

Effects of Al₂O₃ and MgO on Softening, Melting, and Permeation Properties of CaO-FeO-SiO₂ on a Coke Bed



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In ironmaking, maintaining gas permeability in blast furnace with low coke rate operation is essential to reduce carbon emissions. The high pressure loss in the cohesive zone decreases the gas permeability and affects the productivity of blast furnace. In order to increase the gas permeability in the cohesive zone, the thickness of the cohesive layer should be decreased. For this purpose, increasing softening temperature and decreasing dripping temperature of the iron ore are desired. In this study, softening, melting, and permeation of SiO₂-FeO-CaO-Al₂O₃-MgO on a coke bed were investigated. The oxide sample in a tablet form was heated under CO/CO₂ atmosphere, and the shape of the tablet was observed. The softening and melting temperatures of the SiO₂-FeO-CaO system changed with the addition of Al₂O₃ and MgO. Oxide tablets with and without Al₂O₃ softened below and above the solidus temperature, respectively. The melting temperatures varied with the ratio of CO/CO₂ in the gas. The permeation temperature was independent of the melting temperature, but dependent on the wettability.

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I. INTRODUCTION

DEVELOPMENT of new processes for mitigating CO₂ emissions in ironmaking has been widely studied.^[1,2] Coke rate can be reduced from the levels of conventional operation by replacing coke with a CO and H₂-based gas mixture. Coke used in the blast furnace acts as structural material for maintaining gas permeability, and thus, low coke rate operation causes an increase in the pressure loss. With the development of low coke rate operation technologies, it is increasingly important to control the flow of solids, liquids, and gases in the blast furnace.^[3-5] In the cohesive zone, the thickness of the coke slit decreases with the decreasing coke rate. The iron ore melts partially and fills the vacancy in the packed bed, decreasing the permeability. Even for the conventional operation, the pressure loss is strongly dependent on the softening layer in the cohesive zone.^[6,7] Therefore, operation at low coke rate is dependent on increased permeability in the cohesive zone.

The ore layer consists of a solid-liquid mixture in the cohesive zone. The layer thickness can be decreased by

delaying the softening and facilitating the dripping of the iron ore. In a previous study,^[8] softening, melting, and permeation properties of CaO-FeO-SiO₂ on a coke bed were investigated. The following results were obtained: (1) The measured softening and melting temperatures were higher than the calculated solidus and liquidus temperatures, respectively. (2) Deformation of the oxide tablet was affected by the liquid phase ratio. (3) Permeation temperature of the oxide melt was independent of the melting temperature. (4) The permeation of the melt was dependent on the wettability on coke.

The permeability of sinter layer in the blast furnace, including the effects of Al₂O₃ and MgO in the ore, has been extensively studied. It has been reported that the addition of MgO to the sinter ore increases the softening temperature in the cohesive zone.^[9] During the liquid phase formation, addition of Al₂O₃ or MgO can decrease the melting temperature of the ore.^[10] Liquid holdup in the packed bed is increased with the increasing Al₂O₃ concentration.^[11] The sinter ore used in ironmaking contains Al₂O₃ and MgO. An adequate method for decreasing the thickness of cohesive zone can be developed after clarifying the effects of these compounds on the softening and melting properties of the sinter ore.

In this study, we investigated the softening, melting, and permeation of an oxide tablet of SiO₂-FeO-CaO, containing Al₂O₃ and/or MgO, on a coke bed. The changes in the shape of the tablet were observed during heating. The effects of Al₂O₃ and MgO addition, reduction ratio, and CaO/SiO₂ ratio of the oxide were studied. Differences between the softening and solidus temperatures, and the melting and liquidus temperatures were determined by comparing the experimental

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findings with the calculation results based on a thermodynamic database.^[12]

II. EXPERIMENTAL PROCEDURE

Softening, melting, and permeation properties of SiO₂-FeO-CaO-Al₂O₃-MgO oxide tablet were observed. As reported previously,^[8] a small particle of coke was used for the blast furnace burden, and the oxide tablet was placed on the coke bed in an aluminum crucible.

A. Measurement

Reagent grade Fe, Fe₂O₃, CaCO₃, SiO₂, Al₂O₃, and MgO were used to prepare the samples. CaO was prepared by decomposing CaCO₃ at a temperature of 1673 K (1400 °C) for 14 h. Fe and Fe₂O₃ were mixed at a stoichiometric ratio, and the mixture was fully melted and quenched to form FeO. The oxides were mixed at the compositions shown in Table I.

The samples s5-8 were prepared by melting the mixed reagents at 1723 K (1450 °C), which was followed by quenching on a water cooled copper plate in a stream of He gas and then by grounding in an agate mortar. Two grams of oxide mixture was pressed in a die to produce a tablet with a diameter of 10 mm. The sample s1 was used as the standard material.^[8] The samples s2-4, s5-8, s9-10, and s11-12 were used to investigate the effects of additives, *i.e.*, Al₂O₃ and MgO, pre-melting, FeO concentration and the basicity (CaO/SiO₂), respectively.

Coke of 1-2 mm diameter was filled into a shallow alumina crucible (φ 23 mm in diameter), and the oxide tablet was placed on the coke bed. The coke was prepared from screening small particles of coke. The concentrations of fixed carbon, volatile matter, ash, and water in the coke were 87.7, 0.3, 12.1, and 0.3 mass pct, respectively. As shown in Figure 1, a horizontal electric resistance furnace with a mullite tube (φ 35 mm ID) was employed for the observation of the tablet. The sample was placed in the center of the tube, and the changes in the appearance of the sample were observed using a camera placed close to the end of the reaction tube. The temperature of the furnace was controlled by a proportional-integral-derivative (PID) controller connected to

a thermocouple located near the sample. N₂ gas was introduced at a flow rate of 100 ml/min, and the temperature was increased to 1173 K (900 °C) at a heating rate of 10 K/min. The temperature was subsequently increased to 1723 K (1450 °C) at a rate of 5 K/min under a CO/CO₂ gas mixture flow and maintained at 1723 K (1450 °C) for 1800 seconds. The mixing ratio of CO/CO₂ was controlled at 4/6, 5/5, and 6/4 with the use of mass flow controllers. The softening, melting, and permeation temperatures were measured by observing the changes in the shape of the tablet.

B. Observation of Samples

In this study, the softening, melting, and permeation processes were observed. The method employed in the determination of process temperature is explained elsewhere.^[8] Deformation of the shape of the tablet indicated the formation of a liquid phase during the heating. The temperature of softening was defined as the temperature at which the deformation starts. The melting temperature was defined as the temperature at which the softened sample transformed into a smooth droplet. The permeation temperature was defined as the temperature at which the sample in liquid phase gradually permeated into the coke bed after softening, or the temperature at which the droplet suddenly permeated into the coke bed after remaining on the coke bed for a certain period of time. If a temperature of 1723 K (1450 °C) was reached and the droplets did not permeate into the coke bed, the permeation temperature was recorded as 1723 K (1450 °C) regardless of the time taken for the permeation.

III. EXPERIMENTAL RESULTS

A. Effects of Al₂O₃ and MgO on Softening and Melting

The CaO/SiO₂ ratio and FeO concentration of the sample s1^[8] were 1.2/1 and 30 mass pct, respectively. The softening, melting, and permeation temperatures of the CaO-FeO-SiO₂ mixture containing Al₂O₃ and/or MgO heated under a gas flow with CO/CO₂ = 5/5 are shown in Figure 2. Circle, triangle, square, and inverted

Table I. Composition of Sample (Mass Percent)

Sample	CaO	SiO ₂	Al ₂ O ₃	MgO	FeO
s1	38.2	31.8	—	—	30.0
s2	32.75	27.25	10.0	—	30.0
s3	35.45	29.55	—	5.0	30.0
s4	30.0	25.0	10.0	5.0	30.0
s5	38.2	31.8	—	—	30.0
s6	32.75	27.25	10.0	—	30.0
s7	35.45	29.55	—	5.0	30.0
s8	30.0	25.0	10.0	5.0	30.0
s9	49.1	40.9	—	—	10.0
s10	40.9	34.1	10.0	5.0	10.0
s11	16.5	38.5	10.0	5.0	30.0
s12	38.5	16.5	10.0	5.0	30.0

triangle represent the samples s1, s2, s3, and s4, respectively.

The softening temperature of samples s2 and s4, which contain Al_2O_3 , are lower than that of sample s1, indicating that Al_2O_3 decreases the softening temperature. The softening temperature of sample s3 is close to that of sample s1, and higher than that of sample s4, indicating that MgO reduces the influence of Al_2O_3 on decreasing the softening temperature. Similarly, the addition of Al_2O_3 and MgO , respectively, decreases and increases the melting temperature.

The softening, melting, and permeation temperatures of pre-melted samples s5-8 are shown in Figure 3. The compositions of the samples s5, s6, s7, and s8 are the same as those of s1, s2, s3, and s4, respectively. Comparing Figure 3 with Figure 2, it can be seen that the softening and melting temperatures overall decrease with pre-melting for all the samples, except for samples s4 and s8, for which the softening temperature increased by pre-melting. The decrease in melting temperature caused by pre-melting is greater than that in softening temperature.

It can be seen that when the structure of sample is uniform, the difference between the softening and melting temperatures is small. Permeation temperature, on the other hand, does not change with pre-melting, and the droplets of samples s5-8 maintained their shape on the coke bed up to 1723 K (1450 °C). There was no difference in time taken for permeation.

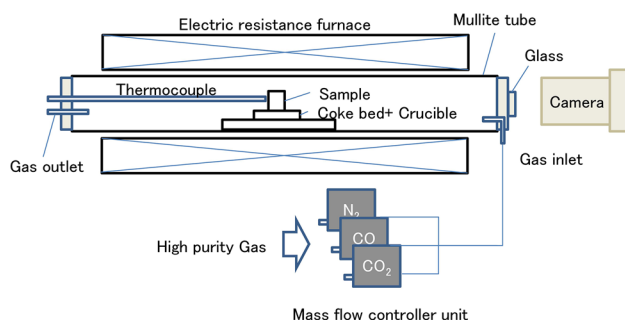


Fig. 1—Schematic diagram of the experimental apparatus.

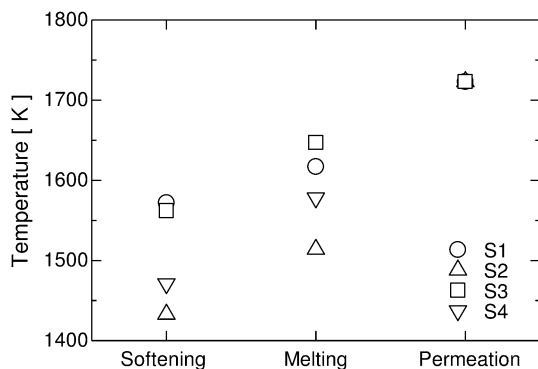


Fig. 2—Softening, melting, and permeation temperature of CaO-FeO-SiO₂-based oxide mixture tablet.

The liquid ratios of CaO-SiO₂-FeO oxide systems containing Al_2O_3 and/or MgO were simulated by a thermodynamic database^[12] with varying temperatures. For each temperature, the oxygen partial pressure (P_{O_2}) was determined under a gas flow with $\text{CO}/\text{CO}_2 = 1/1$. The results are shown in Figure 4. The lines indicate liquid ratio of s1-4 calculated. The solid and open circles denote the softening and melting temperatures measured in this study, respectively. The softening of the tablet started at a different temperature than the calculated solidus temperature. For samples s1, s3, s5, and s7, the softening temperature was higher than the calculated solidus temperature. The softening of pre-melted samples s5 and s7 was observed at a temperature lower and closer to the solidus temperature than the softening temperature of the mixing samples s1 and s3.

For the samples s2, s4, s6, and s8, the softening temperature was lower than the calculated solidus temperatures. According to the phase diagram of the FeO- Al_2O_3 -SiO₂ system,^[13] the lowest eutectic temperature of the system is 1356 K (1083 °C). By contacting SiO₂- Al_2O_3 -FeO in the mixed sample, the liquid phase was formed, and the tablet was softened at a temperature lower than the solidus temperature. Similarly, the

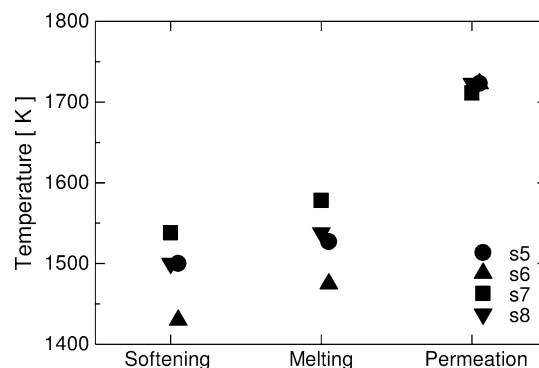


Fig. 3—Softening, melting, and permeation temperature of CaO-FeO-SiO₂-based oxide tablet.

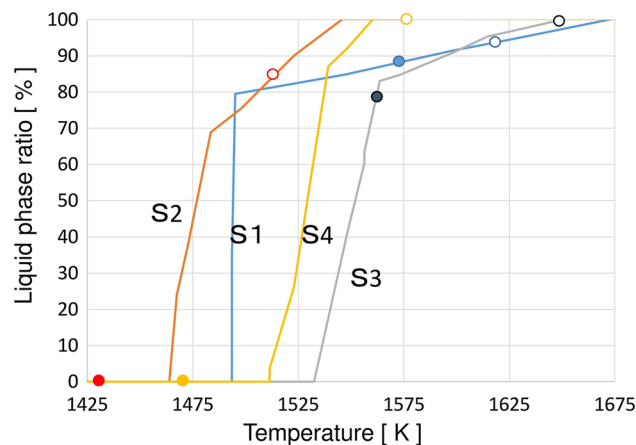


Fig. 4—Comparison of measured and calculated liquid ratio of CaO-FeO-SiO₂-based oxide.

liquid phase in non-equilibrium state might be formed at a temperature lower than the solidus temperature by contacting CaO-FeO-SiO₂-Al₂O₃ and CaO-FeO-SiO₂-Al₂O₃-MgO.

The results showed that samples containing Al₂O₃ formed an initial liquid in non-equilibrium condition. Compared to mixed samples, the softening temperatures of the pre-melted samples were close to their respective solidus temperature, regardless of their composition. For uniform samples, the softening temperatures were closer to the solidus temperatures.

When the liquid phase ratio of the mixed sample at equilibrium was 80 pct or more, melting was observed. The melting temperature was lower for the pre-melted samples, when the liquid phase ratio at equilibrium was between 40 and 90 pct. During melting, the oxide tablet became a droplet and was in a solid-liquid mixed state.

The droplets remained on the coke bed for up to 10 minutes. Then, the liquid permeated into the bed. There was no relation between the temperatures of melting and permeation, and pre-melting did not show any effect on permeation.

B. Effects of FeO Concentration on Softening and Melting

FeO concentration in the sinter ore decreases with the progress of reduction in the blast furnace. In this study, the influence of FeO concentration on the softening and melting behaviors was determined changing the initial concentration of FeO in the sample with fixed CaO/SiO₂ = 1.2/1. The experimental results are shown in Figure 5. Solid and open circles denote CaO-FeO-SiO₂ and CaO-FeO-SiO₂-Al₂O₃-MgO samples, respectively.

The softening temperatures of samples s1 and s4 increased slightly with a decrease in FeO concentration from 30 to 10 pct. Comparing the samples s1 and s9, it was shown that the melting temperature was increased by 40 K (40 °C) with a decrease in FeO concentration. The melting temperatures of samples s4 and s10 were close. For all the samples, permeation was observed after the formation of droplet on coke bed at 1723 K (1450 °C). FeO concentration did not affect the time required for permeation.

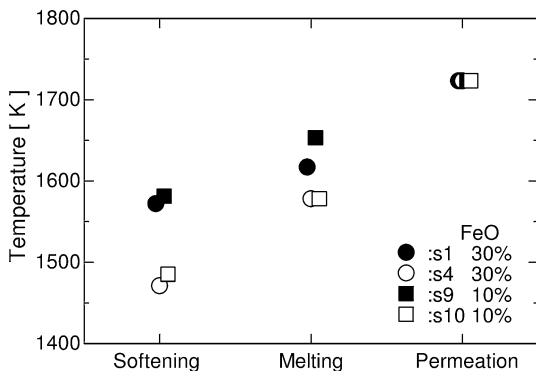


Fig. 5—Influence of concentration on softening and melting temperature of CaO-FeO-SiO₂-based oxide mixture tablet.

The experimentally measured softening and melting temperatures and the calculated liquid phase ratio are shown in Figure 6. Solid and open circles represent the softening and melting temperatures, respectively. The lines indicate calculated liquid ratios of s1, s4, s9, and s10. The data points fell close to the calculated liquid ratio line. Since a liquid olivine phase was formed as an initial liquid from CaO-FeO-SiO₂ samples s1 and s9, the temperatures of initial liquid phase formation and softening were approximately 1573 K (1300 °C). The solid phase equilibrated with the liquid phase was 2CaO-SiO₂. Since the solid ratio with 2CaO-SiO₂ composition increased with the decreasing FeO concentration, liquid formation was delayed.

The calculated solidus temperatures of samples s4 and s10 were 1513 K (1240 °C), which is the eutectic of melilite, FeO, and 2CaO-SiO₂, and 1533 K (1260 °C), which is eutectic of olivine, melilite, and 3CaO-MgO-2-SiO₂, respectively. Solidus temperatures of samples s4 and s10, which contain both Al₂O₃ and MgO, were higher than those of samples s1 and s9. The softening temperatures of samples s1 and s9 were higher than those of s4 and s10, and also higher than the calculated solidus temperatures of samples s1 and s9, respectively. The softening temperatures of samples s4 and s10, which contain only Al₂O₃, were lower than their respective solidus temperatures.

The liquid phase ratios of samples s4 and s10 were significantly different from each other: the calculated liquid ratios of s4 and s10 were 100 and 46 pct at 1573 K (1300 °C), respectively. However, the measured melting temperatures of s4 and s10 were very close to each other. In a previous study,^[8] it was reported that FeO concentration has no effect on the melting temperature but causes a change in liquid ratio. The shapes of s4 and s10 at 1523 K (1250 °C) are shown in Figure 7. The softening and melting temperatures of both samples were approximately 1473 K and 1578 K (1200 °C and 1305 °C), respectively. 1523 K (1250 °C) is higher than the solidus temperature of s4 and lower than the solidus temperature of s10. Deformation in sample s4 is clearly shown in Figure 7. The sample 10 was shrunk down in size and had slight deformation at the surface, but maintained its shape. Even if there was a deformation in the surface caused by the liquid phase formation below the solidus temperature, only non-equilibrium liquid phase was formed, and the liquid phase fraction was not increased.

Figure 8 shows the softening, melting, and permeation temperatures of the samples s1-s4 (CaO/SiO₂ = 1.2/1 and 30 wt pct FeO) at atmospheric pressure with CO/CO₂ ratios of 4/6, 5/5, and 6/4. Influence of CO/CO₂ ratio on the softening temperature was small, and temperature difference between the samples was not evident.

The melting temperatures increased with the increasing CO ratio. The melting temperatures of samples s1 and s3, which did not contain Al₂O₃, increased by 100 K (100 °C) with an increase in the CO ratio from 0.4 to 0.6. The softening behavior in the measurements was changed by changing the CO/CO₂ ratio, more specifically by changing the P_{O₂}. The influence of P_{O₂} on

melting was obtained using a thermodynamic database. Influence of P_{O_2} on the eutectic temperature or liquid phase ratio was not clear. The permeation temperature was 1723 K (1450 °C), and the permeation was observed approximately 10 minutes after the formation of droplet. The effect of P_{O_2} on the permeation behavior was not determined.

The P_{O_2} changed depending on the CO/CO₂ ratio in the atmosphere. In this study, FeO exhibited a stable phase at temperatures over 1173 K (900 °C). Based on experimental results and calculated phase diagram of the CaO-FeO-SiO₂,^[13] it was concluded that the effect of FeO concentration in the samples on the solidus and liquidus temperatures was small. However, the rate of liquid phase formation decreased with the decreasing FeO concentration. FeO in samples would be reduced on contacting the coke bed. Thus, the samples were exposed to both reducing and oxidizing atmospheres.

The oxide phase became a mixture of solid and liquid at temperatures above the softening temperature. Since mass transfer is relatively easy through the liquid phase, reduction with coke would be accelerated. Reduction becomes easier with the decreasing P_{O_2} , while it progresses faster at high CO/CO₂ ratios. FeO reduction after reaching the solid-liquid mixed state decreased the rate of liquid phase formation, decreasing the melting

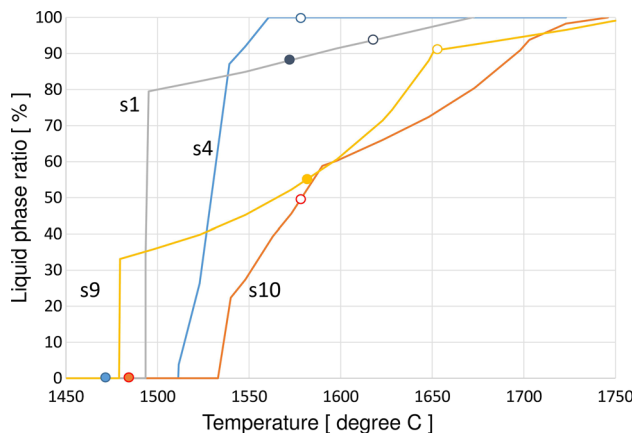


Fig. 6—Comparison of influence of concentration of FeO on measured and calculated liquid ratio of CaO-FeO-SiO₂-based oxide.

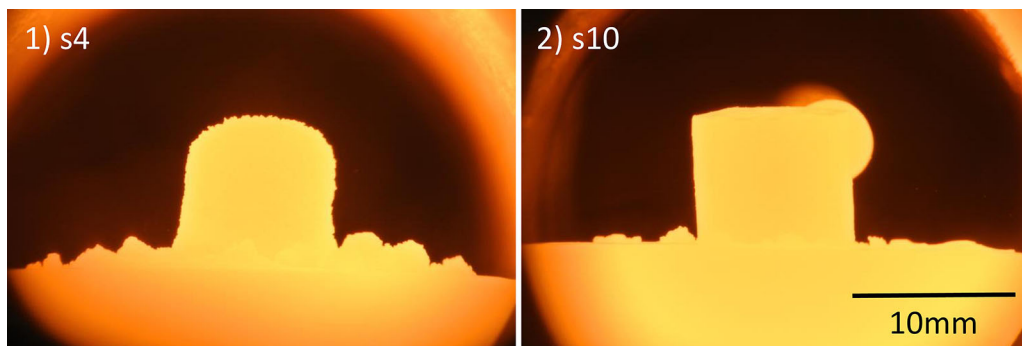


Fig. 7—Influence of concentration of FeO on appearance of CaO-FeO-SiO₂-Al₂O₃-MgO tablet at 1523 K (1250 °C).

temperature. It cannot be confirmed from the experiments; however, there is a possibility of increasing the dissolution rate with the increasing P_{O_2} in atmosphere.

C. Effects of CaO/SiO₂ Ratio on Softening and Melting

The softening and melting temperatures of the CaO-FeO-SiO₂-Al₂O₃-MgO mixture heated under a gas flow with CO/CO₂ = 5/5 are shown in Figure 9. The ratios of CaO/SiO₂ were 0.43, 1.2, and 2.3, and the FeO concentration was 30 mass pct. The influence of CaO/SiO₂ ratio on the softening temperature was small: the temperature changed from 1433 K to 1473 K (1160 °C to 1200 °C). Softening temperature of the CaO-FeO-SiO₂ ternary system changed with basicity.^[8] However, the temperature of the system containing Al₂O₃ and MgO changed little with basicity. The melting temperature increased with the increasing basicity. The permeation into the coke bed was observed after keeping the system at 1723 K (1450 °C) for approximately 10 minutes. There was no relation between basicity and permeation time at 1723 K (1450 °C).

The measured softening and melting temperatures of samples s4, s11, and s12 and the calculated liquid phase ratios are shown in Figure 10. Solid and open circles represent the softening and melting temperatures, respectively. The lines indicate calculated liquid ratio.

The softening temperatures of samples s11 and s12 were higher than their respective solidus temperatures.

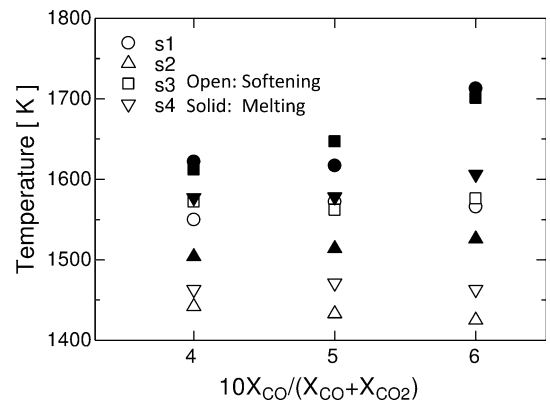


Fig. 8—Influence of atmosphere on softening and melting temperature of CaO-FeO-SiO₂-based oxide tablet.

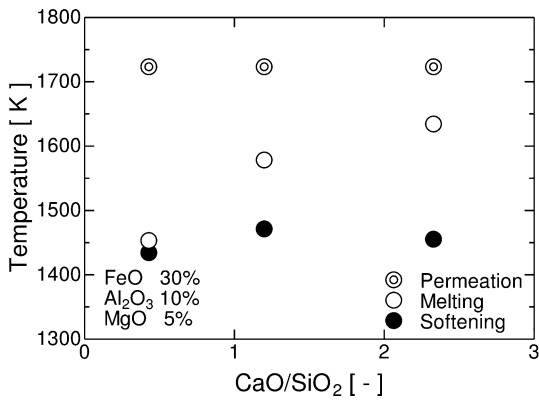


Fig. 9—Influence of concentration of CaO/SiO₂ ratio on softening and melting temperature of CaO-FeO-SiO₂-Al₂O₃-MgO oxide tablet.

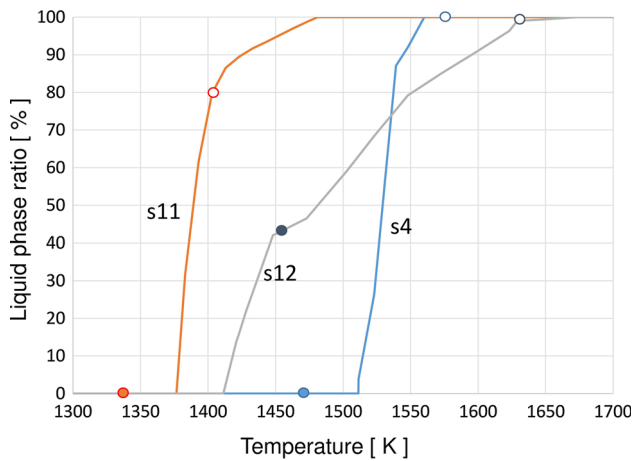


Fig. 10—Comparison of influence of CaO/SiO₂ ratio on measured and calculated liquid ratio of CaO-FeO-SiO₂-MgO-Al₂O₃ oxide.

The softening temperature of sample s4 was lower than its solidus temperature. For all the samples, the melting and liquidus temperatures were close to each other.

Since the weight ratio of CaO/FeO/SiO₂ of sample s11 was close to the composition of eutectic CaCO₃-FeSiO₂-Fe₂SiO₄, even with Al₂O₃ and MgO, the difference between softening and melting temperatures was small.

Since the weight ratio of CaO/FeO/SiO₂ of sample s12 corresponded to CaO-2CaO·SiO₂-FeO composition, CaO-FeO-rich initial liquid was formed at 1403 K (1130 °C). The initial liquid phase of sample s12 would be in equilibrium with CaO and 2CaO·SiO₂. Since the melting temperatures of these solid phases are high, s12 became liquid at or above 1623 K (1350 °C).

IV. CONTROL OF IRON ORE COMPOSITION TO DECREASE COHESIVE LAYER THICKNESS

In order to decrease the thickness of the cohesive zone in the blast furnace, it is needed to increase the softening and decrease the dripping temperatures of the iron ore. Softening caused by liquid formation leads to deformation and blockage of the packed bed. The

liquid phase ratio increases with melting. As the ratio increases, the liquid drops into the coke bed. Experimental results have shown that there is a difference between softening and solidus temperatures, and the permeation or fall of the droplet into the coke bed is determined by wettability and is not affected by the melting temperature.

Addition of Al₂O₃ to the CaO-FeO-SiO₂ causes the formation of an initial liquid with non-equilibrium composition by heating and decreases the softening temperature. Addition of MgO slightly increases the softening temperature of CaO-FeO-SiO₂. When components are non-uniform, there is a difference between the temperature of softening initiation and the solidus temperature at equilibrium. Since the influence of the initial liquid with non-equilibrium composition on the rate of liquid formation is small, it is not clear whether there is a correlation between softening and the clogging of the packed bed. For a more detailed analysis of softening, the softening step should be considered separately from the deformation of iron ore by formation of the initial liquid, and blockage of void of the ore layer by liquid.

Addition of Al₂O₃ and MgO, respectively, decreases and increases the melting temperature of CaO-FeO-SiO₂. It also increases the difference between the softening and melting temperatures. It has little effect on the wettability of the oxide with the coke, since there is no relationship between the melting and permeation temperatures.

Addition of Al₂O₃ and MgO, respectively, increases and decreases the temperature difference between softening and dripping of the iron ore, in the temperature range of solid-liquid coexistence in the cohesive zone. When the P_{O₂} is low, the rate of liquid formation is suppressed, and the temperature difference between softening and melting is increased. Influence of basicity on the softening temperature of CaO-FeO-SiO₂-Al₂O₃-MgO is less than that on the softening temperature of CaO-FeO-SiO₂. On the other hand, since the melting temperature of CaO-FeO-SiO₂-Al₂O₃-MgO is close to the melting temperature of CaO-FeO-SiO₂, higher basicity has the advantage of suppressing the increasing liquid phase formation rate.

When wettability of slag on coke is poor, decreased Al₂O₃ concentration, MgO addition, and increased basicity can decrease the temperature difference between softening and dripping. In addition, decreasing concentration or enhanced reduction of FeO is effective on reducing the liquid formation rate.^[8] When wettability of slag on coke is good, increased concentrations of Al₂O₃ and FeO, and decreased basicity can decrease the temperature difference between softening and permeation of ore into the coke bed.

Dynamic wettability between coke and slag is dependent on the reaction conditions at the interface, the properties of coke, and composition of slag, which cannot be predicted by the available data and conventional methods on wettability. Further research on dynamic wettability is needed to determine the optimum properties of the iron ore.

V. CONCLUSIONS

The softening, melting, and permeation properties of a tablet of CaO-FeO-SiO₂ system containing Al₂O₃ and/or MgO were investigated. The changes in the shape of the tablet were observed by heating under CO/CO₂ gas flow. The following conclusions were drawn:

- From CaO-FeO-SiO₂ oxide containing Al₂O₃, an initial melt at non-equilibrium composition is produced by heating at a temperature lower than the eutectic temperature.
- When Al₂O₃ and MgO are both added to the oxide system, the variation in the softening temperature due to change in basicity is mitigated.
- The initial liquid with non-equilibrium composition has a small effect on the liquid ratio.
- The influence of FeO concentration on softening and melting temperatures is small. However, decrease in FeO concentration in the oxide reduces the liquid phase formation rate.
- The temperature difference between softening and melting is increased with the decreasing P_{O₂}. In addition, P_{O₂} might affect the dissolution rate of solid oxide phase.
- The influence of basicity on the softening temperature of CaO-FeO-SiO₂-Al₂O₃-MgO is smaller than that on the softening temperature of CaO-FeO-SiO₂. Melting temperature of the system increases with the increasing basicity.

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