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Note

# Consideration of Spindle Immersion Depth on Determining the Viscosity of Glass Melts by Rotating Cylinder Method

**Young-Jin Kim, Ki-Dong Kim,† Seung-Heun Lee, and Jong-Hee Hwang***

*Department of Materials Science & Engineering, Kunsan National University, Kunsan 573-701, Korea*
**Korea Institute of Ceramic Engineering & Technology, Seoul 153-801, Korea*
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**ABSTRACT**

The influence of spindle immersion depth on the determination of glass melt viscosity was examined in rotating cylinder method. The exact adjustment of spindle immersion depth into soda lime silicate standard glass melts could be performed by self-constructed electric system. The results showed a slight dependence of viscosity value on the immersion depth change of spindle shaft. The viscosity error per unit length of spindle was 0.4%/mm under the present cylinder dimension.

***Key words : Glass melt viscosity, Rotating cylinder method, Spindle immersion depth***

## 1. Introduction

The viscosity is very important property in the glass manufacturing as well as in the glass science. Especially, the knowledge of viscosity of glass forming melts is not only valuable in optimizing the process operation for melting, fining and forming but also in studying their structure in the melt state if its dependence on temperature and composition is known.

In relation to the temperature dependence of viscosity, it is well known that an equation of Arrhenius type is available for the narrow temperature range. For overall range over glass transition temperature, several equations besides VFT have been suggested to describe the relationship between viscosity and temperature.[1,2] For the measurement of glass viscosity, several methods have been suggested. Especially, two methods called falling ball method and rotating cylinder method have been used to determine the viscosity of glass melts.[3-6] The most widely used method is, however, the rotating cylinder method because of its high accuracy and easy operation. Nevertheless, in this method there are some factors causing errors in viscosity measurement.

In the present work, the rotating cylinder method was discussed more from an aspect of accuracy in measurement. The influence of the immersion depth of inner cylinder (or spindle) on the melt viscosity was examined and the error occurring in the measurement of melt viscosity was suggested quantitatively.

†Corresponding author : Ki-Dong Kim
E-mail : kdkim@kunsan.ac.kr
Tel : +82-63-469-4737 Fax : +82-63-466-2086

## 2. Principle of Rotating Cylinder Method

Fig. 1 shows schematic description of inner cylinder (spindle) and outer cylinder (crucible filled with melt) used typically in rotating cylinder method. Generally the Searle type, namely the crucible is fixed while the spindle rotates, is applied to determine melt viscosity, in other words viscosity is determined via torque arising from the spindle rotation under the constant immersion depth (h) in melt.

According to DIN 53018 Part 1[7] about rotating cylinder method, melt viscosity $\eta$ in dPa·s or Poise is expressed as

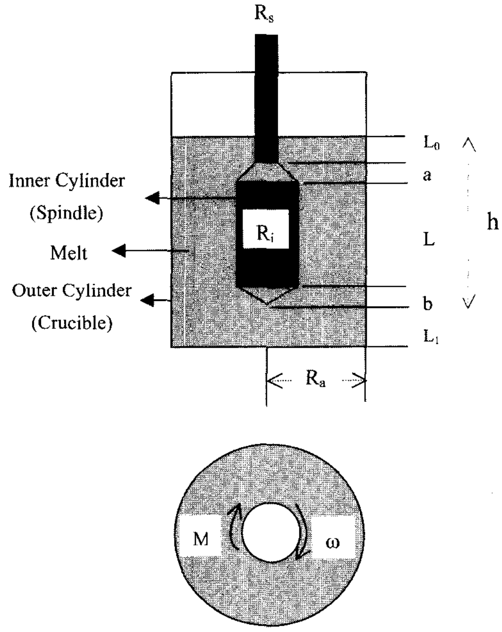


**Fig. 1.** Schematic description of cylinder geometry used in rotating cylinder method.

following equation.

$$\eta = \frac{(R_a^2 - R_i^2)}{4\pi(L+\Delta L)R_i^2 R_a^2} \cdot \frac{M}{\omega} = \frac{\tau_i}{D_i} \tag{1}$$

Here, $R_a$ is the radius of crucible (cm), $R_i$ is the radius of the spindle head (cm), $(L+\Delta L)$ is a fictive immersion depth of spindle (cm) in which $\Delta L$ means a conversion length considering $a$, $b$, and $L_0$ ($\Delta L \neq a+b+L_0$), M is the torque (dyne · cm) and $\omega$ is the angular velocity (rad/sec) expressed as $\omega = \frac{2\pi n}{60}$ in which n is rpm of the spindle.

The equation (1) can be also expressed using shear stress ($\tau_i$ in dyne/cm$^2$ or dPa) and shear rate ($D_i$ in /sec) that have in details following relation with cylinder dimension and torque.

$$\tau_i = \frac{M}{2\pi(L+\Delta L)R_i^2} = AM \tag{2}$$

$$D_i = \frac{2\omega R_a^2}{(R_a^2 - R_i^2)} = \left\{\frac{\pi}{15} \cdot \frac{R_a^2}{(R_a^2 - R_i^2)}\right\} \cdot n = Bn \tag{3}$$

Where $A = \frac{1}{2\pi(L+\Delta L)R_i^2}$, $B = \frac{\pi}{15} \cdot \frac{R_a^2}{(R_a^2 - R_i^2)}$. Equation (3) shows that $D_i$ depends only on the radii of cylinders at n rpm. It is thus easy to calculate $D_i$ at an rpm of spindle if the dimension of spindle and crucible is known. On the other hand, $\tau_i$ cant be determined simply because the term $\Delta L$ in equation (2) is not same as $a+b+L_0$ but a fictive length, although M can be measured by viscometer. Therefore, DIN 53018 Part 2[8] suggests a determination of DL by using two spindles with different $L$ and same $R_i$. However, in DIN 52312[9] and ASTM C965-96[10] related with the measurement of glass melt viscosity it is recommended to determine a so called flow-field-coefficient ($f$) as shown in equation (4) by calibration of viscometer at high temperature using standard glass melts whose viscosity ($\eta_s$) is known.

$$\eta_s = \frac{15(R_a^2 - R_i^2)}{2\pi^2(L+\Delta L)R_a^2 R_i^2} \cdot \frac{M}{n} = \frac{A}{B} \cdot \frac{M}{n} = f\frac{M}{n} \tag{4}$$

As glass melts are regarded as Newtonian fluid the $\frac{M}{n}$ must be almost constant irrespective of n value at one temperature. However, as increase of temperature $\frac{M}{n}$ would decrease due to the decrease of glass viscosity. The f could be also slightly temperature dependent considering thermal expansion of cylinder materials. Thereby, it is desirable to calibrate the viscometer in wide temperature range. The determined f at high temperature does not include the original dimension $\left(\frac{A}{B}\right)$ of cylinders with DL in equation (4) but their changed dimension due to thermal expansion of cylinders and glass melts.

Once the calibration is performed at an arbitrary immersion depth of spindle, f can be applied to determine the viscosity of unknown glass melts only under the same immersion depth of spindle.

## 3. Experimental

The computerized viscometer (RotoVisco "RV30" Hakke Co., Germany) operated by rotating cylinder technique was used to measure the viscosity of glass melts. For the calibration of the viscometer soda lime silicate standard glass I of Deutsche Glastechnische Gesellschaft (DGG) was used after full degassing at 1500°C. Instead of $f$ in equation (4), $\kappa$ was used in the present method as coefficient with following equation $\eta_s = \kappa\eta_h$ where $\eta_h$ is a measured viscosity at a spindle immersion depth of $h$ mm. Based on the absolute viscosity values ($\eta_s$) of standard glass offered by DGG, the calibration of the viscometer was performed at 1450°C under the spindle immersion depth of 35 mm by entering the optimal values corresponding to A and B of equation (4) to the computer program, and the coefficient $\kappa$ was determined at each measuring temperature between 1450 and 1000°C. Subsequently, by applying the same coefficient determined at the spindle immersion depth of 35 mm to the viscosity measurement of DGG glass melt at another immersion depth (30 mm), the influence of depth change was discussed. In order to detect the contact of spindle tip with the melt level, an extra electrical circuit was installed in furnace control box. By such electric system the immersion depth of spindle into melts could be controlled exactly. Table 1 contains the corresponding values of present Pt/20Rh-cylinders with the symbol of Fig. 1 at each immersion depth h.

## 4. Results and Discussion

Table 2 contains $\eta_s$, $\eta_{35}$, $\Delta\eta$ ($\eta_{35} - \eta_s$), and $\kappa(\eta_s/\eta_{35})$ of DGG glass melts at various temperatures where $\eta_{35}$ is a measured viscosity at the spindle immersion depth ($h$) of 35 mm. In Fig. 2 $\Delta\eta$ and coefficient ($\kappa$) versus temperature was plotted. $\kappa$-values show a slight fluctuation against temperature, but almost no temperature dependence and the fitting value of $\kappa$ is about 1.0 at $h$=35 mm. This means that the thermal expansion of cylinders and glass melts does not

**Table 1.** Immersion Depth of Spindle and Size of Cylinder Used in Rotation Viscometer (unit: mm)

| $h$ | $L_0$ | $L_1$ | $L$ | $a$ | $b$ | $R_a$ | $R_i$ | $R_s$ |
|---|---|---|---|---|---|---|---|---|
| 35 | 6 | 8 | 20 | 4.5 | 4.5 | 17.5 | 4.6 | 2 |
| 30 | 1 | 13 | | | | | | |

The symbols correspond to those of figure 1.

**Table 2.** $\eta_s$, $\eta_m$, $\Delta\eta$ ($\eta_{35} - \eta_s$), and $\kappa(\eta_s/\eta_{35})$ of DGG Glass Melts at various Temperatures. $\eta_{35}$ is Measured Viscosity at the Spindle Immersion Depth (h) of 35 mm

| Temperature (°C) | $\eta_s$ (dPa·s) | $\eta_{35}$ (dPa·s) | $\Delta\eta$ ($\eta_{35} - \eta_s$) | $\kappa$ ($\eta_s/\eta_{35}$) |
|---|---|---|---|---|
| 1450 | 102.5 | 100.3 | −2.2 | 1.02 |
| 1402 | 145.7 | 145.0 | −0.7 | 1.00 |
| 1309 | 314.7 | 326.0 | +11.3 | 0.96 |
| 1213 | 808.5 | 830.4 | +21.9 | 0.97 |
| 1112 | 2709.5 | 2674.0 | −35.5 | 1.01 |
| 1016 | 11363.7 | 11140.0 | −223.7 | 1.02 |

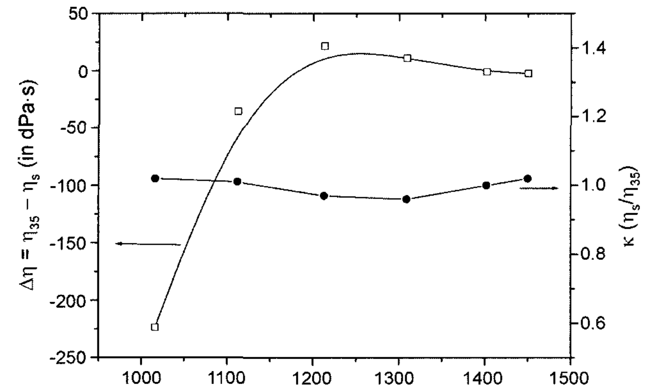


**Fig. 2.** Viscosity difference ($\Delta\eta$) between $\eta_{35}$ and $\eta_s$, and $\kappa(\eta_s/\eta_{35})$ values in the temperature range of 1450~1000°C.

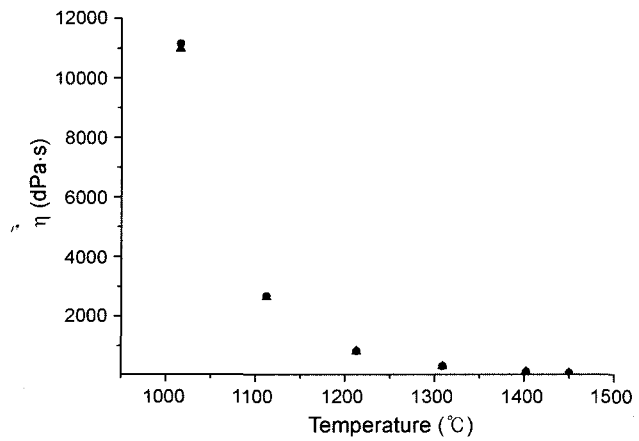


**Fig. 3.** Measured viscosity $\eta_{35}$(,) and $\eta_{30}$(7) of DGG glass melts at the spindle immersion depth of 35 and 30 mm.

give any serious effect on the viscosity measurement and thus $\eta_{35}$ is the same value as $\eta_s$ in calibrated temperature range. However, according the deviation value ($\Delta\eta$) from $\eta_s$ in low temperature range of Table 2, the influence of thermal expansion seems to occur to some degree with decrease of temperature. $\eta_{35}$ lies within ±2 – 3% error range for $\eta_s$.

Fig. 3 shows temperature dependence of measured viscosity $\eta_{35}$ and $\eta_{30}$ of DGG glass melts at $h$=35 and 30 mm, respectively. Because of large scale of Y axis there seems to be no difference between both values. However, it will cause naturally the reduction of viscosity value to apply the same factors ($A$, $B$) and the same coefficient ($\kappa$) determined at $h$=35 mm to the case of $h$=30 mm because the torque ($M$) or the shear stress ($\tau_i$) decreases also with decrease of spindle immersion depth. Fig. 4 shows clearly that $\eta_{35}$ is larger than $\eta_{30}$ since the difference of both values, $\Delta\eta_h=\eta_{35}-\eta_{30}$ is positive always. Especially, $\Delta\eta_h$ value increases with decrease of temperature. It may also derive from additional thermal expansion effect of cylinders and glass melts due to the change of spindle immersion depth. In order to acquire the similar value to $\eta_{35}$ at $h$=30 mm the factor $A$ must be corrected.

Based on the results of $\Delta\eta_h=\eta_{35}-\eta_{30}$ in Fig. 4, it can be dis-

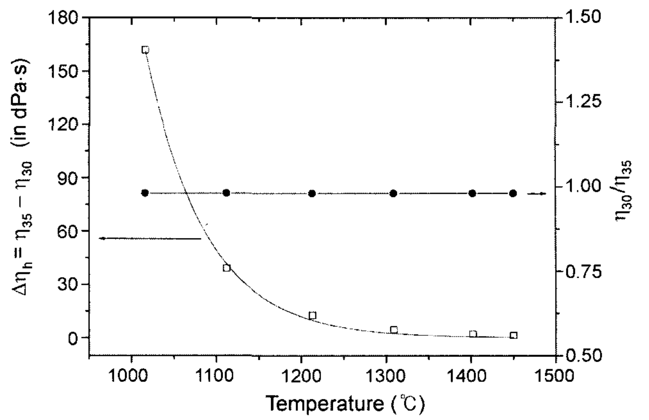


**Fig. 4.** Viscosity difference ($\Delta\eta_h$) between $\eta_{35}$, $\eta_{30}$, and $\eta_{30}/\eta_{35}$ in measuring temperature range.

cussed the influence of spindle depth change on the viscosity. The 5 mm reduction from the standard spindle immersion depth of 35 mm in the present work results in approximate 2% reduction of melt viscosity. As shown on the right side in Fig. 4, $\eta_{30}$ is about 98% of $\eta_{35}$ and thus, the error of 1 mm in immersion depth of spindle shaft under present cylinder dimension would correspond to the error of 0.4% in viscosity measurement. The error induced by immersion depth change will be larger if the spindle shaft is thicker.

## 5. Summary

There are various factors causing errors in viscosity measurement of glass melts by the rotating cylinder method. The immersion depth change of spindle is one of them. In the present work, it was examined its influence on viscosity of glass melts. As glass melts DGG standard glass I was used and the spindle immersion depth into melts could be adjusted by self-constructed electric system. According to the results, viscosity ($\eta_{30}$) at depth of 30 mm showed lower value than the viscosity ($\eta_{35}$) of 35 mm because of torque decrease due to decrease of spindle immersion depth. $\eta_{30}/\eta_{35}$ was about 0.98. Therefore, it is expected that the change of 1 mm in immersion depth of spindle shaft would induce the error of 0.4% in viscosity measurement

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