following for intensive study and implementation during the second Five Year Plan :—

1. Treatment of low-grade manganese, chrome and other Indian ores by ore-dressing methods.
2. Thermal beneficiation of low-grade manganese and chromite ores.
2. Pilot plant on refractories.

It must be recognised that laboratory-scale researches and investigations or patents taken out etc., have to be made full use of through pilot plant trials. We have achieved on laboratory scale some very promising lines of industrial processes that have to be demonstrated to authorities and industrialists on pilot plant scale so that their translation on full commercial and industrial scale can be accomplished. I may perhaps further elaborate

my views a little more. The production of ferro-manganese in a blast furnace requires high grade manganese ores containing 48% Mn, with a Fe : Mn ratio 1 : 6 and above, and low phosphorus content (0.3%). India's resources of low phosphorus, high-grade manganese ores and of low-phosphorus coking coal for ferro-manganese production are limited.

The investigation was carried out to separate iron and phosphorus by thermal reduction from low-grade manganese ore and obtain a slag rich in manganese, which could subsequently be charged into blast furnaces or the electric furnace for the production of ferro-manganese. The process would enable the utilization of low-grade ores and the conservation of high-grade ores and the production of exportable grade of ferro-manganese. The experimental work endorsed the potentialities of the method, but exposed sources of variation which could only be brought under control by extensive pilot plant scale experimentation. This alone could also indicate whether the economics of the method are attractive. I am discussing this subject in another paper.

I may perhaps further point out our other rambling ideas. One of them relates to studies into reducibility of different iron ores. Some laboratory work has lately been done here and can be further extended. On the iron sinter aspects perhaps the well-known CO_2 moulding process applied to silica sands used in foundries, may be investigated with limy additions in order to dispense with Dwight-Lloyd or other forms of sintering iron ore fines. I believe work along the above line is being studied in the U.S.A.

Some of our other thoughts relate to direct reduction of iron pyrites. As we all know India has no indigenous supply of sulphur and has to depend completely on import of this commodity from abroad. In recent years efforts are being made to tap secondary sources of sulphur, viz., pyrite and gypsum, of which India possesses sufficient reserves. Pyrite is generally used for manufacture of H_2SO_4 and pyrite cinders obtained as a by-product, could be utilized as raw material in an iron blast furnace. But utilization of the pyrite cinders in this manner has got two drawbacks : (i) the available quantity of pyrite cinders is set by the requirements of H_2SO_4 plants, and (ii) pyrite cinders require a large input of energy to reduce it to iron in the blast furnace.

These facts make the development of a direct pyrite reduction process attractive. In a series of experiments conducted elsewhere pig iron was obtained by direct smelting pyrite in absence of air with a charge of pyrite and coke mixture in an electric furnace. CS_2 and graphite were obtained

[Continued on page 27]

sintered before export. It will be very inconvenient even to rail these fines as such, without bagging which is costly. Sintering plants require heavy capital outlay and it is quite unnecessary for beneficiation plants to have their own individual sintering units. A sintering plant can serve two or more milling plants in the same region and it should be designed for a capacity to deal with the concentrates from the whole region. Fortunately for the manganese ore industry in India, the mines are concentrated more or less in certain districts, namely Keonjhar (Orissa), Balaghat, Chindwara and Nagpur (Madhya Pradesh), Srikakulam (Andhra) and North Kanara (Bombay); and this suggestion to have regional sintering plants should be most welcome to the industry. They could be financed by the regional producers with or without State aid and located at the nearest railhead or some such suitable place.

During the second Five Year Plan period, all the fine concentrates that will be produced from beneficiation plants will be utilized as such, by the ferro-manganese producers in India in their electric furnaces which can take in such fines. But with the enforcement of the Government policy of conservation of minerals, more concentrate fines will be produced than are likely to be consumed in India and the surplus will need sintering before it can be exported. It is expected that the third Five Year Plan will see the export from India of sintered manganese concentrates.

THE NATIONAL METALLURGICAL LABORATORY IN THE DEVELOPMENT OF MINERAL BENEFICATION AND

Continued from page 22]

as by-products. The pig iron assayed about 4% C, 0.8 to 1.5% S, 0.03 to 0.04% P, 0.60 to 2.35% Si and 1.20 to 0.60% Mn and the pyrite assayed 46.4% Fe, 44.4% S and 7.3% SiO₂. Iron recovery of the process was more than 95% and that of S, 60%. Loss of S was mainly due to leakage in the equipment which is possible to minimise resulting in higher recovery. Power consumption was of the

order of 2700 to 2800 kWh/ton of iron produced.

We are also inclined towards experimenting into autogenous smelting of copper ores to get copper matte of high copper content which may directly be charged in the converters to get blister copper. Sulphur, which is converted to sulphur dioxide can be directly purified, compressed and marketed as liquid sulphur dioxide without the necessity of the usual processing for enrichment and upgrading of the dilute products. The process consists in the introduction through a burner of dry concentrates (20-30% Cu) and flux (silica) to a tall shaft or smelting furnace, where preheated air or oxygen required for combustion is also blown in. The reaction is exothermic due to the burning of sulphur to SO₂; the impurities enter the slag and copper concentrate is obtained as matte having about 70% copper. This matte can be directly charged in the converter for production of blister copper. The gas generated is sufficiently hot and can be used for preheating air and steam raising.

In conclusion let me emphasize that during the short period of six years, though no epoch making discoveries can be claimed by it, the National Metallurgical Laboratory has established its place among laboratories of its kind in the world. By long-range researches, by shorter sponsored investigations, and by offering technical guidance to and undertaking field investigations for numerous small and medium scale industries throughout India, the Laboratory has proved its ability to assist in the country's mineral and metallurgical developments in a significant manner. The good work done so far by the Laboratory has been duly appreciated not merely in this country but outside as well; commendable comments and reviews on its research activities and publications have appeared in the foreign technical Press from time to time. This young Laboratory has, doubtless, gone some way in achieving the objectives for which it was established!