Effect of MnO on High-Alumina Slag Viscosity and Corrosion Behavior of Refractory in Slags

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The influence of MnO on viscosities of CaO–SiO2–MgO–Al2O3–Cr2O3-based slags and the corrosion mechanism of carbon composite brick used in blast furnace hearth by slags was investigated in this work. From the viscosity experimental results, it was indicated that the viscosity decreased with the addition of MnO. The break point temperature decreases from 1 655 to 1 632 K and the apparent activation energy decreases from 217.54 to 185.90 kJ mol−1 with the increasing MnO content from 0 to 3 wt.%. The FTIR spectra analyses revealed that MnO exists as a network modifier and the polymerization degree of the slag decreases with the rising content of MnO. The corrosion of carbon composite brick by molten slag could be a consequence of dissolution, slag penetration and slag-refractory reaction, and the degradation of the brick becomes intenser with the addition of MnO.

KEY WORDS: high Alumina slag; viscosity; refractory; corrosion behavior; MnO.

1. Introduction

With the exhaustion of nickel sulfide ore resources, nickel laterite ores will be the main feedstock for the nickel production, and in China, some laterite ores are smelted in blast furnace.1,2) Compared with the ordinary iron ore used in ironmaking, laterite ore is a low-grade ore which contained a certain amount of oxides, such as TiO2, Cr2O3, MnO and so on. In order to use more laterite ores in blast furnace, it is important to understand the effect of these minor oxides on operations of blast furnace and properties of slag. In our previous study,3) the effect of Cr2O3 on the viscosity and structure of high-Alumina slag has been clarified. However the influence of MnO on the property of high-Alumina slag has not been completely clear.

The viscosity of CaO–SiO2–MnO slags has been measured and calculated using viscosity estimation models by Ji.5) Park et al.5–8) studied the relationship between structure and property of CaO–SiO2–MnO slag, indicating that the properties such as viscosity, density, electrical conductivity and sulfide capacity are significantly dependent on the influence of MnO on the degree of polymerization of slag networks. Kim et al.9) revealed the effect of MnO on viscosity and structure of TiO2–MnO–SiO2 welding fluxes. Deng et al.10) found that the addition of MnO could reduce the viscosity of La2O3–SiO2–Al2O3 slag. Zuo et al.11) investigated the viscosity of CaO–SiO2–MgO–Al2O3–MnO slag systems and the wetting behaviour between slag and refractory, the results showed that the slag viscosity decreased and the erosion rate of refractory increased with the addition of MnO. Although the effect of MnO on the property of slags and fluxes was widely researched, few studies regarding the viscosity of high-Alumina slag containing Cr2O3 after adding MnO have been reported.

In general, the performance of slag not only affects the operation of blast furnace, but also influences the dissolution and erosion of refractory. The wear of refractory is an important factor to limit the blast furnace campaign life. Therefore, it is necessary to investigate the corrosion behavior of refractory by molten slags. A lot of studies focus on the corrosion or dissolution of MgO–C refractory,12,13) MgO–Cr2O3–C refractory,12,13) Al2O3–C refractory,14) Al2O315) in different slags and the wetting behavior of11) or interfacial reaction16) between refractories and slags. However, the corrosion mechanism of refractory by slag formed with the laterite ores has not been systematically investigated.

In this work, the influence of MnO on viscosity and structure of slag was studied and the corrosion mechanism of carbon composite brick used in blast furnace hearth by slags with or without MnO addition was also investigated, which can provide a reference for optimizing blast furnace operations and using more laterite ores.

2. Experimental

2.1. Materials Preparation

Slag samples were synthesized using analytical reagent grade powders of CaO, SiO2, MgO, Al2O3, Cr2O3 and MnO, and all these powders were dried at 378 K for 12 hours in a desiccator. According to the compositions of slags shown in Table 1, the reagents were precisely weighed 125 g and
thoroughly mixed in the agate mortar. The test refractories were cut from commercial carbon composite brick manufactured by Henan Winna Industrial Group Co., Ltd. The main compositions of carbon composite bricks are 74.05 wt.% Al₂O₃, 9.2 wt.% C, 8.18 wt.% SiO₂ and 6.0 wt.% SiC. The apparent porosity and bulk density of the carbon composite brick is 10.9% and 2.98 g cm⁻³ respectively. The brick samples were processed to a cylindrical specimen of 17.5 mm in diameter and 17 mm in height and the center of the cylindrical brick also drilled a through hole of 5 mm in diameter. The slag and brick samples were prepared for the following studies.

2.2. Experimental Apparatus and Procedure

The slag viscosity was measured using rotating cylindrical method, the apparatus and calibration procedure before experiment in detail can be found in our previous study.³) The molybdenum crucible (with inner diameter of 39 mm and height of 60 mm) containing slag samples was placed in the tube of the furnace, then the slags were heated to 1 823 K and held at this temperature for about 2 hours to homogenise the molten slags in an Ar gas atmosphere. The rotation rate of 200 rpm was used and the slag viscosity was measured with the temperature cooled at a rate of 3 K min⁻¹. After the viscosity measurement, the slags were reheated to 1 823 K and quenched by water rapidly. The quenched slags were dried and crushed into powder. The powder samples were verified to be non-crystalline state by X-ray diffraction (XRD; Ultima IV, Japan) using Cu Kα radiation (λ=1.5406 Å) in 2θ range from 10° to 90°, as shown in Fig. 1. The slags and KBr was mixed according to the mass ratio of 1:100, then the FTIR spectra of No. 1 and No. 4 slags were recorded in the range of 4 000–400 cm⁻¹ using a Fourier Transform Infrared spectrometer (470FTIR, USA).

The apparatus used to investigate the corrosion behavior of carbon composite brick and slags is shown in Fig. 2. The carbon composite brick cylinders are mounted on a molybdenum bar with a diameter of 5 mm. Four slag samples with 0 wt.%, 1.0 wt.%, 2.0 wt.% and 3.0 wt.% MnO were used in the corrosion experiment. The slags were contained in the Mo crucible and heated to 1 823 K in the furnace, then held at 1 823 K for about 2 hours to ensure the molten slag homogeneously. After that, the brick sample was immersed into the molten slags and rotated at a speed of 200 rpm for 120 min. During the whole experiment, high-purity argon gas (99.999%, 1.5 L min⁻¹) was used to prevent the oxidation of the crucible and the brick. After the experiment, the brick sample was lifted to the upper position of the crucible and rotated the brick again to remove the slag adhered in the brick surface as much as possible. Then the brick was taken out of the furnace and cooled in air. The brick cylinder diameter was measured at different levels in height using a vernier caliper. The average value of these measurements was employed as the mean diameter of the brick cylinder after the corrosion experiment, then the diameter reduction could be calculated by the difference of the diameter before and after the experiment.

The brick samples after experiment were cut longitudinally using a diamond saw and the cross section was moulded in epoxy resin, ground and polished. The morphology and the elemental distribution of the cross sections was examined with a scanning electron microscope (SEM; FEI MLA250, USA) with energy dispersive spectroscopy (EDS). The XRD was used to analyze the phase composition of the brick surface after the experiment.
3. Results and Discussion

3.1. Effect of MnO on the Viscosity and Structure of Slags

The viscosities of CaO–SiO₂–MgO–Al₂O₃–Cr₂O₃-based slag with different MnO content as a function of temperatures are shown in Fig. 3. The effects of MnO on the slag viscosities at different temperature are presented in Fig. 4. It can be found that the viscosity of the slags gradually increases with the temperature decreasing and decreases with the addition of MnO. It is consistent with some previous studies, although the slag systems are different. According to previous researches, MnO was a basic oxide similar to CaO in silicate melts, which can supply free oxygen ions (O²⁻) and O²⁻ subsequently interact with the bridged oxygen (O³) to produce non-bridged oxygen (O⁻). According to Fincham et al., O⁻ is the oxygen bonded to one silicon, which can formed according to the reaction (1). This transition could promote to simplify the slag structures and improve the fluidity of slag. Therefore, the addition of MnO as a network modifier could increase the amount of O⁻ and depolymerize the complex network structures of the molten slag, then decreasing the viscosity. On the other hand, the high Al₂O₃ content of the present slag system may increase the quantity of complex aluminate or alumino-silicate structural units, and a dynamic balanced reaction as shown in Eq. (2) may exist in the slag. MnO could react with Al₂O₃ to form melilite, then breaking the balanced reaction and promoting the reversal reaction, consequently decreasing the content of AlO₄⁻ anion group as well as the slag viscosity. From Fig. 4, the viscosity of slags decreased significantly with the content of MnO adding to 2 wt.%, while the viscosity slightly decreased with the MnO content increased from 2 wt.% to 3 wt.%%. It means that the effect of MnO additions becomes less pronounced with the slag structure further depolymerized.

\[ \text{O}^{2-} + \text{Si-O-Si} = 2\text{Si-O}^- \]  \hspace{1cm} (1)

\[ 5\text{O}^{2-} + \text{Al}_2\text{O}_3 = 2\text{AlO}_4^{5-} \]  \hspace{1cm} (2)

The break point temperature (Tbr) was the temperature at which the measured viscosity increases abruptly and the slag becomes non-Newtonian in behavior during the cooling cycle. Generally, the break point temperature would likely be lower than the initial crystallization temperature and higher than the fully crystallization temperature. As seen in Fig. 5, Tbr decreases from 1655 to 1632 K with the rising of MnO content from 0 to 3 wt.%. A higher MnO content of the slags corresponds to a lower break point temperature.

The structure information of molten slags was obtained by using FTIR spectroscopy, as shown in Fig. 6. FTIR spectra of the silicate slags are ordinarily focused in the 1200–400 cm⁻¹ range. The vibration band in the range of 1200–800 cm⁻¹ generally represents the symmetric stretching vibration of SiO₄ tetrahedra, and the band within the wavenumber of 1100–1050 cm⁻¹, 980–950 cm⁻¹, 920–900 cm⁻¹ and 880–850 cm⁻¹ was structurally defined as sheets (Q3), chains (Q2), dimmers (Q1) and monomers (Q0), where n in Qⁿ denotes the number of bridging oxygen per unit silicon atom, respectively. The vibration band between 800 cm⁻¹ and 600 cm⁻¹ was identified as the asymmetric stretching vibration of AlO₄ tetrahedra and the vibration around 500 cm⁻¹ corresponds to the Si–O bending vibration. From Fig. 6, the depth of the transmis-
sion trough decreases and the SiO$_4$ tetrahedral stretching vibrations become less pronounced with the addition of MnO, which indicates the slag structures depolymerized and silicate units simplified.\textsuperscript{25,26) While the AlO$_4$ tetrahedral asymmetric stretching vibration and the Si–O bending vibration exhibits a little change with MnO additions. The result of FTIR spectra seems to suggest that MnO depolymerizes the network structure by modifying the silicate units of the slags, consequently resulting in the viscosity decreased.

3.2. Activation Energy for the Viscous Flow of the Slags

The viscosity of the molten slags is a function of composition and temperature, which is dependent on the degree of polymerization.\textsuperscript{17) The relationship between the viscosity and temperature is conventionally expressed by Arrhenius equation:

\[
\eta = A \exp \left( \frac{E_\eta}{RT} \right)
\]

where $\eta$ is the viscosity, $A$ is a pre-exponent constant, $E_\eta$ is the apparent activation energy for viscous flow, $R$ is the gas constant and $T$ is the absolute temperature. The apparent activation energy $E_\eta$ represents the frictional resistance for viscous flow in molten slags, and the variation of that could be reflect the change of slag structures or flow units. The activation energy $E_\eta$ can be obtained by plotting ln$\eta$ vs. 1/T. Figure 7 shows the relationship curve of ln$\eta$ and 1/T. It can be seen that the ln$\eta$ depends linearly on 1/T, indicating the relationship between viscosity and temperature obeys to the Arrhenius equation. The calculated results of activation energy are listed in Table 2. $E_\eta$ decreases from 217.54 to 185.90 kJ mol$^{-1}$ with the increasing MnO content from 0 to 3 wt.%, which means the polymerization degree of the slag decreases and the structural units become simpler with the addition of MnO.

3.3. Corrosion Behavior of Carbon Composite Brick in Slags

Two corrosion indexes, reaction area and radius reduction, can be used to characterize the erosion degree of refractory.\textsuperscript{27) In present study, the diameter reduction was adopted to represent the corrosion degree of the carbon composite brick. The decrease in diameter of the brick is shown in Fig. 8. It can be seen that the value of diameter reduction increases from 0.343 to 0.414 mm with the addition of MnO from 0 to 3 wt.%, indicating that the degradation of carbon composite brick immersed in MnO-containing slag becomes intenser. MnO could decrease the viscosity and break point temperature of the slags, resulting in the erosion of brick increased.\textsuperscript{27)

Table 2. Apparent activation energy of the slags.

| No. | MnO(wt.%) | $E_\eta$(kJ mol$^{-1}$) |
|-----|-----------|------------------------|
| 1   | 0         | 217.54                 |
| 2   | 1.0       | 209.61                 |
| 3   | 1.5       | 200.15                 |
| 4   | 2.0       | 197.55                 |
| 5   | 3.0       | 185.90                 |

Fig. 8. Decrease in diameter of brick after corrosion experiment.
Al$_2$O$_3$–Cr$_2$O$_3$–2 wt.%MnO slag was observed and analyzed using SEM-EDS. The appearance of carbon composite brick immersed in CaO–SiO$_2$–MgO–Al$_2$O$_3$–Cr$_2$O$_3$ slag was shown in Fig. 9. Calcium could be used to characterize the presence of slag because the refractory does not contain the calcium oxides. From the EDS result in Figs. 9(C)–9(D), a slag layer can be observed on the surface of refractory and a number of metallic Cr and some unreacted carbon also presented. The formation of Cr particles was due to the reaction between the carbon of refractory and Cr$_2$O$_3$ of slag according to the reactions (4) and (5). Han et al. investigated the degradation of MgO–C brick by SiO$_2$–Fe$_2$O$_3$–V$_2$O$_5$–TiO$_2$–MnO–MgO–Cr$_2$O$_3$ slag, which indicated that the carbon with non-wettability by slag played an important role in the service life of MgO–C refractory and carbon oxidation as well as MgO dissolution were the main erosion mechanism. Sun et al. studied the wettability and reduction of MnO in slag by carbonaceous materials, indicating the poor wettability of graphite or coke by MnO–SiO$_2$–CaO–Al$_2$O$_3$ slag. Therefore the unreacted carbon could prevent the slag...

Fig. 9. SEM mapping and EDS results of the brick after corrosion by CaO–SiO$_2$–MgO–Al$_2$O$_3$–Cr$_2$O$_3$ slag. (A), (B) and (F): appearance of carbon composite brick; (C)–(E): EDS results of P1, P2 and P3; (G)–(M): EDS mapping results of image (F). (Online version in color.)
penetration due to the non-wettability of carbon by slag.

\[
\text{Cr}_2\text{O}_3(l) + \text{C}(s) = 2\text{CrO}(l) + \text{CO}(g) \quad \ldots \ldots (4)
\]

\[
\text{CrO}(l) + \text{C}(s) = \text{Cr}(s) + \text{CO}(g) \quad \ldots \ldots (5)
\]

From the mapping results in Figs. 9(G)–9(M), it can be seen that the oxides of calcium and silicon are mainly distributed in the surface of refractory, and magnesium oxides are enriched between the slag layer and Al2O3 particles. A certain amount of calcium elements also existed near the boundary of Al2O3 grains. It means that MgO and CaO may preferentially diffuse to the interface of aluminum grain and react with Al2O3 to form intermediate compounds, e.g., MgO·Al2O3 and CaO·2Al2O3, when the slag is infiltrated into the refractory. Sandhage et al.\(^{31}\) and Zhang et al.\(^{32}\) have reported that a layer of MgO·Al2O3 could be formed at the interface between alumina and slag under certain conditions. An intermediate compound of CaO·2Al2O3 has been reported by Li et al.\(^{15}\) on study of Al2O3 dissolution into CaO–Al2O3–CaF2 flux.

The corrosion of refractory (carbon composite brick) by molten slag can be described as follows: when the carbon composite brick in contact with the molten slag, to a certain extent, the slag could infiltrate into the bulk of refractory through open pores, grain boundaries or voids of the brick. The infiltrated slag would change the structure and densification of the brick and cause the brick to swell or shrink, then the disintegration of carbon composite brick will take place.\(^{33,34}\) On the other hand, the molten slag can dissolve and react with the refractory. When the oxides of slag such as Cr2O3, SiO2 directly encounter the carbon of refractory during the infiltration or dissolution process, a decarburized layer will be formed due to the oxidation of carbon. This process is not conducive to corrosion resistance of carbon composite brick by slag penetration. The gas generated by the reaction will escape from the refractory result in convection flows at the contact area, accordingly promoting the dissolution of refractory into molten slag.\(^{15}\) A certain amount of pores or cracks are also produced inside the brick due to the oxidation of carbon, so as to further promote the molten slag penetrate into the brick, result in the severe corrosion of brick.\(^{14,29}\) According to Kumar et al.\(^{14}\) the decrease of aggressive slag components such as Cr2O3 and SiO2 was conducive to reduce the refractory erosion. At the dissolution process of the refractory which consists mainly of Al2O3 into the high alumina slags, it should be noted that the dissolution rate was effected by the solubility of Al2O3 and the dissolution rate was proportional to the thermodynamic driving force \(\Delta(G_{\text{Al}_2\text{O}_3}/\text{wt}.)\) under the mass transfer controlled condition.\(^{15}\) The higher concentration of Al2O3 in the slag may lead to the lower solubility of refractory. The rate controlling step of the dissolution of refractory and the influence of Al2O3 solubility on the dissolution rate need to be further studied.

Besides the extrinsic oxidation of carbon by reducible slag constituents, the in-situ reaction of refractory such as SiO2 and carbon according to reactions (6) and (7) may also lead to the degradation of refractory.\(^{30}\) In comparison with the oxidation of carbon by Cr2O3, the effect of SiO2 on the carbon oxidation is limited and play a minor role.\(^{25}\) In summary, the corrosion of refractory could be a consequence of dissolution, slag penetration and slag-refractory reaction.

\[
\text{SiO}_2(\text{s or l}) + 3\text{C}(s) = \text{SiC}(s) + 2\text{CO}(g) \quad \ldots \ldots (6)
\]

\[
\text{SiO}_2(\text{s or l}) + \text{C}(s) = \text{SiO}(g) + \text{CO}(g) \quad \ldots \ldots (7)
\]

The surface of the carbon composite brick after experiment was analyzed using XRD. According to the XRD results presented in Fig. 10, the products on the refractory surface were mixtures of corundum, carbon, spinel (2 408 K), gehlenite (1 866 K) and akermanite (1 723 K). The solid solution of Al1.98Cr0.02O3 may be formed due to the reaction of Cr2O3 or Cr with Al2O3. The undissolved Al2O3 and C were originated from the refractory. The formation of spinel, gehlenite and akermanite may be due to the reaction of oxides in refractory and slag or the reaction within the slag. Due to the higher melting point and volumetric stability of spinel phase, it can effectively inhibit the penetration of slag into refractory and reduce the corrosion of refractory.\(^{35,37}\) However, the formation of gehlenite, akermanite and other possible products such as anorthite (1 828 K), which melting point is lower than or similar to the temperature of blast furnace hearth, may lead to the softening and disintegration of the refractory.\(^{15,36}\)

The appearance of carbon composite brick immersed in CaO–SiO2–MgO–Al2O3–Cr2O3-2 wt.%MnO slag was shown in Fig. 11. It can be seen that a slag layer was existed in the surface of the brick, a number of metallic Cr, Mn and some unreacted carbon also presented. The formation of Mn particles was due to the reaction between the carbon of brick and MnO of slag according to the reaction (8).\(^{28}\)

\[
\text{MnO}(l) + \text{C}(s) = \text{Mn}(l) + \text{CO}(g) \quad \ldots \ldots (8)
\]

From the mapping results in Figs. 11(E)–11(L), it can be seen that the metallic Cr, Mn and unreacted carbon are distributed in the slag layer, and magnesium oxides are enriched between the slag layer and Al2O3 grains. When the slag containing MnO contacted the carbon composite brick, due to the reducible component MnO, more carbon was oxidized. Which may produce more pores and cracks inside the brick, consequently more molten slags infiltrate...
into the brick, resulting in the severer corrosion of the brick. According to previous studies, the penetration rate of slag into refractory was related to the fluidity of slags and the penetration rate increased with the slag viscosity decreased. In the present work, the addition of MnO could reduce the slag viscosity, therefore promoting the penetration of slag into brick and aggravating the erosion of the brick. In fact, the surface tension of the slag also influences the corrosion of carbon composite brick and lower surface tension may lead to the brick more corrosive. Due to the complex properties of the slag formed with the laterite ores, the effect of physical properties such as surface tension and wettability of the slag on the corrosion of refractory need to further investigated in the future.

4. Conclusions

Influences of MnO on the viscosity and structure of high-Alumina slag and the corrosion mechanism of carbon composite brick by slags have been studied. Some conclusions are obtained as follows:

1. The viscosity, break point temperature and activation energy of the present slag decreases with the addition of MnO content.
MnO acts as a network modifier in present slag and depolymerize the complex network structure, which decreases the polymerization degree and viscosity of the slags.

The corrosion of carbon composite brick by molten slag could be a consequence of slag penetration, dissolution and slag-refractory reaction, and the degradation of the brick becomes intenser with the addition of MnO.

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