Viscosities of Iron Blast Furnace Slags

Mao CHEN,¹⁾ Dianwei ZHANG,²⁾ Mingyin KOU^{1,3)} and Baojun ZHAO^{1)*}

1) School of Chemical Engineering, The University of Queensland, Brisbane, Australia.

2) Shougang Research Institute of Technology, 69 Yangzhuang Street, Beijing, China.

3) School of Metallurgical and Ecological Engineering, University of Science and Technology Beijing, Beijing, China.

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The viscosities of the industrial and synthetic iron blast furnace slags have been measured using a custom designed rotating bob apparatus. This advanced apparatus enables control of the gas atmosphere and rapid quenching of the samples on completion of the viscosity measurements. The experiments were carried out using Mo spindle and crucible under ultra-high purity Ar flow. The microstructures and phase compositions of the quenched slag samples were determined by Electron Probe X-ray Microanalysis (EPMA) after the viscosity measurements.

It was found that the viscosities of the industrial slags are lower than those of the corresponding synthetic slags made from pure Al_2O_3 , CaO , MgO and SiO_2 . The difference in viscosities between industrial slags and synthetic slags will provide useful indications when applying the results of the synthetic slags to the real iron blast furnace slags. The activation energy of both industrial and synthetic slags is 174 kJ/mol and increases with decreasing MgO concentration. Replacement of $(\text{CaO}+\text{SiO}_2)$ or CaO by MgO can decrease the slag viscosity. The viscosity of iron blast furnace slag at 1500°C can be estimated by $V = 0.005 + 0.0262[\text{SiO}_2] + 0.0184[\text{Al}_2\text{O}_3] - 0.0172[\text{CaO}] - 0.0244[\text{MgO}]$ which is derived from the present and previous data.

KEY WORDS: ironmaking; blast furnace slags; viscosity; Al_2O_3 – CaO – MgO – SiO_2 .

1. Introduction

The principal components of current iron blast furnace (BF) slags are described by the system Al_2O_3 – CaO – MgO – SiO_2 .^{1,2)} The targets of the optimum slag compositions are to minimise slag mass and operating temperature whilst satisfying requirements for removal of sulphur and alkali, and slag tapping.²⁾ In modern ironmaking operations, the hot metal and slag temperatures can be controlled within a narrow range to obtain stable operating condition of the blast furnace. More accurate data on the physiochemical properties of the slag, such as viscosity, are required to optimise the operation of the blast furnace.

A large number of data on viscosity of the synthetic Al_2O_3 – CaO – MgO – SiO_2 slags have been reported.^{3–10)} However, significant discrepancies exist for the reported viscosity data due to the difficulties of high temperature viscosity measurement. In addition to the major components Al_2O_3 , CaO , MgO and SiO_2 , up to 5 wt% of other components, such as S, TiO_2 , MnO and FeO may also be present in the BF slags.¹¹⁾ It is important to understand the difference between the actual slags and the synthetic slags before the viscosity data of the synthetic slags can be applied in blast furnace operations.

High temperature viscosity measurements are expensive and time-consuming. Different viscosity models^{12–18)} have

been proposed to predict the viscosities of BF slags in wide composition range. As significant discrepancies are present for the reported viscosity data, these viscosity models are yet to be developed to accurately predict the viscosities of BF slags.

The aim of the present study is to compare the viscosities of BF slags from Shougang and corresponding synthetic slags and also evaluate the accuracy of the existing viscosity models. The systematic and accurate viscosity data will be obtained to optimise the existing viscosity databases and improve the reliability of the modified quasi-chemical viscosity model.

2. Experimental

A high-temperature viscosity measurement apparatus was constructed and commissioned at The University of Queensland, Australia^{19,20)} (Fig. 1).

A Brookfield digital rotational rheometer (model LVDV III Ultra) controlled by a PC was used in the present study. A Pyrox furnace with lanthanum chromite heating elements (maximum temperature 1650°C) and 90 mm uniform temperature zone was employed. The ID of the recrystallised alumina reaction tube is 50 mm. The rheometer placed on a movable platform was enclosed in a gas-tight steel chamber. The rheometer rotated co-axially the alumina driving shaft (8 mm OD) with the cylindrical spindle. The crucible was suspended using an alumina tube (38 mm OD) and held onto the tube with Pt wires and positioned within the hot zone of

* Corresponding author: E-mail: baojun@uq.edu.au

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Fig. 1. Schematic of the high temperature viscosity measurement apparatus used in this study.

the Pyrex tube furnace. A Pt-6Rh/Pt-30Rh (B type) thermocouple was bound to the suspension tube platform, the tip of which remained adjacent to the crucible at the level where the spindle rotated. This enabled accurate temperature of the slag to be measured during the viscosity experiments. The measurements of viscosity were usually carried out in the order from high temperature to low temperature in 50 degree interval. At each temperature, the sample was kept for enough time to ensure the chemical equilibration has been achieved which was confirmed by the stable torque readings. The lowest measuring temperature was limited by the liquidus temperature of the slag.

The main feature of the current experimental setup with top suspension was the possibility of quenching the slag sample immediately after the viscosity measurement. This is important to enable the microstructure of the slag and compositions of the phases present in the slag at the temperature to be characterised directly after the viscosity measurement. In this way the uncertainty associated with the changes of the slag composition during the measurements can be minimised. In addition, the presence of crystal and composition change of the liquid due to the precipitation of the crystal can also be identified precisely.

The viscosity of the fluid can be determined by:

$$\eta = K \frac{M_s}{\Omega} \quad \dots\dots\dots (1)$$

where η [Pa.s] is viscosity of a Newtonian fluid, K is the instrument constant, M_s [N m⁻¹] is the measured torque at a given rotation speed Ω [m.s⁻¹]. The equipment constant K ,

Table 1. Chemicals used in the present study.

Materials	Purity (wt pct)	Supplier
Silicon oxide (SiO ₂)	99.9	Sigma-Aldrich (St. Louis, MO, US)
Calcium Carbonate (CaCO ₃)	99.95	Alfa Aesar (Ward Hill, Massachusetts, US)
Aluminium oxide	99.8	Alfa Aesar (Ward Hill, Massachusetts, US)
Magnesium oxide	99.9	Sigma-Aldrich (St. Louis, MO, US)
Mo spindle/crucible	99.9	Zhuzhou Cemented Carbide Works (Zhuzhou, China)

which is a function of the spindle/crucible geometries and the rheometer, can be determined from calibration of the apparatus with fluids of known viscosity. The viscosities of the standard fluids supplied by Brookfield Engineering Ltd are 0.0092, 0.0484, 0.0968, 0.51 and 0.96 Pa.s that cover the range of the slag viscosities to be measured. The same set of Mo crucible and spindle after calibration was used in the high temperature measurement in argon gas atmosphere. Compositional analyses of the quenched samples showed that negligible amount of MoO₃ (less than 0.3 wt%) was present in the slags. Inspection of the crucible and spindle after the viscosity measurements confirmed that the geometries of the spindle and crucible did not change at high temperatures.

Approximately 35 ml of melted slag was required for each viscosity measurement. Granulated industrial BF slag was used directly for viscosity measurement. Synthetic slag was prepared from pure powders of SiO₂, CaO (calcined from CaCO₃), Al₂O₃ and MgO with desired proportions and pelletised. The chemicals used are listed in Table 1. The pelletised sample was melted under ultra-high purity argon (O₂ < 1 ppm, H₂O < 2 ppm) and the volume of the melt was measured so that the density of the slag can be estimated. The sample was usually melted at a temperature 50°C higher than the liquidus temperature which can be predicted by FactSage 6.2.^{21,22)} After melting, the crucible with sample was slow cooled to room temperature for high temperature measurement.

When the viscosity measurements have been completed, the sample was dropped into water so that phase assemblages at temperature can be retained. The quenched samples were sectioned, mounted, polished and carbon coated for EPMA measurement. A JXA 8200 Electron Probe Microanalyser with Wavelength Dispersive Detectors was used for microstructure and composition analyses. The analysis was conducted at an accelerating voltage of 15 kV and a probe current of 15 nA. The standards used for analysis were supplied by Charles M Taylor Co., Stanford, California, USA: CaSiO₃ for Si and Ca, Al₂O₃ for Al, Fe₂O₃ for Fe, TiO₂ for Ti, MgO for Mg, Mn₃Al₂Si₃O₁₂ for Mn, CuFeS₂ for S and CaMoO₄ for Mo. The ZAF correction procedure supplied with the electron probe was applied. The average accuracy of the EPMA measurements was within 1 wt%.

The uncertainties associated with the viscosity measurements have been discussed in a previous study.¹⁹⁾ It was found that

- 1) The total weight of the spindle used for the rheometer needs to be controlled below 210 g;
- 2) The gap between spindle and crucible wall needs to be greater than 3 mm;
- 3) The distance between the spindle tip and bottom of the crucible needs to be greater than 8 mm;
- 4) The error associated with the thermal expansion of the Mo spindle is below 3 pct.

All efforts have been made to reduce the possible errors associated with high temperature viscosity measurements.

3. Results

Viscosity measurements have been carried out for one industrial blast furnace slag SG1 and three synthetic slags (BF1, BF2 and BF3). The compositions of BF1, BF2 and BF3 were prepared to have constant 17 wt% Al_2O_3 and basicity (CaO/SiO_2) equal to 1.1 with different MgO concentrations in the slag. Synthetic slag BF3 has a composition close to the industrial slag SG1 so that effects of minor components such as FeO, TiO_2 and sulphur on viscosity can be evaluated. All samples were quenched directly after the viscosity measurements at lowest measuring temperature. Inspection of quenched samples confirms that all viscosity measurements were carried out above the liquidus temperatures so that only liquid phase was present during the viscosity measurements (Fig. 2).

The compositions of industrial and synthetic slags were analysed by EPMA after the viscosity measurements (see Table 2). It can be seen from Table 2 that in addition to the major components Al_2O_3 , CaO, MgO and SiO_2 , 0.4 wt% FeO, 0.7 wt% TiO_2 and 1.2 wt% S are also present in the

Fig. 2. Backscattered scanning electron micrographs of quenched SG1 samples after viscosity measurement at 1420°C.

Table 2. The measured compositions by EPMA analysis.

Sample	CaO/ SiO ₂	Measured compositions (wt%)								
		SiO ₂	CaO	Al ₂ O ₃	MgO	FeO	TiO ₂	MnO	S	MoO ₃
SG1	1.15	34.1	39.2	16.1	8.1	0.4	0.7	0.2	1.2	0
BF1	1.1	38	41.6	17.2	3	0	0	0	0	0.2
BF2	1.1	36.5	40.2	17.2	5.8	0	0	0	0	0.2
BF3	1.1	35	38.5	17.3	8.9	0	0	0	0	0.3

BF slag. Less than 0.3 wt% MoO_3 was present in the synthetic slags and no Mo was detected in the industrial slags.

The measured viscosities are listed in Table 3 and plotted in Fig. 3 as a function of temperature. It can be seen that SG1 has the lowest viscosities and BF1 has the highest viscosities in the temperature range investigated.

Table 3. The measured viscosities in the present study.

Sample	Temperature (°C)	Viscosity (Pa.s)
SG1	1420	0.510
	1450	0.406
	1500	0.288
	1550	0.207
	1600	0.156
BF1	1400	1.121
	1450	0.702
	1500	0.482
	1525	0.399
	1550	0.352
	1575	0.295
BF2	1400	0.891
	1450	0.581
	1475	0.478
	1500	0.408
	1525	0.335
	1550	0.290
BF3	1575	0.247
	1425	0.580
	1450	0.479
	1475	0.395
	1500	0.341
	1525	0.286
	1550	0.250
	1575	0.211

Fig. 3. Viscosities of the measured samples as a function of temperature.

The temperature dependence of the viscosity for a given slag composition can be described by Arrhenius-type equation.²³⁾

$$\eta = A \exp\left(\frac{E_a}{RT}\right) \dots\dots\dots (2)$$

where η is the viscosity in Pa.s, T is the absolute temperature in K, R is the gas constant (8.314 J/mol.K). The temperature-independent parameters A and E_a are the pre-exponential factor and the activation energy (kJ/mol), respectively. The Arrhenius-type equation can be re-arranged after taking logarithm:

$$\ln \eta = A + \frac{E_a}{RT} \dots\dots\dots (3)$$

Equation (3) shows that $\ln \eta$ has a linear relationship with $1/T$ and the slope of the line is the activation energy E_a . It can be seen from **Fig. 4** that all slags measured in the present study follow the Arrhenius behaviour.

Activation energies derived from Fig. 4 are 174, 194, 188 and 174 kJ/mol for SG1, BF1, BF2 and BF3, respectively. It seems that presence of the minor components such as FeO, TiO₂ and S in the slag (SG1) does not affect the activation energy. The activation energy decreases with increasing MgO concentration in the slag.

4. Discussions

4.1. Viscosities of Industrial BF Slags

Experimentally determined viscosities of the Baosteel blast furnace slags²⁴⁾ are compared with the present results (**Fig. 5**). It can be seen that the viscosities of Baosteel and Shougang BF slags are in the range of 0.25–0.4 Pa.s at temperatures between 1 530 and 1 470°C. This indicates that the blast furnace slags in Baosteel and Shougang have good fluidity at normal operating temperatures if solid phases are not present.

4.2. Comparison of Viscosities between Industrial Slag and Synthetic Slag

One of the aims in the present study was to investigate the difference between industrial slag and synthetic slag on vis-

cosity. Most of BF slag viscosity has been investigated for four-component slag Al₂O₃–CaO–MgO–SiO₂. Comparison of the viscosities between industrial slag and synthetic slag will enable the measurements and predictions of the viscosities of the synthetic slags to be directly used for industrial BF slags. It is clearly seen from Fig. 3 that the viscosities of the industrial slag SG1 are approximately 20% lower than its corresponding synthetic slags BF3 at a given temperature. It is well known that FeO and MnO can decrease the viscosity.^{25–28)} TiO₂ was also reported to decrease the viscosity based on the experimental study carried out by Shankar *et al.*⁹⁾ and Park *et al.*²⁹⁾ Effect of sulphur on the viscosity of BF slag is still not clear. Lower viscosities of the industrial BF slag comparing to the synthetic slags are combined results of all minor components. Further study is required to investigate individual effect of the minor components on viscosity of BF slags.

4.3. Effect of MgO on Viscosity of BF Slag

The viscosities of the synthetic slags BF1, BF2 and BF3 were measured to evaluate the effect of the substitution of (CaO+SiO₂) by MgO in the slag while controlled a constant Al₂O₃ concentration and CaO/SiO₂ ratio of 1.1 (Table 1). **Figure 6** shows that the viscosity gradually decreases with the increase of MgO and the effect is more significant at low temperature. CaO/SiO₂ ratio in BF slag is usually fixed to maintain the sulphur capacity of the slag. Besides SiO₂, Al₂O₃ may also increase viscosity with association of basic oxides, such as CaO,^{30–32)} which is abundant in the BF slags. The mechanism can be explained as the “charge compensation effect”,³³⁾ in which Al³⁺ can be charge compensated by Ca²⁺ and Mg²⁺ to continue the silicate network. The enriched CaO in BF slag can fully charge compensate the Al₂O₃, so that increased Al₂O₃ concentration in the BF slag results in higher viscosity. One of the strategies to decrease viscosity of the BF slag is to add more MgO in the feed while the CaO/SiO₂ ratio keeps the same. Accordingly, (CaO+MgO)/SiO₂ ratio in BF slag and slag mass will be increased.

Fig. 4. Linear relationship between $\ln \eta$ and $10\,000/T$.

Fig. 5. The comparison of viscosities of SG1 from Shougang slag with Baosteel industrial slags.²⁴⁾ (A5: SiO₂ 35.6, CaO 40.9, Al₂O₃ 15.5, MgO 8.0 wt%; A6: SiO₂ 34.9, CaO 40.1, Al₂O₃ 17.0, MgO 8.0 wt%).

Fig. 6. The comparison of present results at temperature 1450, 1500 and 1550°C.

Fig. 7. Comparison of present results with data from Hofmann⁶⁾ and Machin.⁴⁾

4.4. Viscosities between Synthetic Slags

The viscosities are compared for the synthetic slags between the present study and previous data (Fig. 7). The viscosities measured by Hofmann⁶⁾ and Machin⁴⁾ (Table 4) have the compositions close to the present study. The liquidus temperatures of these samples were predicted by Factsage 6.2.^{21,22)} It can be seen from Fig. 7 that some of the measurements were carried out below the predicted liquidus. However, presence of the solid in the slag during the viscosity measurements was not mentioned in these studies.^{4,6)} The slag compositions reported by Hofmann⁶⁾ and Machin⁴⁾ were designed to keep the constant SiO₂ and Al₂O₃ concentrations, and also constant (CaO+MgO)/SiO₂ ratio. It can be seen from H1 to H3, and M1 to M2 that, substitution of CaO by MgO can decrease the slag viscosity. The compositions of H3,⁶⁾ M2⁴⁾ and BF3 are close. But it can be seen from Fig. 7 that the viscosities of BF3 and M2⁴⁾ are much higher than that of H3⁶⁾ at a given temperature. This demonstrates that not all viscosity data reported previously are accurate. It is important to use only accurate viscosity data to assist in operation and viscosity model development.

4.5. Sensitivities of Viscosity to Major Components

BF slag can be represented by four-component system Al₂O₃–CaO–MgO–SiO₂. It would be easy to control slag composition if the viscosity can be predicted from slag chemistry. However, a complete mathematical viscosity model requires good theoretical base and a large number of accurate measurements. As a first approximation, the following empirical correlation is obtained from the present viscosity data and data reported by Machin⁴⁾ at 1500°C:

$$V = 0.005 + 0.0262[\text{SiO}_2] + 0.0184[\text{Al}_2\text{O}_3] - 0.0172[\text{CaO}] - 0.0244[\text{MgO}] \quad \text{..... (4)}$$

Where [SiO₂], [CaO], [Al₂O₃] and [MgO] are in weight percent and viscosity is in Pa.s. The composition range for the regression includes CaO 25–50, SiO₂ 30–45, MgO 0–15 and Al₂O₃ 5–25 wt%. The overall error range of the coefficients in Eq. (4) is estimated to be less than 10%. The parameters in front of each component indicate the positive and negative effects and their significances to the viscosity. It can be seen from Eq. (4) that the increase of the viscosity

Table 4. The viscosity data reported by Hofmann⁶⁾ and Machin.⁴⁾

Sample	Composition (wt%)				Basicity	Liquidus temperature (°C)
	SiO ₂	CaO	Al ₂ O ₃	MgO		
H1 ⁶⁾	34.3	45.8	16.6	3.3	1.34	1465
H2 ⁶⁾	34.8	41.7	16.9	6.7	1.20	1447
H3 ⁶⁾	35.2	37.6	17.1	10.1	1.07	1423
M1 ⁴⁾	35.0	45.0	15.0	5.0	1.29	1443
M2 ⁴⁾	35.0	40.0	15.0	10.0	1.14	1424

Fig. 8. Comparison of the average relative deviation for different models.

by Al₂O₃ is approximately 70% of that caused by the same weight SiO₂. In real industrial practice, same weight of MgO decreases the viscosity more than that of CaO, which is consistent with the conclusions made by Biswas²⁾ and Machin.^{3–5)} With a lower molecular weight of MgO, replacing the same weight of CaO by MgO means the mole ratio of (CaO+MgO)/SiO₂ is increased and the silicate network is further modified.

Selected BF viscosity models^{13–15)} are reproduced and calculated using the same compositions. The average relative deviations of the present model and other models are calculated and plotted in **Fig. 8**. It can be seen that the empirical correlation obtained from our measurements and Machin's data⁴⁾ can be used as a fast prediction of viscosity for BF slag within the given range.

5. Conclusion

Viscosity measurements have been carried out for granulated iron blast furnace slags from Shougang and corresponding synthetic slags. The microstructures and phase compositions of the quenched slag samples were determined by EPMA and the bulk composition of industrial slag sample was measured by XRF after the viscosity measurement.

It was found that the viscosities of the industrial BF slags are in the range of 0.3 to 0.5 Pa.s at 1500°C. The viscosities of the synthetic slag are higher than that of the corresponding industrial slag which contains minor components. The viscosities measured in the present study are compared with previous data for industrial and synthetic slags. It was found that the viscosity decreases with increasing MgO at constant Al₂O₃ and basicity. The differences of the viscosities between industrial slags and synthetic slags provide useful indications to apply the laboratory-based results of the synthetic slags to the operation of the iron blast furnace.

The effects of CaO, SiO₂, MgO and Al₂O₃ in the BF slag on the viscosity have been evaluated from the present and previous viscosity measurements.

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