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Studies on plasma processing of blue dust

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Abstract. Plasma smelting was carried out using blue dust and petroleum coke mixtures for five different compositions. By altering percentage of reductant and type of plasma forming gas, recovery rate and degree of metallization were calculated in order to examine the extent of reduction of blue dust. The products were characterized by XRD and optical microscopy techniques. The results of these investigations exhibited that highest degree of metallization and recovery rate of about 98% and 86% respectively, were achieved for nitrogen plasma smelted products.

Keywords: Blue dust, waste utilization, plasma smelting, argon and nitrogen, recovery, degree of metallization.

1. Introduction
Currently iron and steel making is dominated by the route comprising a blast furnace and a basic oxygen converter; meanwhile Ferro alloys are formed in submerged-arc furnaces. Production on reliable source of high quality raw materials such as sinter, pellets, and coke are the principal economic constraints of the process. In addition, the inflexibility of the process capacity, high capital and operational costs and energy lost in between stages still a prominent aspect for reduced economy [1]. Blue dust is a high grade soft hematite ore fines containing more than 90 % Fe₂O₃ enormously available today. For transportation problem and environmental hazardous factor concerned, these iron ore fines are dumped at mine sites. Since its particle size lies in the micron range, blue dust cannot be used directly in the blast furnace for iron making [2, 3]. Utilization of these iron oxide fines and applicability of the same for the blast furnace feed is an approach for production of value added product being wasted [4]. Keeping above facts in sight since last two decades thermal plasma has claimed to be an emerging solution to a numerous processes due its some unique features (high enthalpy, high temperature and high reactivity) and hence implemented in various sectors. Plasma finds significant industrial applications as processes like melting, smelting, smelting and reduction, refining, spark plasma sintering, surface modification and surface coating [5,6]. Application of plasma technology seems to be an emerging alternative route for iron and steel making and for many of its advantages over any other processes [7-9]. Carbothermal reduction and smelting of metal oxides were experimented effectively in the past research works to get metal as final product with recovery more than 80% [10-13]. Carbothermal reduction of hematite can be possible in three ways [2] as given below:

\[
\begin{align*}
\text{Fe}_2\text{O}_3^1 \rightarrow & \text{Fe}_3\text{O}_4^1 \rightarrow \text{FeO}^2 \rightarrow \text{Fe} \\
\text{(Single chain reaction)} \\
\text{Fe}_2\text{O}_3^1 \rightarrow & \text{FeO}^3 \rightarrow \text{Fe} \\
\text{(A combination of single and double reaction)} \\
\text{Fe}_2\text{O}_3^1 \rightarrow & \text{Fe} \\
\end{align*}
\]
In this piece of research work, an attempt has been made to study the effect of reductant and type of plasma forming gas on reduction of blue dust and characterization of final product.

2. Experimental details

2.1. Materials
Blue dust of average particle size about 100-150µ whose origin is Koida range, Odisha was collected by DISIR, Rajangapur, Odisha for our study. The major constituents of blue dust was obtained by wet chemical analysis, is given in the Table 1. The loss on ignition was calculated by heating the raw blue dust at 1000°C for 1hr. The XRD pattern of raw blue dust obtained is given in fig.1.

<table>
<thead>
<tr>
<th>Oxides</th>
<th>In wt. %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe₂O₃</td>
<td>96.87</td>
</tr>
<tr>
<td>SiO₂</td>
<td>0.45</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>0.21</td>
</tr>
<tr>
<td>TiO₂</td>
<td>Trace</td>
</tr>
<tr>
<td>MgO</td>
<td>Trace</td>
</tr>
<tr>
<td>LOI</td>
<td>1.48</td>
</tr>
</tbody>
</table>

2.2. Experimentation
The experimental set up used to treat the blue dust consists up a 35 KW DC transferred arc plasma setup, power supply unit, gas feeding system, power and gas control unit etc. On the top, plasma torch is attached in the downward direction. The plasma furnace contains a hollow cylindrical graphite crucible with 145mm outer diameter, wall thickness 15mm and 300mm high that serves as the anode. Hollow graphite rod of 400mm long with 5mm central hole and 35mm outer diameter serves as the cathode. Hollow structure of cathode has designed to have provisions for gas flow. The material to be processed was placed in the anode crucible bed and the arc was initiated by shorting the cathode and the crucible bottom wall (graphite plate). The arc length was increased by raising the cathode up suitably within the crucible to heat the charge placed in the crucible. For charge, material nodules of 8-12mm (diameter) were prepared by taking five different compositions of mixture of blue dust and petroleum coke (5%, 10%, 12%, 15% and 20% of coke). Then nodules were dried at 110°C for one hour for moisture removal. Before initiating the arc, reaction chamber was properly cleaned and plasma forming gas is purged for 2min. Then arcing was done by striking cathode with anode. Plasma arc was generated by passing plasma forming gas (Argon and Nitrogen) through cathode and it gradually spreads in all directions and melted down the charge material. The important operating parameters used in this work are illustrated in table 2.
Figure 1. XRD pattern of raw blue dust.

Table 2. Operating parameters for smelting.

<table>
<thead>
<tr>
<th>Operating Variables</th>
<th>Quantity</th>
</tr>
</thead>
<tbody>
<tr>
<td>Input Voltage</td>
<td>250-300 V</td>
</tr>
<tr>
<td>Input Current</td>
<td>50 A</td>
</tr>
<tr>
<td>Gas Flow rate</td>
<td>2.5 lpm</td>
</tr>
<tr>
<td>Processing time</td>
<td>17 minutes</td>
</tr>
<tr>
<td>Charge quantity</td>
<td>300 g</td>
</tr>
</tbody>
</table>

3. Results and discussion

3.1. Recovery
Since carbothermal reduction of blue dust was made in this study, carbon percentage was increased gradually to examine its effect to the extent of reduction. Percentage of recovery was calculated by taking ratio of weight of final product to weight of metal present in the composition before smelting and reduction. Fig.2 represents the plot of variation of percentage of recovery w.r.t. %C in compositions. The curve representing samples smelted in nitrogen plasma lies above that of argon plasma. Maximum recovery of about 86% achieved for sample with 12% coke by nitrogen plasma. Comparatively higher percentage of recovery has been achieved by utilizing nitrogen as plasma gas.
3.2. Degree of metallization
Following wet chemical analysis route degree of metallization of all smelted samples were calculated by taking ratio of metallic Fe to total Fe. Highest degree of metallization of about 98% is achieved in case of smelted composition of blue dust and 12% coke.

Nitrogen plasma smelted samples shows greater DOM values may be due to the fact that, nitrogen being diatomic gas increases the enthalpy of plasma which in turn helps in reduction of blue dust to a greater extent.
3.3. Microstructural studies

Micrographs of all cold resin mounted polished samples were observed by using Zeiss forced light emission electron microscope under a constant magnification of 200X as shown in Figure 4 and Figure 5 (a-5\%C, b-10\%C, c-12\%C, d-15\%C, e-20\%C) . In most samples micro cracks are observed as shown in Figure 4 (a), (b) and Figure 4 (b), (d). It may be due to high cooling rate of smelted samples as in case of plasma processing, temperature falls from thousands of degree to ambient temperature in very short period of time. Elongated flakes are found in Figure 4 (d) and Figure 5(c), whereas in Figure 5(e), elongated flakes with some voids are seen to be present. Length of flakes are in decreasing order in figures 5(c), 4(d) and 5(e) whereas width of flakes are in increasing order in figures 5(c), 5(e) and 4(d) respectively. Hence Figure 5 (c) possesses flakes of highest L/D ratio. In all other cases homogeneous distribution of two phases are observed.

Figure 4. Optical micrographs of nitrogen plasma smelted samples (unetched).
3.4. Phase characterization by XRD

XRD analysis of smelted samples was made to examine presences of phases in each by using Cu-Kα (1.54Å) radiation. X-ray diffraction pattern of all samples are presented in Figure 6.a and Figure 6.b, and in all cases highest intensity peak shows the presence of iron and other peaks shows the presence of cohenite (cementite). Since graphite crucible was used as reaction chamber, decay of graphite bottom plate (more) and crucible walls (lesser) mainly occurs at high temperature and this extra carbon is dissolved into the molten feed and reduces iron oxide to a little more extent. Formation of cementite is due to reaction of carbon with iron (Fe) and wustite (FeO) [14].

3Fe + C → Fe₃C
3FeO + 5CO (g) → Fe₃C + 4CO₂ (g)

Again reaction of cementite with wustite can give rise to iron and carbon dioxide.
2FeO + Fe₃C → 5Fe + CO₂
Hence the brighter phase observed micrographs were confirmed as cohenite (Fe₃C) as clearly visible in Figures 4(c) and 5(a). Figure 4(c) coincides with ledeburite structure as along with cohenite (brighter phase) a dark phase (pearlite) is present. This may be due to rapid erosion of graphite from crucible and cathode rod.

Figure 6.a XRD pattern of all nitrogen plasma smelted samples.

Figure 6.b XRD pattern of all argon plasma smelted samples.

3.5. Hardness

Vickers hardness of smelted samples was performed by using a LECO micro hardness tester LM248AT taking 300gf load and dwell time of 10 second. Three different values of hardness were obtained i.e. 147, 208 and 312 (average values), is due to presence of ferrite, pearlite and cementite
phases respectively depending on microstructure shown in micrographs. Meanwhile when hardness measured on needle like and elongated phases (cracks and voids) as seen above, sample gets distorted and resulting hardness values varied from 50 to 80 HV.

4. Conclusion
Transferred arc plasma route was used to convert blue dust to industrially useful product like metallic iron. Carbon percentage and type of plasma gas was varied to study its effect on recovery rate and degree of metallization and each case. Nitrogen as plasma forming gas exhibited better rate of reduction as compared to that of argon. Three different phases; ferrite, pearlite and cohenite were identified by optical microscopy, XRD and corresponding hardness values.

References