1. Introduction

The Japanese ironmaking industry has manufactured sintered ores with a low slag content to improve the permeability of blast furnaces operating under high pulverized coal injection. In addition, the strength of the sintered ores must satisfy the permeability requirements of the shaft of the large blast furnace. Hence, low-MgO sintered ores ([MgO] ≤ 1.2 wt%) have become dominant burden materials, with approximately 80% of blast furnaces using them in Japan. MgO is required to lower the fluidity of slag in blast furnaces and for recycling granulated blast furnace slag in cement industries. Currently, lump fluxes are generally used to add MgO to blast furnaces; however other MgO-bearing burdens such as olivine pellets are also effective.

The reduction behavior of olivine pellets in blast furnaces ranks between those of sintered ores and acid pellets.\(^2,4\) Furthermore, it has been reported that olivine pellets are inferior to dolomite-fluxed pellets, when compared among MgO-bearing pellets.\(^5,6\) However, most of these evaluations have been conducted for individual burden materials. The reduction behavior of mixed burden materials is important for the operation of furnace plants. The reduction behaviors that have been extensive researched include the mixing various pellets\(^6,7\) and mixing lumpy ores with sintered ores.\(^8\)

However, there is limited work done on the behaviors in mixing pellets with sintered ores, one of the major burden materials in Japan.\(^9,10\) Particularly, the evaluation of mixture of low-MgO sintered ores and olivine pellets is limited.

Recently, evaluations of blast furnace operation have been performed using experimental blast furnaces (EBFs). One of the results reported was that the melting temperatures of burden materials corresponded to the location of the cohesive zone in the EBF, thereby influencing the operation results of the EBF.\(^11\) Further precise understandings of the reduction behaviors of burden materials were obtained through basket-evaluation tests, in which sample-enveloped baskets were charged in the EBF before quenching, and then...
excavated after cooling.\(^{12}\)

In this study, a basket-evaluation test was performed with baskets containing low-MgO sintered ores and various pellets to investigate the reduction behaviors of these mixed burden materials. We also conducted softening-melting tests under the same mixing conditions to determine their behavior during melting.

2. Experimental Methods

2.1. Basket-evaluation Test in the EBF

The EBF (working volume of 8.2 m\(^3\)) owned by Luossvaara-Liirunavaara AB (LKAB) was used. The operation using 100% olivine pellets was carried out for three days before the EBF was quenched. The operation was stable, with a reducing agent rate of 560 kg/t-HM, pulverized coal rate of 130 kg/t-HM, and productivity of 1.55 kg/h. Ten sets of baskets were charged from the top of the EBF before the end of the operation, then excavated after cooling for two weeks. Three types of burden materials, low-MgO sintered ores, lime-fluxed (LF) pellets, and olivine pellets, were used. All of them were manufactured in commercial plants and sieved to 10–15 mm for sintered ores and to 10–12.5 mm for pellets. The basket was made using steel wires and had a diameter of 65 mm and length of 200 mm. Three baskets were connected in the serial direction to form one set of baskets. Each basket contained approximately 650 g of (A) 100% sintered ores, (B) 50% sintered ores and 50% LF pellets, and (C) 50% sinter and 50% olivine pellets. Two different burden materials were mixed homogeneously in baskets B and C. No flux-addition to achieve constant chemical compositions was performed to provide fundamental information, despite varied values for CaO/SiO\(_2\) and MgO/SiO\(_2\).

The porosities of sintered ores and pellets before reduction were measured. The water intrusion method (JIS K2151) was used to measure the <2 mm porosity. The total porosity including surface concavities was also measured.\(^{13}\) In addition, <200 \(\mu\)m pore volume and pore size distribution were measured with a mercury intrusion porosimeter using particles of 2.8–6.3 mm after roughly crushing samples. Microstructures of the burden materials were observed using an optical microscope.

The contents of baskets excavated from the EBF were identified as sintered ores, pellets, and cohesive masses by their appearances, and subsequently analyzed for size distribution, crushing strength (an average value of 12 particles), reduction degree, <10 \(\mu\)m pore volume, and microstructure of each material.

2.2. Softening-melting Test

The reduction behaviors of the mixed burden materials were analyzed using a small-scale softening-melting tester\(^{14}\) to verify the results of the basket-evaluation tests in the EBF. Samples (120 g) with sizes of 10–12.5 mm were charged to form a layer between the coke (9–13 mm) layers in a graphite crucible. Figure 1 shows the reduction conditions of the samples. The composition of the exhaust gas, bed height, and pressure drop were measured during the heating of the samples.

The temperature at which pressure drop reached 1.0 kPa \((T_s)\), the temperature at which pressure drop decreased to 1.0 kPa \((T_i)\), the temperature at which shrinkage reached 50% \((T_{50})\), the temperature at detection of the first drip \((T_D)\), \(dT\) \((T_E-T_S)\), the time integral of the pressure drop \((S\text{-value})\), and the maximum pressure drop \((dP_{MAX})\) were measured. Changes in the liquid ratio to the sum of oxides during reduction were estimated using FactSage (ver. 6.4). Chemical compositions of the burden materials during reduction \((\text{CaO}, \text{SiO}_2, \text{Al}_2\text{O}_3, \text{MgO}, \text{Fe}_2\text{O}_3, \text{FeO}, \text{Fe})\) were calculated from those before reduction and measured reduction degrees in the softening-melting tests at each temperature.

3. Results and Discussion

3.1. Properties of the Materials before Reduction

Table 1 shows the chemical compositions and porosities of burden materials used in this study, together with the

<table>
<thead>
<tr>
<th>Material</th>
<th>T.Fe</th>
<th>FeO</th>
<th>CaO</th>
<th>SiO(_2)</th>
<th>Al(_2)O(_3)</th>
<th>MgO</th>
<th>CaO/SiO(_2)</th>
<th>MgO/SiO(_2)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sintered ore</td>
<td>58.33</td>
<td>6.45</td>
<td>8.94</td>
<td>5.14</td>
<td>1.44</td>
<td>0.82</td>
<td>1.75</td>
<td>0.16</td>
</tr>
<tr>
<td>Lime-fluxed pellet</td>
<td>65.72</td>
<td>0.93</td>
<td>2.54</td>
<td>2.17</td>
<td>0.58</td>
<td>0.04</td>
<td>1.17</td>
<td>0.02</td>
</tr>
<tr>
<td>Olivine pellet</td>
<td>66.72</td>
<td>0.74</td>
<td>0.48</td>
<td>1.90</td>
<td>0.29</td>
<td>1.53</td>
<td>0.25</td>
<td>0.80</td>
</tr>
<tr>
<td>50% sinter + 50% LF pellet</td>
<td>62.03</td>
<td>3.69</td>
<td>5.74</td>
<td>3.66</td>
<td>1.01</td>
<td>0.43</td>
<td>1.46</td>
<td>0.09</td>
</tr>
<tr>
<td>50% sinter + 50% Olivine pellet</td>
<td>62.53</td>
<td>3.60</td>
<td>4.71</td>
<td>3.52</td>
<td>0.86</td>
<td>1.17</td>
<td>1.00</td>
<td>0.48</td>
</tr>
</tbody>
</table>
chemical compositions of the contents of baskets B and C. Both types of pellets had higher \( < 200 \mu m \) pore volumes and higher \( < 2 \text{ mm} \) porosities than the sintered ores, whereas they had lower total porosities than the sintered ores. Olivine pellets had a slightly higher porosity and smaller pores, compared with LF pellets (Fig. 2).

Figure 3 shows the microstructure of the LF pellets and olivine pellets in the central area (core) before reduction. Both types of pellets exhibited more porous and homogeneous structures than the sintered ores. The microstructures of the olivine pellets had the following characteristics in comparison with those of the LF pellets; 1) finer hematite grain size, 2) round pores representing the formation of a low-viscosity melt during heating, and 3) numerous relict olivine particles.15)

3.2 Analysis of Samples Excavated from the EBF

3.2.1. Excavated Condition

Figure 4 shows the locations of the excavated baskets in the vertical direction of the EBF. All charged basket sets were successfully recovered and numbered along the excavated layer from the top. Only the 13-2 set was located close to the center of the EBF at a radial distance of 0.2 m from the center (The maximum radius of the EBF was 1.0 m). Other sets of baskets were found in the middle or peripheral areas. The EBF was operated under center coke charging; thus the center coke layers were identified during the dissection. Therefore, set 13-2 was exposed to higher temperatures than the other sets. The difference in the vertical positioning between the ends of a set was negligible (less than 89 mm for all the sets). Figure 5 shows the cohesive mass ratio in the excavated baskets. High cohesive mass ratios were found in the centrally located 13-2 set. The cohesive zone in the EBF was located between the 13th and the 18th layers in the middle and peripheral areas.

3.2.2. Lumpy Materials at Lower Temperatures

Figure 6 shows the \( < 10 \text{ mm} \) fraction (a) and crushing strength (b) of samples found in the 5th and 7th layers (lumpy zone in the EBF). The \( < 10 \mu m \) fraction of basket A was always higher than that of baskets B and C (Fig. 6(a)). In addition, the \( < 10 \mu m \) fraction of basket B tended to be higher than that of basket C in the same basket set. Similarly, the crushing strength of basket C was the highest, followed by that of basket B and basket A (Fig. 6(b)). These results implied that the reduction disintegration in size was greatest for sintered ores, followed in decreing order by those for LF pellets, and olivine pellets.
EBF. The high reduction degree found in set 13-2 was due to its central location. A large variety in reduction degrees between two sets of basket, particularly observed at the 18th and 23rd layers, was also due to the difference in their locations. The differences in the reduction degrees between LF pellets and olivine pellets were insignificant in all layers.

Volumes of < 10 μm pores in the sintered ores were always smaller than those of pellets, similar to pore volumes before reduction, as shown in Fig. 2. The pore volume of olivine pellets was higher than that of LF pellets until the 13th layer. However, it decreased in the 18th and the 23rd layers.

3.2.3. Cohesive Masses at High Temperatures

Figure 8 shows the appearances of the cohesive masses found in the 18th layer. Figure 9 shows the images of macro structures of the cohesive masses in the 18th and 23rd layers, together with their reduction degrees. The distance between particles in basket B in the 18th layer was shorter than that in basket C, implying a greater melting extent in basket B. Furthermore, a direct comparison of pellets inside basket 18-1-B (LF pellets) and outside the basket (olivine pellets) revealed that LF pellets tended to undergo significant melting. Similarly, the inside and the outside of basket B in the 23rd layer were directly compared. The melting of both pellets occurred, and partially exuded wustite cores were observed. The material inside the baskets underwent significant melting. In particular, LF pellets were crushed under load. Kaushik et al. reported low-temperature slag exudation because of alkalis in the heating of pre-reduced (80%) olivine pellets. However, the significant exudation of slag from olivine pellets was not observed in this study, mostly because of a low reduction degree (65%).

Figure 10 shows microstructures of the burden materials in area A of Fig. 9. Both LF pellets and olivine pellets
exhibited greater reduction than sintered ores. LF pellets contained a porous metal shell and ‘islands’ of unreduced wustite in the core, even though the reduction front reached the core. In contrast, olivine pellets exhibited a more topochemical behavior with a fine but dense metal shell and an oxide core. The decrease in the volume of <10 μm pores in olivine pellets (Fig. 7(b)) could be due to the densification of the oxide core. These differences in the microstructures during reduction of the pellets might be caused by the differences in the pore structure (Fig. 2) and grain size of the hematite (Fig. 3) before reduction, which would influence the melting behaviors at high temperatures. The physical structure of the metal shell would significantly influence the exudation behavior of core-slag.6)

3.2.4. Interface between Sintered Ores and Pellets

Figure 11 shows the microstructures at the interface between sintered ores and pellets in set 13-2. The reduction degree of baskets B and C was high at 98.4% and 98.8%,
respectively, due to its central location. A large amount of slag formed at the interface between the sintered ores and LF pellets, whereas a small amount formed at the interface between the sintered ores and olivine pellets. Goldring et al. observed FeO-bearing slag forming at the interface between LF pellets (0.7 of C/S and 0.3 wt% of MgO) and sintered ores (1.6 of C/S and 1.6 wt% of MgO) during the dissection of the plant blast furnace after N2-quenching. Chaigneau et al. reported that mixing high-basicity sintered ores and olivine pellets improved their softening-melting behavior. One of the reasons for this effect is the change in the properties of the slag exuded at the interfaces. Nogueira et al. observed an improvement in the inferior shrinking behavior of acid pellets by mixing dolomite-fluxed pellets. They concluded that the reason for the improvement was the increase in the viscosity of the slag exuded at the interface. Furthermore, Kaushik et al. summarized the melting steps of two different kinds of pellets as follows; 1) solid-state sintering of metal shell, 2) initiation of melt, 3) mixing of slag exuded from each kind of pellet, 4) mixing of individual cores. They also suggested the physical structure of the metal shell and viscosity of slag influenced the mixing behavior of the exuded slags. We also observed the metal phase at the interfaces between sintered ores and pellets (Fig. 11), indicating that melting behaviors of mixed burdens are related with the exudation of slag through the metal shell and mixing of the exuded slag at the interface.

3.3. Melting Behaviors in the Softening-melting Tests

Figure 12 shows the results of the softening-melting tests using the same mixing condition for baskets A, B, and C. Table 2 lists specific values of reduction behavior parameters. The $T_S$ value of olivine pellets was higher than that of LF pellets when used individually, which agreed well with the results of previous investigations. The mixing of pellets with sintered ores increased $T_S$ and decreased $T_E$ and $T_D$, which decreased $dT$. The S-value and $dP_{MAX}$ also decreased. These improvements were significant in the case of mixing olivine pellets. $dP_{MAX}$ depends on the liquid ratio and the void structure of the burden layers at high temperatures above 1 200°C. The liquid ratio is determined by the chemical composition and reduction degree. The increase of void in burden layer by mixing different burden materials is insignificant. Therefore, low gangue mineral content mostly caused the low $dP_{MAX}$ for the mixture of sintered ores and olivine pellets.

Figure 13 shows the images of the cross sections of quenched samples at 1 300°C in each case. Olivine pellets contained wustite cores, whereas LF pellets exhibited only a metal shell structure with a large void in the core area after exudation of slag and wustite.

3.4. Melting Behaviors of Pellets Mixed with Sintered Ores

Figure 14 shows the calculation results for the changes in the liquid ratio to the sum of oxides with temperatures for various burden conditions (Table 2). The figure is based on the observations (Figs. 9 to 11) implying that the melting behavior of the mixed burdens initially depended on that of individual burden at lower temperatures, and subsequently depended on that of the complete mixed burden. The initial melt formation temperature of olivine pellets was the lowest, followed by that of LF pellets and sintered
ores, which was similar to results obtained in previous investigations.\textsuperscript{17} The densification of core-oxides found in olivine pellets (Figs. 7(b) and 10) could be explained by the low starting-temperature of melting. However, the increase in the liquid ratio after initiation was significantly different among the burdens. In other words, the liquid ratio of olivine pellets was lowest at 1 300°C. Similar behaviors have been reported previously\textsuperscript{2–4,15} and have been found to be caused by the increase in the melting points of fayalitic olivine slag and coexisting wustite. Mixing pellets with sintered ores decreased the liquid ratio at high temperatures, which was particularly significant in the case of mixing olivine pellets. The melt viscosities at 1 300°C for sintered ores, LF pellets, and olivine pellets were estimated as 0.134 Pa·s, 0.06 Pa·s, and 0.04 Pa·s, respectively.\textsuperscript{18} These results indicated that the mixing LF pellets promoted the exudation of a large amount of low-viscosity slag (Figs. 11 to 13).

Figure 15 shows a schematic explanation of the melting behavior of mixed burden comprising sintered ores and pellets during reduction. In the mixing of LF pellets, abundant slag is exuded from both the pellets and sintered ores. The viscosity of the core-slag in the pellets is low enough to enable penetration of the slag through porous metal-shell. The high liquid ratio is maintained even after complete assimilation of the exuded slags. In contrast, in the case of olivine pellets, only a small amount of the low-viscosity slag is exuded from the pellets and assimilates with the slag exuded from sintered ores. This assimilation causes a decrease in the liquid ratio, which limits melting at the interface. These differences in melting behavior can cause...
the result in the previous studies, which reported that EBF operation using mixtures of olivine pellets and sintered ores was more stable than that using mixture of LF pellets and sintered ores.10)

4. Conclusions

A basket-evaluation test was performed in LKAB’s EBF to investigate the reduction behaviors of mixed burden materials comprising low-MgO sintered ores, LF pellets, and olivine pellets. The following results were obtained.

(1) Both LF pellets and olivine pellets contained more <200 μm pores than sintered ores. Olivine pellets contained smaller pores and finer hematite grains, which influenced their reduction behavior.

(2) Size disintegrations due to the reduction in the lumpy zone in the EBF were smaller for pellets than for sintered ores, particularly for olivine pellets. The volume of <10 μm pores of olivine pellets was higher than that of LF pellets in the lumpy zone. However, it decreased at high temperatures, mostly due to the densification of core-oxides.

(3) LF pellets underwent greater melting than olivine pellets in cohesive masses in the EBF. At the interface with sintered ores, LF pellets formed larger amounts of slag than olivine pellets. The results of the softening-melting tests also revealed the superiority of olivine pellets during melting.

(4) Although initial melt formation occurred at lower temperatures during reduction, olivine pellets exhibited a lower liquid ratio at high temperatures, resulting in the decrease in exuded slag in mixtures with low-MgO sintered ores.

REFERENCES