Effect of High-phosphorous Iron Ore Distribution in Quasi-particle on Melt Fluidity and Sinter Bed Permeability during Sintering

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The effect of high P Brockman ore on the melt fluidity was investigated to make clear the mechanism for the deterioration of permeability of sinter bed during sintering.

Blending high-P ore, characterized by the high porosity and fine size distribution, decreased melt fluidity by the absorption of melt into porous high P Brockman ore. As a result, the pressure drop of sinter bed increased during sintering by the suppression of pore growth, and the sinter productivity decreased.

To improve the sinter productivity using high-P Brockman ore, JFE Steel has developed an advanced granulation process characterized by segregating high-P Brockman ore at the center of quasi-particle, which was coated by coke breeze and limestone. It is important to select the suitable coating thickness of dense hematite ore around porous iron ore for preventing from the melt absorption into iron ore. This process controls the excess melting reaction between iron ore and limestone, owing to the segregation of high-P ore within the quasi-particles.

The commercial plant trials showed that the desirable melt fluidity, resulting from the segregation of high-P ore in a quasi-particle, enhanced the permeability of sinter bed at Fukuyama No. 4 Sinter Plant of JFE Steel. The application of new granulation process remarkably improved both the productivity and reducibility of sinter products, in spite of the recent inferior ore conditions.

KEY WORDS: quasi-particle; granulation; pore size distribution; melt fluidity; productivity, permeability.

1. Introduction

To cope with the growth in steel demand and the need for global warming countermeasures, the ironmaking field in Japan has challenged to achieve a low reducing agent rate at blast furnace in the circumstance of deteriorating iron ore quality.

Blast furnace operation can achieve a low reducing agent rate, but it need to use the sinter with a high strength and a high reducibility as well as increasing the charge ratio of sinter, which is the main burden material charged into blast furnace. European and American steel mills use abundant high-quality hematite ores mined in South America at a low cost. However, geographic location forces Asian steel mills to use iron ore with a deteriorating quality, such as high-porosity ores imported from nearby Australia. Hence, it is important for Japanese mills to develop efficient technologies for production of high-strength and high-reducibility sinter similar to that produced using dense iron ores from South America even while using large amounts of low quality Australian ores.

The last two decades have seen an increasing use of pisolite and marra mamba ores, but the decrease in high-grade hematite ores in Japan is expected to result in the increase in the use of high-phosphorous brockman ores (hereinafter referred to as high-P ores) in the near future. Few reports have been published on high-P ores compared with many papers on pisolite and marra mamba ores. The use of high-P ores started at JFE Steel earlier than at other mills, and it was confirmed that the deterioration in quality and productivity of sinter product is the same as that of pisolite or marra mamba ores.

Increasing the blending ratio of porous iron ores narrowed the range of proper melting level for sintering operation. The conventional sintering process, which is focused on the usage of high-density hematite ores and the melting them by a large quantity of heat source, is not able to fully respond to the recent iron ore trend of porous character. As a result, the deterioration in permeability of sinter bed and strength of sinter product has been exposed. As a countermeasure, various technologies and processes have been developed to optimize the melting reaction by controlling quasi-particle structure as well as granulating fine iron ores such as pellet feed or limonitic marra mamba ores using a pelletizer or high-speed agitating mixer.

For example, Haga et al. studied on the influence of alumina on the formation of melts and they achieved a reduction in coke breeze consumption at Oita Works No. 2 Sinter Plant by avoiding the dispersal of alumina in the sinter mixture by means of pre-granulation of the fine powder...
in high-alumina ores. Another study reported on the effect of limestone distribution in quasi-particle on sintering of iron ores, and the sintering performance was improved at Kashima Works No. 2 Sinter Plant by separately granulating high and low CaO quasi-particles.13)

We previously developed the new granulation process, which is characterized by coating coke breeze and limestone on the surface of quasi-particles, in order to control the excess melting reaction between coarse pisolite ores and limestone.14) However, because the high-P ores are much finer than the pisolite ores, and tend to distribute in the adhering layer of quasi-particle, the control of reaction between limestone and the porous high-P ore becomes difficult when the use ratio of high-P ore increases.

A study was conducted on the optimum distribution of high-P ores for increasing their usage in the near future. This paper presents the experimental results using an electric furnace and a X-ray CT equipment, and the operational test results at a commercial sinter plant, in succession from our previous study14) on the optimum distribution of coke breeze and limestone within quasi-particle.

2. Concept of Quasi-particle Structure Design

Figure 1 shows the concept of quasi-particle structure for the production of high-quality sinter in relation to the trends of iron ore recent resources. It can be seen in the figure that a uniform mixture of SiO2, CaO and MgO, etc. was the primary target when conventional dense hematite ores were used abundantly, and then a coating technology of coke breeze (see Fig. 1(B)) was developed to improve its combustibility15) which later progressed to a coating technology of limestone and coke breeze (see Fig. 1(C)) to cope with the increase of high porosity and coarse pisolite ores.16) Furthermore, a new concept was developed to actively control the melting reaction between the high-porosity ore and limestone in order to process the increased amount of fine and porous marra mamba ores and high-P ores. As shown in Fig. 1(D), the idea is that it is better to segregate the porous ore in the center of the quasi-particle for preventing the absorption of calcium–ferrite melts, and improving the melting reaction of iron ores with limestone by using dense iron ores. An experiment was conducted to investigate this concept.

This study presents the segregation granulation technology of high-P ores for controlling the pore structure of sinter cake as an example of quasi-particle structure coping with the future trends of iron ores resource.

3. Experimental Method

3.1. Test Specimen

Table 1 shows the chemical composition, pore volume of 500 μm or less and harmonic mean size of iron ore samples. Here, the pore volume and pore size distribution were measured using a mercury porosimeter (PoreMaster GT60, Quantachrome Corp.) after heating 10 g of iron ore sieved to 3–5 mm at 773 K for 1 h. Figure 2 shows the pore size distribution of iron ore samples.

The results indicate that pisolite ore (Ore G), marra mamba ore (Ore M) and high-P ore (Ore P) have high combined water content and a high porosity compared with a typical hematite ore (Ore H). Pisolite ore has a large volume of micro pores with a size of 0.1 μm or less; however, marra mamba and high-P ores have a large pore volume ranging from 0.1 to 1 μm and also have a larger volume of total pores in the ore.

3.2. Laboratory Granulation Experiment Method

Table 2 shows the blending ratio of raw materials used in the sintering test. The raw materials used in the experiments were a standard material (Blend 1), which was composed of...
Ore H (hematite ore) only as an iron ore, a material (Blend 2) in which 50% of Ore H was replaced Ore P (high-P ore). In all cases, the limestone and silica sand contents were adjusted so that the SiO₂ content of the blended material was 5.0% and the CaO was 9.5%, and the amount of coke breeze was 5.0%.

Figure 3 shows the granulation experiment method. In the limestone/coke breeze coating method aimed at a uniform distribution of iron ores in quasi-particle, all raw materials except limestone and coke breeze were first charged into the drum mixer and granulated for a specified time, and then the coke breeze and limestone were charged and the mixture was granulated again, as shown in Fig. 3(A). In the another granulation method aimed at segregating the high-P ore within the quasi-particle, Ore P was granulated in a high-speed mixer and then granulated with the hematite Ore H in a drum mixer, after which limestone and coke breeze are charged for the coating and granulation operation, as shown in Fig. 3(B).

Other granulation conditions and measuring methods for size distribution of quasi-particle, etc. were already introduced in the previous report

### 3.3. Measurement Method for Pressure Drop in the Sintering Pot Test

Sintering process was simulated using a test pot of 300 mm in inner diameter and 400 mm in height, in which a sheath-type thermocouple (1.6 mmφ) and a pressure probe (stainless steel pipe; 6 mm in outer diameter×4 mm in inner diameter) were set at a position of 100 mm inward from the sidewall. Pressure drop within the sinter bed was measured as the difference from atmospheric pressure, and the inner temperature was also measured at the same time, at a constant wind velocity of 0.5, 1.0 and 1.5 Nm/s. In addition, the pressure difference from atmospheric pressure was also measured at a height of 230 mm by inserting a pressure probe in the melting zone, because the over 1473 K zone (melting zone) was approximately 30 mm in thickness from measurement of infrared thermal image recorder with a transparent silica tube.

### 3.4. Sintering Pot Test Using X-ray CT Device

Sintering test was conducted using X-ray CT equipment to quantify the influence of iron ores distribution in quasi-particle on the formation of pores with a size of 5 mm and larger, which affect the permeability of sinter bed. The experiment involved sintering at a constant wind velocity of 0.5 Nm/s using the same raw mixture as described in Sec. 3.2. Other sintering conditions and the X-ray CT filming conditions and image analysis method were described in the previous report

### 3.5. Tablet Test for Melt Fluidity

In order to examine the influence of iron ore distribution in quasi-particle on melt fluidity, an electric furnace test was conducted using a two-layer tablet. Figure 4 shows a schematic drawing of the test. The upper tablet (8 mmφ×8 mmH) assuming calcium–ferrite melt was placed on top of the lower tablet (16 mmφ×10 mmH) assuming iron ore, utilizing the method developed by Okazaki et al. The tablets were then charged into the electric furnace heated up to three temperature levels (1498, 1523, and 1573 K) and sintered for the specified time (60, 120, and 180 s), and then the melt flow length was measured after removing the sample from the furnace. In addition, the cross sections of cooled sample were observed using an optical microscope and the chemical composition of the melted material was quantified by use of electron probe micro analyzer (EPMA).

The upper tablet was made by blending a reagent of CaO and Fe₂O₃ to match the chemical composition having a low melting point in the binary phase diagram (CaO=20 mass%,

### Table 2. Blending conditions in pot test (mass%).

| Blend   | Blend 1 | Blend 2 |
|---------|---------|---------|
| Ore H (hematite ore) | 100.00  | 50.00   |
| Ore P (high-P ore)   | 0.00    | 50.00   |
| SiO₂     | 5.00    | 5.00    |
| CaO      | 9.50    | 9.50    |
| Coke breeze | 5.00    | 5.00    |

Fig. 3. Granulation method in laboratory test.
Fe₂O₃=80 mass%) and was pressed at 40 MPa. The lower table was made by mixing Ore H and Ore P, which were ground to 0.5 mm or less in size and first granulated to be a size of 5–8 mm, and was pressure formed at 40 MPa, and then preliminarily sintered at 1,173 K for 1 h. Three types of lower tablets were chosen: two homogeneous types (shown in Fig. 4(A); 100% Ore H and 50% Ore H/50% Ore P) and a segregation type (shown in Fig. 4(B); 50% Ore H/50% Ore P in core).

### 3.6. Operation Test at Commercial Sinter Plant

Figure 5 shows the “segregation granulation” test equipment for high-P ore at Fukuyama No. 4 Sinter Plant in West Japan Works of JFE Steel, which was installed for quantifying the influence of iron ore distribution in quasi-particle on commercial sintering operation.

The process involves the pre-granulation of high-P ore using a high-speed agitating mixer, which aimed at segregating the relatively fine and porous high-P ore at the center of the quasi-particle. The granulated high-P ore was mixed and granulated with other iron ores, and then the coke breeze and limestone transported by a separate high-speed conveyor (50–300 m/min) was injected at the rear of the drum mixer, in order to coat the quasi-particle mainly composed of iron ore with them. In addition, as a reference, conventional sintering operation was conducted with coating limestone and coke breeze around all iron ores.

The blending ratio of burnt lime, bed height and granulation moisture were kept constant during the operation test at the commercial plant. Change in permeability was adjusted by pallet speed so as to maintain a constant exhaust gas temperature (633–653 K) at the second wind box from the discharge section (No. 22 wind box). Furthermore, in order to avoid the deterioration in permeability of sinter bed, operational conditions such as pallet speed and coke breeze ratio, etc. were set to keep the −4 mm fines mass ratio of sinter product to be under 10.0%. SiO₂ and basicity were controlled to be at 5.0 mass% and 1.9, respectively by changing ratio of limestone, Ni slag and silica stone.

Quasi-particles produced at the commercial plant were sampled at the charging equipment, and their size distribution was measured as described in Sec. 3.2. A test specimen was prepared by embedding quasi-particles of 4.76 mm or larger in a resin mold, and the distribution of iron ore or limestone in quasi-particle was investigated by EPMA.

Sintered products were sampled on the final conveyor at the exit side of the sinter plant. Measurement was performed not only on −4 mm fines ratio of sinter product and the FeO content, but also on tumble strength (JISM8712), reducibility (JISM8713) and low-temperature reduction-disintergation (JISM8720). The content of hematite, magnetite and quaternary calcium–ferrite in sinter were quantified using powder X-ray diffraction analysis, and the residue was roughly classified into four compositions as the amorphous silicate quantitative analysis method for various minerals, described in the previous report. In addition, primary hematite (original iron ore) and secondary hematite once crystallized after melting were separately quantified according to shape using both an optical microscope and an image analyzer, and their mass ratio was calculated based on area ratio. As for the pore structure in sinter product, fine pore volume and pore size distribution of 500 μm or less in sample of approximately 10 g in mass and 4–7 mm in size were measured using a mercury porosimeter as in case of the iron ore.

### 4. Experimental Results and Discussion

#### 4.1. Influence of High-P Ore Blending on Permeability and Melt Fluidity in the Sintering Process

Figure 6 shows the pressure drop of each sintering zone in sinter bed at a time of 300 s after ignition. The definition of temperature range is as follows: wet zone ≤353 K; drying and combustion zone 353–1,473 K; melting zone over 1,473 K; and sinter cake zone under 1,473 K in cooling process. The results indicate that the permeability of the wet zone did not basically change with the increase in high-P ore ratio; however, the permeability of the melting zone deteriorated significantly and total pressure drop during the sintering process increased. Okazaki et al. reported that marra mamba ores are difficult to be granulated, however, in the present test the granulation capability of high-P ore was...
did not appear with change significantly compared to hematite ores, because the high-P ore used contained a relatively large amount of clay minerals and the granulated quasi-particle size did not change significantly.

Table 3 shows the calculation results of each permeability resistance coefficient for the laminar flow term and turbulent flow term using Ergun’s equation.\(^{21}\)

\[
\frac{\Delta P}{L \cdot u} = 150 \left( \frac{1 - \varepsilon}{\varepsilon^3} \right) \frac{\eta_g}{d^2} + 1.75 \left( \frac{1 - \varepsilon}{\varepsilon^3} \right) \rho \cdot u \cdot d = k_1 \eta_g + k_2 \rho \cdot u
\]

where \(\Delta P\) is pressure drop (Pa); \(L\) is bed height (m); \(u\) is gas velocity (m/s); \(\varepsilon\) is void fraction (—); \(\eta_g\) is gas viscosity (Pa·s); \(d\) is particle size (m); \(\rho\) is gas density (kg/m\(^3\)); \(k_1\) is permeability resistance coefficient (1/m\(^2\)); and \(k_2\) is permeability resistance coefficient (1/m).

The experimental results show that although any permeability resistance coefficient of the wet zone basically did not increase with the increase in high-P ore ratio, the pores permeability resistance coefficient of the melting zone increased greatly. Furthermore, the contribution from increased turbulent flow resistance \((k_2)\) is greater than that of the increased viscous flow resistance \((k_1)\), and the increase of pressure drop due to branching, growth and shrinkage of pores has a much bigger effect than the pressure drop due to the fluid friction at the solid surface. The complexity of pore structure in the sinter cake caused the deterioration of permeability with blending high-P ore. Accordingly, it is considered that the improvement in melting zone is effective for coping with the deterioration in sinter productivity and permeability of sinter bed at a usage of high-P ore.\(^{22}\)

Figure 7 shows X-ray CT images of sinter cake after completion of sintering, and Fig. 8 shows the effect of blending high-P ore on the change in branch width of pores during sintering. According to these results, it is presumed that the increase in high-P ore resulted in the formation of a complex pore structure due to the reduction in number of pores with a size of 5 mm and larger functioning as gas flow channels in the sinter cake, and due to the increase in finer pores less than 5 mm in size that seem to function as closed gas flow channels, thus leading to an increase in pressure drop.

Figure 9 shows the melt flow length in homogenous type tablets with a high-P ore blending ratio of 0% and 50%. Figure 10 shows the optical microscopic images of the cross section and schematic diagrams of the melt flow part in tablets heated at 1 573 K for 180 s. It is presumed that melt fluidity, which is highly correlated to pore growth rate,
deteriorated with the increase in high-P ore blending as shown in Fig. 9, which resulted in narrower pore width and led to the deterioration in permeability of melting zone. Furthermore, as shown in Fig. 10, the boundary between the calcium–ferrite melts and iron ore particles is clear for dense hematite ores; however, in the case of highly porous high-P ore, the interface between the melt and iron ore is unclear as the melts are absorbed into the iron ore.

Next, the chemical composition of the melt flow part in Fig. 10 was quantified using EPMA and plotted on the binary phase diagram of CaO–Fe₂O₃ system as shown in Fig. 11. It was confirmed that Fe₂O₃ content in the calcium–ferrite melts increased with the increase in blending ratio of highly porous high-P ore. It is presumed that the melt was a solid/liquid mixture because the tablet test temperature was below the liquidus temperature. The viscosity of melts suspending solid phase in them was calculated using Eq. (2), referencing the equation proposed by Mori et al. and assuming that solid spheres with a single size are in cubic packing and the critical solid phase fraction is 0.52. The melt viscosity used in the Eq. (2) was obtained based on measurement data from Endell et al. using Andrade’s equation for the correction of temperature dependence.

\[
\eta = \eta_L \left\{1 + 3/(1/S - 1/0.52)\right\} ................................(2)
\]

where \(\eta\): viscosity of suspensions (Pa·s); \(\eta_L\) is melt viscosity (Pa·s); and \(S\) is volume base solid fraction in mixed system (—). The calculated viscosity of suspensions are shown in Table 4.

Furthermore, assuming the driving force of melt transfer as capillary force, the melt flow length is expressed as in Eq. (3) using the Hagen–Poiseuille’s law. In this formula, the radius of capillary tubes \((R)\) was assumed to be constant, although it was closely related to the size and shape of iron ore particles, and the complexity of pores as capillary tubes. The melt flow length \((h)\) was calculated through a fitting treatment. Solid lines in the above Fig. 9 show the calculated results based on Eq. (3), which correspond well to the measured values.

\[
h = \phi \left(\frac{R \gamma \cos \theta}{2 \eta^{1/2}}\right)^{1/2} t^{1/2} ................................(3)
\]

where \(h\) is melt flow length (m); \(R\) is radius of capillary tubes (m); \(\gamma\) is surface tension (N/m) \((=0.55)\); \(\theta\) is contact angle (°) \((=30)\); \(\eta\) is viscosity of suspensions (Pa·s); and \(t\) is time (s).

Combined with the results shown in Table 4, the reason for the increased pressure drop in the melting zone with the increase in high-P ore blending ratio is considered to be as follows: first, melt fluidity deteriorated as a result of the increase in suspension of solid phases (Fe₂O₃) due to the melt absorption into iron ore; and the number of coarse pores with a size of 5 mm or larger contributing to permeability of sinter bed decreased with the suppression of pore growth due to the deterioration of melt fluidity.

### 4.2. Effect of High-P Ore Distribution in Quasi-particle on Sinter Bed Permeability and Melt Fluidity

In order to suppress the deterioration in permeability of sinter bed at a usage of high-P ore, the segregation type of quasi-particles was granulated as shown in Fig. 4 above mentioned and the measurement of the melt fluidity and observation of melt flow part by EPMA were conducted.

**Figure 12** shows the effect of Ore P distribution in quasi-particle on the melt flow length at 1 573 K. **Figure 13** shows the melt flow analysis using a secondary electron microscope (SEM) and EPMA. Based on these results, it was confirmed that the absorption of melt into iron ore was suppressed and melt flow length was remarkably improved, al-
most at the same level as that without high-P ore, by segregating high-P ore to the center of quasi-particle for the blending ratio of 50% high-P ore.

Figure 14 shows the change of pressure drop distribution at each sintering zone measured using granulated quasi-particle, following the granulation flow as shown in Fig. 3 above mentioned. It was confirmed that by the segregation of high-P ore in the center of quasi-particle, melt fluidity improved, pore growth accelerated, and the permeability of the melting zone decreased to almost the same level as that without blending of high-P ore.

4.3. Operational Test Results at Commercial Plant

In order to confirm the results obtained from earlier laboratory experiments, an sinter plant test was conducted at

| Temperature range (K) |   |
|----------------------|--|
| Sinter cake          | ~1473K |
| Melting zone         | 1473K  |
| Drying and combustion zone | 353K ~ 1473K |
| Wet zone             | ~353K  |

In Fig. 14, effect of Ore P distribution on pressure drop at a time of 300 s after ignition ($\nu_r = 0.5 \text{ Nm/s}$).

**Fig. 12.** Effect of iron ore distribution on melt fluidity at 1 573 K.

**Fig. 13.** SEM and EPMA images and the distribution of Ca and Fe in the melt flow part heated at 1 573 K for 180 s.

**Fig. 14.** Distribution of Ca, Fe and Al in the quasi-particle analyzed by EPMA.
Fukuyama No. 4 Sinter Plant by installing the experimental equipment shown in Fig. 5 above mentioned.

**Figure 15** shows the EPMA analysis result for cross sections of granulated particles with a size of 4.76 mm or larger, which were sampled at the charging equipment. It can be seen from these images that quasi-particle produced by the segregation granulation method shows the successful segregation of high-P ore of relatively high Al₂O₃ in a quasi-particle center as designed, compared to the quasi-particle by the homogenous granulation method.

**Figures 16 and 17** show the results of the sinter plant, in which the blending ratio of high-P ore was set at 15%. It can be seen in Fig. 16 that by applying the “segregation granulation method” for high-P ore, the permeability of sinter bed improved, the No. 22 wind box temperature that is strongly correlated to flame front speed increased, and the burn through point moved up toward the charging apparatus side. Then, after increasing the pallet speed to keep the No. 22 wind box temperature constant, sinter productivity increased by approximately 4%. As shown in Fig. 17, although the mass percentage of fines under 4 mm in size and the low-temperature reduction-disintegration of sinter products did not change greatly by applying the “segregation granulation method”, the tumble strength and reducibility index (JIS-RI) of sinter improved. The improvement in tumble strength, which is the cold strength, is attributed to the reduction number of pores with an intermediate size of 0.5 to 5 mm that adversely affects the cold strength, resulting from the improved melt fluidity shown in the above Fig. 12 due to applying the segregation granulation method.

### 5. Conclusions

Porous, fine high-P ores, which are expected to be increasingly utilized in the near future, was studied through laboratory experiment to determine its effects on sinter bed permeability and melt fluidity during the sintering of iron.
ores. Furthermore, a new granulation method for segregating fine high-P ore in the center of quasi-particle “segregation granulation method” was developed based on the experimental results to prevent adverse effects of high-P ore and the effect of the new method was confirmed by a sinter plant tests.

The following knowledge was obtained in this study:

(1) It was found from laboratory experiment that deterioration in sinter bed permeability by using high-P ore occurred due to the increase in pressure drop of the melting zone in which pore growth rate was suppressed by the decrease of melt fluidity.

(2) Deterioration in permeability of sinter bed was attributed to the increase in viscosity of suspensions due to the absorption of formed melts into highly porous high-P ore.

(3) A new granulation method for segregating fine high-P ore in the center of quasi-particle and coating dense hematite ores around its surface made it possible to improve the sinter bed permeability through improved melt fluidity by preventing the absorption of the melt into high-P ore.

(4) The sinter plant test showed the increase in sinter productivity due to the improvement of sinter bed permeability and in addition, cold strength and reducibility were also improved by applying the “segregation granulation method”.

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