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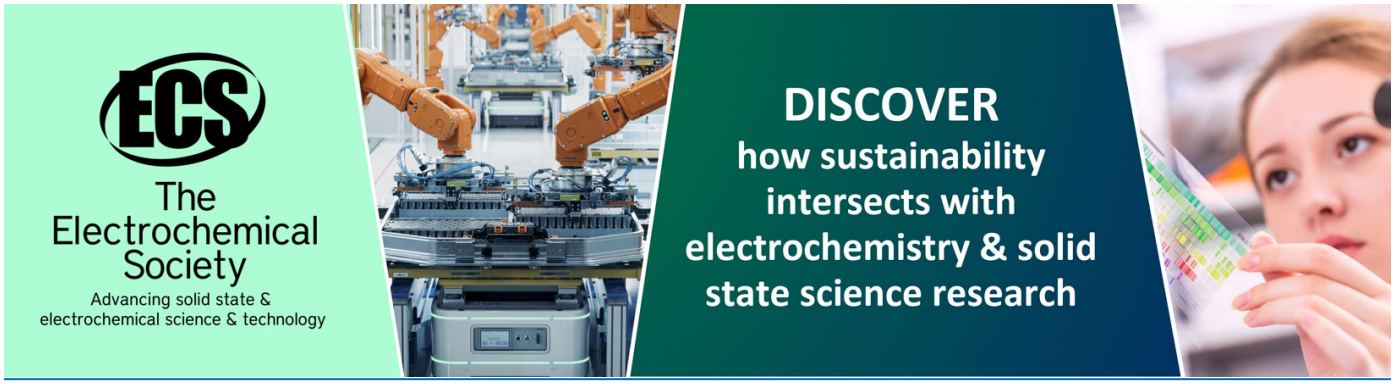

Calibration problems with the viscosity measurement of liquid metallurgical slags

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Abstract. The viscosity of slag is an important characteristic of liquid slags regarding its lubricating effect and mass transfer. For measurement, however, they exhibit considerable differences in the values reported. Therefore, the rotation method, mostly used for high temperatures areas, is investigated regarding the impacts of any geometric inaccuracies. Furthermore, problems in the centering and use of calibration slags are discussed. It appears that, with the use of a more precise rheometer with air bearing, an error of less than +/- 3 % is possible in compliance with geometric critical values and online monitoring of the central operations. The verification was carried out with a blast furnace slag, which is also proposed as a calibration slag.

Keywords: rheometer, viscosity, slags, calibration, geometry errors

1. Introduction

In metallurgy, the viscosity of slags is an important parameter. It plays a major role for transport and reaction velocities, for emulsification of slag in steel or inversely and for emptying or filling of metallurgical vessels. In continuous casting process, viscosity of the casting powder slag is responsible for lubrication and detachment of hot strand from the cold mold [1-3].

For measurements in high-temperature areas, which can be between 900 and 1800°C, values of determined viscosities of metallurgical slags show considerable deviations [4-6]. On one side, this can be explained by different measuring methods and the various crucible materials and other components of the slag contact (graphite, molybdenum, platinum). On the other side, geometrical problems can also cause higher errors in measurement. Due to the large temperature range, the thermal expansion cannot be neglected. Thus, all works have to be performed with a wide gap (more than 2 mm), leading to higher revolution speeds (rpm) and respectively low torque.

Moreover, a disadvantage is that the measuring arrangement is not visible during experiments. It is not sure, if the furnace meets the requirements. Another source of error can be changes within the slag, due to evaporation of fragile components or due to reactions of the slag components. Such reactions can also cause changes in geometry. Therefore, an appropriate inert material, an inert gas and a suitable method have to be chosen depending on the slag investigated.

2. Device

The most commonly used method of measuring viscosity is Couette's rotation method. At the Institute of Iron and Steel Technology (IEST), TU Bergakademie Freiberg, this rotation method is mostly used for slags in addition to the oscillating method [7].

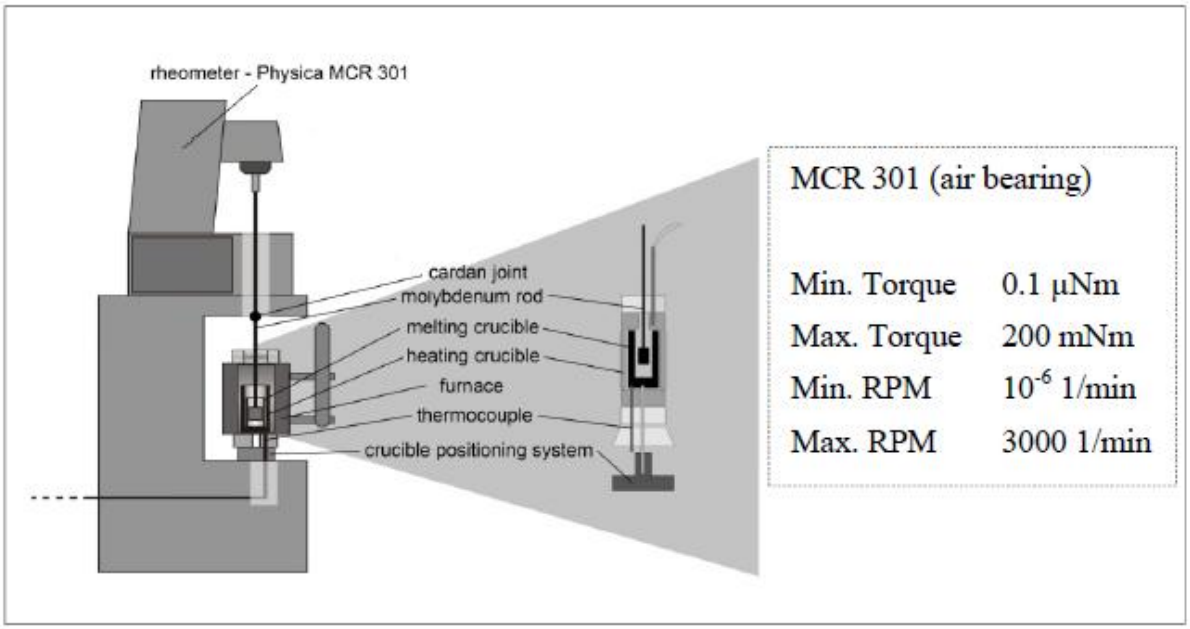

Figure 1. Scheme of the high-temperature-viscometer at IEST

The viscometer consists of a commercial rheometer MCR 301 from Anton Paar and a selfengineered high temperature device. As shown in figure 1, the furnace is approximately 0.7 m away from the rheometer head, which is conditioned by heat reasons. For the measurements of homogeneous slags, the middle shaft is equipped with a cardan joint to reduce the influence of imprecise centering. The MCR 301 is a precision-rheometer with air bearing and can measure small torques down to below 1 µNm over a wide range of revolution speeds. The advantages of this high temperature viscometer are:

- There is no calibration necessary, since the torque and rpm are measured in absolute values.
- Due to the measureable low torque, low rpm are applicable.
- Rotation and oscillation are possible.
- By employing a high frequency induction furnace, temperatures up to 1750 °C are possible.

3. Geometry

In order to estimate the influence of geometrical errors on measurements with a cardan joint, experiments with silicone oils from wacker were carried out at room temperature. These oils were found to be the most stable in regards of time dependencies. Oils between 50 and 20000 mPas were available. The manufacturer's data for these oils was confirmed by a cone-plate- measuring system with Peltier element. Since the viscosity is strongly depending on the temperature, temperature-viscosity-curves were obtained for each oils used. For all of them, a uniform dependency of $dq/dT = 2$ %/K at 25°C could be found and the influence of temperature on cold experiments respected.

Series of measurements were carried out for the influence of a curved shaft, an inclined rotating bob, an inclined crucible, a wrong immersion depth and for a bad alignment/centering. The most important results are displayed in figure 2. On the left side, the impact of an inclined crucible by itself and in combination with a tilted rotating bob is displayed. On the right side, the effect of an imprecise centering is shown.

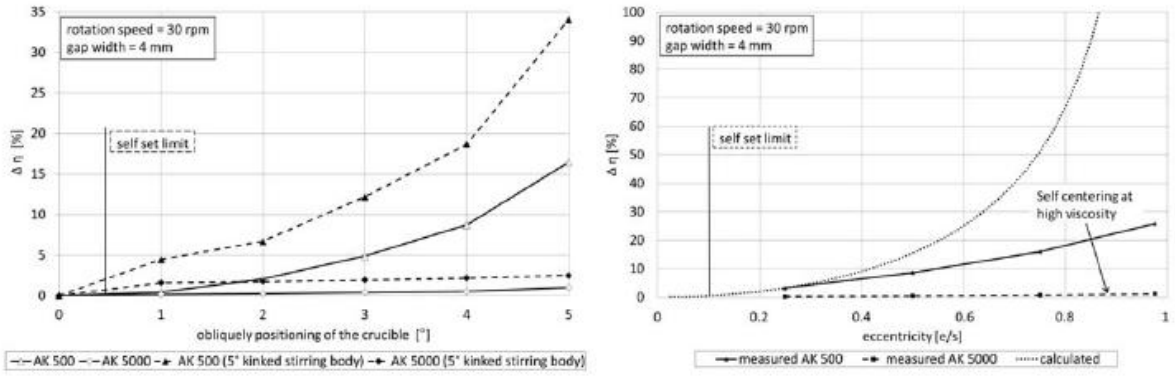

Figure 2. Influence of geometric malposition to measured viscosity

As visible, small malpositions cause acceptable measurement errors. For high viscosities, the error is not extreme since the cardan joint allows a self-centering (Figure 2, curves for AK 5000 with a viscosity of approximately 5 Pas). At low viscosities (AK 500 with approximately 500 mPas), the error quickly exceeds the acceptable limits. Therefore, it is necessary to limit malpositioning below a self-set threshold value, especially for precise measurements of thin slags. Thus, measurements at the IEST are carried out with followinglimits for geometry, since they will lead to an expected geometrical error of malposition of maximum 5%.

- Obliquity of crucible < 0.5° (inclined position)
- Obliquity of rotating bob < 1°
- Eccentricity < 0.5 mm

4. High temperature trials

In order to meet the requirements during hot temperature trials, the following procedure is maintained: prior to the heating of the furnace, the position of the crucible and the centricity of the rotating bob are controlled and adjusted if necessary. For that, a calibration tool is exchanged for the rotating bob which allows an adjustment of the crucible and a centered run. Afterwards, the rotation bob is brought into position by video recording and image processing to ensure no eccentricity or tilting. Figure 3 shows the tool for crucible adjustment on the left side and the image processing applied on the right side. Afterwards, the furnace is put over the crucible without changing its position and another visual control of the centric run of the rotation bob is done. Subsequently, the furnace is closed and the experiment continues with heating and melting the slag.

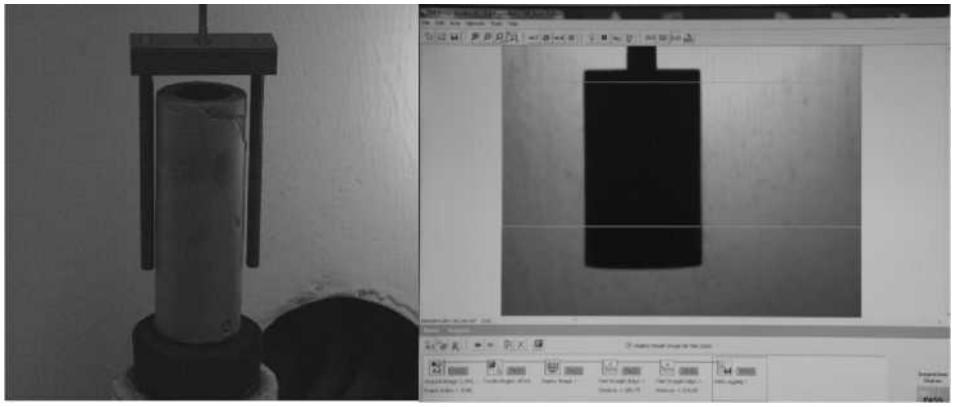

Figure 3. Tool for crucible adjustment (left), image processing (right)

To obtain the correct immersion depth, the rotating bob is slowly immersed in the crucible, while the torque is detected. An increase in torque defines the first moment of touching the slag clearly visible. Based on this height, the measuring depth is calculated (slag 7 mm above bob) and the bob is driven into position. Usually, three cooling curved are measured at one sample, which reach from a defined maximum temperature until solidification, respectively exceeding a maximum viscosity. The cooling rates are commonly 10 or 20 K/min and the rotation speed 15 or 30 rpm.

5. Reproducibility

For demonstrating the reproducibility, measurements were performed on a blast furnace slag using molybdenum as crucible and rotating bob material. Blast furnace slags crystallized stable and does not react with the crucible material. The selected composition (39.98% CaO, 35.93% SiO**2**, 12.32% M**2**O**3**, 8.76% MgO, 0.78% Fe**2**O**3**, 0.16% MnO, 0.06% K**2**O, 0.57% TiO_2, < 0.01% Na_2O) shows a low viscosity and a crystallization that starts below 1250 °C. Thirteen measurements were performed on this slag, as shown in figure 4. Large deviations appear only on two cooling curves. These deviations were due to the not centric rotating bobs. The rotation of the gauge bar was monitored by a video camera and an online image processing in order to record the data of the experiment.

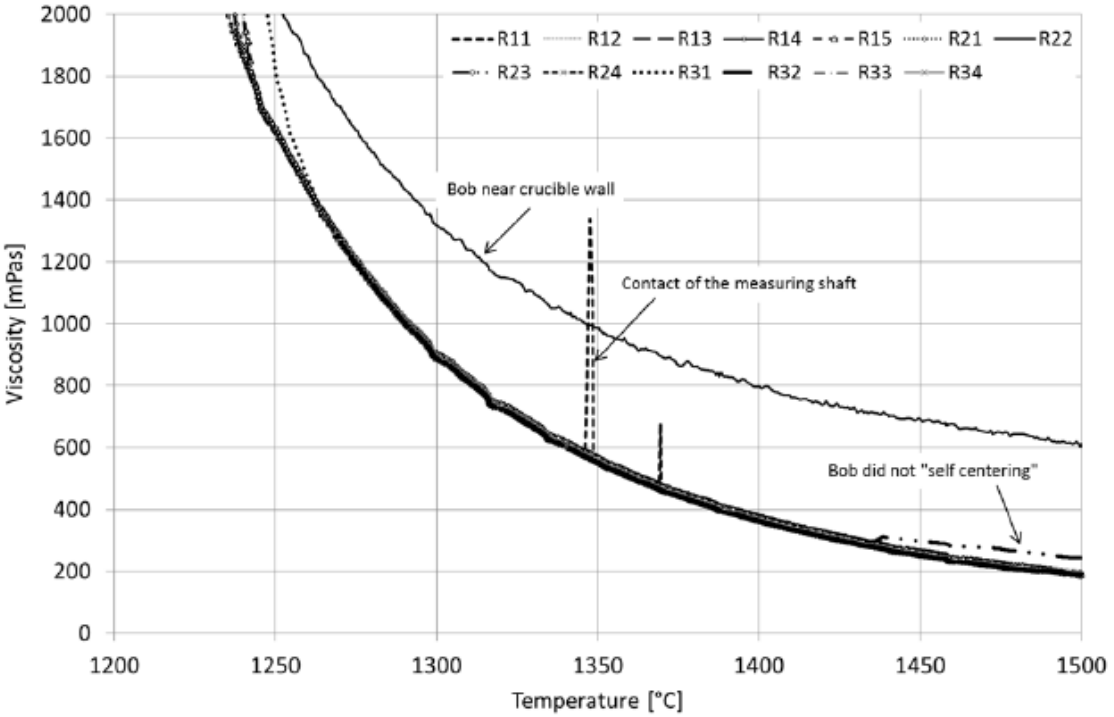

Figure 4. Viscosity temperature curves of a blast furnace slag with some centering problems

When the bob rotated at the crucible wall, an extreme error of 200% is observed. Also, small inaccuracies in centering can produce larger errors. This means that the centric rotation should always be supervised when using a cardan joint. If the incorrect curves are eliminated, the image in figure 5 is generated.

The deviations between the biggest and the smallest data lie below 5%, which appears to be sufficient for high temperature measurements.

Simultaneously, the blast furnace slag proves to be a suitable reference material. Measurements of a reference material should be done at regular intervals. A common granulated blast furnace slag (CaO-SiO_2-Al_2O_3-MgO) with low viscosity (0.2 Pas at 1500°C) should be employed. This way, no changes in composition are expected and some existing models for calculation of viscosity can also be used.

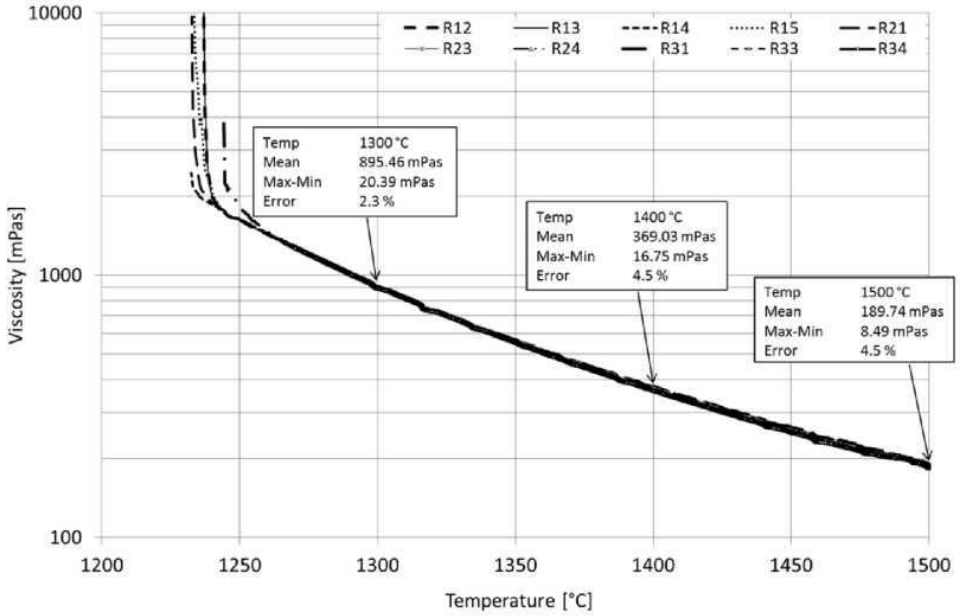

Figure 5. Measured viscosity of a blast furnace slag

Other proposals for reference materials are:

- Calibration glass by Physikalisch-Technische Bundesanstalt.
- There are three kinds available. However, the lowest viscosity is about ~16 Pas at 1400°C and it is containing PbO. Therefore, an oxidizing atmosphere and Pt as material should be used.
- Calibration slag by Mills based on SiO_2-Al_2O_3-Li_2O.
- The viscosity is about 2 Pas at 1400°C, which is the maximum temperature recommended (lowest viscosity 0.5 Pas at 1600°C).
- Continuous casting slags with CaO-SiO_2-Al_2O_3-MgO-CaF_2-Na_2O having a lowest viscosity of < 0.2 Pas at 1500°C. Yet, the high CaF_2 and Na_2O content make changes at high temperatures possible.

All the above being considered, a blast furnace slag seems to be the best choice.

6. Conclusions

Accurate viscosity measurements using the rotation method (Couette) on liquid slags to 1700°C are possible if there is a sufficient adjustment of the crucible and rotating body and a supervision of rotating shaft by online image processing (by using of two cameras for monitoring rotation outside the furnace). Furthermore, inert materials and protection gases have to be used. However measurements of a reference material should be done at regular intervals.

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