1. Introduction

Viscosity is one of the most important properties of the metallurgical melts (slag and steel) and an accurate information about this property is crucial for a better understanding, stabilization and modeling of the metallurgical processes.\(^5\) In addition, viscosity is also essential for the infiltration and gas atomization processes.\(^6,7\) Nowadays the most common method for the determination of viscosity at high temperature is the concentric cylinder method. Upon recent ten years, this method has been widely used for the determination of the slag viscosity.\(^8-10\) However, it is difficult to determine a precise low torque due to the mechanical friction inside the viscometer.\(^17\) Viscometers of this type consist of two concentric cylinders (a bob and a crucible) and the viscosity is determined from the calculations of the torque of bob at constant or different rotations. A few other studies were reported to use the different types of viscometers such as the rotation crucible viscometer and the oscillating plate method.\(^17-20\) The concentric cylinder method was primary used for the determination of slag viscosity. It is important to note that for the liquid metals another type of viscometer should be applied. In the following, several types of viscometer are summarized:\(^21\)

- Capillary method
- Falling body method
- Oscillating method (oscillating cylinder or plate, oscillating vessel method)
- Concentric cylinder (rotation) method

Each method is applicable for the different temperature and viscosity ranges. The capillary method has found an application in the medium temperature range. However, applying this method at higher temperatures brings about some complications related to the selection of a suitable crucible and a capillary material (i.e. dimensional stability and corrosion resistance).\(^21\) Another method is the falling body method which is used for the determination of the glass and slag melts viscosity. In addition, for the determination of viscosities in the centipoise (mPa·s) range e.g. CaF\(_2\) based slags, metals and etc. the oscillating methods have been used.\(^21\) In fact, during recent years the oscillating cylinder and plate methods were successfully applied for the determination of the steel and slag viscosity at high temperatures (\(\leq 1600^\circ\text{C}\)).\(^5,19,20,22,23\)

The present study was performed to experimentally...
investigate an applicability of the vibrating finger viscometer for the measurements of slag viscosity up to 1,600°C and to discuss the high temperature calibration errors such as deviations in geometry of the crucible, the slag stability, the fluid flow, the atmospheric conditions, as well as the difference in both measurement principles. For this purpose CaO–Al₂O₃, CaO–SiO₂ binary systems and an CaO–SiO₂–Al₂O₃–MgO industrial blast furnace slag and a glass as a reference system were chosen.

2. Experimental Procedures

2.1. Alloys and Conditions of Experiments

The determination of the viscosity of CaO–Al₂O₃, CaO–SiO₂, CaO–SiO₂–Al₂O₃–MgO slag systems and reference glass up to 1,600°C was carried out in argon 5.0 (99.999 vol.% Ar, < 2 ppm O₂) using the vibrating finger viscometer and the Anton Paar MCR 301 rheometer in molybdenum crucibles (Fig. 1).

The slags and glass were pre-melted in molybdenum crucibles under argon atmosphere in an induction furnace and the chemical compositions were analyzed afterwards using a Bruker AXS S8 Tiger (XRF). The corresponding results presented in Table 1 are described as follows: CA (CaO–Al₂O₃), CS (CaO–SiO₂), CASM (CaO–SiO₂–Al₂O₃–MgO) and glass. The weight of the slags in the crucibles was ca. 30–35 g. For the main experiments a similar construction of induction furnaces for both viscometers were used (Fig. 2). The temperature control in the furnace and in the melt was accomplished with the help of the bottom thermocouples in both furnaces.

Viscosity of the slags was measured continuously using various cooling rates of 2, 5 and 10 K/min. After the complete melting of slags and obtaining the first measuring temperature, the holding time of ca. 30 minutes was used. A temperature field in molybdenum crucibles was controlled using two thermocouples (Type B). For the determination of viscosity in the vibrating finger viscometer and in the Anton Paar MCR 301 rheometer, two types of bobs were applied which dimensions are presented in Table 2 and Fig. 2.

3. Methods of Viscosity Measurement

3.1. Rotating Bob Viscometer

The viscometer for the determination of slag viscosity at high temperature using the rotating bob method in the present work consists of the following parts: The Anton Paar MCR 301 rheometer, the cardan joint, the molybdenum rod, the melting crucible, the heating crucible (graphite), two thermocouples, the crucible positioning system and the measuring system (Fig. 2). The viscosity can be calculated using Eq. (1):

\[ \eta = \frac{M}{8 \cdot \pi^2 \cdot n \cdot H_b \left( \frac{1}{R_b^2} - \frac{1}{R_{mc}^2} \right)} \]

where \( \eta \) is the dynamic viscosity, \( M \) is the torque, \( n \) - number of rotation per minute, \( H_b \) - the height of the long side of the bob, \( R_b \) and \( R_{mc} \) - the radius of the bob and molybdenum crucible, respectively. Before measurements, only the rotation speed and torque of the Anton Paar MCR 301 rheometer have been calibrated. The immersion depth for measurements of the bobs in the slags system is calculated by Eq. (2):

Table 1. The measured composition of slags by Bruker AXS S8 Tiger (XRF) after pre-melting.

<table>
<thead>
<tr>
<th>Name</th>
<th>Chemical composition, mass.%</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>CaO</td>
</tr>
<tr>
<td>CA</td>
<td>51.62</td>
</tr>
<tr>
<td>CS</td>
<td>40.78</td>
</tr>
<tr>
<td>CASM</td>
<td>38.85</td>
</tr>
<tr>
<td>Glass</td>
<td>9.94</td>
</tr>
</tbody>
</table>

Table 2. Dimensions of the bobs in millimeters for viscosity measurements.

<table>
<thead>
<tr>
<th>Material</th>
<th>Bob (Anton Paar MCR 301), mm</th>
<th>Bob (Vibrating finger viscometer), mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>Molybdenum (99.9% Mo)</td>
<td>diameter of bob 12</td>
<td>diameter of bob 5</td>
</tr>
<tr>
<td></td>
<td>length of bob 20</td>
<td>length of bob 30</td>
</tr>
<tr>
<td></td>
<td>diameter of shaft 3</td>
<td>diameter of shaft 3</td>
</tr>
</tbody>
</table>
\[ \Delta H = H_b \left( 1 - \left( \frac{R_b}{R_{mc}} \right)^2 \right) + H_{rod} \left( 1 - \left( \frac{R_{rod}}{R_{mc}} \right)^2 \right) \quad \cdots (2) \]

where \( \Delta H \) is the immersion depth, \( H_{rod} \) is the standard measuring depth (7 mm) and \( R_{rod} \) is the radius of rod.

In this work, for the determination of the slag viscosity, the bobs were immersed into the molybdenum crucibles by applying two restrictions. Firstly, the distance between the bottom of the molybdenum crucible and the bottom of the bob needs to be more than 10 mm. Secondly, the height of the slag layer above the bob should be 7 mm (Fig. 3). Previous investigations revealed that the mentioned height of the liquid above the bob (i.e. 7 mm) led to a negligible systematic error of 1.43% by changing this depth in the range of \( \pm 3 \) mm. Before the start of each experiment, the inner diameter of molybdenum crucible \( (R_{mc}) \) and the diameter of the bob \( (R_b) \) were measured to determine the accurate height of the slag layer above the bob. The position of the molybdenum crucible relative to the axis of rotating the bob was centered by the crucible position system. Additionally, the bob at 6 rpm was recorded using special software NATIONAL INSTRUMENTS Vision Builder 2012 for Automated Inspection and the deviations from the central line of rotation were continuously measured and controlled. Consequently, the bobs with the deviations less than 1.5° and 1.5 mm from the axis of rotation were used. After the complete melting of slags and obtaining the first measuring temperature, the holding time of ca. 30 minutes was used. For the first 10 minutes, the bob was above the slags and then was gradually immersed in the slag within 5 minutes. The last 15 minutes were allocated to the holding time at a constant temperature and a constant rotation speed of the bob (30 rpm). Within the experiments, the temperature of the molybdenum crucible and the position of the molybdenum rod were continuously measured and recorded. All experiments were carried out under inert argon atmosphere (with flow rate 200 l/min (furnace) and 60 l/min (top cover) (Fig. 2)).

3.2. Vibrating Finger Viscometer

The viscosity measurement equipment was based on the recent invented patent of a new vibrating finger viscometer. The technical scheme of the viscometer is presented in Fig. 4 and the operation concept was recently described in reference. The molybdenum bob (indicated with No. 8 in Fig. 4) is set in an oscillatory motion with constant peak-to-peak amplitude of 0.625 mm. For the stable oscillatory motion an AC-powered field coil (No. 2) and neodymium magnet (No. 3) were used. The path of finger vibration is measured using a highly sensitive laser micrometer (No. 7) and a sharp-edged diaphragm (No. 5). Via a 12-bit ADC microcontroller, the field coil is powered in a rectangular mode using the bipolar transistors (No. 4). An absorber system (No. 1) is installed to avoid any external interferences of the viscometer oscillation. The vibration of the viscometer as a function of the oscillator weight and spring constant (No. 6) was set in its resonant frequency at approximately \( (26 \pm 0.1) \) Hz. In contrast to a vibrating wire viscometer or an oscillating plate viscometer, the recorded amplitude is kept constant in the vibrating finger viscometer in all measurement states.

In the case of the present viscometer, the following behavior of the measurement system was assumed:

- Newtonian behavior of investigated slags,
- Absence of induced turbulent flow within the melt container,
- Full stick condition of finger/melt interface,
- No slipping effects,
- Possible end effects of finger/melt interface are negligible small and are considered, within the pre-calibration using reference fluids,
- Absence of reflective wave interactions on the melt surface.

The viscometer was calibrated using the silicon reference oils of varying dynamic viscosity at 25°C. The product of viscosity and density \( \eta \rho \) is a function of the relative change of the field coil current \( (I_{rel}) \) of the vibrating finger
which is defined as follows:

\[
I_{\text{rel}} = \frac{I_{\text{fluid}} - \frac{I_2 - I_1}{2}}{\frac{I_2 - I_1}{2}}
\]

(3)

where \( I_{\text{rel}} \) is the relative change of the field coil current [dimensionless quantity], \( I_{\text{fluid}} \) is the average coil current at the fixed immersion depth (20 mm) of the finger in the liquid melt at a constant peak-to-peak amplitude of 0.625 mm, \( I_1 \) and \( I_2 \) are the average coil current after the achievement of a constant peak-to-peak amplitude of 0.625 mm in the atmosphere and after the immersion of the finger into the melt.

The relation between \( \sqrt{\eta} \cdot \rho \) and \( I_{\text{rel}} \) provided by using an exponential fit of calibration for the experimental data of silicon oils (Fig. 5) has been shown as Eq. (4):

\[
\sqrt{\eta} \cdot \rho = -27.45 \cdot \exp \left(-\frac{I_{\text{rel}}}{1.77}\right) - 980 \times 540.49 \cdot \exp \left(-\frac{I_{\text{rel}}}{216 \times 55.06}\right) + 980 \times 540.49
\]

(4)

Density temperature functions for the binary systems (CaO–Al\(_2\)O\(_3\) and CaO–SiO\(_2\)) and for the industrial blast furnace slag were taken from the maximum bubble pressure method measurements.\(^24\)

4. Results

The experiments were carried out on the four slags at different temperatures and were subsequently compared to the published literature data (Table 3). The information regarding to the blast furnace slag was taken (selected) from the researches of Han \etal,\(^25\) Kim \etal,\(^26,27\) Suzuki \etal,\(^28\) and Tang \etal.\(^29\) The measurements of blast furnace slags have covered the compositions of 40 to 43.3 mass. pct. CaO, 33 to 45 mass. pct. SiO\(_2\), 10 to 15 mass. pct. Al\(_2\)O\(_3\), 5 to 10 mass. pct. MgO and the experiments were carried out in temperature range from 1300°C to 1575°C. Also, the binary slags systems (CaO–Al\(_2\)O\(_3\) slag and CaO–SiO\(_2\) slag) were compared with the results obtained by Oliveira \etal,\(^30\) Urbain \etal,\(^31\) and Wu \etal.\(^32,33\)

The results of present work for the industrial blast furnace slag are in a good agreement with the data reported by Han\(^25\) and Kim\(^27\). In the work of Kim\(^27\), a Brookfield digital viscometer (rotating cylindrical method) was employed for the viscosity measurements and holding time for reaching equilibrium state was more than 30 minutes at each temperature. The aforementioned condition can be the source of deviation from results of the vibrating finger viscometer and the Anton Paar MCR 301 rheometer. In present study for comparison of the results between the two types of viscometers, cooling rate of 10 K/min was chosen; therefore the equilibrium state could not be reached. As a result, the viscosity values for the two types of viscosimeters are lower than those in the work of Kim \etal\(^27\). The viscosity values reported by Han\(^25\) were collected from the other publications and contain no information about the procedure of viscosity measurement. Meanwhile, the significant deviation from the results of Tang \etal\(^29\) and Chen \etal\(^34\) can be associated with the considerable differences in the slag compositions as well as the measurement techniques. In these two mentioned researches, Brookfield digital viscometers were applied and the viscosity of slags was estimated with an approx. holding time of 30 minutes at each temperature to ensure the chemical equilibration. As previously noted, the equilibrium state of the slag viscosity during the continuous cooling may not be achieved which can cause the incompatibility of the different results. Besides, Chen \etal\(^34\) reported data for a slag that contains 17.3 mass% of Al\(_2\)O\(_3\). This high concentration of aluminum oxide in slag can lead to the increase of viscosity as reported by Persson \etal\(^8\) and Yan \etal\(^35\). Additionally, the basicity (CaO/SiO\(_2\)) of the slag in research done by Tang\(^29\) was less than 1 (this parameter is 1.12 in the present work). It is well known that the change of basicity of slag can cause essential changes in its viscosity.

The data obtained for the CaO–Al\(_2\)O\(_3\) system was compared with the both calculated and experimental data. The experimental data presented in the research performed by Oliveira \etal\(^30\) were estimated using Brookfield LVTd viscometer at 1550°C. Results of the present work for the CaO–Al\(_2\)O\(_3\) system are in a good agreement with the data reported by Oliveira \etal\(^30\). Comparison of the experimental data of the present work using two viscosity measurement techniques with the data of Oliveira \etal\(^30\) reveals that the obtained results for the vibrating (oscillating) finger viscometer is more compatible with the data reported by Oliveira \etal\(^30\). However, there are considerable differences between the experimental results of the present work and the calculated data presented by Wu \etal\(^32,33\). Moreover, the obtained viscosity values for CaO–SiO\(_2\) system have been compared with both the rotation cup viscometer results obtained by Urbain \etal\(^31\) in the temperature range of 1552 to 1903°C and with the viscosity data obtained by simulations reported by Wu \etal\(^32,33\). Surprisingly, computed results of Wu \etal\(^32,33\) depict a better agreement with the present research as compared to the experimentally obtained values of Urbain \etal\(^31\).
5. Discussion

It is generally accepted that the rotational viscometer is the most validated and used viscosity measurement technique comparing to the other techniques. At the same time, the oscillating methods (oscillating cylinder or plate and vessel method) are more suitable for the materials with low viscosities within the viscosity range of $10^{-5}$ to $10^{-2}$ Pa·s, such as liquid steel and metals.

In this research, the vibrating finger (cylinder) viscometer for measurement of the slag viscosity up to 5 Pa·s was applied at high temperatures. In order to study the applicability of this method for the wider range of viscosities, the vibrating finger viscometer was calibrated with the silicon oils up to viscosity of 10 Pa·s. The measuring range of the vibrating viscometer is limited to the capability of a coil field to maintain the stable oscillation motion. At the viscosity of 10 Pa·s a problem with the stable oscillatory motion with the constant peak-to-peak amplitude of 0.625 mm arises. During experiments, voltage of the field coil is always 5 V and the current can be changed by a 12-bit ADC microcontroller to up to value of 10 A. Thus, the measuring range of the oscillating viscometer is essentially limited by the current in the field coil. Therefore, before the start of experiments three types of cylinders: 1. cylinder, 2. cylinder with the spherical nose and 3. cylinder with the conical nose (Fig. 6) were tested in silicon calibration oil (WACKER SILICONEOLE AK 200) to study the influence on accuracy and measuring range of the vibrating finger viscometer. The corresponding results are illustrated in Fig. 7.

The test program was carried out within approx. 2 min and was divided into three operations. Firstly, the vibrating finger was immersed into the calibration silicon oil to the depth of 20 mm. At this depth a relative current change was observed. After that, the cylinder was immersed into the calibration silicon oil to the depth of 20 mm. In the second operation, the same measurements were performed at high temperatures.

Table 3. Comparison between the experimental data and the data published in literature.

<table>
<thead>
<tr>
<th>Source of data</th>
<th>Compositions (mass%)</th>
<th>Viscosity, Pa·s</th>
<th>Temperature, K</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>CaO</td>
<td>SiO₂</td>
<td>Al₂O₃</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Blast furnace slag</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Rotating viscometer</td>
<td>40.30</td>
<td>36.20</td>
<td>12.3</td>
</tr>
<tr>
<td>Oscillating viscometer</td>
<td>40.00</td>
<td>40.00</td>
<td>10.0</td>
</tr>
<tr>
<td>Han et al.</td>
<td>42.90</td>
<td>33.00</td>
<td>15.0</td>
</tr>
<tr>
<td>Kim et al.</td>
<td>43.30</td>
<td>36.70</td>
<td>13.0</td>
</tr>
<tr>
<td>Suzuki et al.</td>
<td>40.90</td>
<td>38.10</td>
<td>10.0</td>
</tr>
<tr>
<td>Kim et al.</td>
<td>40.00</td>
<td>40.00</td>
<td>10.0</td>
</tr>
<tr>
<td>Tang et al.</td>
<td>40.00</td>
<td>45.00</td>
<td>10.0</td>
</tr>
<tr>
<td>Chen et al.</td>
<td>38.50</td>
<td>35.00</td>
<td>17.3</td>
</tr>
<tr>
<td>CaO–Al₂O₃ slag</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Rotating viscometer</td>
<td>51.51</td>
<td>0.32</td>
<td>47.78</td>
</tr>
<tr>
<td>Oscillating viscometer</td>
<td>52.00</td>
<td>48.00</td>
<td>0.28</td>
</tr>
<tr>
<td>Oliveira et al.</td>
<td>52.00</td>
<td>48.00</td>
<td>0.28</td>
</tr>
<tr>
<td>Wu et al.</td>
<td>52.00</td>
<td>48.00</td>
<td>0.28</td>
</tr>
<tr>
<td>Wu et al.</td>
<td>52.00</td>
<td>48.00</td>
<td>0.28</td>
</tr>
<tr>
<td>CaO–SiO₂ slag</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Rotating viscometer</td>
<td>40.44</td>
<td>59.03</td>
<td>0.29</td>
</tr>
<tr>
<td>Oscillating viscometer</td>
<td>40.44</td>
<td>59.03</td>
<td>0.29</td>
</tr>
<tr>
<td>Urbain et al.</td>
<td>37.60</td>
<td>62.40</td>
<td>1.70</td>
</tr>
<tr>
<td>Wu et al.</td>
<td>37.60</td>
<td>62.40</td>
<td>1.70</td>
</tr>
<tr>
<td>Wu et al.</td>
<td>40.00</td>
<td>60.00</td>
<td>0.37</td>
</tr>
<tr>
<td>Wu et al.</td>
<td>40.00</td>
<td>60.00</td>
<td>0.37</td>
</tr>
</tbody>
</table>

![Fig. 6. Geometrical parameters of the investigated bobs in millimeters.](image-url)
was measured. After determination of the relative current change, the vibrating cylinders were lifted.

It has been concluded that cylinders with the spherical or conical nose and low weight should be applied for the experiments. In the first instance, the spherical or conical nose provides a relative minor change of the current in the field coil (in comparison with the molybdenum or aluminum cylinder). At the conical and spherical nose, at the aluminum cylinder and at the molybdenum cylinder the relative current changes reach the approximate values of 0.62, 0.77 and 0.84, respectively (Fig. 7). This trend can be explained by the lower turbulence and lower weight of the fingers. In addition, the mentioned types of noses decrease the probability of so-called “end effect” which has been discussed in literature. Furthermore, the oscillating motions of cylinder can cause the formation of secondary flow and the production of flow instability.

Moreover, all types of applied cylinders (bodies) were calibrated in silicon oils using varying viscosities from 200 to 12 500 mPa·s at room temperature (25°C). Estimated viscosity values for cylinder with the spherical or conical nose can be better described than for the cylinder by mathematical functions. In fact, the mathematical function for estimation of viscosity is more accurate in the case of spherical and conical nose than for cylinder. The determined correlation coefficients are: 1. conical nose - 0.99989, 2. spherical nose - 0.99985, 3. aluminum cylinder - 0.99983, 4. molybdenum cylinder - 0.99947.

Another critical aspect for the selection of cylinders is the weight of the cylinders as the light ones are necessary for the broadening of measuring range. Heavier cylinders, molybdenum cylinder with the approx. weight of 5.6 g, require a higher level of relative current change of ca 0.84 in the field coil for the stable motion than the aluminum cylinder with the approx. weight of 5.0 g (~ 0.77) (Fig. 7).

After performing the calibration and preliminary tests with the cylinders, the main experiments using four different slags were carried out. The results are presented in Fig. 8.

Comparing the experimental data of the vibrating (oscillating) finger (cylinder) viscometer with the results obtained for the rheometer, some differences in the value of viscosity are revealed. These differences can be attributed to the:

1. differences in the geometry of the molybdenum...
cricules,
ii) different chemical composition of the slags,
iii) different temperature fields occurring in two molybdenum crucibles
iv) Taylor vortex in the rotation bob viscometer.

i) During experiments, two types of molybdenum crucibles with different external geometrical parameters but similar inner volume geometries (Fig. 1) were used. Obviously, the hardness of the metal decreases as the applying temperature approaches the melting point. Temperature of experiments (max. 1 600°C) was substantially below the melting point of molybdenum – 2 617°C. Therefore, upon the experiments the hardness limit of molybdenum should not be surpassed.

Another point to be noted is that the geometrical parameters of the molybdenum crucibles and the cylinders can be physically changed at high temperatures due to the thermal expansion. However, this statement is not valid for molybdenum as this metal has a small thermal expansion like tungsten, chromium and zirconium.40) The values of constants $A_1$ and $A_2$ for the pure molybdenum in the temperature range from 25 to 2 341°C amount to $3.91 \cdot 10^{-6}$ and $1.81 \cdot 10^{-6}$, respectively.41)

As the chemical reactions can change the geometrical parameters of crucibles, the inner diameters of them were controlled before and after each experiment. Since this effect was negligible or was not observed in our experiments, it is possible to conclude that the deviations in geometry didn’t have an influence on the accuracy of the measurements.

The main experiments were carried out in an inert argon gas with the flow rate of 200 l/min (bottom) and 60 l/min (top). Such a high level of flow rate is essential to protect the graphite heater and molybdenum crucibles from high temperature oxidation. Using an insufficient flow rate of argon, the graphite heaters and the molybdenum crucibles may react with oxygen at high temperatures which can lead to both the changes of slags chemical composition and to a temperature field change in molybdenum crucibles. Nevertheless, the molybdenum oxide has not been found in slag and the geometry of heaters faced only minor changes. Thus, the chemical composition of slags and the geometrical parameters of heaters after experiments demonstrate the sufficiency of flow rate of argon in our experiments.

ii) Chemical stability of slags may affect the accuracy of measurements. To investigate that, the chemical composition of slags after the experiment was analyzed by (XRF) and the corresponding results are presented in Table 4.

The chemical composition of the CaO–Al₂O₃ and CaO–SiO₂ slag systems are stable, while for the industrial blast furnace slag an insignificant change of the chemical composition is observed. It can be concluded that the small differences of the chemical composition within experiments should not considerably affect the estimated viscosity.

iii) The difference between the experimental data or high temperature errors can mainly be explained by the different temperature fields in molybdenum crucibles and an uncertainty in estimation of the density functions for the vibrating finger viscometer. It has been turned out that the other factors have a minor influence on the estimated values. The applied thermocouples were calibrated using high purity copper and nickel references and the deviations from the real temperature were taken into account. In addition, the density functions were determined using the maximal bubble pressure method which is a fundamental method for the precise determination of thermophysical properties.

iv) Starting of Taylor vortex can influence the accuracy of results of the rotating bob viscometer. For the given geometry of the system crucible-melt-rotating bob the the conditions Taylor vortex initiations were calculated. It was estimated that with the typical slag density of 2.70 g/cm³ and the viscosity of 100 mPa·s Taylor vortex can arise only at 745 rpm of the bob. The experiments were accomplished under 30 rpm, what is very far from necessary preconditions of vortex initiation.

Comparison of the experimental data of two types of viscometer reveals the following phenomenon: for three slags with SiO₂-content the experimental viscosity values obtained by the vibrating finger viscometer are higher than the data obtained by Anton Paar MCR 301 rheometer, whereas for the CaO–Al₂O₃ system (without SiO₂) the experimental viscosity values achieved by the vibrating finger viscometer are higher than for Anton Paar MCR 301 rheometer (Fig. 8). This phenomenon can be explained by the different measuring principle. Using the oscillating finger viscometer, the viscosity has been estimated using an oscillatory motion with constant peak-to-peak amplitude of 0.625 mm. Existence of SiO₂ in the CaO–SiO₂ system, in the blast furnace slag and in the glass leads to the formation of polymeric structure. In polymeric systems, the real viscosity of the complex dynamic viscosity is inversely proportional to the frequency of oscillations.

An alternative frequency-dependent viscosity, so-called dynamic viscosity, has been introduced by Ferry42) and can be calculated by Eq. (65):

$$
\eta^* = \left( \eta_1^2 + \eta_2^2 \right)^{1/2} = \left( G_1^2 + G_2^2 \right)^{1/2} / \omega. \quad \text{......... (5)}
$$

Where $\eta^*$, $\eta_1$ and $\eta_2$ are the dynamic viscosity, real part of real viscosity and imaginary part of complex viscosity, respectively. $G_1$ is the shear storage modulus, $G_2$ is the shear
loss modulus and $\omega$ is frequency.

As the proof of this theory can be mentioned the researches of Shin et al.\(^{43,44}\) which were mainly focused on the viscoelastic properties of calcium silicate-based mold fluxes at $1.623$ K and presents the required values for the storage modulus (elastic property) and the loss modulus (viscosity property) of the SiO$_2$-contained slags.

Thus, it is important to emphasis that another source of discrepancy between the data obtained by two viscometers can be the elastic properties of slags that reduce the measured viscosity. That is, the three cases of CaO–SiO$_2$ system, the blast furnace slag and the glass at high temperature may turn into viscoelastic liquids whose viscosities depend on the frequency.

6. Conclusion

In the current investigation, the viscosities of CaO–Al$_2$O$_3$, CaO–SiO$_2$ and CaO–SiO$_2$–Al$_2$O$_3$–MgO slags and reference glass are measured up to $1.600^\circ$C using the two viscometers. The results are discussed in the context of high temperature calibration errors, *e.g.* deviations in geometry of the crucible, the slag stability, the fluid flow, the atmospheric conditions, as well as the differences in both measurement principles. The main results can be summarized as follows:

- The measured data of viscosity for the binary slag systems (CaO–Al$_2$O$_3$ and CaO–SiO$_2$) and for the blast furnace slag were in good agreement with the literature data.
- Vibrating finger viscometer was successfully applied for the viscosities of up to $5$ Pa·s.
- Measuring range of the vibrating finger viscometer essentially depends on the current in the field coil.
- Increasing the mass of bob or finger decreases the measuring range of the vibrating finger viscometer.
- Type of the bob or finger nose influences the accuracy of the viscosity data.
- Spherical or conical nose can be preferably applied for the measurements of slag viscosity using the vibrating finger viscometer.
- Changes of the geometrical parameters of the molybdenum crucibles at high temperature were negligible.
- Chemical composition of slags was stable and thus had a minor influence on the viscosity data.
- The relative measurement difference between the two types of viscometers at up to $1$ Pa·s and up to $1.400^\circ$C was less than $30\%$.
- Viscoelastic properties of the CaO–SiO$_2$ system, the blast furnace slag and the glass at high temperature can be the main reason for the dissimilarities between the two groups of experimental data obtained by two different viscometers.

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