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Test Method for Melt Viscosity of Glass

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Test Method for Melt Viscosity of Glass

1 Scope

This document describes the test principle of the test method for melt viscosity of glass, and specifies the instruments and equipment, specimen preparation, instrument calibration, test and result, and test report.

This document is applicable to the viscosity test of glass products in high-temperature melt state.

2 Normative References

This document does not have normative references.

3 Terms and Definitions

The following terms and definitions are applicable to this document.

3.1 viscosity

The internal resistance coefficient of fluid against shear deformation.

NOTE: the engineering unit is dPa • s, which is called poise.

3.2 melt viscosity

The viscosity of glass in a molten and flowing state.

3.3 glass melt

A glass that manifests liquid characteristics when it is above its softening point temperature.

4 Test Principle

Based on the principle of the coaxial rotation method, heat the glass to the molten state, immerse the rotor in the glass melt and rotate it at a certain speed; measure its torque and rotational speed, then, the relations between the viscosity of the glass melt, the torque and rotational speed comply with Formula (1).

$$\eta = \frac{K \cdot M}{\omega} \qquad \cdots\cdots(1)$$

Where,

η---the viscosity;

K---the instrument coefficient;

M---the measured torque;

ω---the rotational speed.

5 Instruments and Equipment

5.1 Electronic Balance

The minimum division value of the electronic balance shall be not greater than 0.1 g, and the maximum measuring range should be 500 g.

5.2 Test Device

The schematic diagram of the glass melt viscosity test device is shown in Figure 1. The requirements are as follows.

a) The viscosity test range is $10^{1.5}$ dPa • s ~ $10^{4.5}$ dPa • s.

b) The heating temperature of the heating furnace is not lower than 1,600 °C, the temperature control accuracy is not less than ± 2 °C, and the heating / cooling rate is 1 °C/min ~ 10 °C/min.

c) The rotor and crucible should be made of platinum-rhodium alloy, and the shape of the rotor should be a biconical cylinder, as shown in Figure 2; the crucible should be an open conical structure, with an inner diameter of 50 mm ~ 60 mm at the bottom, an inner diameter of 70 mm ~ 80 mm at the mouth, and a height of not less than 60 mm; the ratio of the average inner diameter of the crucible to the diameter of the rotor shall be not less than 5. The average inner diameter of the crucible refers to the average value of the maximum diameter and the minimum diameter.

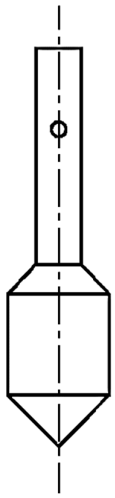

Figure 2 -- Schematic Diagram of Rotor

6 Specimen Preparation

Choose glass with no bubbles, no impurities, no disfigurement, and a clean surface, preferably larger flakes or blocks. The specimen loading should not exceed the crucible mouth, and the loading mass should be controlled between 200 g ~ 300 g, so as to ensure that the rotor can be immersed in the glass melt.

7 Instrument Calibration

It is advisable to choose certified national reference material / reference sample of glass viscosity or reference material of standard viscosity liquid (serial No.: GBW13612, GBW13613, GBW13614) for calibration, see Appendix A.

When using the reference sample of glass viscosity for calibration, carry out the test in accordance with the test conditions specified in the reference sample certificate. When the test results exceed the uncertainty range specified by the reference sample, the instrument shall be systematically corrected and adjusted.

When using the standard viscosity liquid for calibration, place the beaker containing the standard viscosity liquid in the water bath, set the temperature of the water bath to the temperature specified by the standard viscosity liquid, and leave it for no less than 1 hour. When the temperature of the standard viscosity liquid stabilizes at the specified temperature ± 1 °C, put the rotor connected to the head into the standard viscosity liquid, measure the viscosity of the standard viscosity liquid, and calculate the ratio of the test viscosity to the standard viscosity; use it to correct the instrument coefficient K, which is inserted into the device or measurement software.

The instrument calibration frequency should be set to once a year, and the instrument coefficient shall be corrected after each replacement of the rotor.

8 Test and Result

8.1 Test Procedures

The melt viscosity measurement process is as follows.

a) Vertically and densely place the glass specimens to be tested in the crucible.

b) Place the crucible holding the glass specimens on the sample table of the heating furnace, and place it in the center of the heating furnace;

c) In accordance with the heating section, thermal insulation section and cooling section, set the temperature control program. At the heating section and thermal insulation section, the glass specimens can be heated and converted into the melt. The thermal insulation temperature is set at the corresponding temperature when the glass viscosity is 10^2 dPa • s or the temperature provided by the entrusting party. When the temperature exceeds the upper limit of the operating temperature of the instrument, the upper limit temperature of the instrument should be adopted, and the cooling section is used for the measurement of glass viscosity; the heating rate should be 5 °C/min, the thermal insulation time is 10 min ~ 30 min, and the cooling rate is 2 °C/min; for glass with volatile components, the maximum temperature can be appropriately reduced, so as to reduce the volatilization of volatile components.

d) Open the heating furnace, in accordance with the set temperature control program, heat it up;

e) When entering the thermal insulation section, hang the rotor and fall into the furnace for pre-heating.

f) Before the end of the thermal insulation section, put the viscosity measuring head in the operating state, and adjust the head lifting mechanism, so that the rotor slowly drops, meanwhile, use tweezers to gently stir the rotor connecting rod, so that the rotor continuously swings during the slow drop. When the rotor suddenly stops swinging, it indicates that the top of the rotor just came into contact with the glass liquid surface. Record the location of the viscosity measuring head at that moment. Take this location as a benchmark, continue to descend the rotor, until reaching the specified descending distance, so that the main body of the rotor is completely immersed in the glass liquid.

g) After the temperature curve enters the cooling process, start collecting information, such as: specimen temperature, rotational speed of rotor, and torque, etc. The software automatically calculates the instantaneous viscosity value and records it.

h) When the glass viscosity reaches $10^{4.5}$ dPa • s, stop collecting data and save the collected data.

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