Influence of MgO and Al₂O₃ Contents on Viscosity of Blast Furnace Type Slags Containing FeO

Joo Ro KIM, Young Seok LEE, Dong Joon MIN, Sung Mo JUNG¹⁾ and Sang Ho YI²⁾

Department of Metallurgical Engineering, Yonsei University, Seoul 120-749, Korea. E-mail: chemical@yonsei.ac.kr

1) Graduate School of Iron and Steel Technology, Pohang University of Science and Technology, Pohang 790-784, Korea.

2) Technical Research Laboratories, POSCO, Pohang 790-785, Korea.

(Received on October 2, 2003; accepted in final form on April 7, 2004)

The viscosities of CaO–SiO₂–Al₂O₃–MgO–FeO slag were measured under conditions of C/S=1.35–1.45, 10–18 mass% Al₂O₃, 3.5–10 mass% MgO and 5 mass% FeO. The bosh slag with 10 mass% Al₂O₃ content had the lowest melting temperature and the widest solid-liquid coexisting region at about 5 mass% MgO, while in case of 14 mass% Al₂O₃, an increase in a MgO content from about 3.5 to 10 mass% raised the melting point of bosh slag. The viscosity of bosh slag also exhibited a minimum value at about 7 mass% MgO at temperatures above 1723 K. However, it was not significantly changed with varying MgO content. On the hand, with increasing Al₂O₃ content, the viscosity of bosh slag increased at a fixed C/S and MgO content. Based on the melting temperature and the behavior of viscosity at a fixed temperature, it could be proposed that the MgO and Al₂O₃ contents in bosh slag should be maintained around 5 and 10 mass%, respectively, for high pulverized-coal ratio (PCR) and low slag volume operations in blast furnace.

KEY WORDS: viscosity; bosh slag; blast furnace; slag volume reduction; slag structure; FT-IR.

1. Introduction

In view of environmental and energy saving issues, the great efforts to reduce slag volume and to increase the pulverized coal ratio (PCR) in blast furnace (BF) have recently been made at POSCO.¹⁾ In order to stabilize the high PCR/low slag volume operation, the good gas/liquid permeability should be confirmed although slag volume is reduced and PCR is significantly increased. However, the mechanisms of slag formation in BF during high PCR/low slag volume operations are not the same as those in conventional operations. Therefore, slag chemistry in bosh zones, where injected coals are mixed with dropping slags and metals, should be carefully controlled during high PCR/low slag volume operations. Occasionally, reduction of gas/liquid permeability in the bosh zone during high PCR/low slag volume operations has been reported.^{2–4)} For high PCR/low slag volume operation, the gas/liquid permeability should be estimated with considering the slag chemistry, and it can be indirectly predicted by investigating the viscosity of bosh slags.

In previous work,⁵⁾ the viscosities of typical bosh slags (CaO–SiO₂–Al₂O₃–MgO–FeO system) were measured for slag composition at C/S=1.15–1.6 and 0–20 mass% FeO in terms of considering the reduction of iron ore. However, the influence of MgO and Al₂O₃ contents on the viscosity of bosh slags was not investigated. And few viscosities of CaO–SiO₂–Al₂O₃–MgO–FeO slags at bosh level have been reported. Therefore, in the present study, the effects of MgO and Al₂O₃ contents on the viscosities of the CaO–SiO₂–Al₂O₃–MgO–FeO slags were investigated under

bosh slag conditions in order to optimize slag composition in bosh region. Also, the structural role of each component in the polymerization or depolymerization reactions of silicates was clarified by employing the FT-IR measurement technique in order to investigate the relationship between viscosity and slag structure.

2. Experimental

2.1. Measurement of Slag Viscosity

The rotating cylindrical method was employed for viscosity measurement using the experimental apparatus shown in the previous paper.⁵⁾ A Brookfield digital viscometer (model RVDV-II+) was used in the present study. The viscometer head was connected to the working spindle by two Pt-10 mass%Rh hooks. The crucible, suspending wire and spindle used in the experiments were made of Pt-10 mass%Rh in order to prevent the slag contamination. The dimension of each component for measurement is listed in **Table 1**. The viscometer was calibrated by using Brookfield standard oils with 0.485, 0.96, 4.85, 9.75, and 50.4 dPa·s at room temperature.

All slag samples were prepared using reagent-grade chemicals. In order to measure slag viscosity, 120 g of mixed powder comprising each component listed in **Table 2** was held in a Pt-10 mass%Rh crucible. The crucible containing slag sample was placed into the reaction chamber in an Ar (0.4 L/min) atmosphere at 1773 K. The slag sample was molten for more than 3 h. Then, the rotating spindle was immersed into the slag and was held in the middle of the melt. Three different rotating speeds (30, 60 and

100 rpm) were employed in 1 773 K to ensure that viscosity values were independent of rotation speed. All measurements were performed with cooling and holding tempera-

Table 1. Crucible and spindle dimensions used in the measurement of slag viscosity.

Crucible	Material	Pt-10 pct Rh alloy
	Inner diameter (mm)	40
	Inner depth (mm)	60
Spindle	Material	Pt-10 pct Rh alloy
	Bob diameter (mm)	10
	Bob length (mm)	25
	Shaft diameter (mm)	3
	Shaft length (mm)	65
	Degree of taper	45
Suspension wire (lower part)	Material	Pt-10 pct Rh alloy
	Wire diameter (mm)	2
	Wire length (mm)	150
Suspension wire (upper part)	Material	Molybdenum
	Wire diameter (mm)	2
	Wire length (mm)	340

ture. The holding time for reaching equilibrium state was more than 30 min at each experimental temperature. A metal flange was utilized to fill the gap between the viscometer and the mullite reaction tube, and an Ar gas (0.4 L/min) was flown into the reaction chamber. After viscosity measurements were completed, the slag samples were reheated up to 1 773 K, and held for more than 3 h. Then, the slag sample was quenched to analyze slag composition and to measure the infrared spectra of the slag. According to the composition analysis of post-measurement slag using X-ray Fluorescence (Bruker-SRS3400), there was no significant change in any component of slags (less than 1 %).

In order to confirm the reproducibility of viscosity values, some experiments for the same composition were carried out three times. The results show that differences between measurements were lower than 0.2 dPa·s (less than 0.5 %).

2.2. Infrared Spectra Measurement

The structure of the investigated slags was analyzed by FT-IR spectroscopy (JASCO, FT/IR-300 E). FT-IR trans-

Table 2. Experimental results.

Sample Number	C/S	Al ₂ O ₃	MgO	FeO	Viscosity (dPa.s)	
					1 773 K	1 723 K
1	1.45	10	3.5	5		6.8
2	1.45	10	5	5		8.2
3	1.45	10	7	5		9.2
4	1.45	10	10	5		6.0
5	1.45	12	3.5	5	2.6	6.0
6	1.45	12	5	5	2.1	8.1
7	1.45	12	7	5	2.0	8.7
8	1.45	12	10	5	2.0	6.2
9	1.45	14	3.5	5	2.6	6.1
10	1.45	14	5	5	2.3	4.5
11	1.45	14	7	5	2.0	4.4
12	1.45	14	10	5	2.1	2.7
13	1.45	18	3.5	5	2.9	2.8
14	1.45	18	5	5	2.7	15.4
15	1.45	18	7	5	2.1	15.2
16	1.35	10	5	5	2.0	3.4
17	1.35	10	7	5	1.9	3.0
18	1.35	10	10	5	1.8	3.0
19	1.35	12	5	5	2.8	3.8
20	1.35	12	7	5	2.2	3.0
21	1.35	12	10	5	1.7	2.3
22	1.35	14	5	5	2.7	3.7
23	1.35	14	7	5	2.5	3.6
24	1.35	14	10	5	2.7	3.9
25	1.18	13	5	0	2.8	4.2
26	1.18	13	7	0	2.6	4.0
27	1.18	13	10	0	3.3	4.8
28	1.18	13	12	0	2.9	4.3

mitting spectra were recorded in the $4000\text{--}400\text{ cm}^{-1}$ range using a spectrometer equipped with a KBr (deuterated triglycine sulfate with potassium bromide windows) detector. A spectral resolution of 4 cm^{-1} was chosen. Each 2.0 mg sample was mixed with 200 mg of KBr in an agate mortar, and then pressed into 13 mm diameter pellets. The spectrum for each sample represents an average of 20 scans, which were each normalized to the spectrum of the blank KBr pellets. The FT-IR spectra were analyzed by computer software.

3. Results and Discussion

In the present study, the effects of MgO and Al_2O_3 contents on the viscosities of the $\text{CaO-SiO}_2\text{-Al}_2\text{O}_3\text{-MgO-FeO}$ slags were investigated under the conditions of bosh and tapped slag. The experimental results are listed in Table 2.

3.1. Temperature-Viscosity Curve of Bosh Slag

If a slag is melted at high temperature and is then cooled slowly, the slag viscosity linearly increases up to a specific temperature, and then abruptly increases below that temperature. Thermodynamically, the temperature should be the liquidus temperature of the slag, T_{LQ} , that is, the maximum temperature at which liquid and solid phase can coexist in equilibrium. If the slag is cooled further, more solid phase will precipitate and increase the slag viscosity. Under equilibrium conditions, the composition of phase separating as temperature decreases from liquidus temperature to solidus temperature, T_{SL} , will change continuously, but will be defined in composition and amount by the temperature and overall composition of the slag system. The amount of solid separating at each temperature under these equilibrium conditions—and hence, in general, the viscosity—will be the maximum and the course of the viscosity/temperature curve will therefore represent the worst behaviour of viscous flow in blast furnaces. Although this curve is not likely to represent the actual behaviour of most slags under thermal conditions in blast furnaces or even in viscometers, it gives much information, *i.e.* slag formation and operation stability in the blast furnace. Therefore, it is necessary to investigate the viscous behavior at high temperature as well as in the vicinity of the melting temperature of slag.

The effects of MgO content on the viscosities of synthetic bosh slags with $\text{C/S}=1.45$ are shown as a function of temperature for $\text{Al}_2\text{O}_3=10$ and 14 mass% in Figs. 1(a) and 1(b), respectively. In Fig. 1(a), it is observed that the slag with 5 mass% MgO has the lowest T_{SL} and the widest solid-liquid coexisting region. On the other hand, the solid-liquid coexisting region of slag with 14 mass% Al_2O_3 is narrower than that of slag with 10 mass% Al_2O_3 , and T_{SL} decreases with increasing MgO content from 3.5 to 10 mass% in Fig. 1(b). This tendency is also observed in the temperature-viscosity curve of slags with $\text{C/S}=1.35$. (Figs. 2(a) and 2(b)). In Fig. 2(a), it is observed that the width of the solid-liquid coexisting region for each Al_2O_3 content in slags with $\text{C/S}=1.35$ is similar to each other although the MgO content varies from 5 to 10 mass%. By increasing MgO content from 5 to 10 mass%, the T_{SL} of slag with 10 mass% Al_2O_3 increases in Fig. 2(a), and *vice versa* at 14 mass% Al_2O_3 in Fig. 2(b). This tendency of temperature dependence of vis-

Fig. 1. Viscosities of $\text{CaO-SiO}_2\text{-Al}_2\text{O}_3\text{-MgO-5mass\%FeO}$ systems at $\text{C/S}=1.45$ by varying MgO content as a function of temperature: (a) at 10 mass% Al_2O_3 (b) at 14 mass% Al_2O_3 .

cosity could be because the primary solid phase in the present slag composition (except for slag with $\text{C/S}=1.35$ and 14 mass% Al_2O_3) changes from dicalcium silicate ($2\text{CaO}\cdot\text{SiO}_2$) to merwinite ($3\text{CaO}\cdot\text{MgO}\cdot2\text{SiO}_2$) with increasing MgO content and from melilite ($2\text{CaO}\cdot\text{MgO}\cdot\text{SiO}_2\text{-}2\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot\text{SiO}_2$) to merwinite in $\text{C/S}=1.35$ and 14 mass% Al_2O_3 . (The region of primary solid phase was inferred from phase diagrams of the $\text{CaO-SiO}_2\text{-Al}_2\text{O}_3\text{-MgO}$ and $\text{CaO-SiO}_2\text{-MgO-FeO}$ system because the phase diagram of $\text{CaO-SiO}_2\text{-Al}_2\text{O}_3\text{-MgO-FeO}$ system has not been reported, yet.)⁶⁾

Figure 3 shows the variation of the liquidus temperature measured by a differential thermal analyzer (SETARAM, CS92-1618) as a function of MgO content for each slag composition. In Fig. 3, the T_{LQ} of slag with $\text{C/S}=1.45$ and 10 mass% Al_2O_3 exhibits a minimum value at about 5 mass% MgO by increasing MgO content, while that of slag with $\text{C/S}=1.45$ and 14 mass% Al_2O_3 decreases from about 1740 to 1650 K by increasing MgO content from 3.5

Fig. 2. Viscosities of CaO-SiO₂-Al₂O₃-MgO-5mass%FeO systems at C/S=1.35 by varying the MgO content as a function of temperature: (a) at 10 mass% Al₂O₃ (b) at 14 mass% Al₂O₃.

to 10 mass%. In the case of C/S=1.35, the T_{LQ} of slag with 10 mass% Al₂O₃ slightly increases from about 1665 to 1680 K by increasing MgO content from 5 to 10 mass%, while that of slag with 14 mass% Al₂O₃ decreases from about 1725 to 1660 K with increasing MgO content. This behavior of melting temperature of slag plays an important role in BF operation as well as viscosity of slag because the temperature of bosh region, at which slag formation proceeds, is less than 1773 K.^{1,7)} Also, Al₂O₃ content in bosh slag, which is not fully assimilate with ash as source SiO₂ and Al₂O₃ from coke and/or coal, is about 10 mass%.^{1,5)} Therefore, it can be suggested in view of melting temperature of slag that MgO content in bosh slag is preferable to be maintained at about 5 mass% for the stable BF operation.

3.2. Effect of MgO and Al₂O₃ Content on Viscosity of Bosh Slag

Figure 4 shows the effect of MgO and Al₂O₃ content on

Fig. 3. Liquidus temperatures of CaO-SiO₂-Al₂O₃-MgO-5mass%MgO slags as a function of MgO content.

Fig. 4. Effect of MgO content on viscosity of CaO-SiO₂-Al₂O₃-MgO-5mass%FeO slag with C/S=1.45 at 1773 K.

the viscosity of CaO-SiO₂-Al₂O₃-MgO-5mass%FeO slag (C/S=1.45, Al₂O₃=12, 14 and 18 mass%) at 1773 K. Slag viscosity increases with increasing Al₂O₃ content at a fixed MgO content. On the other hand, slag viscosities at a fixed Al₂O₃ content exhibits a minimum value at about 7 mass% MgO. In a previous study,⁵⁾ it was suggested that the MgO would behave as a network modifier at MgO≤7 mass% in the CaO-SiO₂-10mass%Al₂O₃-MgO-5mass%FeO slag (C/S=1.45), while it would contribute to the increase in the melting temperature of slag above 7 mass% MgO. However, as shown in Fig. 3, the liquidus temperature of slag with C/S=1.45 and 14 mass% Al₂O₃ decreases with increasing MgO content. Therefore, the viscous behaviour of slag with Al₂O₃>10 mass% would be different from that of slag at 10 mass% Al₂O₃.

Fig. 5. Infrared spectra of CaO-SiO₂-12mass%Al₂O₃-MgO-5mass% FeO slags (with C/S=1.45) as a function of wave number (cm⁻¹) at different MgO contents.

In general, it is well known that the increase in basicity decreases slag viscosities because the silicate structure changes from a three-dimensional network to discrete anionic groups containing simple chains and/or rings as basic oxides (such as CaO and MgO) increase.^{8,9} For example, the depolymerization reaction of [Si₃O₉]⁶⁻-ring (NBO/Si=2, NBO/Si: Non-Bridging Oxygen per Silicon) units is described by the following equations:¹⁰

where M²⁺ denotes the metal ions such as Ca²⁺ and Mg²⁺. With increasing amount of basic oxides, the spontaneous depolymerization reaction such as in Eqs. (2) and (3) results in the formation of [Si₂O₇]⁶⁻-dimer (NBO/Si=3) and [SiO₄]⁴⁻-tetrahedra (NBO/Si=4). It indicates that the size of silicate flow unit becomes smaller with increasing the contents of basic oxides, resulting in decreasing the slag viscosity.

Figure 5 shows the IR spectra of CaO-SiO₂-12mass%Al₂O₃-MgO-5mass%FeO slag (C/S=1.45) as a function of wave number (cm⁻¹) at different MgO contents. Four major peaks for Si-O bonds are found at about 1030, 980, 940, and 850 cm⁻¹ which are assigned to NBO/Si=1, 2, 3, and 4, respectively.¹⁰⁻¹³ Two kinds of band groups are observed at about 720-630 cm⁻¹ and 500 cm⁻¹ which are assigned to the asymmetric stretching vibration of [AlO₄]-tetrahedra and the Si-O-Al bending, respectively.¹³⁻¹⁵ In Fig. 5, by increasing the MgO content, the IR bands for [SiO₄]-tetrahedra with NBO/Si=3+4 is most remarkable at 7 mass% MgO and those related to aluminate anions do not change significantly. This variation of IR spectra indicates that MgO interacts with the silicate anion group more rela-

Fig. 6. Infrared spectra of CaO-SiO₂-Al₂O₃-5mass%MgO-5mass% FeO slags (with C/S=1.45) as a function of wave number (cm⁻¹) at different Al₂O₃ contents.

tively than other components (except for CaO) in present slag compositions and the size of silicate anion group at 7 mass% MgO is the smallest. Therefore, it can be known that the viscosity of slag with Al₂O₃>10 mass% exhibits the minimum value at about 7 mass% MgO because the size of silicate anion groups is the smallest at 7 mass% MgO.

Figure 6 shows the IR-transmittance of CaO-SiO₂-Al₂O₃-5mass%MgO-5mass%FeO slag (C/S=1.45) as a function of wave number (cm⁻¹) at different Al₂O₃ contents. Three kinds of band groups related with aluminate anions has been reported at about 720-630 cm⁻¹, 570-520 cm⁻¹, and 500 cm⁻¹, which are assigned to the asymmetric stretching vibration of [AlO₄]-tetrahedra (which behaves as a network former), [AlO₆]-octahedra (which behaves as a network modifier) and Si-O-Al bending, respectively.¹⁴⁻¹⁶ In Fig. 6, the absorption of [SiO₄]⁴⁻-tetrahedra does not fluctuate as much as the change of intensity of Al-O band groups. With increasing Al₂O₃ content, the absorption of [AlO₄]-tetrahedra increases but the change of peak for [AlO₆]-octahedra is negligible. The relative intensity of IR-band for Si-O-Al bending also increases. It means that Al₂O₃ behaves as a network former in present slag system although Al₂O₃ has been known as amphoteric oxide. Therefore, it can be understood that an increase in Al₂O₃ content can increase the viscosity of bosh slag as shown in Fig. 4.

Figure 7 shows the viscosities of slag with C/S=1.35 and 5 mass%FeO as a function of MgO and Al₂O₃ content at 1773 K. Slag viscosity increases by increasing Al₂O₃ content at a fixed MgO content. On the other hand, the effect of MgO content on the slag viscosity at a fixed Al₂O₃ content is not significantly. However, within levels of acceptable experimental uncertainty, the viscosity of slag slightly decreases with increasing MgO content at Al₂O₃=10 and 12 mass%, while it exhibits a minimum value at 7 mass% MgO and 14 mass% Al₂O₃. In order to investigate in detail, the IR-transmittance was measured for these slag samples. Figure 8 shows the IR-spectra of the CaO-SiO₂-

Fig. 7. Effect of MgO content on viscosity of CaO-SiO₂-Al₂O₃-MgO-5mass%FeO slag with C/S=1.35 at 1773 K.

Fig. 8. Infrared spectra of CaO-SiO₂-Al₂O₃-MgO-5mass%FeO slags (with C/S=1.35) as a function of wave number (cm⁻¹) at different Al₂O₃ and MgO contents.

Al₂O₃-MgO-5mass% FeO slags with C/S=1.35 and Al₂O₃=10 and 14mass% as a function of wave number (cm⁻¹) at different MgO content. With increasing Al₂O₃ content from 10 to 14 mass%, the relative intensity of the peaks for [SiO₄]-tetrahedra with NBO/Si=3+4 decreases and that of the IR band for [AlO₄]-tetrahedra (about 720–630 cm⁻¹) increases. It means that the silicate and aluminate structures are more complex and the activation energy for viscous flow increases with increasing Al₂O₃ content. In the contrary, it is observed that the relative intensity of the IR bands for [SiO₄]-tetrahedra with NBO/Si=3+4 increases and that for [AlO₄]-tetrahedra decreases with increasing MgO content at 10 mass% Al₂O₃. Especially, the

change of IR band for [AlO₆]-octahedra is noticeable at 10 mass% MgO and 10 mass% Al₂O₃. It indicates that a portion of Al₂O₃ added into slag gradually changes from network former to network breaker with increasing MgO content. It would be because an increase in MgO content relatively reduces the CaO content which is more basic oxide than MgO, resulting in decreasing the basicity in slag system. However, it can be known from the relative intensities of IR bands for [SiO₄]-tetrahedra and [AlO₄]-tetrahedra that the silicate and aluminate networks become simpler by increasing MgO content at 10 mass% Al₂O₃. At 14 mass% Al₂O₃, the intensities of peaks assigned to NBO/Si=3+4 is slightly prominent at 7 mass% MgO with increasing MgO content. Also, the intensity of IR bands for [AlO₄]-tetrahedra and Si-O-Al bending (about 500 cm⁻¹) decrease with increasing MgO content at 14 mass% Al₂O₃. These variations of IR-spectra of slag with 14 mass% Al₂O₃ indicate that the size of silicate anion group is the smallest at 7 mass% MgO and the aluminate network becomes simpler by increasing MgO content. This tendency of IR spectra at different MgO and Al₂O₃ contents is in good agreement with the viscous behavior in Fig. 7.

4. Application to Reducing Slag Volume in Blast Furnaces

One proposal is to reduce the total amount of tapped slag by decreasing the feeding of serpentine (Mg₃Si₂O₅(OH)₄) as a source of MgO which is a magnetite stabilizing element.¹⁸⁾ However, the reduction of serpentine can increase the basicity (CaO/SiO₂) and melting temperature of bosh slag, resulting in an increase of the viscosity at certain temperatures. Therefore, in high PCR/low slag volume operations, the viscosity of bosh slag can be higher than that of conventional operations, while the viscosity of tapped slag can remain at the same level because an additional large amount of SiO₂ is supplied from injected coal particles at the tuyere level and CaO and SiO₂ are added to the sinter to maintain the CaO/SiO₂ ratio like that of tapped level in conventional operation.

Based on experimental results, it can be known that bosh slag with 10 mass% Al₂O₃ content has the lowest melting point and the widest solid-liquid coexisting region at about 5 mass% MgO, while an increase in MgO content from 3.5 to 10 mass% raises the melting point of bosh slag with 14 mass% Al₂O₃ (see Figs. 1 and 2). It is also observed that the viscosity of bosh slag with 7 mass% MgO exhibits a minimum value at higher temperature than 1723 K. However, it does not significantly change with varying MgO content. Therefore, it can be known that good gas permeability in the bosh zone can be maintained although the MgO content in bosh slag is reduced to 5 mass%. Also, the viscosity of tapped slag is not significantly changed even with varying MgO content within 10 mass%, as shown in Fig. 9.

In present POSCO operations, the MgO content has been controlled to about 7–10 mass% in bosh slag because of tapped slag viscosity and sulfide capacity. If the MgO amount in sinter is reduced from 1.2 to 0.8 mass% and hence, the MgO content in the bosh slag is reduced around 5 mass%, the slag volume can be reduced from about 300 to

Fig. 9. Effect of MgO content on viscosity of CaO-SiO₂-13mass%Al₂O₃-MgO slag with C/S=1.18 at 1773 and 1723 K.

270 kg/THM in the POSCO BF, which has a working volume of 3 800 Nm³. Although lowering MgO content in the slag may deteriorate sulfur removal capacity, the sulfide capacity would not be significantly affected by reducing the MgO content.^{19,20)} Therefore, it can be proposed that the MgO content in bosh slag is preferable at about 5 mass% for high PCR and low slag volume operations.

5. Conclusions

Viscosities of the CaO-SiO₂-Al₂O₃-MgO-FeO slag were measured under conditions of C/S=1.35–1.45, 10–18 mass% Al₂O₃, 3.5–10 mass% MgO and 5 mass% FeO. The bosh slag with 10 mass% Al₂O₃ content had the lowest melting temperature and the widest solid-liquid coexisting region at about 5 mass% MgO, while an increase in MgO content from 3.5 to 10 mass% caused an increase in the melting temperature of bosh slag at 14 mass% Al₂O₃. Also, it was observed that the viscosity of bosh slag exhibited a minimum value at about 7 mass% MgO although experi-

mental errors were considered. And, the viscous behavior of bosh slag at different slag compositions was qualitatively confirmed by measuring the infrared spectra for slag structure. To stabilize high PCR/low slag volume operation at POSCO, it was proposed that MgO and Al₂O₃ contents in bosh slag should be maintained at around 5 and 10 mass%, respectively.

Acknowledgment

This work received financial support from POSCO and one of the authors (LYS) was supported by the Brain Korea 21 project.

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