Viscosity of the copper smelting slag in the Mitsubishi Process

Nozomu Hasegawa and Fumito Tanaka
Mitsubishi Materials Corporation
Central Research Institute
1-297 Kitabukuro-Cho
Omiya, Saitama 330-8508
Japan

ABSTRACT

The viscosity of copper smelting slag in the Mitsubishi Continuous Smelting and Converting Process at Naoshima was measured over the temperature range 1150 °C to 1250 °C by using a rotating cylinder viscometer. Combining these results with other reference data on synthesized basic slags, an empirical equation for estimating slag viscosities from the slag composition and temperature was derived by using viscosity modulus Kv and an Arrhenius-type equation. The equation covers a narrow composition range only, but the estimated viscosities more closely reflect actual operating conditions than values determined from previously proposed equations. This derived equation is very useful for ensuring low slag loss because the slag composition and temperature are hourly and continuously monitored in the process and the slag viscosity in the furnace can be automatically estimated in every hour.

INTRODUCTION

The Mitsubishi Continuous Copper Smelting and Converting Process is currently employed at Naoshima in Japan, Gresik in Indonesia, Onsan in Korea and Timmins in Canada (1). The process has many advantages, for example, its slag loss of copper can be kept at a very low level (2) by its continuous and steady operation using high efficiency top-blowing lances. The flowsheet of the process is schematically shown in Figure 1. The smelting, slag cleaning and converting furnaces are connected by launders carrying melt flows continuously. The mixture of matte and slag formed at the smelting furnace overflows to the slag cleaning furnace and separates by gravity difference. The slag overflows and is granulated by water while the matte is siphoned out and sent to the converting furnace. There, converting slag overflows and blister copper siphons continuously to the anode furnaces. At Naoshima, the temperature of the smelting furnace melt and the cleaning furnace slag is continuously measured at the outlet of the furnaces by using immersed thermocouples. Matte and slags are sampled hourly and immediately analyzed by fluorescent X-ray analyzer (1). Consequently, furnace operators can keep the operation very stable.

However, Naoshima is a custom smelter similar to Onsan and Gresik, where many different feed-stock materials are treated. Unavoidably the concentration of minor components in the smelting slag, such as alumina and magnesia, can fluctuate in short term basis resulting in changes of slag viscosity. This sometimes increases the slag loss. In the process, the silica sand and limestone flux additions can be easily adjusted, and melt temperature can be precisely controlled. Therefore, the slag viscosity can be kept within a specified range to minimize slag loss, provided an easy and fast estimation method of slag viscosity by using operational data can be developed.

Furthermore, Hasegawa and Kaneda have performed numerical heat and fluid flow analysis of the slag cleaning furnace to optimize its operation and design (3). The actual slag viscosity in the furnace , where temperature distribution is not always uniform and slag viscosities are not even, is one of the most important parameters in this analysis.

However, existing viscosity measurement data on industrial copper smelting slags is insufficient and the slag compositions are not specific to the Mitsubishi process. To allow precise estimation of slag viscosities, many viscosity measurements of Naoshima smelting slags were made by using a rotating cylinder viscometer. An equation for estimating slag viscosity from slag composition and temperature was then developed.

BRIEF REVIEW OF THE PREVIOUS STUDIES

The slag viscosity is of interest not only for slag loss but also for mass transfer at the slag/matte interface, heat transfer through the slag and the degree of refractory wear, and a lot of studies have been made on the effect of slag composition and temperature on viscosity. Those related to copper smelting were reviewed and summarized by Diaz (4) and Mackey (5).

Many empirical and a few theoretical parameters for estimating the viscosity-composition correlation were previously suggested (6) (7) (8). Most of the previous studies tried to derive viscosity-estimating equations which can be applied for a relatively wide range of slag composition and temperature by using the empirical and simple parameters in multi-component systems. However the viscosity in these systems is complicated and is not fully understood theoretically although studies for relatively simple and synthesized systems continue (9). For example, it is well known that the viscosity of the Fe-O-Si melts shows a maximum around the fayalite composition (10). The addition of MgO in the SiO_2-FeO-Al_2O_3-CaO system causes a decrease in viscosity but it makes the temperature at which a steep increase in viscosity higher, and the replacement of CaO by MgO in the same system causes a decrease in viscosity up to about 6% MgO above which MgO increases viscosity (4) (5). Similar amphoteric character of alumina was reported in the SiO_2-Al_2O_3-CaO system (4). Accordingly, it seems difficult to estimate the viscosity in these complicated multi-component systems precisely over a wide range of slag composition and temperature by using only empirical parameters.

Toguri et al. (6), based on viscosity data reported by Johannsen and Wiese (11) and Winterhager and Kammel (12), have proposed a viscosity modulus Kv to fit the observed values. The Kv is almost similar to the " base – to – acid " ratio used by the steel industry.

$$Kv = \frac{wt\%(FeO + Fe_3O_4 + CaO + MgO)}{wt\%(SiO_2 + Al_2O_3)} \qquad (1)$$

The relationship between Kv and viscosity is shown in Figure 2. The Kv value range of the slags at Naoshima is 1.2 to 1.6 as shown later, but the agreement is not good for such acid slags, and these viscosities are much more sensitive to changes in composition than basic slags as pointed out by Diaz (4).

Higgins and Jones (7) formulated an index, termed " modified viscosity ratio ". They classify slag constituents into " network-forming " ions, which increase viscosity, and " network-breaking " ions, which have the opposite effect. The ion-oxygen attraction of each

particular cation was taken as a measure of its effect on viscosity. This index seems more theoretical than Kv, however, its usefulness is not always confirmed by other researchers.

Battle and Hager (8) measured the viscosity of a typical lead blast furnace slag, whose contents of ZnO, CaO, MgO and CaS are much higher than those of the copper smelting slags, and analyzed their data accompanying with other reference data (12) (7) (13) including the data on the copper smelting slags. They proposed a weight parameter WP to fit these data. This parameter is just an extension of Kv and essentially empirical.

$$WP = \frac{wt\%(\,CaO + MgO + ZnO + PbO + CuO \ + \ FeO + Fe_2O_3 + CaS\,)}{wt\%(\,SiO_2 + Al_2O_3\,)} \qquad (\,2\,)$$

Plots of viscosity as a function of WP at $1250\,°C$ are shown in Figure 3. The WP range of interest in this study is 1.3 to 1.7 as shown later, and in this range the agreement is not always good. Battle et al. presented an analytical expression for the fitting curve and estimated the accuracy of the expression as 1 poise, but some of their measurement data around the WP value of 1.5 were higher than the error limit as shown in the figure.

Considering all of the above, it was decided to actually measure the viscosities of the smelting furnace slags experimentally in order to derive a viscosity-estimating equation to be used for process control.

EXPERIMETAL MEASUREMENTS

Viscosity was measured by using a concentric cylinder viscometer. The measuring apparatus is schematically shown in Figure 4. The viscometer was made by TOKI-SANGYOU corporation and consisted of a rotating-motor, a torque-meter and an elevating-mechanism. The rotating shaft was made from stainless steel(18Cr-8Ni). The slag sample was heated by the use of a electric resistance furnace and the uniform temperature region of 50 mm could be obtained with the accuracy of $\pm 3\,°C$. Two Pt-Pt.Rh thermocouples protected by an alumina tube were inserted through the furnace inner tube from the both ends of the furnace. The tip of the upper one was positioned very close to the middle of the sample crucible and used for controlling the furnace temperature. The tip of the lower one was attached to the bottom of the crucible to measure the sample temperature. Readings of these thermocouples differed by less than $3\,°C$.

The 97% magnesia, flat-bottomed crucible measured 34mm inner diameter, 40mm outer diameter and 150mm in height. The viscometer spindle was made from similar fused magnesia crucibles of smaller size. A schematic of the spindle is shown in Figure 5. The crucible with the size of 6mm outer diameter was inserted in a 10mm outer diameter crucible, and the rotating shaft of 4mm diameter was inserted to the smaller crucible. They were united by using magnesia cement. Each spindle and sample crucible was calibrated against standard oil, contained in a crucible, placed in a constant $30^o C$ water bath before every slag viscosity measurement.

Water granulated smelting slags at the plant were sampled and the viscosity of five master slags was measured. Other samples were doped with reagent grade Fe_2O_3, SiO_2, CaO, MgO and Al_2O_3 powders in order to spread the composition range of the samples a little bit wider than that in the operation. The comparison of slag composition range in this work with that in

the normal operation is shown in Table 1. The doped samples were prepared by melting the mixtures of the master slags and reagents contained in alumina crucibles at 1250°C for about two hours in argon atmosphere, and then quenched, crushed and mixed in a laboratory-scale ball mill. Some were premixed with coke before melting to reduce Fe_2O_3 content.

In the plant operation, slag loss was much more dependent on the smelting furnace melt temperature than the slag temperature at the outlet of the cleaning furnace (2). Normally the smelting furnace temperature was controlled between 1220 °C and 1230 °C with an hourly standard deviation of less than 10 °C (1). Therefore, a temperature range of 1150 °C to 1250 °C was selected for the experimental work.

In each measurement, a sample of 150 g was put into the crucible and the thickness after the measurement was around 40mm. After setting the crucible on the fixed refractory stand in the furnace, the sample was heated to around 1300 °C in high purity (99.99%) argon stream with the rate of 700 ml/minutes. The melt was then cooled to 1250 °C and held at this temperature for 30 minutes. During this period, the spindle was kept above the sample. Then, the spindle was immersed into the melt sample and the torque measured. After each measurement, the spindle was pulled up above the sample, and the sample cooled to 1220 °C and held for 30 minutes, and the torque was again measured. In this way, viscosity measurements were made at 1190 °C and 1160 °C. The viscosity of a few samples was measured at 1250 °C, 1200 °C and 1150 °C. After each measurement, the sample was cooled to room temperature in the furnace, then crushed and chemically analyzed.

RESULTS AND DISCUSSION

Analysis of the samples is shown in Table 2. Samples of No.3, 10, 11, 12 and 13 are the industrial slags and the others are the doped ones. Some of them were analyzed before and after the viscosity measurement, and in the table the upper lines for each sample show the analysis before the measurement and the lower ones show the others. Comparing both analyses, a decrease of FeO content and an increase of MgO content during the measurement in the sample were seen because of the use of magnesia crucibles. However, the sum of SiO_2 and Al_2O_3 contents was not significantly changed, namely the values of Kv and WP were little changed. This will be shown to be important in later discussion. It was noted that most of these composition changes occurred during the sample melting at high temperature and before the start of viscosity measurement. Thus the change was relatively small during the measurement and cooling to the room temperature. Accordingly, the analysis after the measurement is used in the later discussion. It was confirmed that the Fe_2O_3 content was little changed during the measurement. In the operation, the oxygen potential in the slag at the outlet of the slag cleaning furnace is from 10^{-9} to 10^{-8}(normalized at 1200 °C) (3). It was estimated that the oxygen potential in the sample could be kept at a similar level in spite of the use of the inert argon atmosphere.

The viscosity measurement data is shown in Table 3. Generally, the correlation between the viscosity of liquids and temperature can be described by the Arrhenius-type equation as shown below (4).

$$\mu = A \exp (E / RT) \qquad\qquad (3)$$

Where, μ : viscosity
      A : constant
      E: activation energy of flow
      R : gas constant
      T : absolute temperature

The relation between the measured viscosity and temperature is not shown in the paper, but it was confirmed all of the data shown in Table 3 could be well fitted to the Arrehenius-type correlation.

In deriving a viscosity-estimating equation, the applicable range of slag composition was limited as shown in Table 4 by considering the range in the actual operation and experimental work shown in Table 1. Only data from this work and those of the synthesized basic slags measured by Johannsen and Wiese (11), which is within the limit shown in Table 4, were used for this analysis. Winterhager and Kammel (12) measured the viscosity of many different copper industrial slags but most of their slag compositions are out of the limit and incomplete, hence none of these data was used for this study.

Figure 6 shows the relation between the viscosity data used in the work and Kv calculated from equation (1) at 1250 ^{o}C. Data of this study is in good agreement with that of Johannsen et al. below the Kv value of 1.6. The agreement between the data and the fitting curve is better than that in the fitting by Toguri et al., which is shown in Figure 2. Similar analysis for another parameter, the modified viscosity ratio suggested by Higgins and Jones (7), was performed but the agreement was not better. In the Kv value defined by equation (1), minor components such as ZnO, Na_2O, K_2O and so on are not included, but these levels are very low and stable in the process as shown in Table 1. Therefore, Kv is practical enough for the objective of this work. The relations between the viscosity and Kv at 1220°C and 1190 ^{o}C are shown in Figure 7 and Figure 8, respectively. The agreement between the data and the fitting curves is also good but it becomes worse at lower temperatures. The data at 1160 ^{o}C was not used in later analysis. The analytical expressions for these fitting curves are as follows:

At 1250 ^{o}C μ (poise) = $11.243 Kv^{-3.481}$ (4)
At 1220 ^{o}C μ(poise) = $13.835 Kv^{-3.5234}$ (5)
At 1190 ^{o}C μ (poise)= $15.797 Kv^{-3.4322}$ (6)

By using the equations (4) through (6), the viscosity at each temperature can be calculated for a certain Kv value (slag composition). The Arrhenius-type plot of these calculated viscosites, the logarithm of viscosity versus the reciprocal of absolute temperature plot, is shown in Figure 9. The linearity of these fitting lines in the figure is very good, and it is confirmed that the Arrhenius-type relation defined by equation (3) is applicable for analyzing the temperature dependence of viscosity. The activation energy of flow, E in the equation (3), can be derived from the slopes of the fitting lines in the figure, and the relation between E and Kv is shown in Figure 10. It is interesting that the linearity of the fitting line in the figure is fairly good although Kv is not essentially a theoretical parameter. The analytical equation for the fitting line is as follows:

$$E = 10.671Kv + 94.971 \qquad (7)$$

Similarly, the constant in equation (3), A, can be derived from the intercept of the fitting lines in Figure 9 and the relation between A and Kv can be well described by equation (8).

$$A = 0.0601\exp(-3.2788Kv) \qquad (8)$$

Consequently, substituting equation (7) and equation (8) in equation (3), a viscosity-estimating equation can be derived as follows:

$$\mu(poise) = 0.0601\exp((1283.45/T - 3.2788)Kv + 11422.6/T) \qquad (9)$$

The standard deviation of the experimental results and the reference data sets used in the analysis to the calculated values from equation (9) is 0.6 poise. The error of the measurement data based on the change of composition (Kv of 0.02) and temperature (5 ^{o}C) during the measurement can be evaluated to be less than 8% by using equation (9). Other error factors such as the deformation of the spindle were not quantified but estimated to be minor (8) (10). Accordingly, the measurement error is less than 10% and the accuracy of equation (9) can be estimated to be ±0.7 poise. The composition range applicable to the equation is limited to that shown in Table 4. However, the upper limit of the temperature range may be lifted a little bit higher than 1250 ^{o}C, because it was confirmed that the Arrhenius-type relation can be almost perfectly applied to the system.

Figure 11 shows the comparison of the estimated viscosity with previous estimation by using Kv at 1200 ^{o}C. The estimated value in this work is lower than that estimated by Toguri et al., especially at smaller Kv of interest, the difference is not negligible. The comparison of the measured viscosity used in the analysis with the previous estimation by using WP defined by equation (2) at 1250 ^{o}C is shown in Figure 12. The WP for the measured data was calculated by assuming the content of ZnO and CuO were constant at 1% and 0.5%, respectively, and that of PbO and CaS could be negligible in equation (2). Most of the measured data agree with the estimated ones by using the equation presented by Battle et al. (8) within the error of 1 poise, but almost all of the data are higher than the estimation at the WP value of interest, less than 1.7, and therefore it is considered that the difference can not be ignored.

Furthermore in previous estimations, the temperature dependence of viscosity was not always formulated. Toguri et al. presented only two fitting curves at 1200 ^{o}C and 1300 ^{o}C as shown in Figure 2. And Battle et al. presented some analytical equations at a certain temperature changed by 50 ^{o}C. Accordingly, previous estimations are not truly applicable to the Mitsubishi process, whose continuous nature requires simple and fast viscosity estimation.

The chosen range of the estimating equation presented in this study is relatively narrow. It can however be applied to the slags in the Mitsubishi processes, at Gresik and Onsan, where the operating conditions are similar to that at Naoshima. It may also be applied to the slags in other smelting processes operating within the same applicable range.

We expect the viscosity estimations to be more accurate, easier and faster than previously found in the literature. As aforementioned, respective slag compositions and temperatures are hourly and continuously monitored, therefore the slag viscosity in the furnace can be estimated every hour by using the simple equations (1) and (9), and the viscosity can be

controlled within an appropriate range, normally less than 5 poise, by adjusting flux and coal additions and the lance blowing conditions. In the process Fe^{3+} are not hourly analyzed, but oxygen potential, matte grade and Fe^{2+}/Fe^{3+} ratio are kept very steady. Therefore, slag viscosity estimations are valid if the Fe_2O_3 content is fixed at say 5% in the calculation of Kv. Consequently, the viscosity-estimating equation derived in this work will prove very useful for ensuring low slag loss in the Mitsubishi process.

CONCLUSIONS

It was confirmed that the viscosity modulus Kv and the Arrhenius-type equation are useful for estimating slag viscosities from slag compositions and temperature. By combining these parameters, a simple viscosity-estimating equation was derived from the measurement data on copper smelting slags in the Mitsubishi process at Naoshima smelter and other reference data on synthesized basic slags. The Kv is essentially empirical and it seemed difficult to estimate the accurate viscosity by using the parameter in the wide range of slag composition. Therefore the applicable range of the estimating-equation was deliberately chosen and kept tight to conform to plant process conditions. This viscosity-estimating equation will become a very important tool in ensuring low copper losses in slag and further enhance the cost effectiveness of the Mitsubishi process.

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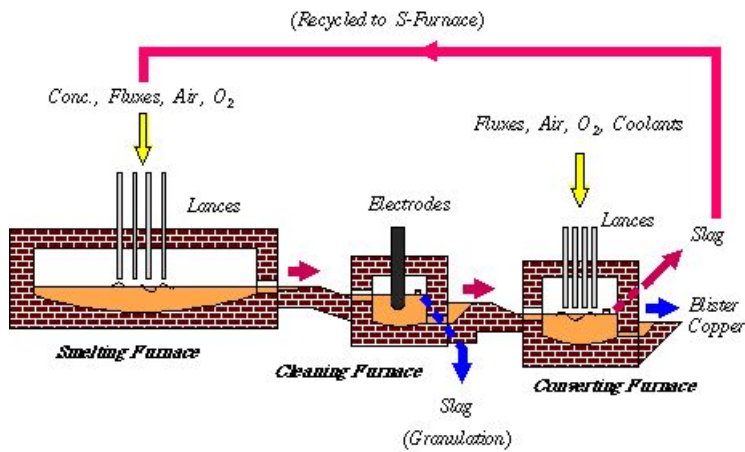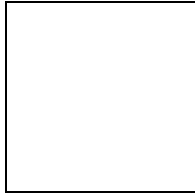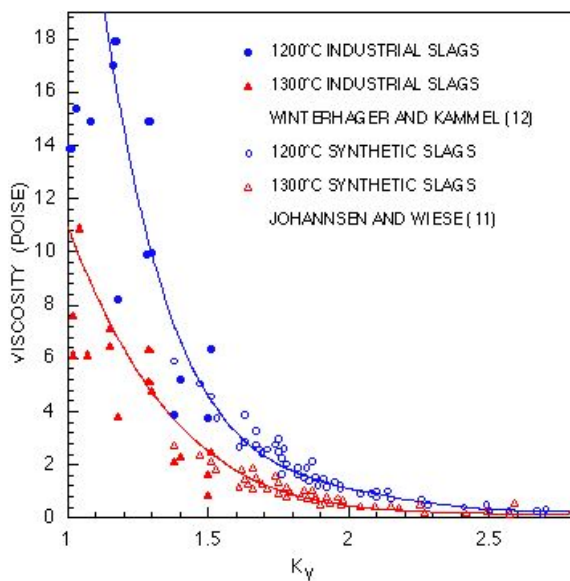
Figure 1   Flowsheet of the Mitsubishi Process

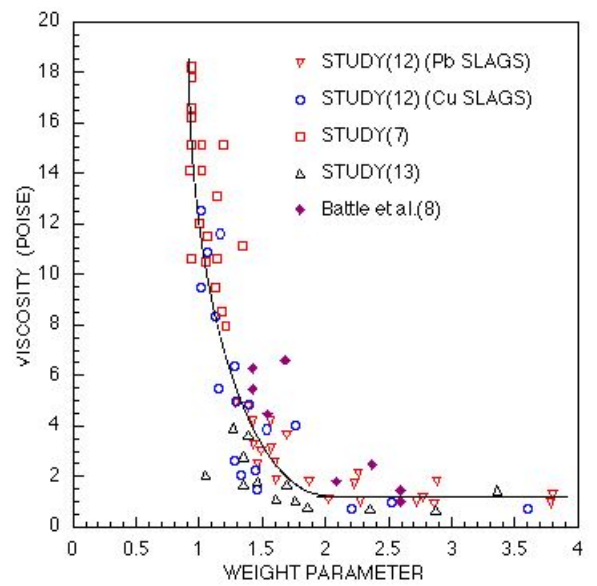Figure 2   Viscosity as a function of K_V by Toguri et al.[6]

Figure 3   Viscosity as a function of WP at 1250°C by Battle et al.[8]
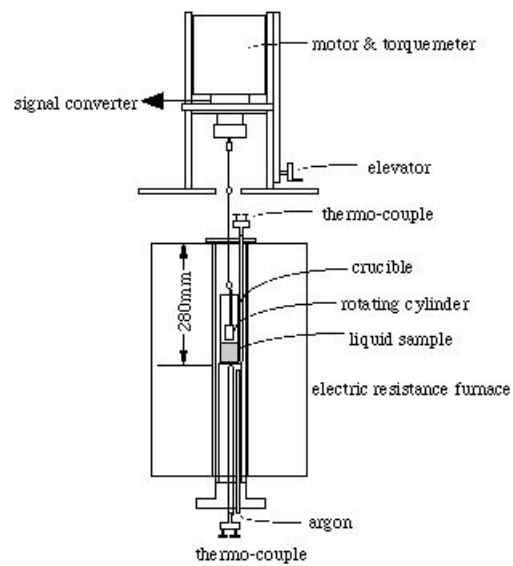
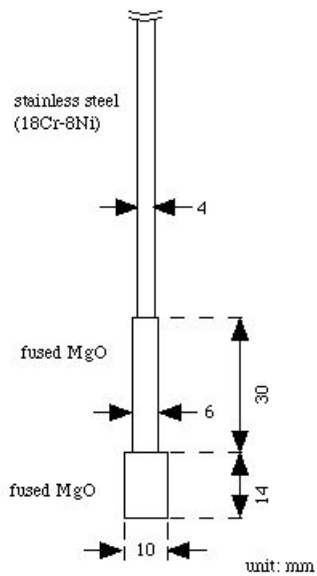Figure 4   Schematic of viscosity-measuring apparatus
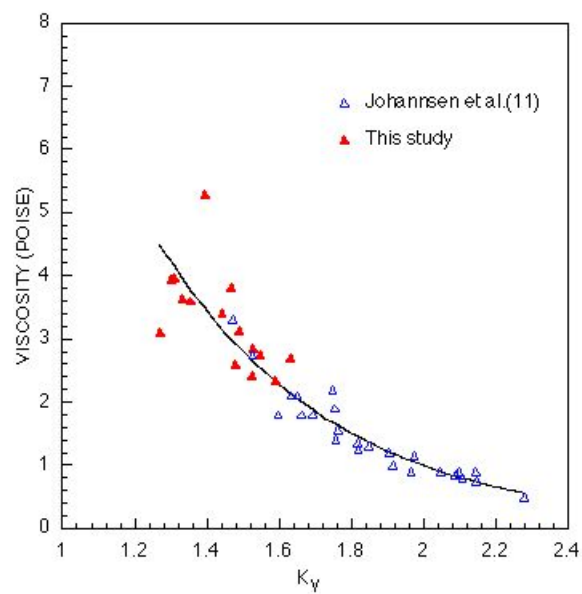

Figure 5   Schematic of viscometer spindle

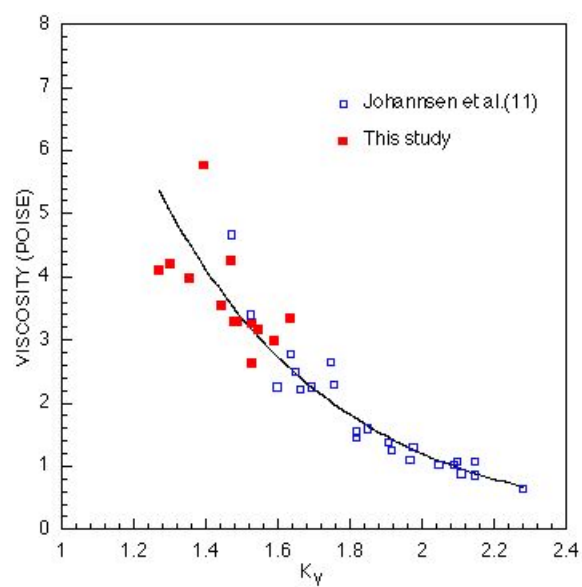Figure 6   Relationship between the viscosity and K_V, 1250℃

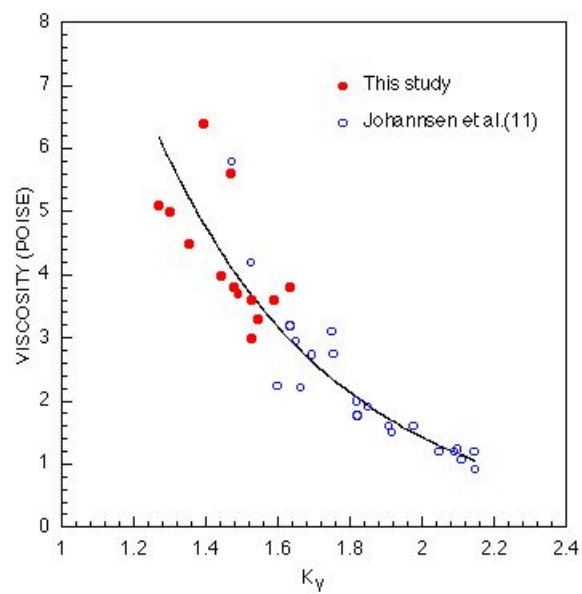Figure 7   Relationship between the viscosity and K_V, 1220℃

Figure 8   Relationship between the viscosity and K_v, 1190°C
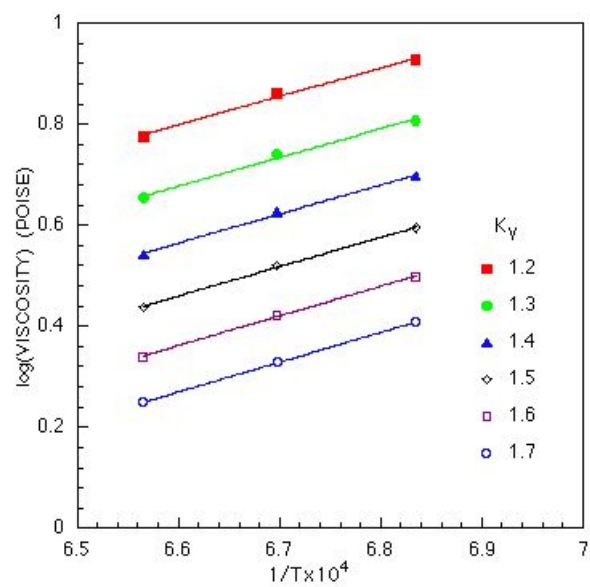

Figure 9   Arrenius-type plots of the estimated viscosity
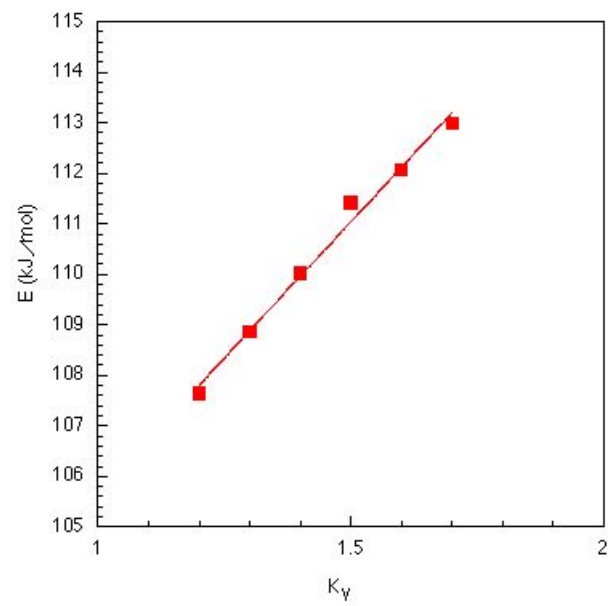
Figure 10   Relation between the activation energy and K_v
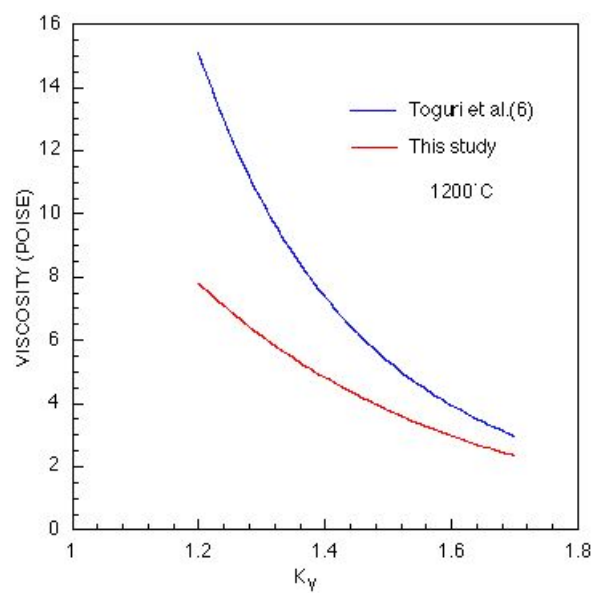
Figure 11   Comparison of the estimated viscosity
with previous estimation by using K_v at 1200°C
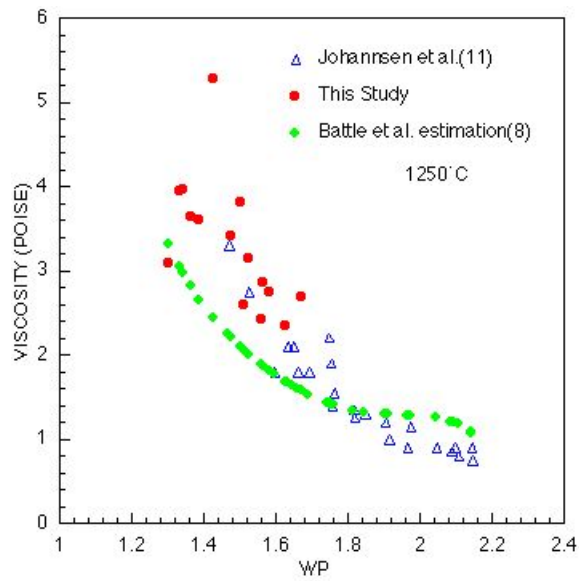
Figure 12   Comparison of the estimated viscosity
with previous estimation by using WP at 1250°C

Table 1 Slag composition range

	Normal Operation (%)	This Study (%)
SiO_2	32 – 36	31.0 – 37.9
FeO	40 – 45	38.9 – 45.9
Fe_2O_3	3 – 6	1.3 – 7.3
Al_2O_3	3.5 – 6.5	3.3 – 7.1
CaO	4.0 – 7.0	3.9 – 7.5
MgO	0.9 – 1.8	3.2 – 4.5
ZnO	1.0 – 1.3	0.7 – 1.3
Cu	0.5 – 0.8	0.4 – 1.1
K	0.7 – 1.1	NA
Na	0.4 – 1.0	NA
S	0.3 – 0.5	NA
Pb, As	< 0.1	NA
Cr, Co, Ni, Sb, Sn	< 0.05	NA

NA: Not analyzed

<div align="center">Table 2 Analysis of the samples</div>

No.	Assay (%)							
	Cu	FeO	Fe_2O_3	SiO_2	Al_2O_3	MgO	CaO	ZnO
1	-	-	-	-	-	-	-	-
	0.41	42.09	4.30	34.42	7.05	3.46	4.09	1.25
2	-	-	-	-	-	-	-	-
	0.52	43.97	2.39	34.62	4.98	3.26	4.05	1.27
3	0.57	46.11	4.72	36.14	3.37	0.75	4.17	1.49
	0.54	43.77	4.72	35.50	3.50	3.62	4.13	1.28
4	-	-	-	-	-	-	-	-
	0.55	43.60	3.56	34.62	3.39	3.96	5.49	1.30
5	-	-	-	-	-	-	-	-
	0.50	43.16	4.42	33.64	3.68	3.44	5.97	1.30
6	-	-	-	-	-	-	-	-
	0.53	42.06	3.70	33.32	3.51	3.72	7.48	1.27
7	-	-	-	-	-	-	-	-
	0.49	41.55	4.78	34.34	3.32	4.06	7.04	1.18
8	-	-	-	-	-	-	-	-
	0.49	43.05	3.85	34.98	5.99	3.73	3.91	1.24
9	-	-	-	-	-	-	-	-
	0.55	42.76	4.45	34.50	7.15	3.29	4.09	1.24
10	0.75	44.31	7.43	30.74	3.65	0.96	4.70	1.21
	1.11	42.13	6.88	32.26	3.66	4.54	5.07	1.14
11	0.61	42.16	2.66	36.80	4.16	2.05	4.76	0.73
	0.47	40.00	4.53	37.90	3.79	3.82	4.59	0.68
12	0.79	42.63	4.06	32.18	3.78	1.10	7.62	1.21
	0.69	38.92	6.26	34.26	3.67	3.66	7.23	1.07
13	1.01	44.92	6.69	32.20	5.96	1.80	7.04	1.42
	0.70	41.67	7.28	31.90	4.58	3.55	5.53	1.00
14	0.50	48.73	1.39	34.48	5.04	0.70	4.54	1.26
	0.43	45.93	1.29	34.94	4.68	3.38	4.63	1.17
15	0.61	46.22	4.59	35.68	3.54	0.78	4.60	1.37
	0.54	42.99	4.25	34.76	3.41	4.32	4.47	1.25

upper lines: before measurement
lower lines: after measurement

Table 3 Viscosity measurement data (poise)

No.	Temperature (°C)					
	1150	1160	1190	1200	1220	1250
1		6.96	4.99		4.22	3.94
2		5.25	4.50		3.98	3.60
3		4.96	3.98		3.56	3.42
4		4.71	3.71		3.29	3.14
5		4.97	3.61		3.27	2.86
6		3.87	3.30		3.16	2.74
7		4.29	3.00		2.64	2.43
8	7.06			4.57		3.65
9	8.53			4.87		3.97
10		4.50	3.80		3.35	2.70
11		7.20	5.10		4.10	3.10
12		4.75	3.80		3.30	2.60
13		4.60	3.60		3.00	2.35
14		7.04	6.39		5.77	5.28
15		6.63	5.60		4.25	3.81

Table 4 Slag composition range applied for deriving the viscosity-estimating equation

	wt%
$FeO+Fe_2O_3+SiO_2+Al_2O_3+MgO+CaO$	≥ 93.0
$Al_2O_3+MgO+CaO$	< 15.0
SiO_2	$30 - 38$
Al_2O_3	≤ 7.3
CaO	≤ 10
MgO	≤ 4.5
Fe_2O_3	$1.2 - 8.3$
ZnO	≤ 1.3