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Iron and Steel Heritage of India : Contributions from the National Metallurgical Laboratory*

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ABSTRACT

The National Metallurgical Laboratory (NML) has done pioneering work in establishing the heritage of India in Iron and steel making. Archaeometallurgical studies conducted by NML in its early days have been presented. Some other more recent pursuits of NML in this direction, as in archaeomaterials science of glasses, has also been discussed. In furtherance of the iron and steel heritage of India, NML has contributed profusely to the development of modern Indian iron and steel industry. Notable amongst such contributions are its work on characterization and beneficiation of raw materials for the iron and steel industry, production of sponge iron, improvements for foundry grade iron technologies, pilot scale studies on steel making, production of ferro-alloys etc. The work done at NML in these areas is briefly presented.

Key words : Archaeometallurgy, Archaeomaterials science, Beneficiation, Sponge iron, Cokeless cupola, foundry grade iron, Ferro-alloys.

INTRODUCTION

The National Metallurgical Laboratory (NML) was established in 1950 as a constituent laboratory of the Council of Scientific and Industrial Research. In his inaugural address, Jawaharlal Nehru made a reference to the glorious traditions of India and stated, '...to ignore the past, to throw it away, means to throw away

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the whole foundation on which we have grown'. It was but natural that the laboratory got interested in the study of heritage not only in the area of iron and steel but also in the nonferrous melting and casting area. In this presentation, a summary of the results of investigations on a comparative study of the corrosion behaviour of the Delhi Iron Pillar and Adivasi irons of nearby regions is presented. We also highlight the contributions from NML to the iron and steel industry of our country.

STUDIES ON ARCHAEOMETALLURGY

The early investigations laid a greater emphasis on the corrosion aspects of traditional steels in view of the interest and excellent facilities created at NML in this important area.

The Samples

A small piece of the Delhi iron pillar was cut and brought to the laboratory. Chemical analysis of the sample is shown in Table 1. It is now well established that West Bengal and Bihar independently contributed to the introduction of iron to the subcontinent. These regions were well endowed with rich deposits of copper and iron ores. The iron age dated back to chalcolithic period and the era of 1050-950 BC has been considered to be Ferro-Chalcolithic age[1,2]. Bahiri, Hatikira, Mangalkot, Pandu Rajar Dhibi in West Bengal and Barudih in Bihar have yielded a large number of iron artifacts and samples of ores and slags. The tradition of smelting iron continues to this day in some the regions. NML had collected samples from the present day Adivasi iron makers and has used these for a comparative study. Compositions of one such sample together with that of a mild steel of modern times is also shown in Table 1. Some independent studies were also made by Chakraborty[3] on samples of steel made by the Asur/Birzia tribals in the Hadu village. The place continues the tradition of iron making and several studies have been made of this process[4,5] in association with Vikas Bharati of Bishnupur, a voluntary organization. Studies at NML[6,7] were mainly focussed on the microstructural aspects of the Hadu/Bishnupur iron. Some attempts were also made in the laboratory towards modifications to the traditional furnace with a view to employ low grade coke as a fuel.

Table 1 : Chemical analyses of various samples studied

	Adivasi steel	Delhi Iron steel	Hadu Pillar	Mild steel
Carbon (%)	1.3	0.28	0.05	0.12
Silicon (%)	0.03	0.056	0.18	0.42
Manganese (%)	nil	nil	0.008	0.42
Phosphorus (%)	0.019	0.155	0.21	0.037
Sulphur (%)	0.006	0.003	0.008	0.043

The Tests

Besides chemical analysis, microstructural characterization and hardness determination, NML conducted a study of the carbides extracted from the Adivasi steel sample. Atmospheric corrosion tests were conducted over a period of time. A careful study of the constituents of the rust formed on various samples was undertaken.

RESULTS AND DISCUSSION

A few of the salient results are presented and discussed in a comparative fashion.

Microstructure

Almost all of the traditional iron making samples showed a high content of slag. The slag was also non-uniformly distributed. In the case of the sample from the Delhi iron pillar, consideration of the three dimensional distribution of the slag indicated that it coats and envelopes the individual grains. The metallic part also showed considerable variation in microstructure. Annealed, normalized and Widmanstatten structures were observed in various locations. Existence of slip bands and distortions indicated that the deformation was also very non-uniform. Similar observations were made with respects to the Hadu steel and the Adivasi steel sample also. In both of them, microstructural variations indicated that the distribution of carbon in the sample is highly variable. Some of the observed

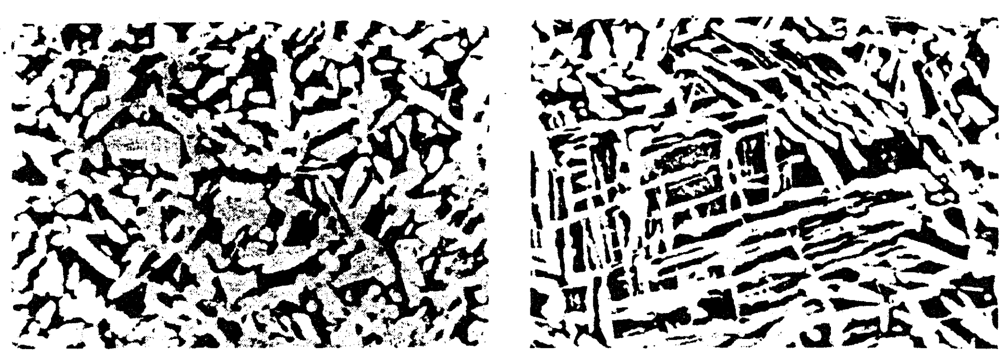

Fig. 1 : (a) Microstructures of samples from Delhi Pillar : showing regions with higher carbon (on the left) a regions with as cast structure (on the right).

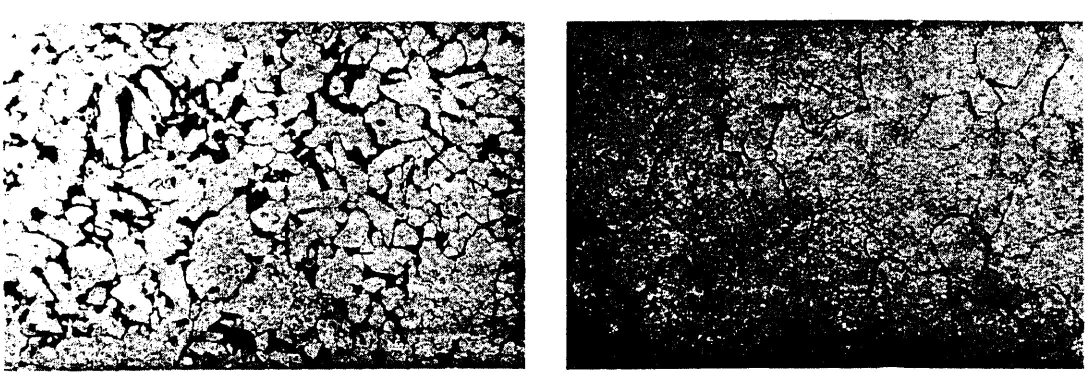

Fig. 1 : (b) Microstructure of Huda steel showing inclusions (on the lef) and ferritic grains (on the right).

microstructures are shown in Fig. 1. The carbides in the Adivasi steel were electrolytically extracted and subjected to X-ray diffraction studies. Results indicated that the carbide was essentially cementite.

Hardness

In all the samples the Vickers hardness depended upon the location and varied considerably. Some of the data are presented in Table 2.

Table 2 : Vickers Hardness number at 30 kg load

Material	Minimum	Maximum
Delhi Pillar*	81	106
Adivasi steel	110	235
Hadu steel (forged bar)	133	195

*from the data of Ghosh[4]

Corrosion Rates and Products

Atmospheric corrosion rates of the Delhi pillar and the Adivasi steel were comparable. Adivasi steel with scale did not rust while the mild steel sample did. Rates of corrosion over different periods of time are indicated in Table 3. An analysis of the scale formed was also conducted and it was found that in all

Table 3 : Corrosion rates observed in different samples at the end of indicated periods

Material	Corrosion rate in gm/dm^2		
	2 months	18 months	42months
Sample from Delhi Pillar	–	–	1.48
Adivasi steel without scale	0.81	1.32	–
Adivasi steel with scale	–	2.26	–
Mild steel without scale	0.79	1.30	–
Mild steel with scale	–	1.80	–

cases the corrosion product consisted of Lepidocrocite ($Fe_2O_3.H_2O$), Hydrogoethite ($Fe_2O_3.H_2O$) and Geothite (FeO.OH). In the case of mild steel some minor concentrations of Fe_2O_3 and FeO were also found. In general, the studies revealed the following aspects:

(a) The composition and microstructure of the traditional steels are highly heterogeneous in any given sample,

(b) the process of forge welding the sponge often gives rise to inhomogeneous deformation,

(c) the corrosion resistance of traditional steels appears to be related to the method of fabrication and arises out of the coating of the slag on individual grains of the metal,

(d) high concentrations of phosphorous could also have contributed to the corrosion resistance.

CURRENT ACTIVITIES IN THE AREA OF ARCHEOMATERIALS SCIENCE

Materials science particularly with reference to the synthesis of special glasses and glazes has occupied the attention of our forefathers. In the book "Amsubodhini" of Maharshi Bharadwaja mention has been made of glasses which are transparent to infrared. In the introductory passage it is said that in the original text of Bharadwaja, the aphorisms of Amsubodhini are divided into twelve chapters having one thousand sections. In order to make it understandable, Bodhananda wrote a commentary on it. The available manuscript is only the first Chapter of the work, having a commentary on the first fifty aphorisms of Maharshi Bharadwaja. The title of this chapter is Srstyadhikrah. Combing through the literature one finds, that this commentary appears to describe details of evolution of universe and our solar system. A description of various instruments and materials of special properties along with their preparation is also provided. In the work undertaken, with support from the Indian National Science Academy and in association with Dr. NG Dongre, H.C.P.G. College, Varanasi a glass with the composition : 8 parts silica, 5 parts calcium oxide, 4 parts magnetite and 6 parts phosphoric acid was melted and tested for it's ability to transmit infrared radiation. Fig. 2 shows that the glass does have some promise. Refinements are being made to this basic composition. Additional studies on glaze materials are also in progress with the compositions being as those recommended in our ancient texts.

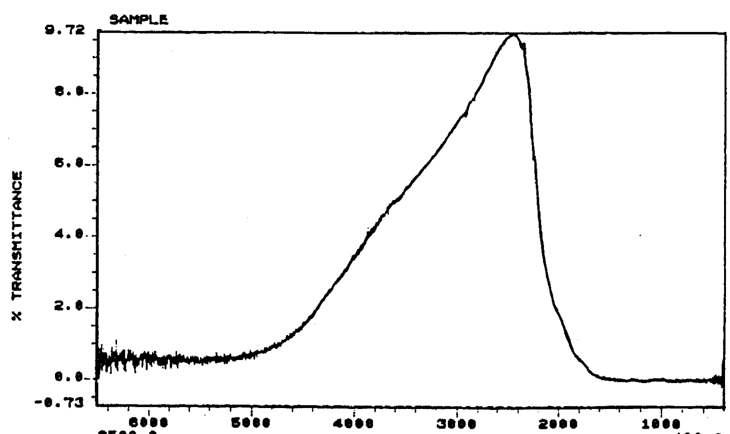

Fig. 2 : Transmittance of a glass made on the basis of compositions suggested in Amsubodhini of Maharshi Bharadwaja

CONTRIBUTION OF NML TO THE DEVELOPMENT OF MODERN INDIAN IRON AND STEEL INDUSTRY

Since it's inception NML has been constantly endeavouring to provide essential research inputs to the steel sector. In the formative years of the laboratory as well as the industry, efforts were essentially directed towards the establishment of pilot facilities for the beneficiation of raw materials as also iron and steel making processes. Some of these are briefly mentioned here.

Raw Material Characterization and Beneficiation

The National Metallurgical Laboratory devoted much of its early efforts to the beneficiation of indigenous iron ores, coking coal and limestone. These studies were conducted on representative samples on a tonnage scale and encompassed virtually all the iron ore, limestone and coal deposits being exploited by the public sector and private steel plants for mineral beneficiation during it's formative years. The pioneering studies of P.I.A. Narayanan and his dedicated band of associates have been greatly appreciated and implemented by the industry. Many of the facilities have since been augmented. The present plans for the expansion of the iron and steel sector have meant a natural resurrection of these activities and several such studies are once again being pursued by the laboratory. This section briefly summarizes the many interesting results obtained in our studies.

Iron Ore Beneficiation

Beneficiation of the iron ores involves both enrichment of the metal content and the improvement of the physical characteristic through crushing, sizing and agglomeration. Several of the plants experienced operational difficulties in crushing and sizing of the iron ore, particularly during the rainy season, due to sticking of the wet fines and blinding of the screens. Over 200 studies in the laboratory have indicated that washing of the ores is an absolute necessity to solve the problem of sticky and wet ores and to yield sized lumpy ore for efficient blast furnace operation. Attempts were made to optimize the moisture content through the conduct of appropriately designed screenability between 5-15% water content for different ores and enabled the optimization of operating parameters at several plants. Heavy media separation and jigging of the washed material, if employed optimally, can decrease their silica and alumina content. Mineralogical studies have indicated that alumina, control of which is essential for obtaining a fluid slag in the blast furnace, may be present in the Indian ores in several forms.

It may exist as fine clay and adherent material or as lumpy lateritic material. While the clay materials can be washed, the lateritic material may pose greater problems as it may be interspersed in the ore body. Studies at NML were mostly aimed at tackling this problem. Amongst several methods explored, the use of gravity separation and/or jigging has been found to be most efficient.

The generation of a large quantity of fines produced in the course of modern mining practice has necessitated the installation of sintering facilities for utilization of the fines. These fines which may sometimes constitute as much as 45% of the produce are invariably high in insolubles. The fines need to be beneficiated before sintering for making them acceptable as charge material and for a reduction in the coke rate. Studies conducted at NML therefore addressed the twin issues-beneficiation of fines and optimization of the conditions for sintering of the fines with and without fluxes.

Several parameters such as chemical analysis of raw materials, their size distribution, moisture content, fuel content and flux additions were varied and optimized. It has been found that the coke content which controls the sintering temperature of the bed has an optimum value below which the sinter is too soft and above which the sinter is fused with large voids and glazed surfaces with little porosity. Moisture content, which determines the rate of sintering through the control of the permeability of the sinter bed was also found to have an optimum value below which the porosity and permeability of the bed cannot be maintained and above which incomplete sintering occurs. Such extensive studies conducted in the laboratory have yielded valuable information on the sintering characteristics of Indian iron ores. Some of these results are summarized in Table 4.

Table 4 : Summary of some selected results on iron ore beneficiation at NML

Ore location	Conc. Assay (%)			Remarks
	Fe	SiO_2	Al_2O_3	
Noamundi (TISCO)	65.1	1.2	3.16	Washing (–25mm)
	66.5	0.9	2.42	HMS (50mm)
	66.7	1.28	2.2	
Joda (TISCO)	63.3	1.9	3.45	Washing (–25mm)
	64.2	1.61	3.0	HMS (–50mm)
Joda flaky (TISCO)	66.5	1.7	1.95	
Khond band (TISCO)	66.5	1.11	2.38	
Bolani (SAIL)	63.4	–	–	Sintering
Reject slimes	60.42	2.6	4.22	Pelletisation
Barsua (SAIL)	63.00	–	–	Sintering
Kiriburu (SAIL)	65.57	–	–	HMS (75mm+10mm)
Meghatuburu (SAIL)	62.52	–	–	
	57.37	–	–	Sintering
Pale (Goa)	64.3	2.76	2.25	Pelletisation
Sesa Goa	61.5	1.26	6.6	Washing, HMS&Jigging
Salem (Magnetite)	70.4	–	–	Pilot plant
Kavuthimalai	67.17	–	–	Wet mag. sep.
Donimalai	68.1	1.1	1.7	
	67.5	1.8	2.2	Classified fines
Kudremukh	67.9	2.9	1.0	Pelletization
Bailadila	66.9	–	–	Sintering tests with fines

Physico-chemical Characterization of Raw Materials

NML, in the last two decades, has been striving to characterize the raw materials for iron making processes. The studies include reducibility, thermal and reduction degradation and softening point of iron ore and the physical tests like shatter, tumbler and abrasion index of iron ore, coke, limestone and dolomite. The performance characteristics of reductant such as char reactivity and coal ash softening point have also been studied. The work undertaken at NML has provided the sponsors *apriori* knowledge of the physicochemical characteristics of the raw materials and helped them in selecting the suitable raw material combination for the production of desired quality of iron.

Beneficiation of Coking Coal

India has 29 billion tonnes of coking coal reserve of which 18 billion tonnes is minable. Only 18.5% of it is of prime variety. Indian coals are of drift origin and contain high proportion of fine grained and intergrown impurities. These are generally high in ash and are characterized by difficult washing characteristics. During washing of such coals, high proportion of near gravity material results in low yield of clean coal. This necessitates fine crushing of coal. The top size for washing was reduced from 75mm (1950-1970) to 38/20mm (1970-1980) and to −13mm (after 1980). Now attempts are being made to decrease the top size to 6/3mm to achieve better liberation and cleaning characteristics of coal. This in turn is going to increase the load in cleaning circuit for fine coal. The present

Table 5 : Summary of results on beneficiation of some coking coal samples carried out at NML.

Location	Feed (%) ash	Concentrate	
		Yield (%)	Ash (%)
Amadoba	29.7	43.0	15.8
West Bokaro	30.6	5.0	18.2
	25.3	51.2	13.8
	23.9	60.2	17.0
	19.9	60.9	13.0
Dugda	39.3	47.0	16.7
Gidi I	27.4	51.0	8.0
	–	66.2	11.9
	–	77.0	16.8
Gidi II	28.0	76.2	16.4
Patherdih	16.4	61.9	7.8
Kedia seam IV	33.8	48.6	21.5
Kedia seam V	33.0	66.0	18.4
Moonidih	26.5	65.2	17.0
Hurliadih	27.5	62.4	17.0
Nandan	38.0	34.4	14.6
Damua	37.3	40.2	17.1

washeries are not equipped to handle coals of such characteristics. NML has carried out beneficiation studies on coking coal from several locations. Based on these studies, NML has provided know-how for the flotation circuit at Jamadoba and West Bokaro to TISCO, for Gidi to Central Coalfields Ltd. And for Dugda to Bharat Coking Coal Ltd. Beneficiation results on various coking coal samples obtained at NML are summarized in Table 5.

NML has also studied on production of non–coking coal with less than 20% ash as a partial substitute for coking coal in the DR processes of iron making. The low ash (10%) non–coking coal can also be used for direct injection in blast furnace to reduce the requirement of coking coal. NML is in constant touch with coal industry and planning to undertake research projects which are going to benefit them.

Beneficiation of Limestone

Limestone is an important raw material for the steel industry and Indian reserves are of sufficient quality and quantity. Current estimates put the total reserves of limestone at about 69 billion tonnes of which about 20% can be exploited for use in the iron and steel industry. Limestone used in this industry has to conform to certain conditions in terms of physical and chemical characteristics. These conditions are different for iron making and steel making. While limestone for iron making is charged into the blast furnace either in lump form or as a constituent of sinter and can have a certain flexibility with respect to the desirable level of silica, it's composition in terms of silica, sulphur and phosphorous has to be carefully controlled in steel making. Each additional 1% of silica reduces the available lime by 2.5%, increase slag volumes and forms a thick insulating layer of very low levels of silica are thus desirable in limestone used as flux in steel making. The insoluble content of the limestones being mined for the steel industry is continuously rising. Simultaneously, the alkali content in the fluxes is increasing leading to an adverse impact on the coke strength in the lower regions of the blast furnace. Situation is further being compounded by the selective mining of better grades to meet the stringent requirements of the industry. Consequently, an accumulation of low grade limestone dumps is posing a problem.

Some of the above trends and problems were clearly visualized by the scientists at NML even in the early sixties and both bench scale and pilot scale investigations were initiated. Studies conducted at NML have shown that low grade limestone can be beneficiated by flotation. In many of the limestones of interest, calcite is the principal carbonate mineral with small quantities of dolomite. The gangue essentially consists of quartz with the other constituents being chlorite, phlogophite, muscovite and feldspars. The siliceous material can be separated by flotation using fatty acids or their soaps as collectors. The results that have been achieved are indicated in Table 6.

The limestone concentrates are in the form of fines and need to be briquetted or pelletised. NML developed the necessary know-how for this purpose and conducted several large scale trials in steel making furnaces.

Table 6 : Summary of results on beneficiation of limestone.

Locality	Feed Assay (%)		Conc. Assay (%)		Remarks
	CaO	Insol.	CaO	Insol.	
TISCO I	4.28	–	50.49	3.30	SMS Grade
TISCO II	45.90	–	50.30	1.90	SMS Grade
Dungri Limestone	33.70	34.34	49.03	8.70	BF Grade
Purnapani I	37.70	–	47.60	3.53	SMS Grade
Chrk	33.45	–	47.06	–	BF Frade
Toli	35.40	–	51.40	–	BF Frade
Tal	38.07	–	46.40	–	BF Frade
Rohtas	38.68	21.24	49.29	8.05	BF Frade
Khargaon	41.52	–	49.58	–	BF Frade
Chanaka Gurbi	43.44	–	48.93	–	BF Frade
Manipur	44.40	12.38	50.60	–	BF Frade
Kerala	43.23	8.41	53.70	–	Carbide Grade

Production of Sponge Iron

The Indian raw materials has certain unfavourable features. NML has been engaged in the R&D efforts for the last 38 years towards developing alternative routes utilizing the favourable characteristics of our raw materials which otherwise cannot be used in conventional iron making processes. The laboratory was the first to produce tonnage quantity DRI in India through a 4 ton/day capacity rotary kiln. NML later concentrated all its developmental efforts in the Vertical Retort Direct Reduction route. The process has been developed on 250 kg/day scale using solid reductants like non-coking coal, wood and other agricultural wastes. The process has a distinct advantage that it can be easily and successfully adopted by Mini Steel Plants to produce sponge iron sufficient for consumption in their own electric furnaces. In addition, it has a productivity 3–4 times of rotary kiln, degradation 1/3 to rotary kiln, investment cost ~60% of rotary kiln and energy consumption 25% less than rotary kiln.

Foundry Grade Iron Technologies

Low Shaft Furnace

A 10–12 ton/day low shaft furnace pilot plant was installed with the object of conducting extensive industrially oriented investigations and developmental work on the production of pig iron with substandard grade of raw materials particularly non-metallurgical fuels. During the 10 years *i.e.,* 1959-69 of intermittent operation, several campaigns were conducted with raw materials collected from the different parts of India. Besides the variations in the physico-chemical characteristics of raw materials employed, alterations in operational conditions such as variation in hot blast temperature, wind rate, basicity of slag, dolomite addition to the burden, oxygen enrichment of the blast were imposed for comprehensive assessment.

The process parameters were evaluated and optimized to produce the suitable grades of pig irons. It may also be mentioned that several hundred tonnes of foundry grade iron was produced using NML's low shaft furnace technology. The pioneering work on the direct injection of highly volatile and inflammable liquid naphtha directly in the smelting zone of iron making furnace with simultaneous enrichment of the blast with oxygen was shown to be technically feasible and commercially acceptable. The extensive investigations have amply demonstrated the possibility of manufacturing of acceptable grade of pig iron ore fines and non-metallurgical fuels in industrial scale. The process of iron smelting in small scale was found to be suitable to the developing countries lacking in raw materials which can be used in conventional blast furnace.

Cokelss Cupola

NML is presently conducting research and innovation on the design and development of cokeless cupola for the production of foundry grade pig iron using LD oil and natural gas. The process is energy efficient and environment friendly. The cokeless cupola designed and developed at NML has been operated by using either liquid fuel oil (LDO) @60 lt/thm or almost sulfur free @30 m^3/thm to produce 1 thm/hr. (foundry grade). The operation promises extremely low levels of SPMs 35 mg/m^3, $SO_2 \leq 88$ mg/m^3 for LDO and SPM ≤ 9 mg/m^3, $SO_2 \leq$ 150 mg/m^3 for LPG as compared to SPM = 400–3000 mg/Nm^3 with SO_2 of the order of 750–1500 mg/Nm^3 in case of normal cupola operated with coke.

VRDR–SAF Route

The technology developed at NML can produce low S, low P hot metal suitable for direct conversion to high quality grey, SG, CG iron castings. Several hundred tonnes of this iron with <0.03% and P<0.06% produced at NML was sold to foundries producing malleable and SG castings.

Pilot Studies on Steelmaking

Successful pilot plant trials were conducted at NML using a special type of converter designed and fabricated in the laboratory. The steel of composition: C = 0.02%; P = 0.035%; Si = 0.019%; Mn = 0.69%; S = 0.03%; was produced from the pig iron of composition: C = 3.2%; P = 0.39%; Si = 1.36%; Mn = 0.79%; S = 0.039%.

Investigations on top blowing with oxygen were also conducted for standardizing the Double Slagging Technique of refining ores with normal high silicon and medium phosphorous contents. The scope of this pilot study was carried out in early sixties to establish a 3 ton converter and study the applicability of Indian basic refractories including tarred dolomite to this process. A side blown converter (1500 kg) to undertake dephosphorization of the iron without the use of oxygen was installed. A pilot scale experimentation unit for the continuous casting technique both for nonferrous and ferrous alloys was installed. The research and development work on refractories *vis-a-vis* steel plant requirements including aerodynaic studies of combustion and flow of gases in a metallurgical furnaces such as the open hearth steel furnace was also undertaken at NML.

NML's Role in R&D of Ferro-alloys Industry in India and Abroad

The National Metallurgical Laboratory has carried out extensive work on various aspects of alumino-thermic reactions and has to a considerable extent mastered the technique. Successful experiments have been carried out and the following alloys and metals have been produced: (i) ferro-titanium, (ii) carbon free ferro-chrome, (iii) chromium metal, (iv) manganese metal, (v) chromium-manganese alloy, and (vi) ferro-vanadium from vanadium pentoxide.

NML has recently carried out the smelting trials of ferro-silicon production (70-74% silicon content) in its pilot plant scale 500 KVA submerged arc furnace for M/s. Bhutan Ferro Alloys Ltd., Bhutan. A number of compositions of charge mix were tried, decreasing the percentage of charcoal in the mixed reductants upto about 5% and process parameters were optimized. NML results will form a basis for their smelting trials in the industrial plants of M/s. Bhutan Ferro Alloys, Bhutan. The experience gained in the production of ferro-alloys on small scale has helped in setting up a unit for the production of these alloys.

CONCLUSION

In keeping with the expectations of it's founding fathers, NML has constantly striven to understand and respect our past and build on it for the future. It is hoped that the monumental contributions made in the areas of raw material characterization and beneficiation for the modern steel plants, the help rendered towards the establishment and nurturing of the ferro-alloy industry, the operation of several pilot plant for the benefit of the designers and operators will long be remembered.

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