Study of effective utilization of iron ore sinter through arc plasma

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Abstract
Generation of fines is common in mining, sizing, and beneficiation and also in high-temperature metallurgical processes as the disintegration of agglomerate/compact occurs. Extraction of metallic iron from ore fines is one of the challenging aspects of iron making industries as the liberation of fines blocks, the charge burden porosity and hence hinders the reduction rate. Along with size factor, mineral composition plays a vital role in the extraction process; particularly silica. As silica has the very high tendency towards iron oxide, at comparatively low temperature, the activity of silica should be suppressed to prevent silicate phases. Adjustment of such conditions is controlled by addition of lime, but sometimes excessive slag generation increases the cost of production. In the present work, carbothermic reduction of partially reduced iron bearing pellets has been melted through 20 KW DC arc plasma furnace, and a comparative study has been made for considering different slag chemistry approaches. Pellets as aforementioned are made available from Patnaik Steel and Alloys Ltd, Odisha, having high silica content ore fines (of about 8.6%) as obtained from the chemical analysis. X-Ray analysis and optical image analyzer result of sinter thus obtained reveal that fayalite phase has major fractional value. Smelting works were done for sinter with/without adjustment of slag chemistry, where argon and nitrogen were used as plasma forming gases. A range of recovery rates (between 87-94%) is achieved by charge composition, ionizing gases, and smelting duration. It is observed that use of nitrogen as plasma forming gas increases the recovery rate than that of using only argon plasma; due to high energy flux of nitrogen which increases the enthalpy due to its diatomicity. A maximum recovery rate of about 94% is achieved for process duration of 13 minutes utilizing nitrogen plasma. Smelting of charge with the addition of hydrated lime targeting melilite as final slag resulted in the formation of metallic iron as confirmed from XRD and XRF analyses. In the other hand, ferrosilicon is liberated in the metallic parts where smelting of charge was done without adjustment of slag chemistry. Both metal and slag thus obtained are characterized by XRD, XRF, microhardness and wet chemical analysis suitably.

Keywords: siliceous iron ore, arc plasma furnace, argon and nitrogen, recovery.

1. Introduction
With increasing global demand for iron ore, the production has increased by initiating steps to utilize the low-grade iron ores, fines and slimes [1]. Generation of fines is common in mining, sizing and also in high-temperature metallurgical processes as the disintegration of agglomerate/compact occurs. These fines being of low grade, cannot be used in the blast furnace directly and if used then it reduces process efficiency [2, 3]. However, this difficulty can be overcome with the application of plasma technology as it possesses several advantageous features [4, 5 and 6].

Along with size factor, mineral composition plays a vital role in the extraction process; particularly silica. As silica has the very high tendency towards iron oxide, at comparatively low temperature, the activity of silica should be suppressed to prevent several iron-silicate phases. Silica hinders the reduction
reaction rate by lowering surface area of pellet samples due to the formation of liquid fayalite phase at comparatively low temperature [7, 8, 9, 10 and 11]. In the thermal plasma process, high density of ionic charges makes uniform heat transfer to the charge material the reactions are completed in short duration [12]. Ferrosilicon is a ferroalloy which is used as a deoxidizer in steel industries [13]. In the present work an attempt has been made for the comparative study of smelting of sinter obtained from siliceous iron ore; with/without adjustment of slag chemistry.

2. Materials and experiments

2.1. Materials
Iron ore sinter was collected from Pattanaik steels & Alloys Ltd, Odisha, India for our present study. The chemical analysis of sinter was done through wet chemical analysis and is listed in table 1 given below. It is clear from the compositional analysis that content of all gangue oxides contributes about 18.23 wt. % in which only silica content is 8.6 wt. %.

| Oxides | Al₂O₃ | SiO₂ | MgO | CaO | TiO₂ | Fe₃ | Others |
|--------|-------|------|-----|-----|------|-----|--------|
| In Wt.% | 7.2   | 8.6  | 0.63| 1.4 | 0.4  | 72.2| 9.57   |

2.1.1. Elemental analysis of sinter:
Elemental analysis of sinter was done by EDS analysis and is given in table 2.

| Elements | Fe | Al | Mg | Si | Ca | Ti | O  | C  |
|----------|----|----|----|----|----|----|----|----|
| In Wt.%  | 63.2| 3.79| 0.38| 3.99| 1.01| 0.53| 18.65| 8.44|

The electron image and EDS spectra of powdered sinter sample are shown in figure 1. In the EDS spectra, the major peaks identified are Fe, Al, and Si. Besides oxygen, other elements are found to be in trace amount.

2.1.2. Optical micrograph of sinter:
Further, the polished surface of sinter sample was observed by using a Zeiss light emission electron microscope under a constant magnification of 200X, and area-fraction analysis was obtained by using Axio vision software of version 4.8 as shown in figure 2.
Two distinct phases viz. bright and dull phases are seen to be widely distributed along with the very small fraction of dark/black phases. Bright phases are mostly interconnected and surrounded by dull phases which signifies the possibility of formation and flow of slag phases over metallic phases. From area-fraction analysis, it is found that bright phases have lesser fractional value by about 13%.

2.1.3. \textit{x-ray analysis of sinter:}

Further X-Ray analysis of sinter was done to detect the presence of phases formed in the sinter by using a Philips X-Ray Diffractometer with Cu-Kα (1.54 \text{ Å}) and is shown in figure 3.

![X-ray diffractogram of sinter](image)

\textbf{Figure 3.} X-ray diffractogram of sinter

Wustite (FeO), fayalite (Fe2SiO4), iron (Fe) and hercynite (Al2FeO4) are found to be the major phases present in the XRD pattern as shown above. Bright and dark phases seen in optical micrograph are thus confirmed to be metallic and Fe-silicate/Fe-aluminium-silicate, i.e., fayalite and hercynite phases respectively. The existence of any other major phases is not detected in X-ray diffractogram as other compounds are in trace amount which is evident form chemical analysis results. Fayalite is a stable phase that not only consumes more reductant and energy to reduce but also requires a higher reducing atmosphere, i.e., high working temperature. The requirement of high energy flux at elevated temperature can be fulfilled by thermal plasma and hence implemented for the smelting of sinter.

2.2 \textit{Experiments}

A 20KW DC extended arc plasma arc furnace was employed for the smelting experiments. Specification of the above mentioned set up is illustrated in table 3 as given below. The generation of plasma was made through arcing mode and is identical as followed and described by S K Samal et al. [4]. The composition of charge feed along with some of the critical smelting parameters are listed in table 4.

| Table 3. Specification of plasma setup. |
|-----------------------------------------|
| Crucible (graphite) | Outer diameter- 145 mm | Wall thickness- 15 mm | Height- 300mm |
| Hollow tapered rod (graphite) | Length- 400 mm | Inner dia- 5 mm | Outer dia- 35 mm |

| Table 4. Important operating parameters. |
|------------------------------------------|
| Sample No. | Plasma forming gas used | Gas flow rate (in LPM) | Process duration (min.) |
| 1 | Ar | 2.5 | 7.0 |
| 2 | Ar | 2.5 | 13 |
| 3 | Ar+ N2 | 2.5 | 7+2 |
| 4 | N2 | 2.5 | 7 |
| 5 | Ar | 2.5 | 13 |
| 6 | N2 | 2.5 | 9 |

*Ar-argon, N2-Nitrogen
First four samples were smelted as such with the addition of coke in 25% excess of stoichiometry. Voltage and current values as maintained throughout the process are 50V and 250A respectively. The weight of charge was in the range of 300-500gm. Further, two more smelting works, i.e., samples 5 and 6, were done with the addition of 8.6% hydrated lime as flux after complete melting of charge. In both cases, petroleum coke was used as reducing agent.

3. Results and discussion

3.1 Recovery:
Recovery rate is the ratio of the weight of the metallic fraction of smelted product to the weight of metal present in the charge feed before smelting. The of the recovery rate of each sample w.r.t smelting duration is shown in Figure 3.1.

![Figure 4. Percentage of recovery of samples smelted by argon and nitrogen plasma](image)

Points representing recovery rates of nitrogen plasma smelted samples lie above to that of argon plasma as evident from figure 4. The highest recovery rate of about 93.82% was achieved for sample smelted with an addition of lime by nitrogen gas.

2.2. SEM study of final product:
Further well-polished surfaces of metallic fractions of smelted products (metal) were observed in scanning electron microscope and are shown in figure 3.2. EDS analysis at different appearing phases was done and is given by Wt% in table 5.

![Figure 5. Scanning electron micrographs and EDS spectra of metals at various region of samples.](image)

In micrograph of sample-5, i.e., smelted in argon plasma for 13 minutes with adjustment of slag chemistry, elongated flakes are seen to be present randomly in between another bright phase as shown...
in figure 5(b). Samples smelted in nitrogen plasma with slag-adjustment for the comparatively lower duration of 9 minutes exhibited a single phase which is confirmed as Fe from EDS analysis. Almost similar type of microstructure, i.e., dark to bright phases is observed in samples 1 to 4 as shown in figure 5(c). Elemental analysis (EDS) on three separate looking phases shows that silicon content in dark, dull and bright phases are in decreasing order. For dark phases, silicon content varies in the range of 5.35 to 0.99 % while for bright phases it varies from 0.32 to 0.15%. Dull phases contain silicon intermediate to that of bright and dark phases.

| Points | Fe    | Si    | C   |
|--------|-------|-------|-----|
| 1      | 96.03 | 0.18  | 3.79|
| 2      | 90.52 | 0.50  | 4.23|
| 3      | 85.45 | 0.70  | 9.56|
| 4      | 88.74 | 8.75  | 3.50|
| 5      | 90.46 | 3.02  | 6.40|
| 6      | 99.63 | 0.37  | 0.45|

2.3. X-Ray analysis:
XRD patterns of metallic fractions of all smelting products are shown in figures 6. Metallic parts as obtained from the smelting of sinter by Argon plasma for 7 min is detected as ferrosilicon (Fe₃Si). Here time for smelting is less, so recovery rate is lowest, i.e., 88%. Further smelting of sinter by Argon plasma gas for 13 min resulted in the formation of Fe and Fe₃Si phases. Although no metal carbide phase is identified in the XRD pattern, carbon was found to be present in fewer percentages as obtained from EDS analysis results. Here smelting duration is comparatively more, so recovery rate is enhanced.

Metallic yield is enhanced by nitrogen plasma smelting of charge over argon plasma where both Fe and Fe₃Si phases were detected in the respective XRD patterns. Although recovery rate is a function of smelting period, samples smelted with the application of nitrogen plasma has shown higher recovery than that of argon plasma in lesser operation length. The principal reason for increased recovery is diatomicity of nitrogen that increases enthalpy of the system which in turn increases reducibility. Further, the lime addition was made after complete melting of charge targeting melilite as final slag with employment of argon and nitrogen plasma. The objective of flux addition was to suppress the activity of silica to improve reducibility. Although recovery rate in both cases is comparatively similar, the formation of phases is different as confirmed from X-Ray analysis results. Along with Fe, cohenite (Fe₃C) phase is detected in case of sample smelted through argon plasma whereas single metallic iron phase is identified in case of nitrogen plasma smelted product.
2.4. Microhardness:

Another characterization technique hardness test as separately appearing phases was applied for confirmation of phases. A LECO microhardness tester LM248AT was implemented for hardness testing where load and dwell time were 100gf and 10 seconds respectively. Three distinct hardness values (average) viz. 618.5, 524.7, 493.2 HV with a maximum deviation of ±5 were obtained; is due to presence of iron carbide, metallic iron and ferrosilicon phases respectively depending on microstructure as shown in figure 5.

From the above results, it is seen that from minerals having high silica content, both metallic iron, as well as ferrosilicon alloy, can be produced. For extraction of iron it requires fluxing agents which in turn increases the final slag volume; hence heating of additional slag volume consumes more energy. On the contrary; initial silica in the ore minerals can be considered for value addition if smelted as such for producing ferrosilicon alloy. In the latter case additional material (flux) cost can be eliminated; hence can be considered as economic in comparison to the other case.

3. Conclusions

1. Ferro-silicon alloy and hot metal (molten iron) can be produced by plasma smelting of siliceous iron ore sinter by varying various plasma forming gases.
2. Recovery rate is a function of plasma forming gas viz. argon, nitrogen or mixture of gases and smelting duration.
3. A maximum recovery rate of about 93 percentages is achieved with employment of argon and nitrogen plasmas for durations of 13 and 7 minutes respectively.
4. XRD patterns of metal show presence of the three distinct phases viz. Fe, Fe$_3$C, and Fe$_3$Si per operating conditions.
5. Dissimilar type of microstructures is observed for samples subject to different operating conditions.
6. Silicon content at distinct phases in the alloy thus produced varies from 0.18 to 8.75 %
7. Flux addition helped in suppressing the activity of silica; hence silicon picks up in the metal is reduced to a significant extent.
8. As both production of iron and ferrosilicon is feasible through the arc plasma, conversion of lean grades into ferrosilicon alloy puts more importance.

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