Influence of Iron Ore Characteristics on Penetrating Behavior of Melt into Ore Layer

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It is important to maintain a certain amount of melt during sintering of iron ores to improve product yield and strength of sinter product. Spreading phenomena of the initial melt within adhering layer in pseudo-particles are essential to achieve efficient agglomeration. Penetration tests have been conducted to demonstrate the spreading behavior of the initial melt. Paired samples consisting of ore tablet and initial melt tablet were heated and penetration length was measured. Penetration behavior of melt during sintering was discussed on a base of capillarity. Determining factors of penetration were morphology of the ore surface before and after dehydration and chemical composition of ores. Product yield and the strength of sinter were reflected by the penetration length of blended ores in a sintering plant.

KEY WORDS: raw material; iron ore; iron ore sinter; agglomeration; penetration; melt.

1. Introduction

The mining situation of Australian iron ores, which occupies about 50% of all import iron ores in Japan, has been changing. Marramamba ores, porous and containing high combined water (CW), were newly developed¹⁾ and have started to export to Japan instead of low phosphorous Brockman ores. This situation should result in an increase in the blending ratio of ores containing high CW.

It is known that the high CW ores form brittle and porous structure in sinter products, as a result of dehydration of CW during sintering.²⁾ In addition, recent tendency to manufacture low slag sinter for decreasing reducing agents rate of blast furnace would result in a more brittle structure of sinter product.

Considering the situation above mentioned, we think that the proper structure designing of pseudo-particles in the sinter mixture is essential to maintain or increase product yield and the strength of sinter.

In the sintering process the quality and quantity of melt largely influence product yield and the strength of sinter product due to the limited reaction time.

It is assumed that the sintering reaction proceeds following two steps:

1. Firstly, the initial melt forms within adhering layer in pseudo-particles.
2. The initial melt spreads out through the adhering layer and promotes assimilation reaction between ores or fluxes and melt.

As for the initial stage of sintering, theoretically the initial melt forms at 1205°C, the eutectic temperature between $\text{CaO} \cdot \text{Fe}_2\text{O}_3$ and $\text{CaO} \cdot 2\text{Fe}_2\text{O}_3$ in $\text{CaO}-\text{Fe}_2\text{O}_3$ system

in air.³⁾ Furthermore in the past mineralogical investigation, we designed pseudo-particles of ores containing low CW as nuclei⁴⁾ and ores containing low Al_2O_3 as adhering fines⁵⁾ in a point of view of the melt formation at the initial stage of sintering.

However, little information have been obtained concerning the behavior of the initial melt within the adhering layer in pseudo-particles and the influence of the behavior of melt on product yield and the strength of sinter. In addition, the ore characteristics concerning with its assimilation with the spreading out melt have been unknown even though many brands of ores are mixed in the actual plant sintering.

Hence in the present study, the behavior of the initial melt and its influence on product yield and the strength of sinter were centered. To clarify them, penetration tests using several kinds of ores were performed and results were discussed on the base of ore characteristics such as surface morphology of ores and gangue mineral compositions.

2. Past Investigations

Many investigations have been conducted on melt in sintering process, especially on 1) formation of melt, 2) fusibility of ores and 3) fluidity of melt.

As for the formation of melt, Soma *et al.*⁶⁾ interrupted the sintering reaction by flowing N_2 in pot tests. They found that the initial melt formed within fine region in pseudo-particles and assimilated with coarse ores, resulting in an increase in amount of melt.

In addition, Matsuno found that melt formed from Calcium ferrite (CF), formed by solid-state reaction between ores and limestone, and assimilated with gangue

minerals.⁷⁾ This result is agreed with the result by one of the authors, showing that the forming region of the initial melt was 'small granular compounds' forming at about 1160°C by solid-state reaction and having the composition of near $\text{CaO} \cdot \text{Fe}_2\text{O}_3$.⁸⁾

Based on those findings, in the present study we focused the ore characteristics on melt formation and evolution.

As for fusibility of ores, to evaluate coarse ores as nuclei particles in pseudo-particles the assimilation degree⁹⁾ was measured. Recently three points bending strength of sandwich samples consisting of lumpy ores and fluxes was measured as well.¹⁰⁾

On the other hand, to evaluate fine ores as adhering layer the appearance change after heating of tablets consisting of CaO and ores was measured in many investigations.^{11–15)} They reported the influence of porosity of ores and chemical composition, especially of gangue minerals, on fusibility of tablets.

The fluidity of forming melt was reported as well. Past investigations strongly suggested large influence of chemical compositions of ores and structural change during heating on fluidity of melt. Kasai *et al.*¹⁶⁾ measured dripping temperature and dripping weight of melt during heating to evaluate melt forming behavior. They found large influence of CaO content of specimen and gangue content of ores on the dripping behavior. As for the influence of Al_2O_3 and SiO_2 , fluidity of melt decreased with an increase in Al_2O_3 and SiO_2 contents in melt^{17,18)} and assimilation of limestone with ores was depressed by SiO_2 .¹⁹⁾

As for structural change of ores during heating, pores originated from porous structure of ores decreased fluidity of melt.¹⁴⁾

Relating those findings in past investigations to empirical changes in product yield and the strength of sinter, we noticed that product yield and strength of sinter must depend on the melt properties, reflected by ore type and reaction quantity of ore with lime.

Figure 1 shows a result of microstructural observation of adhering layer in plant pseudo-particles. Some particles of CaO source distributed near ore particles, whereas some particles distributed near coke breeze and return fines indicated as circle in the figure. Hence, melting process in sintering was assumed as follows:

- 1) The initial melt forms locally in the adhering layer as a result of a reaction between ores and CaO source.
- 2) The melt assimilates with gangue minerals and ores, resulting in an increase in melt amount.

The rapid spreading out of the initial melt through adhering layer in pseudo-particles seems to be essential to achieve solid bonding phase in microstructure in sinter because the sintering reaction takes place within few minutes. In the present study to demonstrate the spreading behavior

of melt sintering tests using samples consisting of a ore tablet and a tablet of initial melt source were conducted and 'penetration length' was defined as an evaluating index.

3. Experimental Method

3.1. Chemical Compositions of Ores

Table 1 shows the chemical compositions of ores used in the present study. Each ore was from different source of mining and had different chemical composition and mineralogy. The particle size distribution of ores, were 50 mass% 0.25–0.5 mm and 50 mass%–0.25 mm, was prepared.

Ore A was Brazilian dense ore, containing low Al_2O_3 . Ore B was Brazilian porous ore, containing low Al_2O_3 . Ore C and Ore D were Australian hematite ores containing high Al_2O_3 . Ore C was more porous than Ore D. Ore E and Ore F were Australian pisolitic ores. Al_2O_3 content of Ore E was higher than that of Ore F. Ore G was Indian ore containing high goethite.

3.2. Experimental Procedure

The fine ore was shaped into a cylinder tablet. The tablets were made by using a steel mould under a pressure of 4 MPa. The green ore tablet had a height of 5 mm, a diameter of 15 mm and a porosity of 30%. The starting materials of the tablets demonstrating the initial melt were chemical reagent of Fe_2O_3 and CaO. The chemical composition of the tablets was fixed at $\text{CaO}=26.0$ mass% and $\text{Fe}_2\text{O}_3=74.0$ mass%, the composition of $\text{CaO} \cdot \text{Fe}_2\text{O}_3$.

After mixing chemical reagent of CaO and Fe_2O_3 for 20 min, the mixture fine was shaped into cylinder tablet under a pressure of 4 MPa. The green tablet had a height of 5 mm and a diameter of 5 mm. **Figures 2 and 3** show the

Fig. 1. Distribution of the limestone in adhering fines of pseudo-particles.

Table 1. Chemical compositions of ores.

	T.Fe	SiO ₂	Al ₂ O ₃	CW	Ore type	Source
Ore A	62.8	8.34	0.54	0.52	Hematite	Brazilian ores
Ore B	65.7	0.86	0.85	1.45		
Ore C	63.2	3.66	2.11	3.01		
Ore D	62.8	4.76	2.47	2.01	Pisolite	Australian ores
Ore E	59.1	5.88	2.92	7.28		
Ore F	58.8	4.94	1.44	7.93	Goethite	Indian ore
Ore G	62.4	3.44	1.47	4.91		

experimental procedure and apparatus. The ore tablet and the $\text{CaO-Fe}_2\text{O}_3$ tablet were paired and charged in a Ni crucible whose diameter was 20 mm and height was 15 mm, subsequently sintered in air stream by using an electric furnace. Heating temperature condition of samples was demonstrated that of actual plant sintering machines, *i.e.*, heating from 1100 to 1290°C in 1 min and cooling from 1290 to 1100°C in 3 min. Samples were quenched at 1100°C by removing outside of the furnace.

After heating, polished surface in the vertical direction of samples was observed and the penetration length was measured. **Figure 4** shows an example of samples in a 5 times magnification. The penetration lengths at the center (③) and at a quarter of the diameter from rim (② and ④) were measured respectively. The penetration length was expressed as the average value of them.

Microstructural observation by using optical microscope

(1) Ore tablet	
(a) Particle size distribution	0.25mm-0.5mm=50mass% -0.5mm =50mass%
(b) Tablet size	5mmH × 15mm φ
(2) Reagent tablet	
(c) Chemical composition	$\text{Fe}_2\text{O}_3=74\text{mass\%}$ $\text{CaO} =26\text{mass\%}$
(d) Tablet size	5mmH × 5mm φ
(3) Heating conditions	
(e) Heat pattern	1100°C-1290°C: 1min 1290°C-1100°C: 3min
(f) Atmosphere	Air

Fig. 2. Experimental conditions of the melt penetration test.

and EPMA analysis of penetrated region were mainly conducted as well to discuss the penetrating behavior of melt.

4. Results and Discussion

4.1. Penetration Length of Melt and Microstructure of Sintered Tablets

Figure 5 shows the photo images of vertical cross section of samples after penetration tests. Penetrating limit in each sample was indicated as dot lines. It was clearly shown that the penetration of melt using Brazilian ores (Figs. 5(a), 5(b)) was larger than that using most of Australian ores (Figs. 5(c)–5(f)).

Figure 6 shows the measurement result of penetration length. The result indicated that penetration length of melt depended on ore types. The penetration length of melt using Brazilian ores (Ore A and Ore B) was about 4 mm, 80% of the height of green ore tablet. In contrast, the penetration length of melt using Australian ores was about 1.2 mm, 25% of the height of green ore tablets, despite of different ore types. The penetration length of melt using Ore G and Ore M was about 2.5 mm (50%) and 3.0 mm

①⑤ : Edge of melted region in the horizontal direction
②③④: Measurement region

Fig. 4. Measurement positions of melt penetration length.

Fig. 3. Schematic diagram of experiment apparatus for the melt penetration test.

Fig. 5. Macrostructure of cross section after melt penetration test.

Fig. 6. Measurement result of the melt penetration length of the specimen.

(60%), respectively.

Looking at morphology of melted region, we noticed that the structure of melted region was largely influenced by ore types. In the case using Ore C and Ore D, the melted region lost its initial tablet shape and covered the surface of the ore tablet. On the other hand, in the case using pisolitic ores (Ore E and Ore F) the melted region maintained its initial shape and contained many pores.

4.2. Factors Determining Penetration

In this chapter, factors determining penetration based on the analysis results are discussed

4.2.1. Influence of Morphology of Ore Surface on Penetration

The initial melt contacts the surface of ore during penetration. Therefore the morphology of ore surface was thought to be one of major factors determining penetration behavior. **Figure 7** shows SEM images of ore surface before heating. Ore A and Ore B had smooth surface compared with other ores. The surface of Ore A seemed to be smoother than that of Ore B. Other ores had rough surface due to many adhering ore particles whose size was 1–5 μm . No significant difference was observed among them.

Fig. 7. SEM images of the ore surface before heating.

To clarify the difference of the morphology of ore surface quantitatively, surface area of ores was measured by using N_2 absorb method. **Figure 8** shows the relationship between penetration length and measured surface area. The penetration length decreased with an increase in surface area. The result implies following mechanism of penetration:

- 1) The initial melt contacts with ore.
- 2) Hematite and gangue mineral dissolve into the initial melt thorough contacting region.
- 3) Fluidity of melt decreases.

Hence the penetration length would decrease with an increase in contact area between melt and ores.

This fact concludes that morphology of ore surface largely influences the penetration behavior.

4.2.2. Influence of Chemical Composition

Not only physical properties of ores, chemical composition was also thought to be one of the major factors determining penetration behavior *via* changes in melt property. **Figure 9** shows the relationship between chemical compositions of ores and penetration length. The influence of T.Fe content on penetration length was small (Fig. 9(a)). In contrast, penetration length tended to decrease with an increase in SiO_2 content except for Ore A (Fig. 9(b)). This result was agreed with the result in the past investigation.¹⁸⁾ The reason behind the different tendency in the case of Ore A is most likely due to peculiar textures of gangue minerals and

hematite mentioned in following chapter.

A clear relationship between Al_2O_3 content and penetration length was found. The penetration length decreased with an increase in Al_2O_3 content. A decrease in the penetration length with increasing CW was also found, however the tendency of the decrease by CW was rather smaller compared with that with Al_2O_3 content. CW content seems to influence penetration behavior via a change of surface structure of ore after dehydration.

After the analysis, it was clearly shown that both of SiO_2 content and Al_2O_3 content inhibited the penetration of melt. The reason behind the inhibition by Al_2O_3 content is a decrease in fluidity of melt after dissolution of Al_2O_3 .

On the other hand, influencing mechanism of SiO_2 on the penetration behavior seems to be rather complex. To clarify the mechanism, microstructural observation in a high magnification was performed. Ores having different level of SiO_2 content were selected. **Figures 10(a)** and **10(b)** show microstructures after penetration tests using Ore B and Ore F, respectively. Ore B was low SiO_2 ore and Ore F was high SiO_2 ore and penetration length of Ore B was larger than that of Ore F. The Al_2O_3 contents of both were small.

In the case using Ore B, calcium ferrite formed at the tip of penetrated region. In contrast, in the case using Ore F silicate slag formed. **Figure 11** shows the results of EPMA analysis. In the case using Ore F, Si condensing zone was found at the tip of the penetrated region. This zone contained less Ca and Fe. Therefore this zone was silicate slag found in the microstructural image in Fig. 10. The viscosity of silicate melt is more than three times higher than that of calcium ferrite melt.²⁰⁾ Hence the reason behind the low penetration length in the case using Ore F is a formation of silicate slag at the tip during penetration of melt, resulting in an increase in viscosity of melt. One of the reasons behind the formation of silicate slag is high content of SiO_2 in Ore F.

However, Ore A had large penetration length despite of its large SiO_2 content ($SiO_2=8$ mass%). This fact represents that the penetration behavior is not fully explained by the chemical composition of ores and other factors must be considered.

Fig. 8. Relationship between the melt penetration length and the surface area of ores.

Fig. 9. Relationship between the chemical compositions of ores and the melt penetration length.

Fig. 10. Microstructures of tablets after penetration test (circles indicate observation part).

Fig. 12. Microstructure of the surface of Ore A.

Figure 12 shows the microstructure of Ore A before heating. The major gangue mineral of Ore A was quartz, having large particle size (50–100 μm) and dense structure. The quartz distributed among hematite grains and at the surface of the ore as a single particle. Furthermore, the hematite grains had smooth surface and dense structure.

From the observation result, the reason behind the large penetration length of Ore A is limited assimilation between melt and quartz and between melt and hematite due to the large particle size and dense structure of quartz and hematite grains. As a result, melt could maintain high fluidity and readily penetrate into ore layer.

This fact implies that microstructure of ores before penetration test is one of the factors determining the penetration behavior. It is well known that pisolitic ores have particular microstructure before heating. Therefore in the following chapter, melt penetration behavior into pisolitic ores was closely examined.

4.3. Penetrating Behavior of Melt into Pisolitic Ore Layer

As shown in Fig. 5, in penetration tests using pisolitic ores (Ore E and Ore F) peculiar penetration behavior was found, *i.e.*, penetration of melt did not proceed and the melted region maintained initial tablet shape and contained many pores. The pore formation must be related to structur-

Fig. 13. Changes in combined water content in ore tablets after heating with heating temperature.

al change of goethite or cracking of the ores during dehydration. To clarify the influence of dehydration on penetration behavior, some fundamental tests were conducted.

The direct contribution of CW to pore formation in melt was one of the possibilities of pore formation mechanism. Therefore, ore tablets without paring initial melt tablet were heated. Ores having different level of CW, Ore C (CW=3.01 mass%), Ore E (CW=7.28 mass%), Ore F (CW=7.93 mass%) and Ore G (CW=4.91 mass%), were selected. After heated from room temperature at a rate of 200°C/min up to 1000°C, 1100°C and 1300°C respectively, the ore tablets were cooled in air and CW content of the tablets was measured. Figure 13 shows the CW content in a relationship with the maximum heating temperature.

CW content after heating was small in all cases and less than 0.15 mass% in heating up to 1100°C and less than 0.04 mass% in heating up to 1200°C. The initial melt was thought to be formed at 1205°C³⁾ in penetration tests. This fact concludes that dehydration of ores had been almost completed before a formation of initial melt in penetration tests. Therefore, the direct contribution of CW to a pore formation in penetrated region of pisolitic ores seems to be small.

In a heating of pisolitic ores cracks form in the goethite region as a result of dehydration.²⁾ To clarify the relation-

Fig. 11. Element distribution of the penetrated region by EPMA.

Fig. 15. Schematic overview of classification of melt penetration behavior.

Fig. 14. Microstructures of ore tablets after heating up to 1200°C.

ship between pore formation in melted region and the structural change during dehydration, microstructural observation of pisolitic ore tablets after heating up to 1200°C was conducted. For references, the microstructures of Ore C and Ore G were observed as well. **Figure 14** shows the microstructures of ores after heating.

No crack was formed in a heating of Ore C (a). This is mostly due to a rapid diffusion of the gas formed during the decomposition of goethite, because of a porous structure of ore particles. Ore G had no cracks after heating either (b), due to a self-densification of martite particles during heating.⁴⁾ On the other hand, many cracks were found in a heating of pisolitic ores, Ore E and Ore F ((c), (d)). This fact implies that the origin of pores formed in melt is cracks and pores in pisolitic ores after dehydration.²¹⁾ Fluidity of melt decreases by containing pore.¹⁸⁾ Therefore the reason behind the low penetration length of pisolitic ores is pore formation in melt due to their peculiar structural change during dehydration.

4.4. Mechanism of Melt Penetration into Ore Layer

Penetration behavior of melt into ore layer in the present study can be explained on the base of penetration phenomena derived by capillary pressure. Therefore penetration length is expressed by the following equation.²²⁾

$$h(m) = [(r\sigma \cdot \cos \theta) / (2\eta)]^{1/2} \cdot t^{1/2} \dots \dots \dots (1)$$

where h : penetration length (m),
 r : capillary radius (m),
 σ : surface tension (N/m),
 θ : contact angle (rad),
 η : viscosity (Pa·s),
 t : penetration time (s).

In the present study, penetration time and temperature were constant due to a constant heating condition. In contrast, melt properties such as viscosity, surface tension and contact angle were varied by changes in chemical composition of melt during penetration. Surface tension of melt

should be maintained constant because those of calcium ferrite melt and silicate melt are almost the same level,²³⁾ whereas viscosity of melt is largely changed by its SiO₂ content. The data of contact angle of melt in the present study is limited.

In addition, capillary radius was changed as well by pore structural change such as cracking or self-densification of ores during heating despite of constant porosity of green ore tablets.

As mentioned in previous chapters, penetration behavior in the present study depended on three factors, 1) morphology of ore surface before heating, 2) amount of gangue minerals, especially Al₂O₃ and SiO₂ and 3) morphological change in ore surface as a result of dehydration. This proposal mechanism was comparable with analysis result based on capillarity.

Results in the preset study clearly showed that ore characteristics largely influence on penetration of melt ores layer. **Figure 15** shows schematic overview of classification of penetration behavior of melt into ore layer as a summary of the results.

In the case of ore types representing large penetration of melt, two types of mechanisms are considered.

a) In the case of ores containing a small amount of gangue minerals, melt maintains high fluidity, resulting in large penetration of melt.

b) In the case of ores containing large and dense quartz as gangue minerals, melt maintains high fluidity due to a slow assimilation, resulting in large penetration of melt.

In the case of ore types representing small penetration of melt, two types of mechanisms are considered.

c) In the case of ores containing a large amount of gangue minerals, melt fluidity decreases with dissolution of SiO₂ and Al₂O₃ into melt, resulting in small penetration of melt.

d) In the case of pisolitic ores, melt fluidity decreases by a formation of pores in melt due to cracks and pores after dehydration, resulting in small penetration of melt.

Fig. 16. Changes in (a) shatter index and (b) product yield with calculated melt penetration length of blended ores in sintering plant.

4.5. Relationship between Product Yield and the Strength of Sinter and Penetration Length

As mentioned above, product yield and the strength of sinter must be relating to melt quantity and penetrating behavior. Figure 16 shows the analysis result of operating data at TOBATA No. 3 sintering plant. Changes in product yield and the strength of sinter Shatter index with calculated penetration length were analyzed. In a calculation of penetration length of blended ores, following equation was adopted considering mass fraction of fine particles of ores.

$$Pb = \left(\sum X_i \cdot a_i \cdot W_i \right) / \left(\sum X_i \right) / 100 \quad \dots\dots\dots (2)$$

where Pb: penetration length of blended ores (mm),
i: brand of ores,
 X_i : mass fraction of Ore_{*i*} (mass%),
 a_i : penetration length of Ore_{*i*} (mm),
 W_i : mass fraction of -1 mm in Ore_{*i*} (mass%).

Product yield and the strength of sinter increased with an increase in the penetration length of blended ores. This fact supports that calculated value of penetration length of blended ores can be used for prediction of product yield and the strength of sinter in sintering plant. Nevertheless, a certain deviation from the relationship was still found. It implies that other factors such as the property of nuclei ores and the influence of a permeability of sintering bed must be considered.

5. Conclusions

To clarify the melt evolution process within the adhering layer in pseudo-particles, penetration tests have been conducted. Paired tablets consisting of ore tablet and the initial

melt tablet were heated to demonstrate the penetration behavior of melt into ore layer. Following results were obtained.

(1) Determining factors of penetration behavior of melt were (1) morphology of the ore surface before and after dehydration and (2) chemical composition of ores.

(2) Penetration length of melt decreased with an increase in surface area of ores as a result of an increase in contact area between melt and hematite and gangue minerals.

(3) Penetration length of melt decreased with an increase in Al₂O₃ and SiO₂ content in ores. However, the influence of SiO₂ was depressed in the case using ores having large and dense quartz as gangue minerals.

(4) Pisolitic ores exhibited peculiar penetration behavior due to pores involved in melt as a result of their structural change during dehydration.

(5) Product yield and the strength of sinter were reflected by the penetration length of blended ores in a sintering plant.

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