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Glass Viscosity Measurement – Rotational Viscometer Method

(ISO 7884-2:1987, Glass -Viscosity and Viscometric Fixed Points

– Part 2: Determination of Viscosity by Rotation Viscometers, MOD)

玻璃黏度测定 旋转黏度计法

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Glass Viscosity Measurement – Rotational Viscometer Method

1 Scope

This Document describes the rotational viscometer test method for the glass viscosity measurement.

This Document is applicable to the measurement of Newtonian liquid glass melt viscosity in the range of $1\text{Pa}\cdot\text{s}\sim 10^4\text{Pa}\cdot\text{s}$.

2 Normative References

The provisions in following documents become the essential provisions of this Document through reference in this Document. For the dated documents, only the versions with the dates indicated are applicable to this Document; for the undated documents, only the latest version (including all the amendments) is applicable to this Document.

GB/T 16839.1 Thermocouples – Part 1: EMF specifications and tolerances (GB/T 16839.1-2018, IEC 60584-1:2013, IDT)

3 Terms and Definitions

For the purposes of this Document, the following terms and definitions apply.

3.1 Newtonian liquid

A liquid that obeys Newton's law of viscosity.

NOTE: The viscosity of this type of fluid has nothing to do with shear rate. The viscosity is constant at a certain temperature.

3.2 Field of flow

The space filled by the glass melt and its boundaries with the inner surface of the crucible, the rotating shaft and the outer surface of the rotor.

3.3 Special instrument constant; k^*

Instrument constants integrated by taking into account the flow field coefficient, dimensional coefficient, viscometer head coefficient, and rotor viscosity coefficient.

NOTE: k^* has nothing to do with torque, speed and viscosity.

3.4 Melting temperature

The temperature corresponds to the viscosity of $10\text{Pa}\cdot\text{s}$.

3.5 Working temperature

The temperature corresponds to the viscosity of $10^3\text{Pa}\cdot\text{s}$.

3.6 VFT-equation

An empirical formula that describes the relationship between the viscosity of glass and temperature.

NOTE: VFT is the abbreviation of Vogel-Fulcher-Tammann.

4 Principles

When a rotational viscometer is used to measure the high-temperature viscosity of glass melt, the motor drives the rotor to continuously rotate in the glass melt, and friction force is generated between the rotor surface and the contacting glass melt interface. The friction force is related to the viscosity of the glass melt. Therefore, changes in the rotation speed and torque of the rotor reflect the viscosity of the glass melt. The greater the torque is, the greater the viscosity of the glass melt is. The higher the rotation speed is, the smaller the viscosity of the glass melt is. vice versa. The torque and rotational speed of the rotor in the glass melt are measured experimentally, and the viscosity of the glass melt is calculated by using Formula (1).

$$\eta = k^* \times \frac{\tau}{n} \dots\dots\dots (1)$$

Where:

η – viscosity, in $\text{Pa}\cdot\text{s}$;

k^* - constant of special instrument;

τ – torque applied to the rotator, in $\text{N}\cdot\text{m}$;

n – speed of rotator.

The high-temperature viscosity of the glass melt is mainly related to the composition and temperature of the glass. The viscosity plot of the glass melt with a specific composition measured at different temperatures constitutes the viscosity-temperature curve of the glass.

by the reference glass and the same viscosity value in Appendix C is greater than 10 °C, the instrument status shall be adjusted to meet the usage requirements.

7.2 Testing

7.2.1 Place the crucible containing the glass sample to be measured into the high-temperature furnace of the viscometer. Make sure that the crucible is placed stably on the base.

7.2.2 Set the test condition parameters, including the heating rate of the high-temperature furnace (recommended 5°C/min~10°C/min), upper limit temperature, lower limit temperature, holding time, cooling rate during the test (recommended 1°C/min~3°C/min), reheating rate after testing. When setting the upper limit temperature, in addition to considering the possible chemical reactions between the glass melt and the crucible and rotor materials, it is also necessary to consider the volatilization and devitrification tendencies of the glass.

For volatile glass, the upper limit temperature should not be too high, as long as the bubbles can be fully discharged (refer to the temperature corresponding to 100Pa·s of the glass melt). For easily devitrified glass, it should use a faster cooling rate during measurement. The temperature change rate should refer to the data set when calibrating the instrument using standard glass. When setting parameters, measurements should be made within a short period of time to avoid changes in the chemical or physical properties of the glass during long-term measurements. The viscosity measurement can be carried out under uniform cooling conditions; or the insulation setting can be performed at a specific temperature during the cooling process, and the viscosity can be measured continuously.

7.2.3 Start heating and temperature-rising the sample to the set upper limit temperature, and keep it warm for no less than 10 min.

7.2.4 Set the initial speed of the viscometer.

7.2.5 Slowly lower the rotor and start the rotor rotation. Once the bottom of the rotor contacts the molten glass, the torque on the viscometer head starts to rise from 0, indicating that the rotor has contacted the surface of the glass melt. Then lower the rotor to ensure that the rotor is completely immersed in the glass melt and ensure that the location where the rotor is immersed into the glass melt is consistent during each test.

7.2.6 The measurement officially starts, and the rotor speed and torque are continuously read during the cooling process.

7.2.7 Stop the rotor rotation after the furnace temperature drops to the set lower limit temperature according to the set testing procedure or when the measured viscosity reaches the maximum set value. However, if it is found that the rotor speed decreases sharply, the rotor rotation can be stopped when the furnace temperature is still higher than the set lower limit temperature to avoid deformation of the rotating shaft due to excessive viscosity of the glass melt.

7.2.8 When the furnace temperature quickly returns to the set value, lift the rotor to completely leave the surface of the glass melt. After the residual glass melt drips from the rotor, lower the crucible base, take out the crucible and pour out the glass melt.

In the event of a sudden failure of the instrument or phase separation or crystallization of the glass, the rotor rotation shall be stopped manually; and the heating program shall be reset immediately to increase the glass melt temperature and facilitate the lifting of the rotor and meter head.

7.2.9 After lifting the crucible base and resetting it, stop the instrument heating system.

7.2.10 Shut down the instrument or after continuing to collect sample temperature and furnace temperature to shut down the instrument.

7.3 End of test

7.3.1 After the test sample is cooled to room temperature, its appearance non-uniformity (such as devitrification, traces of dissolution of crucible and rotor materials, bubbles, discoloration) shall be observed. All unusual changes in materials in contact with the glass melt shall be considered and recorded.

7.3.2 Soak and clean the cooled platinum rotor with hydrofluoric acid. If a platinum crucible is used, the residual glass frit can be removed by pickling.

8 Data Processing

8.1 Data calculation

According to the instrument parameters and the rotor speed and torque recorded by the system, the glass viscosity value corresponding to each temperature is calculated and given by Formula (1). The temperature error corresponding to the same viscosity during two parallel tests on the same sample shall be no greater than 10°C. Otherwise, the re-test or retest after the instrument is calibrated.

8.2 Result presentation

The measurement result shall be test data or viscosity-temperature curve, with temperature as the abscissas and viscosity as the ordinates (as shown in Figure 3). Among them, the temperature unit is °C, and the data is accurate to integer digits; the viscosity unit is Pa·s, and the η value is accurate to one digit after the decimal point. When taking the $\lg\eta$ value, it is accurate to two digits after the decimal point. As needed, use the VFT equation to fit the temperature points corresponding to 10 Pa·s and 10³ Pa·s on the curve obtained to determine the glass melting temperature and working temperature.

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Contact: Wayne Zheng, Sales@ChineseStandard.net

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