Reduction Properties of Raw Materials for Direct Reduction Shaft Furnace

By Dentaro KANEKO,** Yoshio KIMURA,** Mamoru ONODA** and Isao FUJITA**

Synopsis

The reducibility, fine generation during reduction and clustering of pellets and ores were discussed on the basis of the results of the experiments made by reproducing the reducing conditions for a direct reduction shaft furnace.

Pellets produced at an actual pelleting plant and lump ores as well as pellets fired at the test plant were reduced at a temperature ranging from 760°C to 960°C, with the reducing gas composed of H₂: 55%, CO: 36%, CO₂: 5% and CH₄: 4%.

The reducibility and clustering characteristics of pellet are related fairly well to the amount of gangue or that of lime added, and the high iron grade pellet is superior to the low grade one in respect of the reducibility; however, on the contrary it tends to form clusters.

The decrease in reducibility with an increase in slag volume or lime addition is dependent on the formation of liquid phase during the pellet firing process, where a certain amount of iron oxide is included.

The formation of cluster during the reduction process depends mainly on such factors as temperature, load on the burden bed and properties of raw material. The shrinkage ratio of pellet bed of the reduction underload test related to the strength of cluster, and the higher the shrinkage ratio is the stronger the cluster becomes. Addition of a small amount of lime to the high iron grade pellet is effective to obstruct the formation of cluster.

I. Preface

The direct reduction iron making process by shaft furnace is characterized by the high utilization of reducing gas and low energy consumption owing to the heat exchange and reduction taking place between the gas ascending and the solid descending in the furnace. In the shaft furnace process, as well as other ironmaking processes, the properties of raw materials to be used have not a little effect on the process stability and productivity, so it is important to select the materials of good qualities.

The authors have conducted the reduction experiments using various materials under the reducing conditions similar to those adopted for direct reduction shaft furnace by which sponge iron had been produced by reformed natural gas. This paper describes the experimental results regarding mainly the reducibility and clustering of raw materials in the reducing gas comprised of a large amount of H₂ and CO.

II. Properties of Raw Materials for Shaft Furnace

Since the majority of the sponge irons produced by the direct reduction process are used as the burden materials for electric arc furnaces, it is proper to use the high iron-grade raw materials containing a small amount of such impurities as P and S. The raw materials charged into shaft furnace are generally natural lump ore, but high-grade hematite ore or magnetite ore concentrate in the form of pellet is frequently used.

According to the papers reported by Ahrend[3] and other researchers[4,5] on the reduction of raw materials for direct reduction shaft furnace, the important properties of raw materials except the chemical components are as follows:

I) High reducibility
II) Less fine generation during the reduction process
III) No cluster formation during the reduction process

Item I: This relates to the time required for raw materials to reach a certain reduction degree after charging them into a shaft furnace and has an effect on the productivity.

Item II: Excessive fine generation is unfavorable because it increases the gas pressure drop in the furnace and the dust loss.

Item III: Clustering means the adhesion of materials in the high temperature zone near the tuyere of shaft furnace and it is possible to cause the so-called hanging phenomenon.

Clustering, if excessive, makes great troubles because it prevents the smooth descending of materials. In order to improve the productivity of shaft furnace, it is effective to raise the temperature of reducing gas blown through the tuyere, but it is restricted due to the cluster formation.

III. Samples and Experimental Methods

1. Samples

The chemical and physical properties of tested raw materials are shown in Tables 1 and 2, respectively.

The properties of pellets produced at a commercial pelleting plant and of typical lump ores, which are not only the direct reduction feed but also the materials for ordinary blast furnaces, are shown in Table 1.

The properties of pellets produced at the Kobe Steel Pelletizing Test Plant are shown in Table 2. The pellets are made up of, in order to give variety to the iron-grades and CaO contents, high iron grade ores from Brazil, low iron grade ores from Australia and home-yielded lime stone which was blended at various ratios, balled and fired in a batch kiln after being preheated in a pot grate furnace.

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Table 1. Chemical and physical properties of raw materials

<table>
<thead>
<tr>
<th>Brand</th>
<th>Process*</th>
<th>Chemical composition (wt% )</th>
<th>Compression strength (kg/p)</th>
<th>Tumbler** index (%)</th>
<th>Porosity (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>T. Fe  FeO  SiO₂  CaO</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Pellet</td>
<td></td>
<td>T. Fe  FeO  SiO₂  CaO</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Hamersley G</td>
<td>G</td>
<td>63.25  0.29  4.08  0.33</td>
<td>309</td>
<td>4.2</td>
<td>20.5</td>
</tr>
<tr>
<td>Savage River S</td>
<td>S</td>
<td>67.86  0.79  1.41  0.18</td>
<td>159</td>
<td>7.7</td>
<td>24.9</td>
</tr>
<tr>
<td>Chorogle G</td>
<td>G</td>
<td>65.90  0.10  2.58  0.58</td>
<td>227</td>
<td>8.9</td>
<td>29.0</td>
</tr>
<tr>
<td>Robe River G</td>
<td>G</td>
<td>62.76  0.29  5.69  0.78</td>
<td>274</td>
<td>7.1</td>
<td>29.3</td>
</tr>
<tr>
<td>LKAB S</td>
<td>S</td>
<td>68.27  0.40  1.16  0.15</td>
<td>272</td>
<td>—</td>
<td>17.0</td>
</tr>
<tr>
<td>Whyalla GK</td>
<td>GK</td>
<td>68.22  0.14  3.59  0.55</td>
<td>220</td>
<td>4.3</td>
<td>28.1</td>
</tr>
<tr>
<td>Kakogawa GK</td>
<td>GK</td>
<td>61.22  0.27  4.02  4.82</td>
<td>276</td>
<td>3.4</td>
<td>25.1</td>
</tr>
<tr>
<td>CVRD G</td>
<td>G</td>
<td>67.60  0.57  1.56  0.35</td>
<td>399</td>
<td>5.7</td>
<td>26.7</td>
</tr>
</tbody>
</table>

* G: Straight Grate, GK: Grate Kiln, S: Shaft
** - 3.3 mm

Table 2. Chemical and physical properties of sample pellets produced at test plant (Fired at 1280°C, 25 min)

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Chemical composition (wt% )</th>
<th>Compression strength (kg/p)</th>
<th>Porosity (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>T. Fe  FeO  SiO₂  CaO</td>
<td></td>
<td></td>
</tr>
<tr>
<td>A</td>
<td>68.35  0.29  0.79  0.09</td>
<td>281</td>
<td>21.2</td>
</tr>
<tr>
<td>B</td>
<td>66.74  0.29  2.17  0.12</td>
<td>276</td>
<td>22.3</td>
</tr>
<tr>
<td>C</td>
<td>65.62  0.22  3.40  0.10</td>
<td>284</td>
<td>23.6</td>
</tr>
<tr>
<td>E</td>
<td>63.81  0.22  5.66  0.12</td>
<td>213</td>
<td>25.7</td>
</tr>
<tr>
<td>J</td>
<td>66.74  0.1   1.25  0.90</td>
<td>377</td>
<td>21.3</td>
</tr>
<tr>
<td>K</td>
<td>66.60  0.22  1.16  1.11</td>
<td>336</td>
<td>20.2</td>
</tr>
</tbody>
</table>

2. Experimental Methods

With regard to the reduction characteristics in a shaft furnace, three kinds of reduction experiments were performed. The reducing gas used for these experiments is composed of H₂: 55%, CO: 36%, CO₂: 5%, and CH₄: 4%.

1. Reducibility

The sample weighing 500 g suspended in a retort with a balance was reduced in an electric furnace at a fixed temperature. Throughout the experiment the reduction progress of iron oxide was observed by the change in the retort weight, and after the reduction the properties of sponge iron were investigated.

2. Characteristics of Fine Generated during the Reduction Process

As shown in Fig. 2 (a), the specimens in the rotating Linder barrel were reduced in an electric furnace at a fixed temperature. Taking out the samples after reduction, the amount of fine generated during the reduction process was measured. The test conditions were: sample weight: 500 g, flow rate of reducing gas: 15 Nl/min, reduction time: 5 hr, temperature: 760°C, and rotation rate of Linder barrel: 10 rpm.

3. Measurements of the Shrinkage Ratio of Sample Bed and the Cluster Strength

The reduction underload test apparatus used is shown in Fig. 2 (b). The sample weighing 500 g or 1 kg was reduced in the reduction tube (75 mm diameter) at a fixed temperature. The shrinkage ratio of sample bed was automatically recorded throughout the reduction process. The cluster was sometimes formed in the sample after this experiment.

The cluster strength was measured with the apparatus illustrated in Fig. 3. The measurements were conducted by rotating the steel drum (120 mm diameter, 700 mm length) containing clusters at the rate of 30 rpm for 5 min and by sieving the clusters.

The cluster strength C.S. can be given by the following formula:
IV. Reduction of Lump Ore and Commercially Produced Pellets

The results of the reduction test made at 760°C for 2 hr are summarized in Table 3. Though any complete adhesion phenomenon was not observed in all samples treated at this temperature, the samples which were visually confirmed to have the traces of cluster formation are noted in this table.

1. Results of Static Bed Reduction Test

Figure 4 shows the relation between the reduction time and the reduction degree obtained from the static bed reduction test at 760°C.

According to the reduction curves of pellets and lump ores indicated by solid and dotted lines, respectively, it can generally be said that the reducibility of lump ore is inferior to that of pellet, and that the hard lump ore has the lowest reducing speed. Also it shows that the high iron-grade CVRD and Savage River pellets have high reducibilities, that Robe River and Hamersley pellets containing a large amount of gangue have poor reducibilities, and that Kakogawa pellets which are the self-fluxed pellets used as the burden materials for blast furnace have a high reducibility in the range from the initial stage of reduction to the midst, as well as CVRD pellets; however, the reaction becomes less active after 70 min from the beginning of reduction.

According to the relation between the iron-grade of raw materials and the reduction degree shown in Fig. 5 (a), it is known that the higher the iron-grade is, the higher the reduction degree becomes, though the tendency of pellets is a little different from that of ores.

2. Results of Linder Reduction Test

The amount of fine generated during the reduction process is shown in Table 3. Looking at the amount of fine having the size of -3 mm after Linder reduction, it can be known that the ores generate more fine than the pellets do. This is easily presumed from the irregular shape of the materials. Among the pellets, however, Chowgle, Savage River and Kakogawa pellets have a fairly high fine generation ratio. The fine generation in the reduction process can be caused, besides the thermal degradation of lump ores, by the sliding friction resulting from the revolution the barrel, and by the change in the crystal structure, from hematite to magnetite, in the reduction process as pointed out by Offroy.4)

For the pellets with a little lower cold compression strength, such as Savage River and Chowgle pellets the former seems to be the main cause, but the latter for the self-fluxed Kakogawa pellets.

3. Results of Reduction Underload Test

Figure 5 (b) shows the relation between the iron-grade of raw materials and the final shrinkage ratio of sample bed after the reduction underload test.

The lump ores are split into pieces in their edges during the reduction underload test and the packing condition of sample bed becomes so dense that they normally present a high shrinkage ratio; therefore, the cause of the shrinkage of lump ores must be considered independently of that of the pellets. The lower the iron-grade is, the more porous the lump ore is, so the described splitting occurs in the reduction underload test to present a high shrinkage ratio as
Table 3. Results of reduction test (Reducing temperature: 760°C)

<table>
<thead>
<tr>
<th>Brand</th>
<th>Chemical composition of sponge after static bed reduction test (wt%)</th>
<th>Static bed reduction test</th>
<th>Linder reduction test</th>
<th>Reduction underload test</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>TFe  FeO  MFc  C</td>
<td>Degree of reduction (%)</td>
<td>Strength after reduction (kg/p)</td>
<td>Degree of reduction (%)</td>
</tr>
<tr>
<td>Pellet</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Hamersley</td>
<td>80.74  30.20  55.29  0.03</td>
<td>79.6</td>
<td>128</td>
<td>98.0</td>
</tr>
<tr>
<td>Savage River</td>
<td>91.22  9.60  80.40  0.12</td>
<td>87.8</td>
<td>80</td>
<td>97.4</td>
</tr>
<tr>
<td>Chowgle</td>
<td>88.80  6.35  82.40  0.28</td>
<td>85.6</td>
<td>46</td>
<td>96.9</td>
</tr>
<tr>
<td>Robe River</td>
<td>79.49  31.37  54.75  0.01</td>
<td>73.8</td>
<td>189</td>
<td>90.5</td>
</tr>
<tr>
<td>LKAB</td>
<td>90.48  16.79  73.95  0.06</td>
<td>86.4</td>
<td>182</td>
<td>96.6</td>
</tr>
<tr>
<td>Whyalla</td>
<td>85.47  14.85  75.35  0.20</td>
<td>83.8</td>
<td>76</td>
<td>95.6</td>
</tr>
<tr>
<td>Kakegawa</td>
<td>78.97  9.82  68.14  0.51</td>
<td>82.4</td>
<td>50</td>
<td>91.0</td>
</tr>
<tr>
<td>CVRD</td>
<td>91.78  6.49  83.96  0.40</td>
<td>89.8</td>
<td>100</td>
<td>97.5</td>
</tr>
<tr>
<td>Ore</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Iscor</td>
<td>88.43  27.54  61.99  0.03</td>
<td>63.1</td>
<td>—</td>
<td>94.3</td>
</tr>
<tr>
<td>Hamersley HG</td>
<td>90.85  23.28  68.94  0.07</td>
<td>81.5</td>
<td>—</td>
<td>97.9</td>
</tr>
<tr>
<td>Newman</td>
<td>93.05  9.87  81.80  0.09</td>
<td>79.1</td>
<td>—</td>
<td>96.4</td>
</tr>
<tr>
<td>Pico</td>
<td>89.19  28.75  59.49  0.05</td>
<td>70.2</td>
<td>—</td>
<td>79.5</td>
</tr>
</tbody>
</table>

*— 3.3 mm

Fig. 4. Results of static bed reduction test

shown in Fig. 5 (b).

On the other hand, as to the pellets, the final shrinkage ratio of sample bed increases in proportion to their iron-grades, and the samples with high shrinkage ratios exhibit the clustering phenomenon.

4. Effect of Reduction Temperature

In order to investigate the effect of reduction temperature on the reducibility of raw materials and the quality of sponge iron, the static bed reduction tests were performed on CVRD pellet, Chowgle pellet and Pico ore at 700°C, 860°C and 960°C.

Photograph 1 shows the microstructures of sponge iron, which were obtained by reducing CVRD pellet and Pico ore, observed with a Scanning Electron Microscope.

In case of pellet reduced at 760°C, the iron particles maintain their initial shape before reduction but a lot of micro pores are observed on the surface of particle.
Reduction temperature (°C)

760 860 960

CVRD (Pellet)

PICO (Lump ore)

Photo. 1. Micro structures of reduced materials observed by SEM (x780)

However, these pores disappear at 860°C and the borders among the particles become indistinct. At 960°C, the surface of reduced iron becomes solid and the spaces between the particles are enlarged, on the whole, to increase the macro pores, besides the sponge structures with filaments-like iron crystals are partially recognized.

In case of lump ore, the above characteristics are also observed, but at 960°C, the formation of filament-like iron crystals becomes more conspicuous than the case of pellet.

Figure 6 shows the relation between the reduction temperature and the metallization or the compression strength after a 2 hr reduction. Between 700°C and 800°C, the metallization of pellet increases remarkably and at 960°C, the metallization reaches the value more than 95%.

It is noticeable that the metallization of hard lump ore, i.e., Pico ore, increases remarkably in the region above 800°C, and that the reducibility of ore becomes the same as that of pellets at a temperature more than 800°C.

The compression strength after reduction decreases with a rise in reduction temperature.

At a reduction temperature below 1000°C at which the sintering of reduced iron does not occur enough, the compression strength of pellet decreases with an increase in porosity caused by the swelling phenomena which enlarge the spaces among the iron particles, as shown in Photo. 1.

Fig. 6. Effect of reduction temperature on metallization and compression strength of sponge

V. Reduction of Pellets Produced at the Test Plant and Discussion

The reduction experiments were performed by using the pellet produced at the test plant, shown in Table 2 in order to obtain the further information on the reducibility and the clustering characteristics during the reduction process of raw materials in a shaft furnace.
1. Investigation on the Reducibility

The experiments were performed by means of the foregoing static-bed retort reduction method. The conditions adopted are: reduction temperature; 860°C, reduction time; 4 hr, and gas flow rate and others; the same as mentioned in the foregoing paragraph. The reduction time was determined by taking account of the fact that the final reducibility is important to assess the reducibility of burden for the direct reduction process.

According to the reduction curves of pellets with various iron-grades shown in Fig. 7, the reduction rate on the progress of reduction increases with a rise in porosity of fired pellets; however, the reduction degree at the final reduction increases with a rise in iron-grade of raw materials.

As shown in Fig. 8 indicating the effect of additional lime on the reducibility, the more the CaO content of pellets is the lower the final reduction degree becomes. The reduction behavior of the above commercially produced pellets was confirmed by these experiments.

The lime-added fired pellets are mainly composed of hematite, CaO·nFe₂O₃ in the form of solid solution or slag, and CaO·Fe₅O₉·SiO₂ in the form of slag.

The iron oxides fixed in the slag phase are hard to reduce at the temperature adopted for direct reduction shaft furnace, so the increase in the amount of lime added to the pellets tends to reduce the final reducibility.

The reducibilities of self-fluxed pellets are known to be superior to those of oxide pellets from the result of the reduction test for blast furnace burden such as JIS Reducibility Test. This superiority depends on the increase in pellet porosity caused by the limestone added to the raw materials. In case of such reduction test, the reducibilities are generally evaluated in the reduction degree range of 50 to 80%, and the reduction degree higher than the above mentioned range in the shaft portion is not required in practice for the blast furnace burden which is reduced to iron oxides directly and indirectly by solid carbon.

As shown in Fig. 9 indicating the effect of firing temperature at the pellet-indurating stage on the reducibilities of high-grade A pellets and lime-added K pellets, there are no noticeable difference in the final
reducibilities of high-grade pellets fired at 1240°C, 1280°C and 1320°C, and on the other hand, the reducibility of lime-added pellet is apparently influenced by the firing temperature that is, the final reducibility increases with a decrease in firing temperature. This supports the assumption that the rise of firing temperature increases the amount of iron oxide fixed in the slag phase and thus retards the reduction.

2. Clustering Characteristics of Iron Oxide during Reduction

Considering three factors for clustering of raw materials in the shaft furnace, i.e., temperature, load and properties of materials, the clustering characteristics were investigated by using the above-mentioned reduction underload test apparatus.

According to Fig. 10 showing the effect of load on the shrinkage ratio for the pellet with the iron grade of 68.4% at 960°C, the pellet bed expands a little for 10 to 20 min after the beginning of reduction and then it shrinks gradually. The higher the pressure is, the higher the shrinkage ratio is.

From Fig. 11 showing the effect of reduction temperature on the shrinkage ratio under the fixed pressure of 2 kg/cm², it is obvious that the final shrinkage ratio remains below 10% at a reduction temperature below 860°C, and on the other hand, it reaches above 20% at a temperature above 900°C and the clustering phenomenon is observed in the sample taken out after reduction.

Photograph 2 (a) shows the samples reduced at 860°C. Some of them are partially adhered but are easily exploited by finger.

Photograph 2 (b) shows the hard clusters which are formed during reduction at 960°C and are maintained in the form of “bed.”

As shown in Fig. 12 indicating the relation between the final shrinkage ratio and the cluster strength, no cluster remains in the sample after the rotating test when the final shrinkage ratio is less than 15%, and the cluster strength increases remarkably when the shrinkage ratio is more than 15%. Therefore, the degree of cluster adhesion can be judged by measuring the shrinkage ratio of sample bed after the reduction underload test.

Figure 13 shows the shrinkage behavior of the pellets with various iron-grades and different amounts of additional lime when it is reduced at 910°C under the load of 2 kg/cm². According to this figure, the higher the iron-grade is, the higher the shrinkage ratio is. This tendency agrees with the results of the above reduction test for actual plant pellets. And it is also noticeable that the shrinkage ratio of lime-added pellets is too low.

It can be said that at an early stage of reduction, the high-iron grade pellets induce the swelling phenomena which are not allowed to withstand the load, so they begin to soften and shrink and then the formation of clusters is promoted because of the increase in contact area among the pellets.

Concerning the mechanism of cluster formation of which the theory has not been established yet, it can be inferred from the results of these experiments that the adhesion of the materials which form the clusters is caused by the mutual diffusion of reduced metallic irons and if the temperature, load and other conditions are the same, the high iron-grade raw materials easily form the clusters, and the gangue or other

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**Fig. 10.** Effect of load on the shrinkage ratio of sample bed

**Fig. 11.** Effect of reduction temperature on the shrinkage ratio of sample bed
(a) Reduction temperature: 860°C
Load: 2 kg/cm²
Final shrinkage ratio: 10.5%

(b) Reduction temperature: 960°C
Load: 2 kg/cm²
Final shrinkage ratio: 31.2%

Photo. 2. Samples taken out after reduction underload test

VI. Conclusion

The reducibility, fine generation during reduction, and clustering of pellets and ore were discussed on the basis of the results of the experiments made by reproducing the reducing conditions for a direct reduction shaft furnace.

It was recognized that in respect of the reducibility, the high iron-grade raw materials are superior to the low iron-grade ones, but tend to form the clusters. It was also recognized that the addition of lime to the pellets lowers the final reducibility at the same reduction temperature, but this is not necessary to mean that the lime-added pellets are disadvantageous for the shaft furnace burden because high temperature may be brought about by the reducing gas supplied through tuyere without forming clusters. A quantitative examination on this problem is left for the further study.

REFERENCES