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Viscosity and flow behaviour of TiO_2-containing blast furnace slags under reducing conditions

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The viscosity of TiO_2-containing blast furnace slags may increase rapidly (commonly referred to as thickening) under strong reducing conditions in the blast furnace. To elucidate the mechanisms for slag thickening, measurements have been made of changes in the composition and rheological properties of TiO_2-CaO-SiO_2-Al_2O_3-MgO slags during the reduction process. The slag composition covered the range of 17–33 wt% TiO_2, 9–19% Al_2O_3, 8–16% MgO and the CaO/SiO_2 from 0.8 to 1.4. The viscosities were measured using the rotating cylinder method at temperatures up to 1550ºC and the fusibility temperatures were determined from 'breaks' in the viscosity-temperature curves. The reduction of the slag was carried out in graphite crucibles at 1500ºC for 2–6 hours.

The rheological properties of the slags during reduction were predominantly influenced by the slag basicity (CaO/SiO_2), and the effect of other components depended largely on basic or acidic behaviour of the oxide. The reduction of TiO_2 in the slag by carbon was initially fast to form Ti_2O_3, followed by further reduction to TiO and TiC. Correspondingly, the viscosity showed initially a slight decrease, followed by a gradual and then rapid increase. The thickening was found to be accelerated by an increase in TiO_2 content and a decrease in the CaO/SiO_2 in the slag. In addition to the existence of 1~3 wt% TiC, the thickened slags also had a significant amount of TiO (3~7 wt%), suggesting that TiO may play an important role in the 'thickening phenomena', possibly by forming solid solutions with TiC to significantly increase the number of solid particles in the heterogeneous slag.

Keywords: TiO_2 slags, viscosity

Introduction

It is well known that the viscosity of the blast furnace slags containing high levels of TiO_2 (>15 wt%) may increase rapidly under strong reducing conditions in the blast furnace. The reduction of TiO_2 is responsible for the thickening but the prevailing mechanisms have not been well understood due to the complexity of the processes and lack of sufficient data.

In comparison with conventional blast furnace slags, TiO_2-containing slags have a relatively higher fusibility temperature of about 1400ºC and low viscosity before reduction. The fusibility temperature is defined and determined from the 'breaks' in the viscosity-temperature curves below which slag viscosity increases rapidly due to crystallization of oxide phases. It is worth mentioning that, for a reduced TiO_2 slag containing TiC solid particles as a heterogeneous slag, the fusibility temperature is not strictly liquidus temperature.

Under strong reducing conditions in the blast furnace, TiO_2 is reduced by carbon to form a series of reduction products such as Ti_2O_3, TiO and TiC (and often TiN when exposed to air in the blast furnace), accompanied by a rapid increase in viscosity. Numerous studies have been conducted in the past to investigate the behaviours of TiO_2 slags under reducing conditions and the mechanisms for slag thickening[1-9]. Previously published results on the subject have been reviewed by Sommerville and Bell[10].

As solid particles in the slag, the final reduction products, TiC and TiN, are commonly attributed to be the main reason for causing slag thickening[10]. It is difficult, however, to explain the 'rapid' increase in the slag viscosity as TiC content is relatively low, at a few weight percent (accurate estimate of volume proportion is not readily available) in reduced and thickened slags. Attention was also drawn to possible effects of intermediate reduction products, Ti_2O_3 and TiO, on rheological properties of the slags. As found by Handfield *et al.*[8], addition of Ti_2O_3 and TiO slightly reduced slag viscosity, but when added above 10 wt%, resulted in a sharp rise in the fusibility temperature of the slag in the composition range of 35 wt%CaO-37SiO_2-18MgO-4Al_2O_3. (Weight percent is used throughout the present paper unless specified otherwise). This might provide another explanation for thickening phenomena. However, the observation by Handfield *et al.* was made for slags with addition of Ti_2O_3 and TiO. As such, the behaviour may not be directly applicable to reduced slags where the reduction products produced *in situ* may differ from the added oxides in terms of both the form and relative quantities.

Thus, despite numerous studies carried out in the past, the mechanisms for the thickening of TiO_2 slags under reducing conditions are yet to be fully understood. The present work was carried out to clarify key issues, in particular the effects of intermediate reduction products

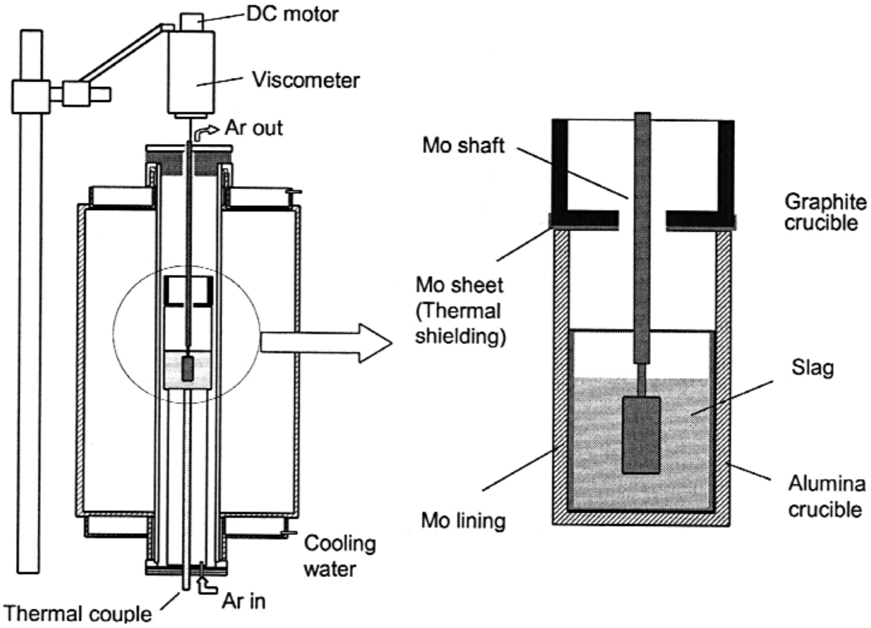


Figure 1. Experimental arrangement

Ti_2O_3 and TiO and final product TiC, through a comprehensive investigation into simultaneous changes in the slag composition and properties during the reduction processes. To achieve these objectives, experiments were conducted under controlled conditions of moderate and prolonged reduction to establish the characteristics of the reduction and thickening process and the effects of the initial TiO_2 content, slag basicity (CaO/SiO_2) and other components.

The viscosities of the TiO_2-containing slags used in the present study under neutral conditions have been published elsewhere[11] and the results are used here for comparison with those measured under reducing conditions. Due to space constraints, this paper only reports the experimental results and the main findings with a brief discussion. A detailed discussion and interpretation will be published elsewhere.

Experimental

The experimental arrangement is schematically shown in Figure 1. A vertical, molybdenum-wound resistance furnace was used for both reduction and viscosity measurement. An alumina tube of 60 mm I.D. was used as a working tube with a hot zone of about 70 mm (±10ºC). The temperature was controlled by a DWK-702 controller in conjunction with a $PtRh_{10}$-Pt (type S) thermocouple placed very near to the base of the crucible. The temperature of the slag was measured before and after the experiment by another thermocouple inserted from the top and immersed 5 mm below the slag surface. The uncertainty of the experimental temperature was ±10ºC.

Slag viscosity was measured by the rotating cylinder method using a ND-2 rotating viscometer operating under an atmosphere of argon. The suspended system of the viscometer rotated at a constant speed of 6 rpm and the torque was measured from the twist angle of a steel wire by optical signal and converted to digital readings. The molybdenum spindle of 10 mm diameter by 20 mm long was screwed on to the recessed tip of a molybdenum shaft (5 mm diameter) as shown in Figure 1. The recessed tip section was 3 mm diameter by 10 mm long, the middle of which was positioned at the slag surface during viscosity measurement to minimize the error caused by uncertainty in the immersion depth of the spindle. The viscometer was regularly calibrated before and after the experiment using a series of standard silicone oils of known viscosities (0.8759 to 54.517 poises). The errors in viscosity measurement depended on the experimental conditions and will be discussed later. Other experimental details were given elsewhere[12].

The viscosity-temperature curve was determined by commencing the viscosity measurement from 1500ºC when the slags were completely molten and thoroughly mixed, to about 20–40ºC below the fusibility temperature. Viscosity was measured every 40ºC in the high temperature range and then at smaller intervals of 20ºC (or even 10ºC) in the temperature range close to the fusibility temperature. At each temperature, the slag bath was held for about 10 min to allow thermal equilibrium, and then viscosity readings were taken. Duplicate experiments were conducted for selected conditions to verify the reproducibility of the results. The viscosity versus temperature curves showed distinct 'breaks' at the fusibility temperature, from a gradual increase in viscosity with decreasing temperature to a rapid rise due to crystallization of oxide phases. The 'break' and fusibility temperature were determined from the interception of two straight lines drawn through the viscosities measured over higher and lower temperature ranges, respectively. For the present slags with around 25%TiO_2 and CaO/SiO_2 of 0.8~1.4, the fusibility temperature could be determined with an uncertainty of about ±10ºC.

Synthetic slags were prepared from reagent grade chemicals ($CaCO_3$, SiO_2, Al_2O_3, MgO and TiO_2). Weighed chemicals were thoroughly mixed and pressed into cylindrical pellets with the addition of a small amount of water. The pellets were dried before being used in the experiments. About 240 g of sample was used in the reduction and viscosity measurements, which gave a slag bath of at least 45 mm deep contained in crucibles of 42 mm internal diameter.

Table I
Slag compositions in wt%

Slag #	TiO_2	CaO	SiO_2	Al_2O_3	MgO	CaO/SiO_2	Grouping ID
1*	25.0	27.8	25.2	14.0	8.0	1.10	T25, B11, A14, CM8
2	17.0	30.8	27.9	15.5	8.9	1.10	T17
3	33.0	24.8	22.5	12.5	7.2	1.10	T33
4	25.0	23.6	29.4	14.0	8.0	0.80	B08
5	25.0	30.9	22.1	14.0	8.0	1.40	B14
6	25.0	30.1	27.3	9.0	8.7	1.10	A09
7	25.0	25.5	23.1	19.0	7.3	1.10	A19
8	25.0	23.8	25.2	14.0	12.0	0.94	CM12
9	25.0	19.8	25.2	14.0	16.0	0.79	CM16

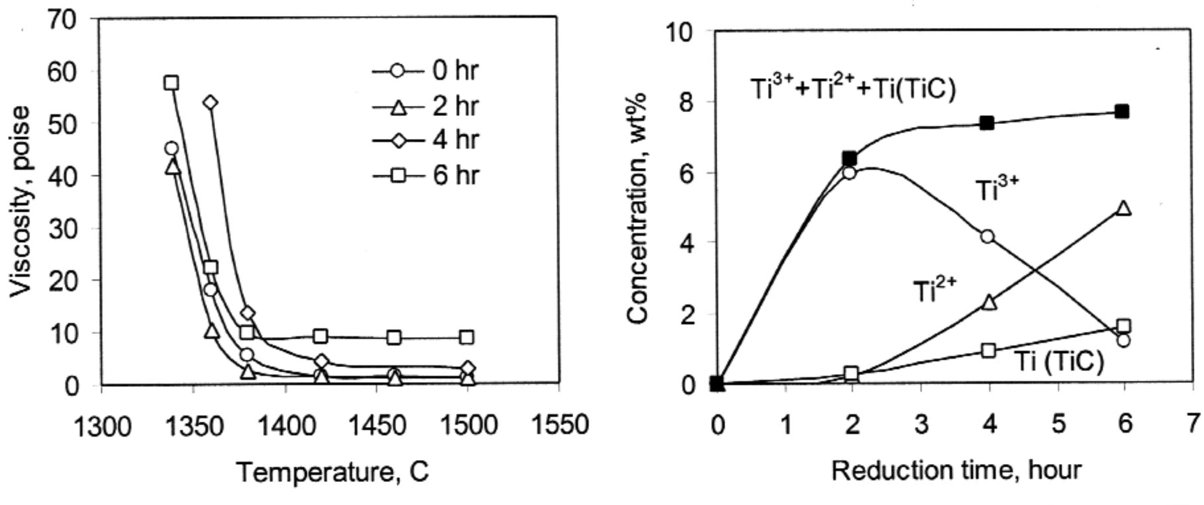
* Base composition

The chemical composition of the slags are shown in Table I. The compositions covered the range 17–33% TiO_2, 9–19% Al_2O_3, 8–16% MgO and the CaO/SiO_2 ratios from 0.8 to 1.4. The right column of Table I indicates the grouping of relevant slags. For example, Group T (including T17, T25 and T23) focused on the effect of TiO_2 while keeping the proportions of other components constant. The TiO_2 content was kept at 25% in all other groups. Group B (B08, B11 and B14) aimed at investigating the effect of basicity (CaO/SiO_2) while Group A the effect of Al_2O_3 content. The effects of MgO were investigated in the CM group focusing on the effect of substituting CaO with MgO on an equal weight basis.

Reduction was carried out in graphite crucibles of 42 mm internal diameter and 60 mm height at 1500ºC under argon. Two procedures were employed in the reduction and viscosity measurement based on the degree of reduction, namely moderate reduction and prolonged (or deep) reduction.

Procedure A, which was used for moderate reductions of two hours, focused on the effects of initial reduction products such as Ti_2O_3 etc. After two-hour reduction in the graphite crucible, the slag bath was stirred thoroughly using a Mo rod and then the furnace power was switched off to allow quick cooling to 1000ºC, at which time all the slag had solidified and the crucible was removed. After further cooling to room temperature, the slag sample was separated from the graphite crucible and transferred to the Mo lined alumina crucible. The viscosity was measured with a thermal shielding as illustrated in Figure 1. The variation of the slag composition during the viscosity measurement was checked and found to be negligible. The relative error in viscosity measurement ranged from 5 to 12% (but typically 8% in most cases).

Procedure B was designed to investigate prolonged reduction (6 hours) and the effect of final reduction products such as TiC on the viscosity. After a 6-hour reduction the slag was left in the graphite crucible and, after thoroughly stirring and mixing the slag bath using the spindle, the viscosity was measured immediately. No thermal shielding was used as the slag bath had to be well mixed before viscosity measurement. The gas bubbles evolved from the slag bath interfered with the viscosity measurements. To obtain stable readings, an Mo sheet was used to cover the crucible bottom to eliminate the gas bubbles evolving from the bottom and thereby to restrict the reactions to the crucible wall. This helped to significantly reduce the disturbance. Although the readings still fluctuated considerably more than those using Procedure A, the results were found to be reproducible through repeat experiments with a relative error in the viscosity measurement of about 10–20% (typically 15% in most



(A) Viscosity-temp curves

(B) Concentrations of reduction products☐

Figure 2. Effects of the reduction time at 1500ºC on the viscosity-temperature curve (A) and the concentrations of the reduction products (B) of Slag #3

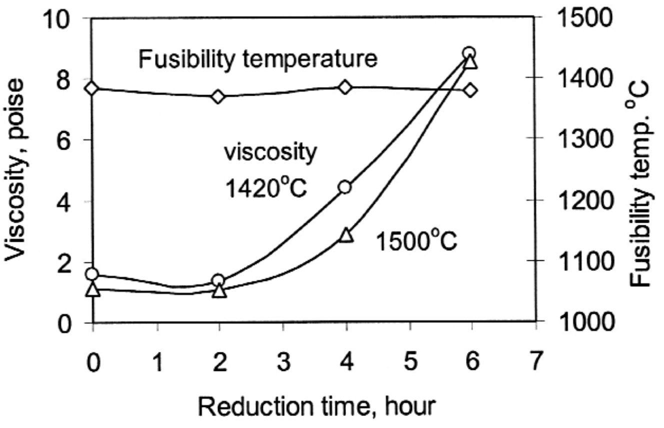


Figure 3. Effects of the reduction (1500°C) time on the viscosity at 1420°C and 1500°C and the fusibility temperatures of Slag #3

cases). The *in situ* viscosity measurement took approximately 1 hour (decreasing from 1500°C until slag solidified).

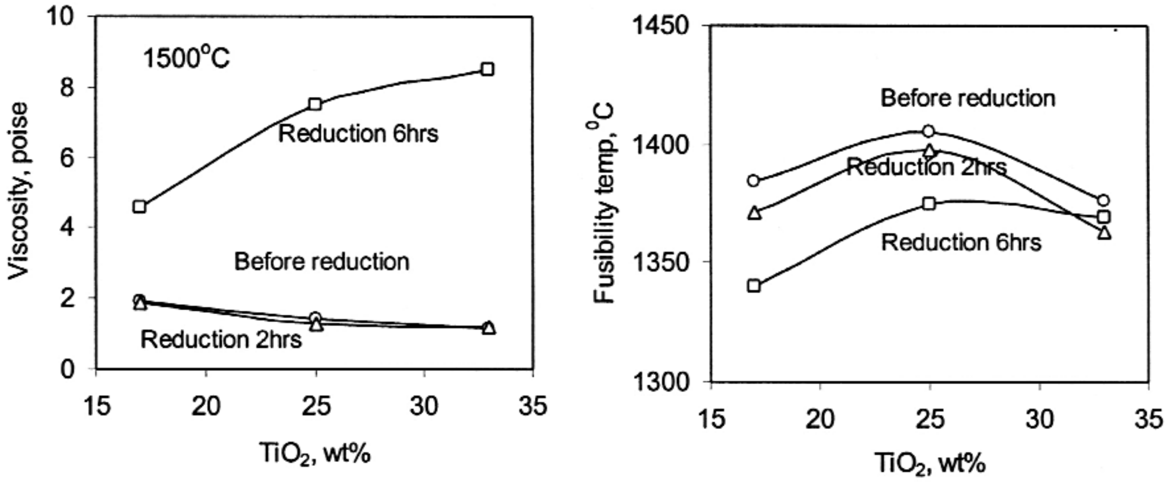
The slag bath was sampled before the measurement of the viscosity-temperature curve by sucking about 10 g of slag into a quartz tube (5 mm ID). Samples were ground into fine powders and analysed for Ti^{4+}, Ti^{3+}, Ti^{2+} and TiC using the procedures and techniques developed by Du[13-14].

Results

Characteristics of the reduction/thickening process

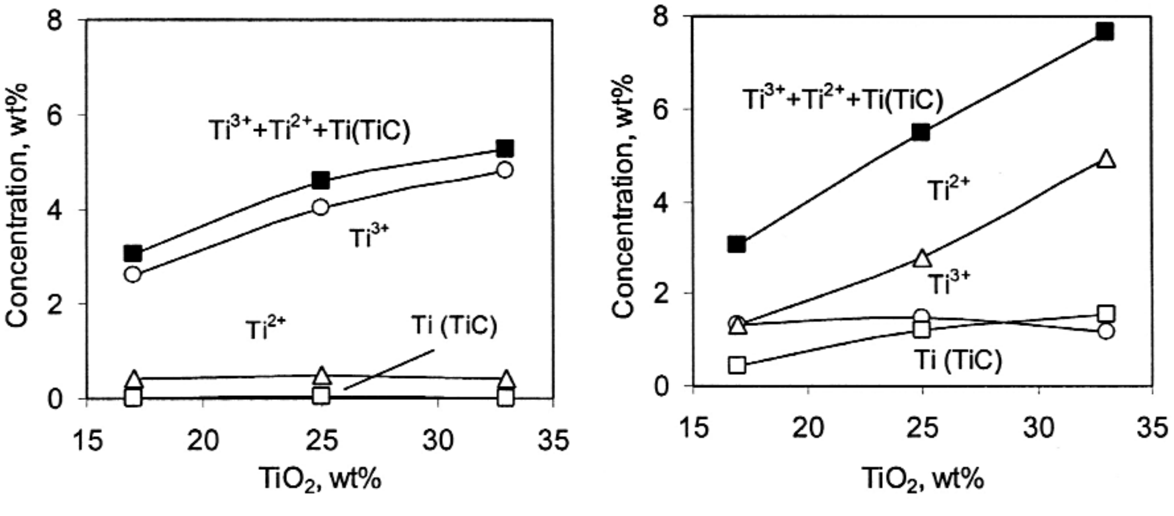
A series of experiments were conducted for Slag #3 ($33TiO_2$-$24.8CaO$-$22.5SiO_2$-$12.5Al_2O_3$-$7.2MgO$) using Procedure B (*in situ* viscosity measurement) to investigate the simultaneous changes in slag composition and rheological properties of the slag as a function of reduction time (2, 4 and 6 hours). The results are shown in Figures 2 and 3.

The reduction of TiO_2 to form Ti_2O_3 was initially fast and then slowed down considerably. Further reduction of



(A) Viscosity

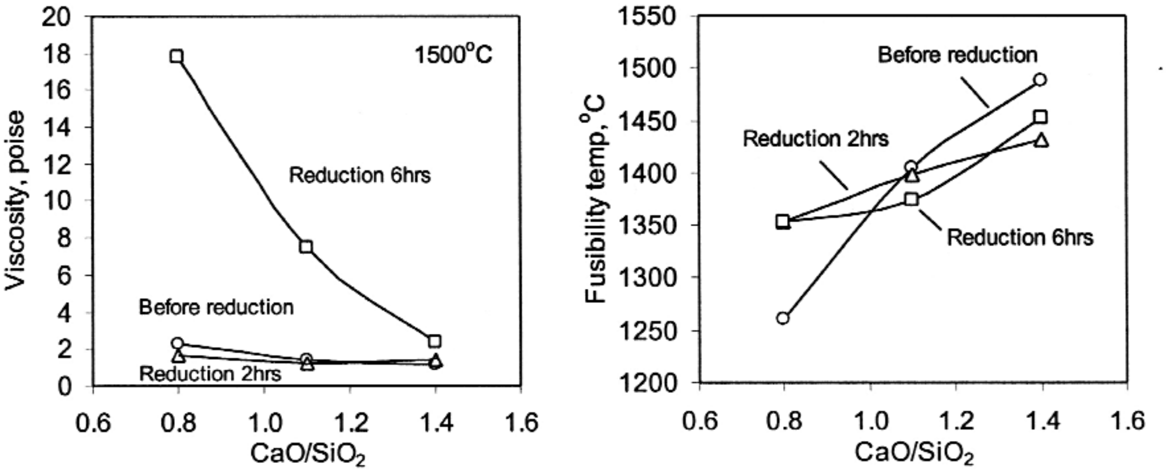
(B) Fusibility temperature



(C) Reduction 2 hours

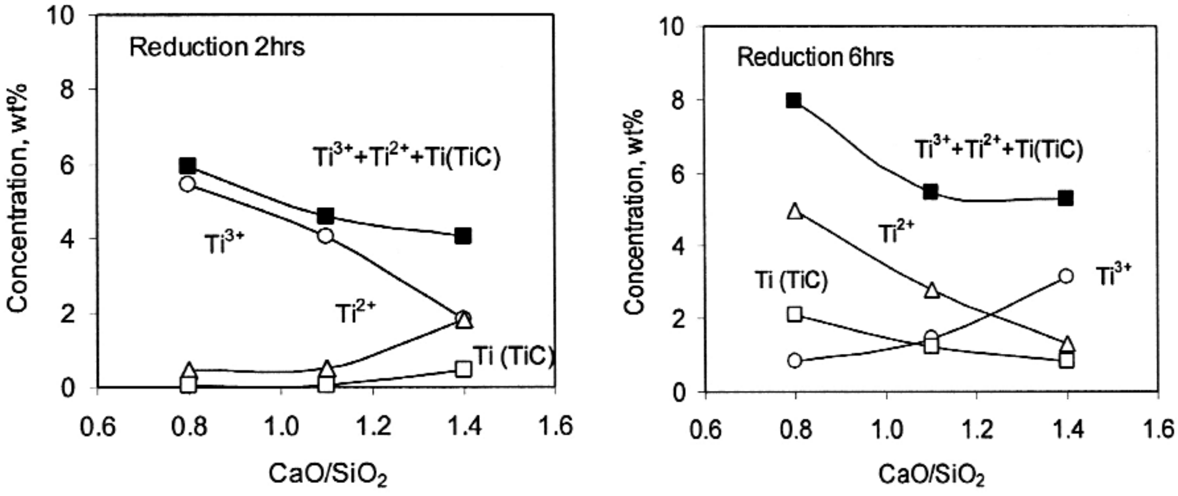
(D) Reduction 6 hours

Figure 4. Effects of the TiO_2 concentration on variation of viscosity (A), fusibility temperature, (B) and slag composition after reduction for two hours (C) and six hours (D), respectively



(A) Viscosity

(B) Fusibility temperature



(C) Reduction 2 hours

(D) Reduction 6 hours

Figure 5. Effects of the basicity (CaO/SiO_2) on viscosity, fusibility temperature and slag composition after reduction for two hours and six hours, respectively

Ti_2O_3 to form TiO and TiC occurred after two hours (Figure 2B). A viscous layer at the crucible wall was detected after reduction for four hours when the slag bath was stirred using the spindle before viscosity measurement. The layer was found to be thicker and more viscous after the six hour reduction.

The viscosity-temperature curves (Figure 2A) showed little changes after moderate reduction, and a significant increase in the viscosity (Figure 2A and Figure 3) occurred only after two hours, corresponding to further reduction from Ti_2O_3 to form TiO and TiC. It is interesting to note that the rapid increase in the viscosity coincided with not only an increase in the TiC content, but also the formation of a considerable amount of TiO. As will be shown later, the co-existence of TiC and a significant amount of TiO was the common feature found in deeply reduced and thickened slags.

The fusibility temperature (Figure 3) determined from the viscosity-temperature curves did not change much (1370~1385ºC) during the reduction.

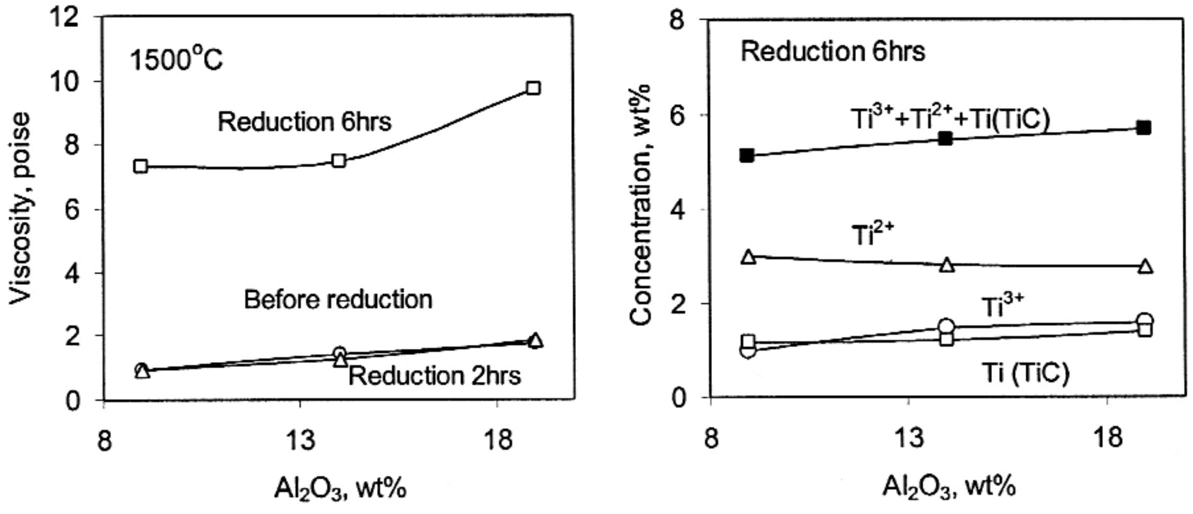
Effect of TiO_2 content

Effects of TiO_2 content were investigated through Slags #1–3, with TiO_2 varying from 17% to 33% while the proportion of all other components remained constant. The results are summarized in Figure 4 (A–D).

Before reduction (under neutral conditions) and after moderate reduction (2 hours), an increase in TiO_2 resulted in a slight decrease in the viscosity (Figure 4A); hence TiO_2 and the initial reduction product Ti_2O_3 behaved as network modifiers similar to basic oxides. Under prolonged reduction of 6 hours, higher TiO_2 slags showed a greater increase in the viscosity (Figure 4A).

The fusibility temperature appeared to show a small peak at 25 wt% TiO_2 and reduction resulted in a decrease in the fusibility temperature (Figure 4B).

The variation in the concentration of reduction products (Figure 4C and D) was in general consistent with the observations made with Slag #3 (T33), which were described earlier. The reduction was marked by an initial fast reduction of TiO_2 to form Ti_2O_3 followed by further reduction to TiO and TiC. The thickened slags showed co-existence of a considerable amount of TiO and TiC. As expected, an increase in TiO_2 accelerated the reduction and thickening. The 17 wt% TiO_2 slag showed only moderate thickening after 6-hour reduction, corresponding to a low concentration of TiC and TiO.



(A) Viscosity (B) Reduction 6 hours

Figure 6. Effects of the Al_2O_3 content on viscosity (A) and composition after 6-hour reduction (B)

Effect of CaO/SiO_2 ratio

The effect of basicity (CaO/SiO_2) was investigated through Slags #1, 4 and 5, all of which had 25 wt% TiO_2 and constant Al_2O_3 and MgO content (Table I). The results are summarized in Figure 5. The basicity had a strong effect on the fusibility temperature before reduction (Figure 5B), varying from 1260°C for the acidic slag (CaO/SiO_2=0.8) to 1487°C for the basic slag (CaO/SiO_2=1.4). Reduction resulted in a decrease in the fusibility temperature of the basic slags and an increase in that of the acidic slags. As shown in Figure 5B, the fusibility temperature of reduced slags varied from 1350°C to 1450°C and was obviously not the reason for slag thickening.

Before reduction, the viscosity decreased with the basicity (Figure 5A) as expected. The point for the slag of CaO/SiO_2=1.4 referred to the viscosity measured at 1540°C as the fusibility temperature of this slag was close to 1500°C before reduction. The moderate reduction for two hours resulted in a slight decrease in the viscosity and the trend observed at neutral conditions remained (viscosity decreased with increasing basicity). Slag thickening after prolonged reduction differed significantly between acidic and basic slags, and the acidic slags showed more severe thickening than the basic slag (Figure 5A).

Slag basicity had a major effect on the reduction process as shown in Figures 5C and 5D for both moderate and prolonged reduction. Reduction degree (the amount of TiO_2 reduced) showed a marked dependence on the basicity and increased significantly with decreasing basicity. The variation of the TiO and TiC concentration produced showed some interesting trends. Under moderate reduction, Ti_2O_3 dominated in the reduction products in acidic slags, while a noticeable amount of TiO and TiC was formed in more basic slag. In deeply reduced acidic slag (CaO/SiO_2=0.8), most Ti_2O_3 formed was converted into TiO and TiC, accompanied by a sharp increase in the viscosity. For the basic slag of CaO/SiO_2 =1.4, however, prolonged reduction resulted in only a moderate increase in the concentrations of reduction products (the total increased from 4 wt% after 2-hour reduction to 5.3 wt% after 6-hour reduction). It is worth noting that the formation of TiC was accompanied by only a small amount of TiO and the corresponding increase in the viscosity was moderate. The results suggested that the existence of a considerable amount of TiO played an important role in causing slag thickening.

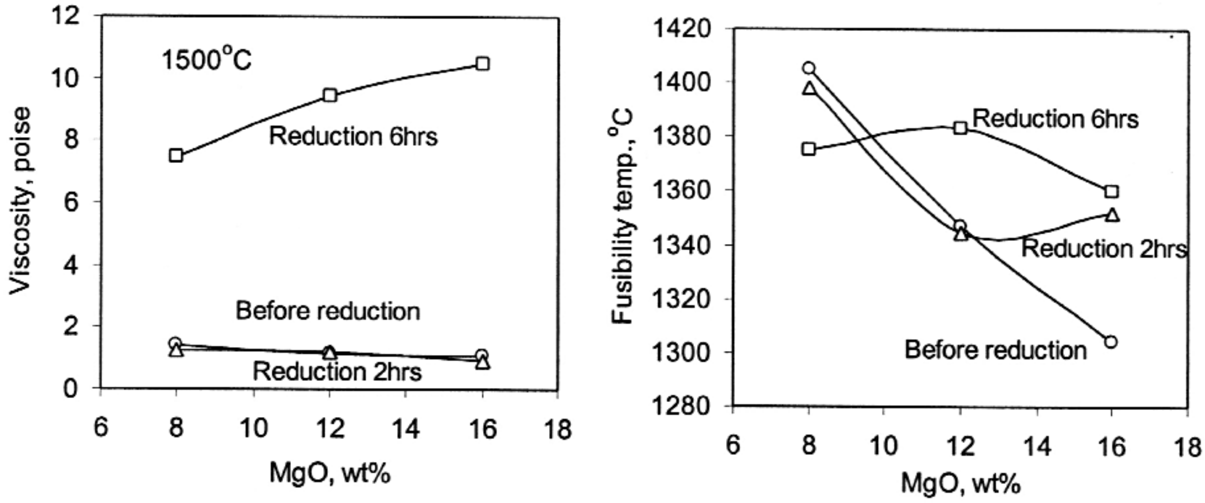
Effect of other components

Effects of Al_2O_3 content varying from 9 to 19 wt% on the reduction and thickening were investigated by Group A (Slags #1, 6 and 7). Increasing Al_2O_3 caused only a slight increase in the viscosity of the slags under neutral and reducing conditions, as shown in Figure 6, and had only some limited effect on accelerating the reduction and thickening of the slag. In general, Al_2O_3 behaved in a similar way to acidic oxide but its effect was much weaker in comparison with SiO_2. The fusibility temperature varied from around 1410°C before reduction to about 1390°C after 6-hour reduction, showing a trend of a gradual decrease with increasing Al_2O_3.

The effects of substituting CaO with MgO while keeping the sum (wt%) of CaO and MgO constant was investigated through the CM group (Slags #1, 8 and 9). The results are shown in Figure 7. Substituting CaO with MgO reduced the slag viscosity under neutral conditions and moderate reduction (Figure 7A), possibly as a result of a net increase in moles of basic oxide when CaO was replaced with MgO on an equal weight basis. A substantial decrease in the fusibility temperature under neutral conditions was observed (Figure 7B) when CaO was replaced by MgO because of a decrease in the CaO/SiO_2 ratio. The fusibility temperature increased during the reduction. Under prolonged reduction, substituting CaO with MgO resulted in a moderate increase in the reduction and thickening. These results were broadly consistent with the effects of decreasing basicity as MgO was slightly weaker than CaO as basic oxide.

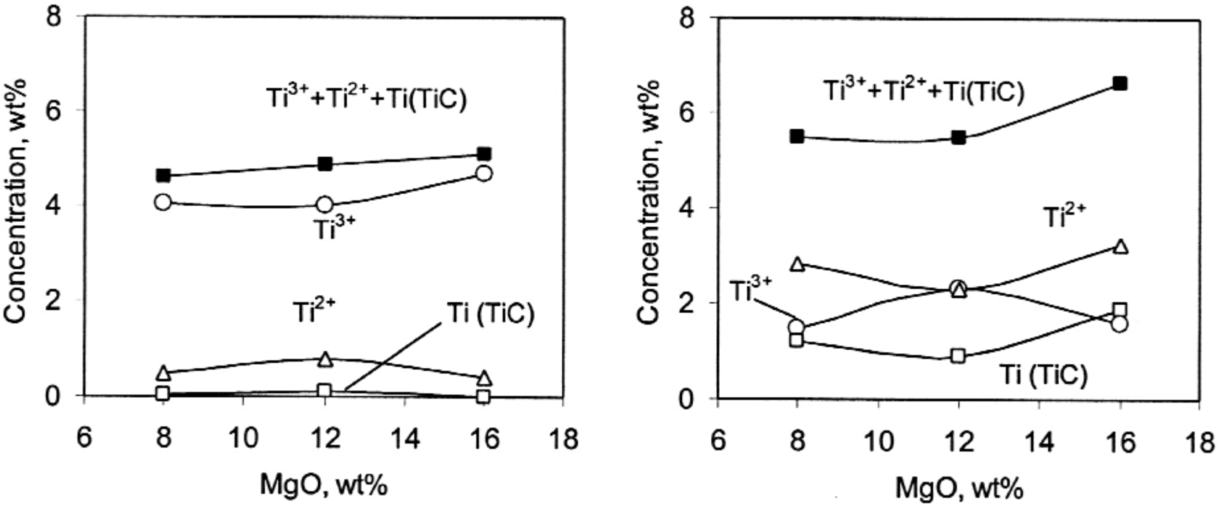
Discussion

The experimental results demonstrated that moderate reduction of TiO_2 slags resulted in the formation of Ti_2O_3 accompanied by a slight decrease in the viscosity and that slag thickening only occurred after prolonged reduction, which produced a small amount (about 1~3 wt%) of TiC,



(A) Viscosity

(B) Fusibility temperature



(C) Reduction 2 hours

(D) Reduction 6 hours

Figure 7. Effects of substituting CaO with MgO on viscosity (A), fusibility temperature (B), and slag composition after reduction for two hours (C) and six hours (D)

particularly in acidic slags. The solid particles suspended in the molten slag did not seem to cause a rapid increase in the fusibility temperature of reduced slag, which in most cases was below or near 1400°C. The flow behaviour of the reduced slags was apparently influenced by the solid particles in a heterogeneous slag phase. It was difficult, however, to explain slag thickening based on the existence of a small amount of TiC. Strictly speaking, the effect of solid particles on liquid viscosity relates to volume proportion of the solid particles (and other factors). Accurate estimate of the volume proportion is, however, not readily available due to complexities in the form and shape of *in situ* formed TiC particles (not as dense crystals).

All thickened slags showed the existence of about 3~7 wt% TiO in addition to 1~3 wt% TiC. The result suggested that TiO might play an important role in the slag thickening. For the highly basic slag ($CaO/SiO_2=1.4$), the formation of TiC (0.5 wt%) was not difficult even after the moderate reduction (when most slags had less than 0.1 wt% of TiC) and, after 6-hour reduction, the appearance of 1 wt% TiC (accompanied by TiO at only 1.8 wt%) did not result in a significant increase in the viscosity.

In a homogeneous molten slag, TiO was found to behave as a basic oxide and was shown to reduce slag viscosity as demonstrated by the work of Handfield *et al.*[8] on the effect of TiO addition on slag viscosity mentioned earlier. The opposite effect observed in the present study, that TiO was associated with slag thickening in a heterogeneous slag, strongly suggested that the TiO, which was formed *in situ*, might be tied up with solid TiC and hence had adverse effect on the viscosity. It is well known that TiO, similar to TiN, can form solid solutions with TiC[15,16]. Present results and observations were consistence with this mechanism although direct evidence on the existence of TiC-TiO solid solution particles in reduced and thickened slags was not readily available due to difficulties in sample preparation and analysis.

The effect of solid particles suspended in a liquid on the viscosity is an extremely complex issue involving many factors such as volume proportion, size and shape of the solid particles, and interaction at the particle surface. Detailed discussion and interpretation are beyond the scope of the present publication. However, as a broad indicator, assuming the formation of a solid solution that retained all

TiO, the amount of the solid particles in the thickened slag would be about 4~9 wt%, which was a factor of 3 higher than that when taking only TiC (1–3 wt%) as solid particles.

Conclusions

The rheological properties of blast furnace slags containing 17~33 wt% TiO_2 under reducing conditions have been studied through measurements of changes in the concentration of reduction products and the slag properties during the reduction process. The results are summarised below.

Slag thickening occurred under prolonged reduction when a considerable amount of both TiO (3~7 wt%) and TiC (1~3 wt%) was formed, particularly in acidic slags. The existence of a considerable amount of TiO suggested that TiO played a key role in causing slag thickening, possibly by forming solid solutions with TiC and effectively causing a significant increase in the amount of solid particles in the heterogeneous slag.

Slag properties (viscosities and fusibility temperatures) and behaviour during the reduction and thickening were dominated by the basicity (CaO/SiO_2). Decreasing basicity resulted in accelerated reduction and thickening.

Reduction proceeded in stages marked by the formation of Ti_2O_3 initially, accompanied by a slight decrease in viscosity, followed by further reduction to TiO and TiC and a significant increase in viscosity.

Fusibility temperature of basic slags decreased and that of acidic slags increased during the reduction. The fusibility temperature of reduced slags was found to be below or near 1400℃ in most cases. The changes in fusibility temperature during reduction were not the reason for slag thickening.

Acknowledgment

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