Accuracy of viscosity measurement using rotation viscometer

Lijia Wu[1,2], Peter Heller[1], Xuegong Bi[2], Piotr R. Scheller[1]

1) *Institute of Iron and Steel Technology, Freiberg University of Mining and Technology, 09599, Germany*
2) *College of Material and Metallurgy, Wuhan University of Science and Technology, 430081, China*

Abstract: In the present work the effect of these parameters was measured and quantified in model fluids at room temperature with wide range of viscosity and the movement of the spindle and shaft were recorded by a camera. Considering some factors such as the type of combination between rotor and viscometer, angle between spindle and shaft, rotating speed, side movement during testing it was indicated that bigger side movement will introduce larger error and the instable side movement was the main effect to generate error, especially in low viscosity fluid, whereas the angle variation between spindle and shaft during testing affected the viscosity hardly. The articulated combination between rotor and viscometer benefited to reduce side movement significantly in high viscosity fluid, whereas it had less influence in low viscosity liquids. Therefore strongly side movements should be avoided during the test especially in low viscosity tests. High temperature measurements were performed with several groups of TiO_2 containing slags in the CaO-SiO_2-TiO_2 and CaO-SiO_2-TiO_2-Na_2O-Al_2O_3 systems. The viscosity-temperature relationships of these TiO_2 containing slags have been given out in the present work as well. The chemical composition effects on viscosity were discussed such as basicity, TiO_2 content, Na_2O content and Al_2O_3 content. Additionally the mechanisms of how the viscosity is influenced in the homogeneous state and in the heterogeneous state with precipitated solid oxides.

Key words: rotation viscometer, geometric parameter, error analysis, TiO_2 containing slags

1. Introduction

Viscosity has a decisive influence on the fluid flow in metallurgical process especially in continuous casting process. One of the main tasks of mold fluxes is forming a lubricant film between strand and mold, where it is strongly depended on viscosity of the mold flux. Thus, viscosity measurement plays an important role in studying physical property of the slags. Even so the variation in values reported by different studies or laboratories is greater.

In order to receive a relative accurate viscosity values especially at high temperature for slags measurements, these following factors should be taken into consideration: geometry of the measuring system, eccentric rotation, temperature controlling, rotate speed (torque) setting, counting interval, sample preparation etc.[1,2] Since the experiments of this paper were preceded in the coaxial cylinder measuring system, the influencing factors will be especially discussed here within coaxial cylinder measuring system.

Viscosity in metallurgy is determined by the structure of the slag, which is highly depended on the composition, temperature and pressure.[3] As the measurement of viscosity is always taken, the slags may be transformed from homogenous melt to heterogeneous one under different temperature or with a certain cooling rate. Thus, discussing the effect of composition should be separated to two parts based on the transformation

2. Room temperature experiments

2.1 Experimental procedure

The relationship between non-coaxial rotation and the errors has been investigated in this room temperature experiments in silicon oil AK200, AK10000 and AK30000 with short (see Fig.1) and long shaft, respectively. The movement of the shaft and spindle were recorded by a camera.

(a) (b)

Fig.1 Rotators (a) short shaft in a fixed statue; (b) short shaft in a articulated statue with articulated combination

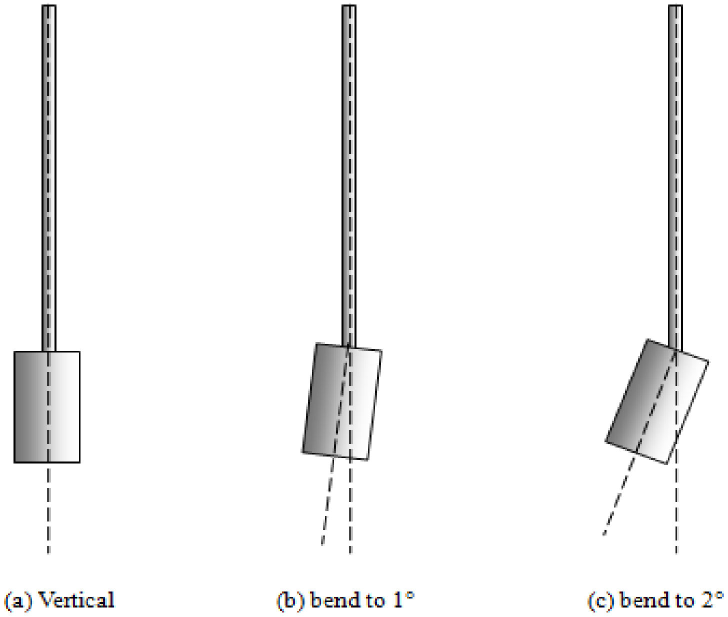

Fig.2 Schemas of the rotor in room temperature experiments

Considering of the practical high temperature measuring system, which includes an articulated combination to transport the rotation from shaft to equipment, this combination has been involved in room temperature experiments as well, in order to study the effects to the movements and results. The experimental was divided into two parts by short and long shaft. For the short shaft series the bottom shaft where is closed to the spindle has been knocked into 1° and 2°, respectively. (Fig.2)

Each type of the rotors above has been conducted in the room temperature experiments with short shaft under different rotating speeds: 6U/min, 15U/min and 30U/min. Silicon oil AK200, AK10000 and AK30000 were selected as the substance to be tested. All of the tests were preceded in crucible and spindle measuring system with the same demission as in high temperature measurement.

The values tested by cone-and-plate measuring system were taken as the reference values of the viscosity to compare the effects of the different factor. Because the cone-and-plate measuring system (CP measuring system) has a better condition such as better temperature controlling, accurate geometry profile, compared to the crucible spindle system. The viscosity curves of the above three silicon oils can be found in the attachment. The movements of the spindle have been recorded by camera as well to research the effects of movement on the viscosity errors. Fig.3 is the schema of the recorded parameters.

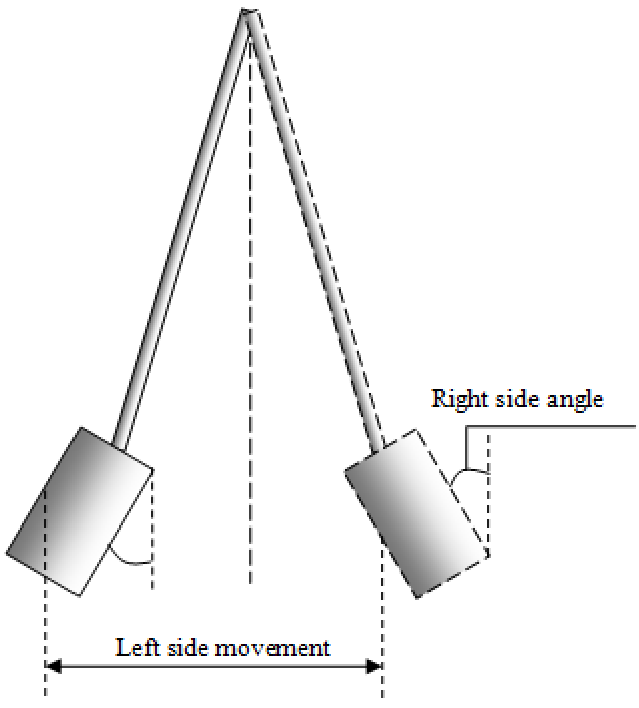

Fig.3 Schema of the recorded parameters

Long shaft series experiments were conducted in crucible and spindle measuring system as well, which is practically closer to the high temperature measurement. As the results showed in the short shaft series, the positive effect of the articulated combination has been well proved. It will be more interested to know, how this component behaves with a long shaft when it rotates non-coaxially. A middle bended long shaft was selected as one variable of geometry factor to be studied (Fig.4) and the included angle between spindle and shaft 0° (vertical) and 1.5° were selected in the long shaft series.

As is not simple to measure the temperature directly in the oil samples, the instant room temperature were recorded into the viscosity-temperature curves, which may be advanced in the later experiments.

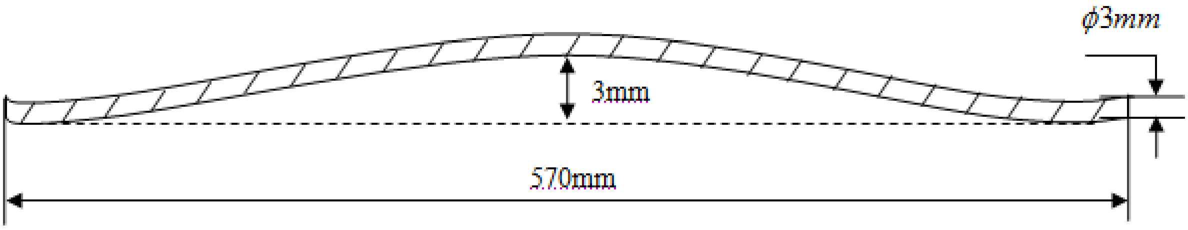

Fig. 4 Schema of the middle bended long shaft

2.2 Results of short shaft series

A series of experiments were conducted in AK10000 and AK30000 silicon oil using fixed shaft (Fig.1a) and articulated shaft (Fig.1b) with vertical and bend spindle (Fig2 a, b, c). During the testing the side movements of the spindle and angle variations were recorded by camera.

Fig.5 and Fig.6 illustrate clearly that the articulated combination has a positive and main effect on reducing errors both in AK10000 and in AK30000. The effect of the included angle between spindle and shaft is not so clearly in AK10000, but in AK30000 (higher viscosity silicon oil) it has showed up a trend, that it introduced a larger error to the results about 1~2%.

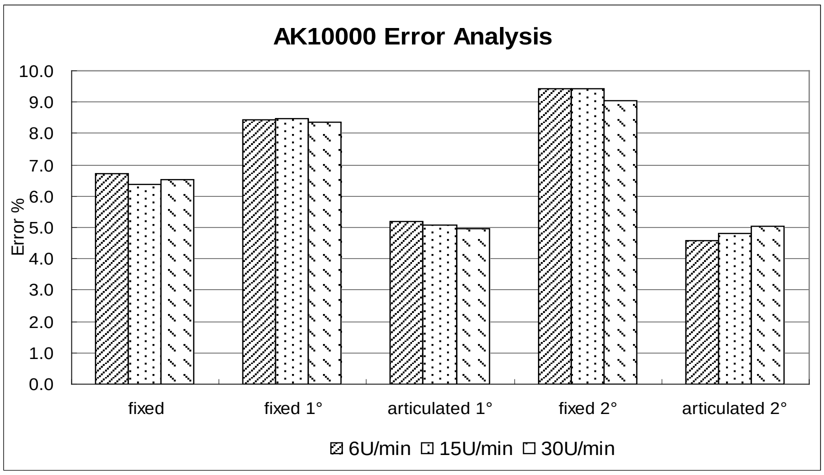

Fig.5 Error analysis of AK10000 with short shaft

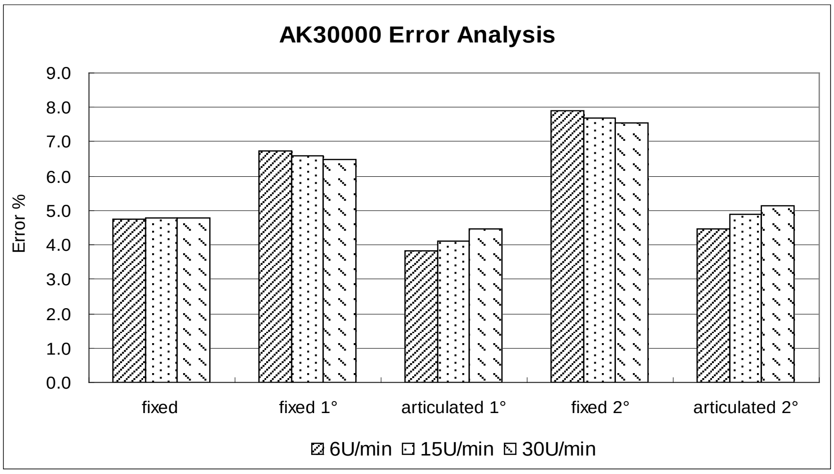

Fig.6 Error analysis of AK30000 with short shaft

The camera has recorded the side movement and angle variation of each spindle during the testing. It was shown that the angle variation changed with the included angle between spindle and shaft, which has no influence from the type of combinations, but didn't show relationship with measuring errors, whereas the side movements illustrated all most the same variation and trend as the measuring error, which means that the main effect on the error of the viscosity results is the side movements (Fig.7 and Fig.8). Side movement is one of the main factors reflecting the rotating statue of the rotator. And the articulated mechanical combination has a positive effect on keeping the inner cylinder steadily and smoothly rotating and in the middle of the crucible staying.

Since the produced centrifugal force will be bigger with longer shaft than shorter one, it is interested in studying the rotating behavior with long shaft as well.

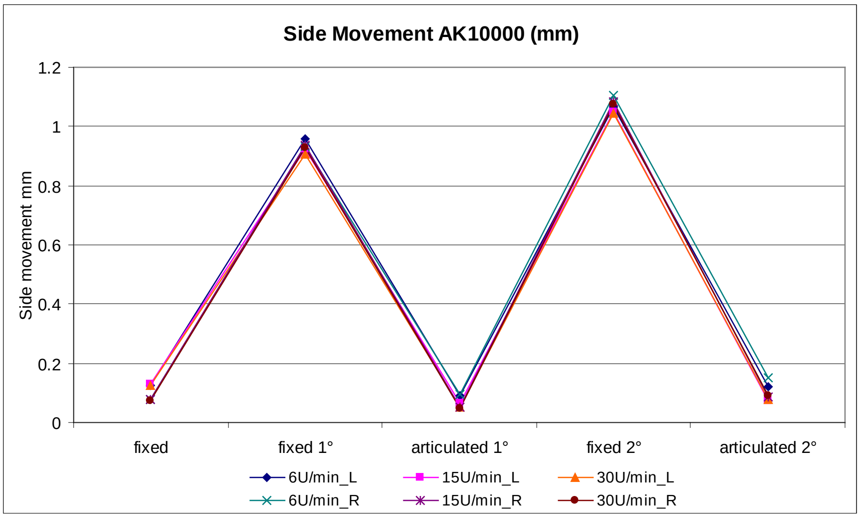

Fig.7 Angel variation of different types of spindle in AK30000

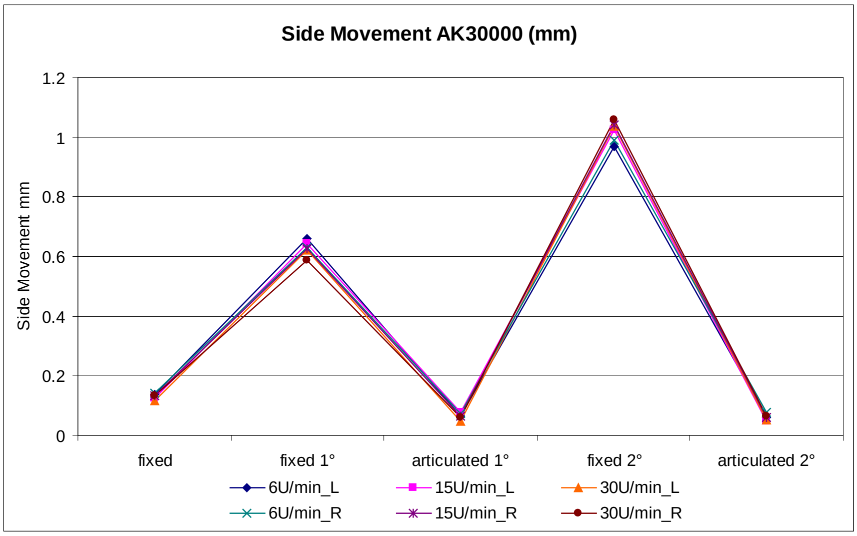

Fig.8 Side movements of different types of spindle in AK30000

2.3 Results of long shaft series

AK200 represented low viscosity substance, of which the error has been magnified as is shown in Fig.9, which indicated a strong request of small fluctuation of side moving and steadily rotating during entire measuring process, especially in testing low viscosity liquids.

According to the error analysis of AK30000 such error magnifying didn't appear and the effect of rotating speed turned out hardly (Fig. 10).

Longer observing time has been taken into the experiments as well to study the change of the movement and angle variation with vertical and bended spindle during the experiments in AK200 and AK30000. It is illustrates both in AK 200 and AK30000 that the include angle between spindle and shaft will not change with the time.

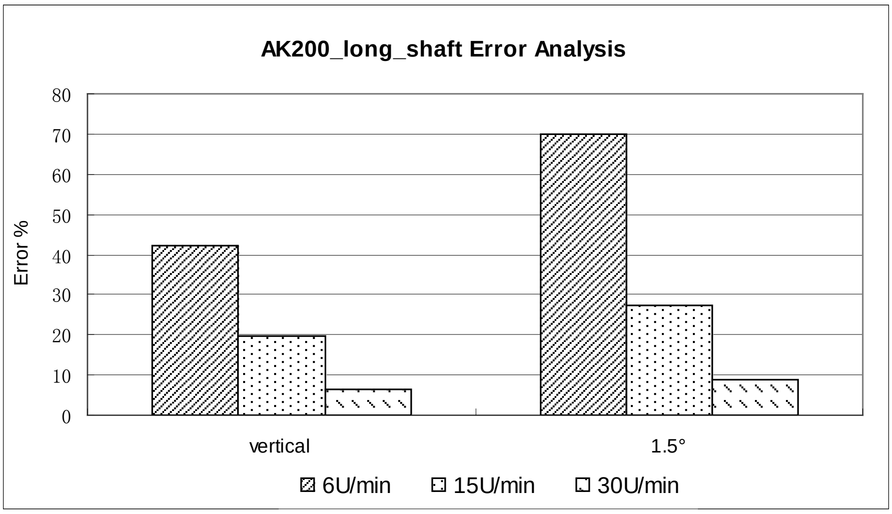

Fig.9 Compared error analysis in AK200

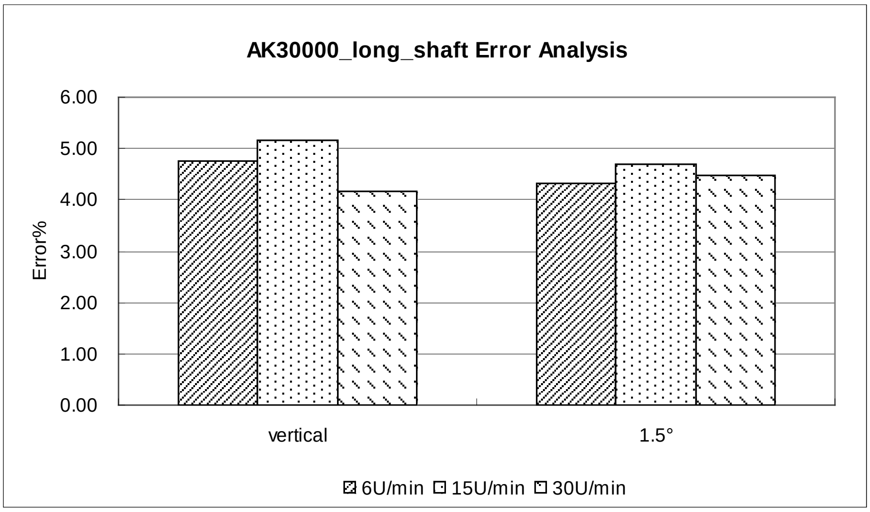

Fig.10 Compared error analysis in AK30000

The Angle variations changed hardly with time but the side movements is time variant. It can be found that the measuring point with each rotating speed stays in the same level as shown in Fig.11, whereas Fig.12 appears a significant reduction of the side movements, i.e. in higher viscosity tests the rotator has a trend to centralize in the middle. The fluctuation of the viscosity with vertical type as well as with 1.5° type spindle turned out to be centralized, which means the inner cylinder rotates steadily in the end. This is well in agreement with the Time-Viscosity curve of long shaft rotator in AK30000, which shows the viscosity as function of time, of which the curve turned out to be quit fluctuated at the beginning but smoother in the end, whereas in low viscosity tests (in AK200) such trend turned out hardly. The effect of the included angle can not be concluded here through time-viscosity, because the two curves stand for different temperatures, thus they are incomparable.

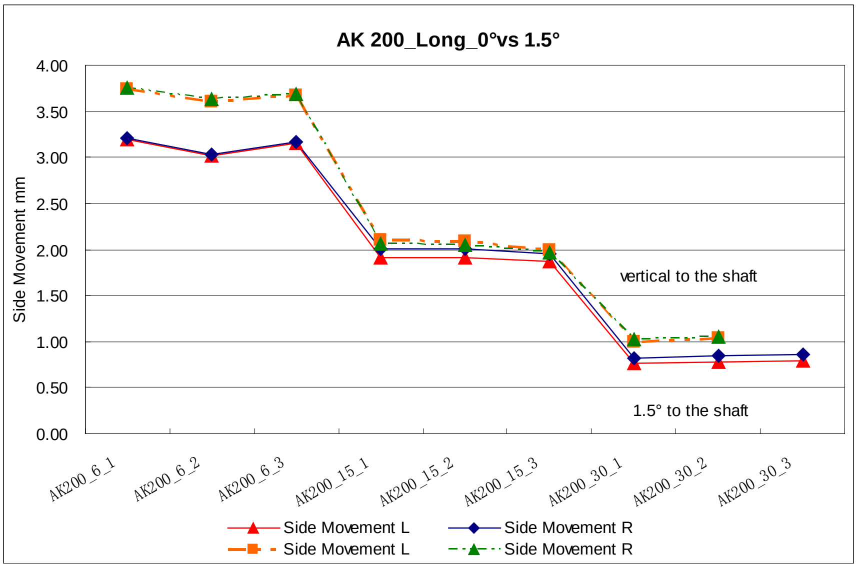

Fig.11 Side movements comparison of vertical type spindle to 1.5° bended spindle in AK200

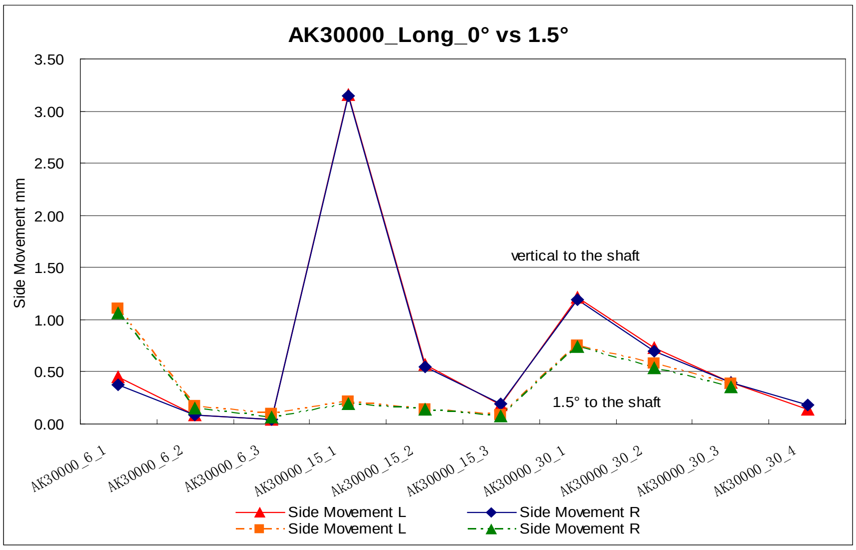

Fig.12 Side movements comparison of vertical type spindle to 1.5° bended spindle in AK30000

Since the side movements and the variation of the instant viscosity come out the same trend, it can be concluded that the fluctuated side movement is the main effect on introducing errors. Strongly side movements should be avoided during the test especially in low viscosity tests.

3. Viscosity measurements of mold fluxes

Viscosities of two series of mold fluxes have been measured in this paper. One was the basic CaO-SiO_2-TiO_2 (CST) ternary system. The other was the CaO-SiO_2-TiO_2-Na_2O-Al_2O_3 (CSTNA) system, which is closer to industry mold fluxes.

3.1 Slag preparation

For the CaO-SiO_2-TiO_2 slags (CST slags) the pre-melting process were not very complex, because the components of this ternary system slags varied hardly during high temperature. The chemical compositions of each sample and the liquid temperature based on Factsage are shown in Table1.

Sample was pre-melted in a Mo-crucible installed with top and bottom thermo couples. Since the bottom thermo couple was located nearer than top one and inserted into the crucible, its reading was recorded and used in the curves as instant temperature, whereas the top thermo couple was a controller to indicate the distribution of temperature field. The shear viscosities of liquids were determined using the concentric cylinder method under argon. Both the inner cylinder and the crucible were made of Molybdenum. The viscosities were measured with Antonpaar MC301 under rotation speeds of 15U/min. After the viscometry was completed, each sample was removed from the furnace, reheated and removed from the crucible into another graphite crucible. The Mo-crucible was cleaned in a mechanical way by sand spray and used for the next sample. After the measurements samples were reheated and removed from the Mo-crucible into a graphite crucible. The recycled slags were analyzed again by XRF (X-Ray Fluorescence Spectrum) and the results are shown in Table 1.

Table1 Chemical composition of ternary system mold fluxes

	Composition before measurement (mass %)					Composition after measurement (mass %)			
	CaO	SiO_2	TiO_2	R*	Liquid T °C	CaO	SiO_2	TiO_2	R*
A0	48.31	51.10	0.00	0.95	1541	47.24	51.15	0.00	0.92
A1	46.10	47.25	6.65	0.98	1488	44.36	48.92	5.29	0.91
A2	41.62	42.60	15.79	0.98	1429	41.87	45.28	11.21	0.92
A3	41.10	29.10	29.80	1.41	1551	39.79	32.20	25.61	1.24
B0	58.00	42.00	0.00	1.38	1735	54.58	44.44	0.00	1.23
B1	55.98	39.79	4.23	1.41	1612	53.83	41.08	3.70	1.31
B2	50.94	34.10	14.95	1.49	1520	51.47	36.17	11.67	1.42
B3	49.91	33.22	16.23	1.50	1529	47.30	37.50	13.69	1.26

* R=CaOwt%/SiO_2wt%

For the other slags $CaO-SiO_2-TiO_2-Na_2O-Al_2O_3$ slags (CSTNA slags) the procedure of preparation was more complicated than CST series slags because of the evaporation of sodium oxide. A basic slag $CaO-SiO_2$ with basicity 0.8 was first to be produced and then added with TiO_2 to achieve required based CST slags. These CST slags were grinded to powder. 2/3 of the based slags were mixed with required Na_2CO_3 and covered with the rest of the based slags in the crucible to reduce the evaporation of sodium oxide. Following table is the chemical composition of $CaO-SiO_2-TiO_2-Na_2O-Al_2O_3$ slags (CSTNA slags), which is quite close to the theoretical composition. Since this series slags contained sodium oxide, samples shouldn't be heated over 1400°C in case it boiled and formed foam, except for CA, CS and CST slags, which were heated to 1600°C for higher fusing temperatures.

Table2 Chemical composition of CSTNA slags

	Analyzed composition (mass %)				
	CaO	SiO_2	TiO_2	Na_2O	Al_2O_3
CA	45.13				52.78
CS	42.71	55.92			
CST	36.76	46.31	17.92		
CSTNA_1	34.77	38.88	14.1	9.54	2.97
CSTNA_2	30.88	36.99	16.16	15.64	0.14
CSTNA_3	33.55	41.98	16.3	8.17	0.12
CSTNA_4	31.44	39.41	15.4	7.03	6.8

3.2 Results and discussion

All of the viscosity tests were determined from high temperature to measured toque over 2mN·m, through which the viscosity was measured in homogenous zone as well as in heterogeneous zone.

3.2.1 CST series slags

The following figures are the viscosity-temperature relationships of A and B series slags throughout the cooling process, respectively. And lgη-1/T relationship can be found in appendix.

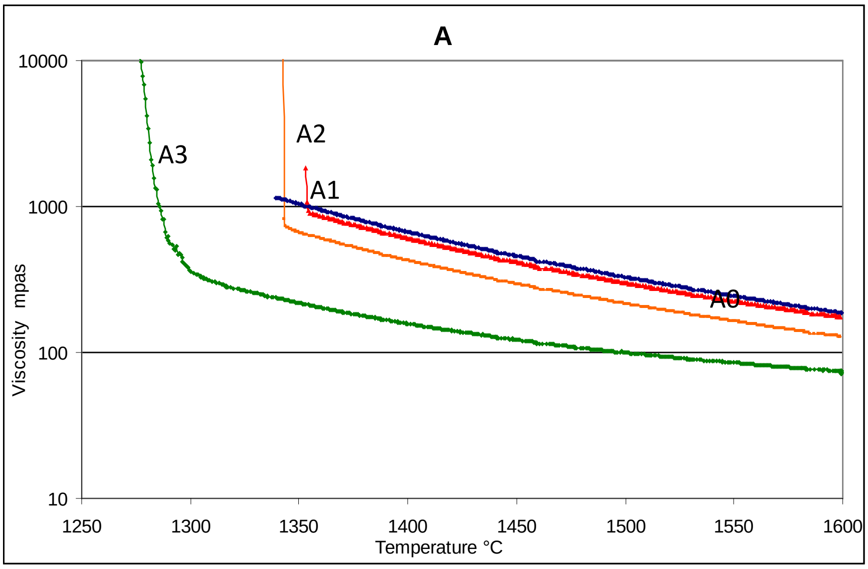

Fig.13 Viscosity-temperature curves of A series slags in a lager rang

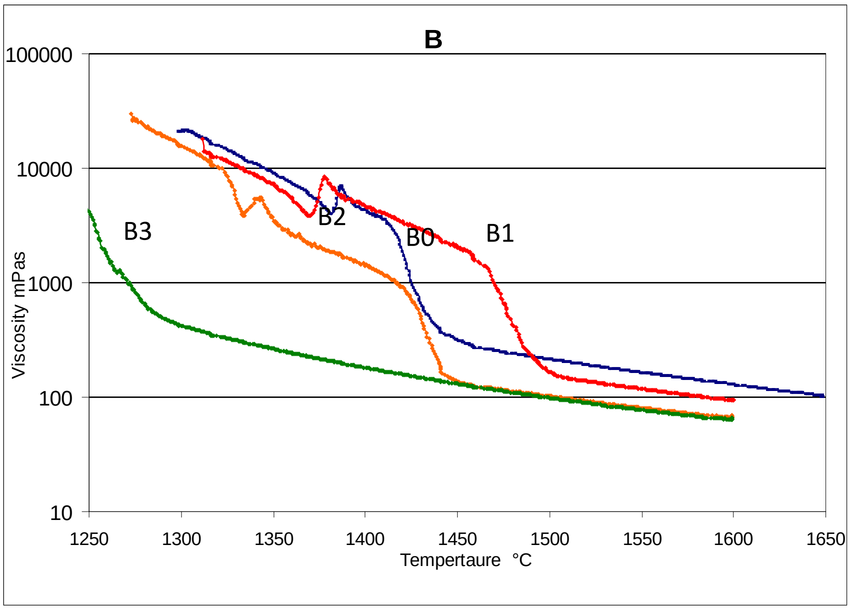

Fig.14 Viscosity-temperature curves of B series slags in a lager rang

3.2.1.1 Effect of basicity on viscosity

As is presented before, basicity has a great effect on viscosity due to its breaking up the structure by network modifiers such as CaO, MgO, Na_2O, K_2O, PbO, BaO above liquid temperature i.e. in homogeneous zone. Fig.13 and Fig.14 show A0, A2 and B0, B2 which have the same amount of TiO_2 but with different basicity ($A \approx 0.92$ and $B \approx 1.3$). Viscosities of A slags are significantly higher than B slags. On the other hands it can be concluded as well that either in basic slags or in acid slag TiO_2 promote reducing the viscosity and the mechanism will be discussed later.

3.2.1.2 Effect of TiO_2 content on viscosity

The general mechanism of the network modifiers has been presented before. Due to the breaking up of the long range order of SiO^{4-} structure by network modifiers the viscosity will drop. The schema of the general reducing mechanism has been given out by P. Koazkevitch[4,5]. If the TiO_2 acts as a basic oxide or an amphoteric substance is not shown clearly in these tests (Fig.15 and Fig.16), since TiO_2 decreases the viscosity in acid slags significantly and promotes to reduce in basic slags with lower TiO_2 but increases at higher content of TiO_2. Thus it is noteworthy that the melting temperatures of the acid slags are much lower than basic slags. If TiO_2 is a network modifier or network former should only be discussed in homogenous zone, in other words, slags should be complete melted. So the effect of TiO_2 on basic slags should be discussed considering the influence of solid phase.

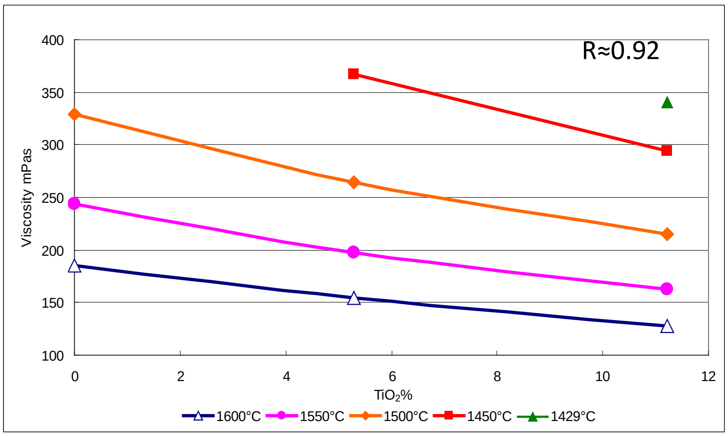

Fig.15 Viscosity variation as function of TiO_2 wt% in basicity 0.92 slags

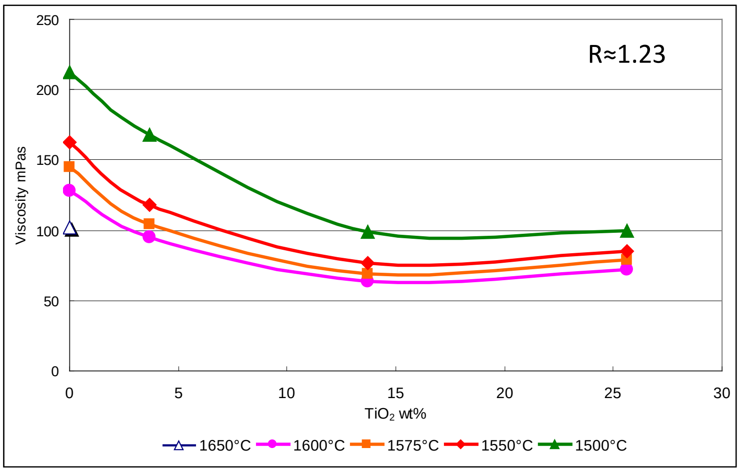

Fig.16 Viscosity variation as function of TiO_2 wt% in basicity 1.23 slags

3.2.2 CSTNA series slags

CSTNA slags have been treated in the same way as CST slags. For the easily evaporation of sodium oxide it is very important to know the chemical composition after measuring. Compared with Table 12 it is ensured that the component changed hardly during the testing at the beginning temperature of 1400°C even the slags containing sodium oxide.

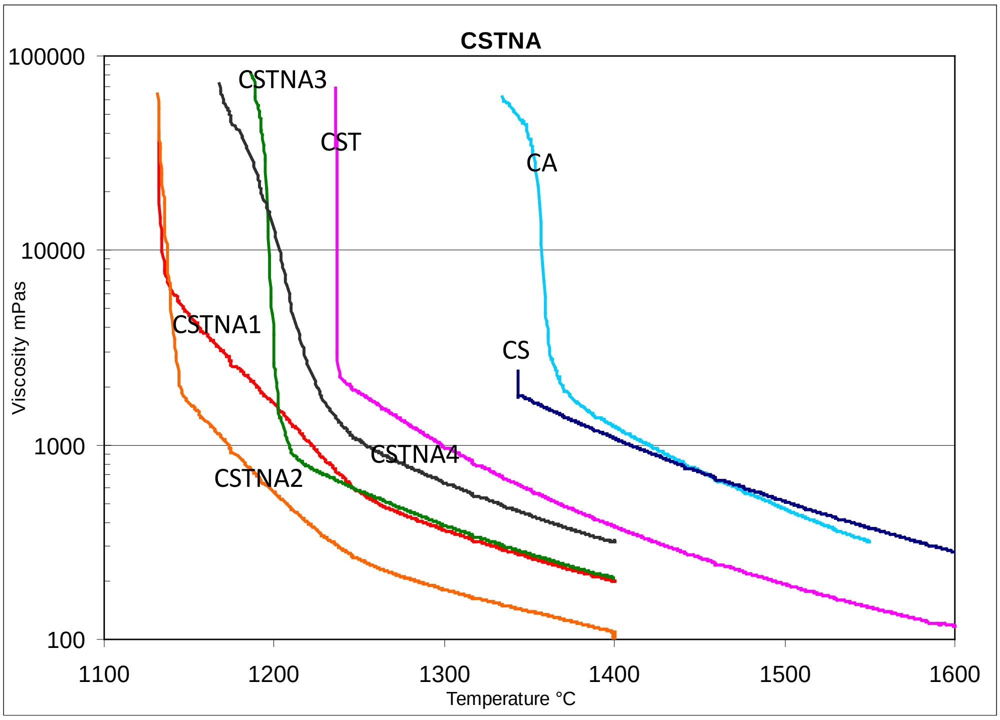

Fig.17 Viscosity-temperature curve of CSTNA series slags

3.2.2.1 Effect of Na_2O content on viscosity

Fig.17 shows all the tested viscosities and indicated that the addition of Na_2O decreases viscosity and the fusing point (Fig. 18). The mechanism of the viscosity reducing has been illustrated by P. Koazkevitch[4,5] as a network modifier. The tested results are shown in Fig.18 as function of Na_2O%, which means that the addition of sodium oxide will significantly decrease the viscosity and melting point of the slags.

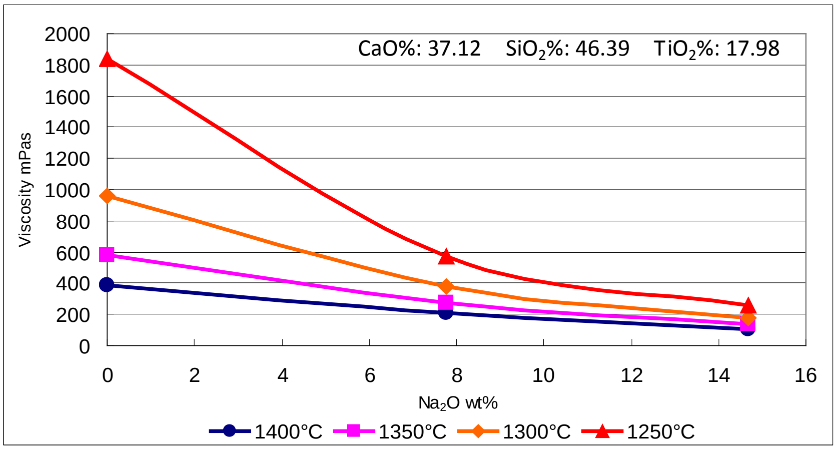

Fig.18 Viscosity variation as the function of Na_2O

3.2.2.2 Effect of Al_2O_3 content on viscosity

Based on the CaO-Al_2O_3 phase diagram only a small rang of CaO/Al_2O_3 can have a low melting temperature, otherwise it increases rapidly with little change of CaO or Al_2O_3 wt%, for example, the melting point of $12CaO_7 \cdot Al_2O_3$ phase is only 1413°C much lower than other phase. Besides Al_2O_3 is also an amphoteric substance, i.e. it acts as a network former in an acid slag and breaks up the structures in basic slags as other network modifiers. Fig.19 shows the viscosity variation as function of Al_2O_3. Small addition of doesn't seen to change viscosity a lot but with the increasing of content, the viscosity raises.

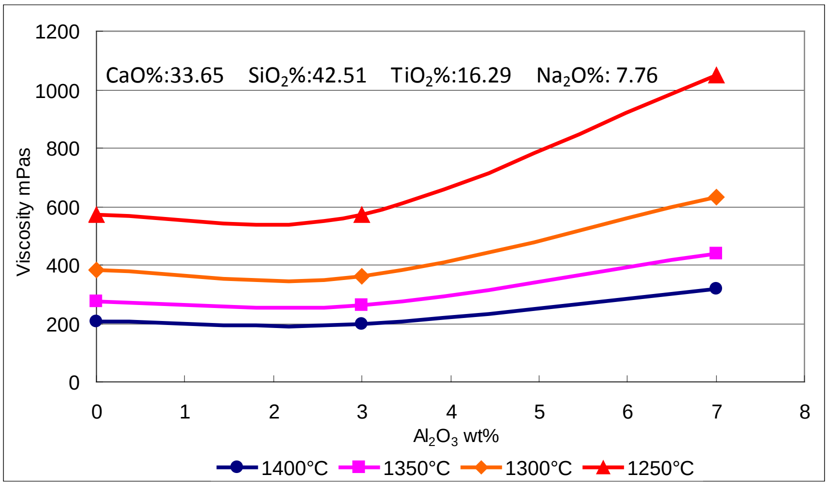

Fig.19 Viscosity variation as the function of Al_2O_3

4. Conclusions

The main effect on the error of the viscosity results is the side movements, whereas the influence of included angle between spindle and shaft appeared not very clearly. Tests low viscosity fluid revealed a magnified error, which indicated a strong request of small fluctuation of side moving and steadily rotating during entire measuring process, especially in testing low viscosity liquids.

This study has tested two series of slags containing TiO_2, Na_2O and Al_2O_3 and the viscosities were given out in a large temperature rang including homogenous zone and heterogeneous zone. For the melts high above the melting temperature viscosity is influenced by the content of network modifier, whereas the viscosity of melts near the melting temperature is affected by proportion of solid and liquid phase. Addition of Na_2O reduces the fusing temperature and viscosity as well, whereas increasing Al_2O_3 content raises rapidly viscosity

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