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Test methods for physical properties of liquid metals - Part 3: Determination of viscosity

液态金属物理性能测定方法 第3部分:黏度的测定

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0 Introduction and position in the series

The foreword records that this is part 3 of GB/T 41079, Test methods for physical properties of liquid metals, and that parts 1 to 3 had been published: density, electrical conductivity and viscosity.

The introduction explains that liquid metals are a broad class of alloys that are liquid at room temperature or at their working temperature, with a wide liquid range, high thermal conductivity and strong electrical conductivity, used in thermal control and energy, printed electronics, biomedicine and flexible machines; that because of their particular physico-chemical properties the existing method standards for metallic materials or for liquids are largely unsuited to them; and that the series is meant to build a set of methods for the physical parameters that production and trade in liquid metal products call for. It states that GB/T 41079 establishes eight methods for the properties in common use and is planned to run to eight parts: density, electrical conductivity, viscosity, thermal conductivity and thermal diffusivity, coefficient of thermal expansion, specific heat capacity, surface tension and contact angle.

The introduction adds that flow and deformation underlie many of the uses of liquid metals and that viscosity characterizes their rheological behaviour; that the capillary, oscillating vessel, rotational and oscillating plate methods have all been used and that their results differ considerably; and that, apart from the rotational method, all of them rest on the assumption that the sample is a Newtonian fluid, whereas in practice the oxide layer that is hard to avoid on the surface gives liquid metals a marked non-Newtonian character. The rotational method was chosen for this reason, and what it measures is the apparent viscosity.

3 Terms and definitions

3 adopts the terms of ISO 3219-1:2021 and adds five of its own.

3.1 shear strain: the deformation rate of the sample caused by a tangential displacement; the note states that it is dimensionless.

3.2 shear rate: the rate of change of the shear strain with time; the note gives the unit as reciprocal seconds.

3.3 shear stress: the ratio of the tangential force to the sheared area; the note gives the unit as the pascal.

3.4 non-Newtonian fluid: a fluid whose shear viscosity changes with the shear rate, the shear stress or the time, or with more than one of them.

3.5 apparent viscosity: the internal friction characteristic of a fluid flowing under a given shear stress and shear rate, its value being the ratio of the shear stress to the shear rate. Note 1 gives the unit as the pascal second. Note 2 explains that when a coaxial cylinder rotational viscometer is used the shear rate in the fluid is not the same along the radius, so that for a non-Newtonian fluid the result is not a true viscosity in the strict sense and is usually called an apparent viscosity.

4 Principle

4 states the principle: a rotational viscometer with the properties laid down is used, the motor torque turns the rotor in the liquid metal so that a shear strain of a given rate is produced in it, and the viscosity is calculated from the shear rate and the torque that corresponds to it.

5 Apparatus

5.1.1 calls for a coaxial cylinder rotational viscometer or rheometer meeting ISO 3219-2:2021. The measuring system is to have two rigid symmetrical coaxial surfaces with the fluid under test between them, is to accept interchangeable inner and outer cylinders, is to produce a series of rotational frequencies laid down, stepwise or continuously, and is to measure the corresponding torque, or conversely to produce a given torque and measure the corresponding rotational frequency.

5.1.2 recommends that the inner and outer cylinders be treated against corrosion or made of a material that does not dissolve in or react with the sample at the test temperature, their dimensions meeting Figure 1. The legend of that figure names the sample, the outer cylinder and the inner cylinder, and the figure fixes a set of ratios between the dimensions and an expression for the sample volume. Those expressions are broken by the extraction and are not reproduced here; the symbols they use are: the radius of the shaft, in centimetres; the radius of the inner cylinder, in centimetres; the inner radius of the outer cylinder, in centimetres; the flare angle of the bottom face of the inner cylinder, in degrees; the length of the inner cylinder, in centimetres; the distance from the lower edge of the inner cylinder to the bottom of the outer cylinder, in centimetres; the depth to which the shaft is immersed in the sample, in centimetres; the ratio of the inner diameter of the outer cylinder to the outer diameter of the inner cylinder; and the volume of sample in the cylinder, in cubic centimetres.

5.1.3 limits the torque measuring error to +/-1 % of the full range and the rotational frequency error, within the normal working range of the instrument, to +/-1 % of the measured value. 5.1.4 requires a torque resolution better than 0.01 micronewton metre, an angular displacement resolution of not more than ten to the power minus eight radians per second, and a lowest measurable viscosity of not more than 0.5 millipascal second. 5.1.5 limits the torque consumed by system friction when the inner cylinder turns to 1 micronewton metre.

5.1.6 requires the instrument to acquire, store and display the torque, the rotational speed, the shear rate, the temperature and the time, and to be able to derive the viscosity, the shear stress and other useful quantities from those signals. 5.1.7 requires a controlled temperature environment for the sample readable to within +/-0.1 °C over a range from room temperature to 200 °C, with a temperature fluctuation of not more than +/-0.5 °C and a temperature uniformity of not more than +/-1.0 °C.

5.2 calls for a protective atmosphere device able to supply an inert gas that keeps the sample from oxidizing too quickly during the test; the gas may be nitrogen or argon with a volume fraction of not less than 99.999 %.

6 Calibration of the instrument

6 requires the viscometer or rheometer to be calibrated by measuring the torque parameters or by using reference fluids of known viscosity. The reference fluid may be a non-Newtonian fluid of viscosity close to that of the sample, or at least three Newtonian fluids of different viscosities lying within the viscosity range of the sample.

7 Sample

7.1 requires the oxide layer to be removed from a liquid sample before the test, or the sample to be drawn from below the oxide layer when it is taken; a solid sample may be made into small pieces or granules easy to load into the cylinder, the surface oxide layer being scraped off before the test.

7.2 refers the volume of sample needed to Figure 1, the amount of a solid sample being calculated from its density. Table 1 pairs four inner cylinder diameters in millimetres with the corresponding sample volumes in cubic centimetres. The four pairs are printed one under the other and the two rows have the same number of entries, but the third pair does not agree with the volume the geometry of Figure 1 gives for that diameter; because the disagreement cannot be resolved from the document alone, the figures are not reproduced here.

8 Test conditions

8.1 requires the lowest test temperature to be more than 5 °C above the melting temperature of the sample.

8.2 states that because of the surface oxide layer that cannot be avoided the liquid metal may be treated as a shear-thinning non-Newtonian fluid, so that the shear rate to which the apparent viscosity refers has to be fixed. Unless otherwise required, and so that measurements made on different instruments can be compared, the shear rate is chosen from the series 1.0, 2.5, 5.0 and 10.0 reciprocal seconds. Note 1 warns that as the angular speed of the inner cylinder rises a Taylor vortex may appear in the liquid metal, which limits the highest shear rate at which the measurement is still valid. Note 2 defines shear thinning as the property by which the steady shear viscosity falls as the shear rate or the shear stress rises.

9 Test procedure

9.1 admits the inert gas at the bottom of the outer cylinder to drive out the air, at a flow rate of 20 mL/min to 100 mL/min for not less than 10 min.

9.2 loads the liquid metal sample into the outer cylinder under the protective atmosphere, heats it to the test temperature and holds it there for at least 10 min; for a solid sample the holding time is to be long enough for the sample to melt completely.

9.3 lowers the inner cylinder slowly into the sample to the depth shown in Figure 1 and holds it at the test temperature for at least 10 min.

9.4 selects the shear rate according to 8.2 and starts the measurement, each measurement lasting not less than 60 s; where several shear rates are used the measurement starts at the lowest and works up to the highest.

9.5 continues measuring at the chosen shear rate until two consecutive results differ by not more than 3 %, and takes the mean of those two; if the apparent viscosity keeps fluctuating and does not settle, the mean of five consecutive results is taken.

10 Treatment of the test data