

Article

The Effect of Titanium Carbonitride on the Viscosity of High-Titanium-Type Blast Furnace Slag

Hongen Xie^{1,2}, Wenzhou Yu^{1,3,*}, Zhixiong You^{1,3}, Xuewei Lv^{1,3} and Chenguang Bai³

- ¹ College of Materials Science and Engineering, Chongqing University, Chongqing 400045, China; pzhxiehongen@163.com (H.X.); youzx@cqu.edu.cn (Z.Y.); lvxuewei@163.com (X.L.)
- ² State Key Laboratory of Vanadium and Titanium Resources Comprehensive Utilization, Pangang Group Research Institute Co., Ltd., Panzhihua 617000, China
- ³ Chongqing Key Laboratory of Vanadium-Titanium Metallurgy and Advanced Materials, Chongqing University, Chongqing 400044, China; bguang@cqu.edu.cn
- * Correspondence: yuwenzhoucd@cqu.edu.cn; Tel.: +86-23-6511-2631

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Abstract: In this paper, the effect of titanium carbonitride (Ti(C,N)) on the viscosity of high-titanium-type blast furnace slags was investigated. The different Ti(C,N) contents were achieved by adjusting the reduction degree of TiO_2 to reflect the real characteristics of the high-titanium slag. The results show that the viscosity of the slag increased with the increasing Ti(C,N) content and decreased with the rising temperature. A deviation between the measured and the fitted viscosity appeared as the content of the Ti(C,N) was beyond 4 wt%. Furthermore, the apparent viscous flow activation energy of the slag ranged from 106.13 kJ/mol to 235.46 kJ/mol by varying the Ti(C,N) contents from 0 wt% to 4.97 wt%, which was evidently different from the results of previous studies. The optical microscope and energy dispersive X-ray spectroscopy (EDS) analysis show that numerous bubble cavities were embedded in the slags and the Ti(C,N) particles agglomerated in the solidified samples. This phenomenon further indicates that the high-titanium slag is a polyphase dispersion system, which consists of liquid slag, solid Ti(C,N) particles and bubbles.

Keywords: blast furnace slag; TiO₂; titanium carbonitride; viscosity

1. Introduction

Viscosity of TiO₂-containing slags is an important factor influencing the processes of ironmaking, steelmaking and Ti-recycling industries [1–3]. The content of titania (TiO₂) in high-titanium-type blast furnace slag is always more than 20 wt%. Therefore, it is inevitable that part of the TiO₂ in this slag was reduced to titanium carbonitride (Ti(C,N)) particles by coke during blast furnace production. The Ti(C,N) particle is harmful for the fluidity of the slag because it has a high melting point and can significantly increase the viscosity of the slag. According to Einstein's theory [4] about the relation between the volume fraction of solid particles and the viscosity of extremely dilute solutions, Roscoe presented a type of equation (Einstein–Roscoe type equation) to describe the viscosity of liquids containing high concentrations of solid suspensions [5]:

$$\eta = \eta_0 (1 - af)^{-n} \tag{1}$$

where η and η_0 are the viscosity of solid-containing and solid-free liquid, respectively; *f* is the volume fraction of solid particles in the liquid; *a* and *n* are constants with regard to the volume fraction and geometrical shape of solid particles in liquid, and are 1.35 and 2.5 for spherical particles with a uniform size, respectively. This equation indicates that the viscosity of the melt should be related to



the volume fraction and geometrical shape of solid particles in liquid. To explore this relationship in metallurgical slags, some research has been carried out by adding small amounts of solid particles to the slags. Wright et al. [6] studied the viscosities of CaO-MgO-Al₂O₃-SiO₂ melts containing spinels with different sizes at 1646 K; Liu et al. [7] studied the effect of Ti(C_{0.3}N_{0.7}) particles of 1.0 μ m on the viscosities of CaO-MgO-Al₂O₃-SiO₂ blast furnace slag and Zhen et al. [8] discussed the effect of TiC particles on the viscosity of CaO-MgO-Al₂O₃-SiO₂-TiO₂ slag. Their results suggested that the viscosity of the solid-containing melt increased with the addition of particles, and the Einstein–Roscoe type equation can well describe the viscosity variation behavior by allowing the parameters *a* and *n* to vary. To investigate the flow behavior of high-titanium-type slag, Jiang et al. [9] studied the effect of TiC solid particles on the rheological behavior of Ti-bearing blast furnace slag with different TiN contents. Both of them pointed out that the slags will convert to non-Newtonian fluids if the volume fraction of the solid particles beyond certain values and the Einstein–Roscoe type equation could be not suitable at that condition.

In the last few decades, a large amount of research [11–19] has attempted to establish an accurate description about the viscosity of the high-titanium-type slag and a lot of fruitful achievements have been obtained. However, the existing empirical and semi-empirical models still cannot describe the viscosity precisely. One of the possible reasons for this may be the improper method for preparing the experimental slag. For example, most of the previous studies prepared the high-titanium-type slag by adding the solid particles directly to the TiO₂-containing slags. However, this could not be enough to reflect the real characteristics of the on-site slag (slag in blast furnace). The morphology and distribution of TiC, TiN, and Ti(C,N) in on-site slag should be different from those directly added to the slag. Additionally, on-site slag is also different from the slag prepared by high purity reagents because there are some gas bubbles in on-site slag, which makes the structure of molten slag more complicated. Up to now, there is still a lack of accurate knowledge of viscosity properties for the high-titanium-type blast furnace slag. In order to control the iron-making process of titanium-vanadium-magnetite more efficiently, a further understanding of flow behaviors in high-titanium-type blast furnace slag should be necessary.

In the present study, to discuss the effect of titanium carbonitride on the viscosity of the high-titanium-type blast furnace slag, on-site slags with different contents of titanium carbonitride (reduced from TiO_2) were prepared. Additionally, the viscosities of these slags were measured to clarify the relationships between slag fluidity and the contents of titanium carbonitride.

2. Materials and Methods

On-site blast furnace slags were used as the raw materials in the experiment. The residual metal iron in the slag was removed by magnetic separation, and then the slag was crushed to less than 0.1 mm. The chemical compositions of the slags were analyzed by X-ray fluorescence spectrometry (XRF, Shimadzu XRF-1800, Kyoto, Japan), as shown in Table 1. Coke was employed as the reductive agent for reducing the TiO₂ to Ti(C,N) in the experiment (compositions are shown in Table 2), which the particle size was controlled between 10 mm and 15 mm.

CaO	SiO ₂	MgO	Al ₂ O ₃	TiO ₂	TiC	TiN
25.12	24.63	9.50	14.00	22.50	0.49	0.50

Table 1. Chemical compositions of on-site slag, mass%.

Table 2. Chemical con	positions of the	coke, mass%.
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FCad	St	Vadf	Ad	CaO	SiO ₂	MgO	Al ₂ O ₃	Fe ₂ O ₃
86.26	0.54	1.23	12.67	0.49	7.01	0.20	3.05	0.95

A muffle furnace (Teenpu CO. LTD., Jiangyin, China) was used to prepare experimental slags with different Ti(C,N) contents. The experimental schematic diagram is shown in Figure 1. The heating element is a U-shaped silicon molybdenum rod, the heating process is controlled by PID (Proportion Integral Differential) program, and the temperature is measured by a B-type thermocouple. About 140 g coke was firstly placed in a graphite crucible (52 mm inner diameter, 160 mm length), and about 210 g slag was placed on the top of the coke. The graphite crucible was then put into a larger corundum crucible to reduce the oxidation degree of the graphite crucible during the experiment. The temperature of the chamber increased to 1773 K at 10 K/min, and then held for 15 min, 30 min, 45 min and 60 min, respectively. After that, the samples were cooled down together with the furnace. The residual cokes in the slags were separated using a hammer and cleaned up by hairbrush. The obtained slags (experimental slags) were crushed to less than 0.1 mm, some of which was used to analyze the chemical composition, and the rest was used to measure the viscosity. The contents of TiC and TiN in the experimental slags were obtained by chemical analysis, and the results are shown in Table 3. The content of Ti(C,N) could not be obtained directly by chemical analysis, thus it was represented by the total contents of TiC and TiN. Number 1 in Table 3 indicates the raw slag without reduction by coke, which the content of Ti(C,N) was 0.99%. The Ti(C,N)-free slag was obtained by roasting the raw slag at 1573 K for 60 min, as represented by 0 in Table 3. The contents of TiC and TiN in this slag were lower than 0.001 mass%, respectively. In order to further confirm the composition of the morphology in the slag, a microstructure analysis was carried out by using the optical microscope (OLYMPUS BX51, Hatagaya, Japan) and scanning electron microscopy with energy dispersive X-ray spectroscopy (SEM-EDS) (FEI, Hillsboro, OR, USA), and the results are shown in Figure 2 and Table 4.



Figure 1. Schematic diagram of the muffle furnace.

Samples	Time/min	TiC	TiN	Ti(C,N)
0	-	< 0.001	< 0.001	0
1	-	0.49	0.50	0.99
2	15	1.08	1.09	2.17
3	30	1.79	1.55	3.34
4	45	2.01	2.04	4.05
5	60	2.16	2.81	4.97

Table 3. The contents of TiC and TiN in experimental slags, mass%.



Figure 2. Microstructure of the slag sample (A is Perovskite, B is Tianium carbonitride, C is metal iron.).

Points	С	Ν	0	Ca	Ti	Fe
Point A	-	-	34.75	29.33	35.92	-
Point B	10.77	5.94	-	-	83.29	-
Point C	-	-	-	-	-	100.0

Table 4. Energy dispersive X-ray spectroscopy (EDS) analysis of the points in Figure 2, mass%.

The EDS (EDAX, Mahwah, NJ, USA) analysis (Table 4) shows that lots of brick-red Ti(C,N) particles were produced in the slag. Additionally, the white iron phase and the grey/white perovskite phase were also observed in the slag.

A schematic diagram of the viscosity measurement apparatus is shown in Figure 3, which includes a Brookfield digital viscometer, a heating system, an automatic lifting system, a temperature control system, and a gas supply system. The heating element is a U-shaped MoSi₂ rod, which has a maximum working temperature of 1923 K. The inner diameter of the high purity alundum tube of the shaft furnace is 55 mm, and the height of the constant temperature zone is about 60 mm. The temperature is controlled by PID program, and temperature is measured by a B-type thermocouple with Pt-6 wt pct Rh/Pt-30 wt pct Rh. Argon gas with a purity of 99.99% was employed as the protection gas. During the experiment, the argon gas flowed in from the bottom of the alundum tube and flowed out from the top, and the gas flow was maintained at 1.5 L/min.



Figure 3. Schematic diagram of the viscosity measurement apparatus.

The viscosity of experimental slag was measured by the rotating-cylinder method. About 170 g experimental slag was put into a high purity graphite crucible (50 mm outer diameter, 40 mm inner

diameter, 120 mm length). A metal molybdenum sheet with a thickness of 0.01 mm was put closely inside the graphite crucle to avoid the reaction between slag and graphite. This graphite crucible was put into another larger graphite crucible (62 mm outer diameter, 52 mm inner diameter, 160 mm length) to prevent slag from spilling to damage the alundum tube. The molybdenum spindle (15 mm diameter and 20 mm height) was connected with a molybdenum rod (5 mm diameter and 475 mm length), which linked with the viscometer by a piano wire (carbon spring steel wire). After the molybdenum spindle was slowly immersed into the slag to the predetermined depth, the total height of the liquid slag was about 50 mm. The viscometer was calibrated using three kinds of standard liquids, of which the viscosities were 222.41 mPa \cdot s, 528.20 mPa \cdot s and 1073.3 mPa \cdot s at 293 K respectively. The viscosity was measured when the temperature reached 1773 K. The rotation speed of the spindle was controlled at 12 r/min. The measurement time was 30 min and two viscosity. After the measurement at 1773 K, the temperature was decreased to 1733 K, 1693 K and 1653 K, respectively, and the viscosity at these temperatures was measured by the similar methods.

3. Results and Discussion

3.1. Effect of Ti(C,N) on Viscosity of High-Titanium-Type Blast Furnace Slags

The viscosities of the slags are shown in Table 5. It can be seen that the viscosities of the slags increased as temperature decreased. Simultaneously, with the increase of Ti(C,N) content at the same temperature, the viscosities of the slags increased gradually. This indicates that the viscosity of high-titanium-type blast furnace slags can be influenced not only by temperature, but also by the content of Ti(C,N) particles.

Samples	1653 K	1693 K	1733 K	1773 K
0	0.194	0.166	0.145	0.123
1	0.289	0.233	0.201	0.170
2	0.570	0.391	0.324	0.272
3	1.052	0.656	0.553	0.452
4	1.794	1.170	0.927	0.783
5	3.178	1.489	1.233	1.017

Table 5. Viscosities of the slags, Pa·s.

In the blast furnace ironmaking process of titanium-vanadium-magnetite, it is inevitable that a lot of Ti(C,N) particles are produced. When the Ti(C,N) particles enter the slag, the viscosity of the high-titanium-type blast furnace slag will be influenced significantly. By modifying *a* and *n* in Equation (1), or only *a* and fixed *n*, the dependence of the measured viscosity on the content of the solid particles could be described by the Einstein–Roscoe type equation. The constant *n* is related to the geometrical particle shape and can be assumed to be 2.5 for spherical solid particles. The reciprocal value of *a* represents the maximum amount of solid (f_{max}) that the melt could accommodate before the viscosity becomes "infinite". By fitting the measured values using the Einstein–Roscoe type equation, the values of *a* can be optimized and the maximum amount of solid (f_{max}) can be obtained, and the results are shown in Table 6. The measured and fitted viscosities of the slags are shown in Figure 4.

Table 6. Values for parameters *a*, *f_{max}* and apparent volume fraction at different temperatures.

Parameters	1653 K	1693 K	1733 K	1773 K
п	2.5	2.5	2.5	2.5
а	13.79	12.25	12.02	11.93
fmax	0.072	0.082	0.083	0.084



Figure 4. The measured (scattered points) and fitted (lines) viscosities vs. the content of Ti(C,N).

It can be seen from Figure 4 that the higher the content of Ti(C,N) in the slag, the greater the deviation between the fitted viscosity and the measured viscosity. This becomes obvious when the content of Ti(C,N) was over 4 wt%. The main reason for this phenomenon is that the molten slag changed to a non-Newtonian fluid and a shear thinning behavior appeared as the content of the solid particles rose beyond a certain value [9]. It was found from Table 6 that the values of the maximum volume fraction of solids (f_{max}) in this study should be significantly lower than that in the studies of Liu [7] and Zhen [8]. The possible reason for this is that the rotation speed of the spindle in this study (12 r/min) was lower than that used in the other studies (>100 r/min).

However, in addition to the content of Ti(C,N), the influence of bubbles on the viscosity of the on-site slag should not be ignored. It is well known that the smelting of high-titanium-type blast furnace slag can always be accompanied with the formation of bubbles (foam slag). If there are too many bubbles in the molten slag, even if the content of titanium carbonitride is low, the viscosity of the slag will be high [20]. In most cases, the bubbles in the slag were not enough to turn the slag into foam slag. However, there are always more or less bubbles in the slag. The high-titanium-type blast furnace slag should be regarded as a polyphase dispersion system, which consists of liquid slag, solid Ti(C,N) particles, and bubbles. When the foam characteristic value was less than 0.74, the viscosity of the foam can be expressed as [21]:

$$\eta = \eta_0 (1 + 4.5\Phi) \tag{2}$$

where η_0 is the viscosity of foam-free liquid and Φ is the foam characteristic value.

Figure 5 shows the microstructure of the solidified slag. It demonstrated that some micro bubble cavities were embedded in the slag, which proves that the bubbles can stably exist in the slag even though the slag is cooled down. This may be another reason that the deviation between the measured viscosity and the fitted viscosity by Einstein–Roscoe type equation.



Figure 5. Optical micrograph of the bubble cavity in slag.

3.2. The Effect of Ti(C,N) on the Apparent Viscous Flow Activation Energy E_{η}

The relation between the viscosity of slag and the temperature was usually expressed by Weymann–Frenkel's equation [22]:

$$\eta = A \cdot T \cdot \exp(E_{\eta} / (RT)) \tag{3}$$

where *A* is a proportionality constant, E_{η} is the apparent activation energy for viscous flow, *R* is the gas constant and *T* is the absolute temperature.

According to Equation (3), the curve representing the relationship of $\ln(\eta/T)-10^5/T$ is shown in Figure 6. The scattered points are the measured results, and the lines are the fitted results according to Weymann–Frenkel equation. It can be seen that the relationships between $\ln(\eta/T)$ and $10^5/T$ of slags 0 and 1 were almost completely linear, while those for slags 2 to 5 were gradually deviated from the Weymann–Frenkel equation. When the temperature decreased from 1693 K to 1653 K, the differences between the measured values and the fitted values were even greater.



Figure 6. Relationships between $\ln(\eta/T)$ and $10^5/T$ of slags 0 through 5.

The apparent viscous flow activation energies at different contents of titanium carbonitride are shown in Table 7. As the content of titanium carbonitride increases, the apparent viscous flow activation energy increased significantly. This is different from the results of Zhen et al. [8], which showed the apparent viscous flow activation energy experienced no change with increases in the volume fraction of solid particles. It is well known that the apparent activation energy represents the frictional resistance for viscous flow. In Zhen's [8] study, the solid particles were added to the slag, and the interaction between the solid and the slag should be weak. Thus, they suggested that the composition of liquid slag should be the main factor affecting the activation energy of a suspension system. However, in our study, the Ti(C,N) particles in the slag were produced by reducing TiO₂, and thus the interaction between the solid and the slag should be strong. The strong interaction can result in a more complicated structure in the real slag. Therefore, much more energy should be required to overcome the viscous fluid activation energy as the content of Ti(C,N) particles increases.

Table 7. Apparent viscous flow activation energy at different total contents of Ti(C,N), kJ/mol.

Parameters	0	1	2	3	4	5
E_{η} , kJ/mol	106.13	120.30	161.41	179.73	180.61	235.49
Adjusted R-Square	0.9978	0.9953	0.9605	0.9329	0.9527	0.8440

3.3. The Distribution of Ti(C,N) in Slag

Figures 7–9 show the microstructures of slag samples quenched in different ways (cooling with liquid nitrogen, water cooling, and natural cooling, respectively). The microstructure was analyzed using the optical microscope (OLYMPUS BX51, Tokyo, Japan) and the phase composition

was confirmed by SEM-EDS. It can be seen that the perovskite phase, metal iron and Ti(C,N) particles were observed in the solidified samples. It is well known that the perovskite phase has a high melting point and can precipitate preferentially from the melt during the cooling process. When decreasing the cooling rate, the crystal size of the perovskite gradually increased, as shown in Figures 7–9. Additionally, a significant agglomeration of Ti(C,N) particles were seen in the slag no matter which cooling methods were adopted. As compared to the samples of water cooling and cooling with liquid nitrogen, the agglomeration of Ti(C,N) particles was more serious in the natural cooling samples. The solid particles adhered together to form the large agglomeration area. The distribution of titanium carbonitrides in this study were evidently different from those works of Liu, Zhen and Wright et al. [6–8], in which solid particles were evenly distributed in the slag. This implies that the properties of the real slag differs from that of synthetic slag. Additionally, it can be seen in Figures 7–9 that the observed Ti(C,N) particles were always adhered with the embedded metallic iron in the slag. The main reason for this phenomenon is that the TiO₂ in the slag can react with the carbon dissolved in the metallic iron. This also proves that the high-titanium-type blast furnace slag has a complicated structure and the Ti(C,N) particles have a strong interaction with the slag, which should be the main influence on the viscosity of the slag.



Figure 7. Microstructure of slag sample quenched in liquid nitrogen.



Figure 8. Microstructure of slag sample quenched by water cooling.



Figure 9. Microstructure of slag sample quenched by natural cooling.

4. Conclusions

This study investigated the viscosity characteristics of high-titanium-type blast furnace slags. Slag samples containing different contents of Ti(C,N) particles were prepared, and the influences of Ti(C,N) on the viscosity of the slag were studied in detail. The main findings can be summarized as follows:

(1) When increasing the content of Ti(C,N), the viscosity of slag samples gradually increased. The measured viscosity and the fitted value by Einstein–Roscoe type equation deviated gradually as the content of Ti(C,N) increased. Additionally, the apparent viscous flow activation energy of the slag also increased with increasing the content of Ti(C,N).

(2) Microbubbles can exist in the slag stably and a serious agglomeration of Ti(C,N) particles was observed in the slag samples. Thus, the high-titanium-type blast furnace slag is actually a polyphase dispersion system, which consists of liquid slag, solid Ti(C,N) particles, and bubbles.

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