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**Fine ceramics (advanced ceramics,  
advanced technical ceramics) —  
Measurement of viscosity of  
ceramic slurry by use of a rotational  
viscometer**

*Céramiques techniques — Mesure de la viscosité des céramiques en  
suspension au moyen d'un viscosimètre rotatif*

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ISO copyright office  
CP 401 • Ch. de Blandonnet 8  
CH-1214 Vernier, Geneva  
Phone: +41 22 749 01 11  
Fax: +41 22 749 09 47  
Email: [copyright@iso.org](mailto:copyright@iso.org)  
Website: [www.iso.org](http://www.iso.org)

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## Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see [www.iso.org/directives](http://www.iso.org/directives)).

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For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: [www.iso.org/iso/foreword.html](http://www.iso.org/iso/foreword.html). (standards.iteh.ai)

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# Fine ceramics (advanced ceramics, advanced technical ceramics) — Measurement of viscosity of ceramic slurry by use of a rotational viscometer

## 1 Scope

This document specifies a method for measurement of the viscosity of a ceramic slurry using a rotational viscometer.

## 2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories*

ISO 20507, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Vocabulary*

## 3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 20507 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

### 3.1

#### shear stress

stress acting on planes parallel to the direction of flow when the fluid is subject to laminar flow

Note 1 to entry: The SI units are pascals (Pa).

### 3.2

#### shear rate

gradient of laminar flow rate perpendicular to the fluid flow

Note 1 to entry: The SI units are s<sup>-1</sup>.

### 3.3

#### viscosity

ratio of shear stress to shear rate

Note 1 to entry: This ratio is representative of the internal resistance of the fluid to flow.

Note 2 to entry: The SI unit of viscosity is Pa × s.

Note 3 to entry: mPa × s = 1 cP in terms of c.g.s. units [where 1 P = 1 g/(cm × s)].

Note 4 to entry: Viscosity is sometimes called 'dynamic viscosity' or 'shear viscosity' for clarification. The measured viscosity is conventionally called 'apparent viscosity' since the gradient of shear rate is not identical for all parts of the spindle.

### 3.4

#### **Newtonian fluid**

fluid in which shear stress is proportional to shear rate

Note 1 to entry: The ratio of shear stress to shear rate is viscosity.

### 3.5

#### **non-Newtonian fluid**

fluid in which shear stress is not proportional to shear rate

Note 1 to entry: The non-Newtonian behaviour can be determined by measuring shear stress using varying shear rate (or angular velocity). Viscosity of the non-Newtonian fluid changes with the shear rate, in contrast with Newtonian fluid, which has constant viscosity with the shear rate.

### 3.6

#### **thixotropy**

flow behaviour of fluid that shows time dependence, such that the apparent viscosity decreases with time for a constant shear rate, and recovers slowly with withdrawal of the shear force

### 3.7

#### **shear thickening**

<dilatant> increase of shear viscosity of fluid when the shear rate (or stress) is increased

### 3.8

#### **shear thinning**

<pseudoplasticity> decrease of shear viscosity of fluid when the shear rate (or stress) is increased

### 3.9

#### **Bingham plastic**

linear shear stress/shear rate relationship starting from a finite yield stress value below which the fluid does not flow

### 3.10

#### **flow curve**

curve showing the relationship between shear rate and shear stress

Note 1 to entry: See [Figure 1 a](#)).

### 3.11

#### **viscosity curve**

curve showing the relationship between shear rate (or shear stress) and viscosity

Note 1 to entry: See [Figure 1 b](#)).

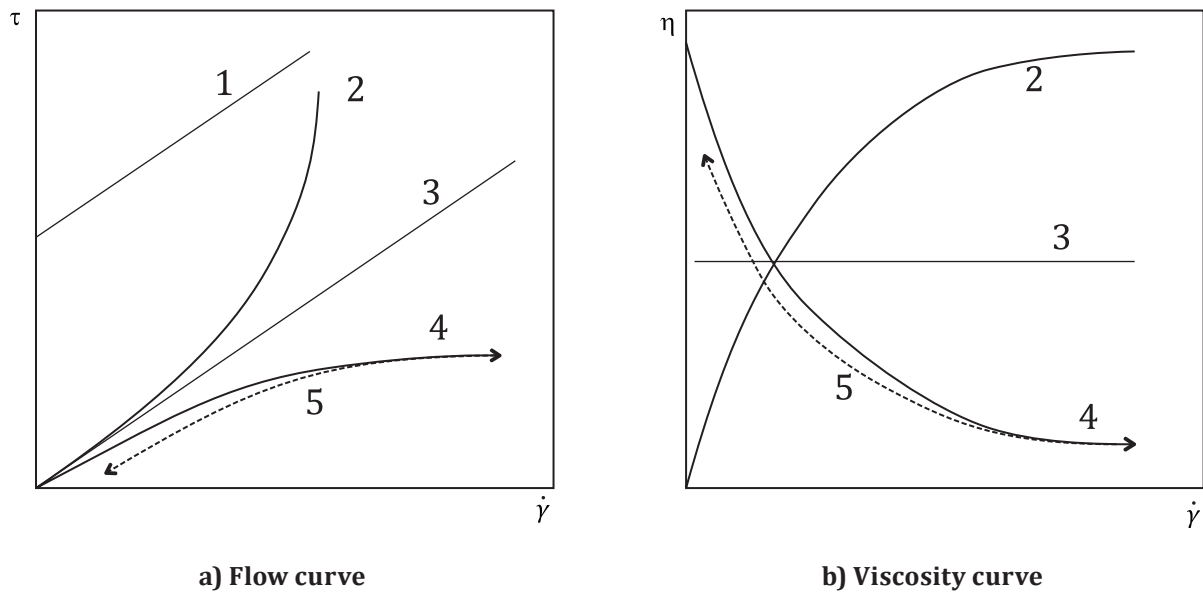
### 3.12

#### **ceramic slurry**

suspension of grinding frit, clay or ceramic powders mixed or dispersed in water or other liquid

Note 1 to entry: ceramic slurry is used in ceramic processes such as dip coating, spray coating, screen printing, slip casting, tape casting, spray drying and polishing.

Note 2 to entry: The category may include low-viscosity colloids with small particles with sizes in the nanometre or micrometre range, and high-viscosity pastes with inhomogeneous mixtures.

**Key**

$\dot{\gamma}$	shear rate	2	shear thickening (dilatant)
$\tau$	shear stress	3	Newtonian
$\eta$	viscosity	4	shear thinning (pseudoplastic)
1	Bingham plastic	5	thixotropy (time dependent)

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**Figure 1 — Summary of rheological behaviours**

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## 4 Principle

### 4.1 Rotational viscometer with defined shear rate

The viscosity,  $\eta$ , of a ceramic slurry measured using a rotational viscometer with a defined shear rate is determined using [Formula \(1\)](#):

$$\eta = \frac{\tau}{\dot{\gamma}} \quad (1)$$

where

$\eta$  is the viscosity, in Pa  $\times$  s;

$\tau$  is the shear stress, in Pa;

$\dot{\gamma}$  is the shear rate, in s<sup>-1</sup>.

NOTE Symbols are in accordance with ISO 80000-4.

### 4.2 Single cylinder viscometer

A spindle of cylindrical or disk-like shape is driven in the ceramic slurry at a fixed angular velocity. Torque is developed from the fluid resistance, and it depends on the viscosity of the slurry. Theoretically,

shear rate (or shear stress) cannot be determined, but viscosity can be measured as a function of angular velocity.

NOTE The gradient of shear rate is not identical for all parts of the spindle. Therefore, the measurement result is conventionally called apparent viscosity because it is not the viscosity determined from the gradient of a known shear rate.

## 5 Apparatus

### 5.1 General

Rotational viscometers have various measuring geometries. Representative rotational viscometers having a defined shear rate are a coaxial cylinder system ([Annex A](#)), a cone and plate system ([Annex B](#)), and a parallel plates system ([Annex C](#)). In contrast, single cylinder viscometers without a defined shear rate ([Annex D](#)) are able to give an apparent viscosity relatively quickly and reproducibly, and to provide a comparison between samples.

### 5.2 Rotational viscometer with defined shear rate.

#### 5.2.1 Measuring system

The measuring system shall consist of two rigid, symmetrical, coaxial surfaces between which the fluid whose viscosity is to be measured is placed. One of these surfaces shall rotate while the other remains at rest. The measuring system shall be such that the shear rate can be defined for each measurement. A torque-measuring device shall be connected to one of the surfaces, thus permitting determination of the torque required to overcome the viscous resistance of the fluid. Suitable measuring systems are the coaxial double cylinder system ([Annex A](#)), cone and plate system ([Annex B](#)), and parallel plate system ([Annex C](#)), as prescribed by JIS 8803 and ISO 20507. The dimensions of each measuring system are detailed in [Annexes A, B, and C](#), which are designed to ensure a geometrically similar flow field for all types of measurement and all common types of basic instrument.

#### 5.2.2 Basic capacity of the instrument

The basic instrument shall be designed to permit alternative rotors and stators to be fitted, for the generation of a range of defined rotational frequencies (stepwise or continuously variable), and for measuring the resulting torque, or vice versa (i.e. measurement of the necessary angular velocity to generate a defined torque). The apparatus shall have a torque-measurement accuracy of 2 % of the full-scale reading. Within the regular working range of the instrument, the accuracy of rotational-frequency measurement shall be 2 % of the measured value. The repeatability of viscosity measurement shall be  $\pm 2$  %.

NOTE By using different measuring systems and rotational frequencies, most commercial instruments cover a viscosity range of at least  $10^{-2}$  Pa  $\times$  s to  $10^3$  Pa  $\times$  s.

#### 5.2.3 Installation of rotational viscometer

##### 5.2.3.1 Coaxial double cylinder viscometer

- a) The axis of rotation of the viscometer is installed vertically.
- b) The surface of the sample is maintained at a lower position than the surface of the thermostat or the top of the liquid jacket in the outer cylinder.
- c) The inner cylinder is placed with its axis coinciding with the axis of the outer cylinder; the distance from the bottom of the inner cylinder to the bottom of the outer cylinder is more than 5 mm. The upper surface of the inner cylinder is more than 5 mm below the surface of the sample.



### 5.2.3.2 Cone and plate viscometer

- a) The axis of rotation of the viscometer is installed vertically, and the plate horizontally.
- b) The surface of the sample is maintained at a lower position than the surface of the thermostat or the top of the liquid jacket in cone and plate.
- c) The cone is adjusted so its tip lies on the axis of rotation, and the centre of the parallel plate is also positioned on the axis.

NOTE The tip of the cone may cause friction if it is in contact with the plate. The tip means the imaginary tip of the cone obtained by extrapolation if the end is cut flat.

## 5.3 Single cylinder viscometer.

The single cylinder viscometer has two main components relevant to this standard.

### 5.3.1 Main body of single cylinder viscometer

It is necessary to select the proper model of viscometer for the viscosity to be measured, each model being suited to a specific range of viscosities. Brookfield provides single cylinder viscometers that are classified as LV (low viscosity), RV (medium viscosity) and HA/HB (high viscosity), as prescribed in ISO 2555. Other manufacturers also provide equivalent systems. The suitable uses of cylinder-type viscometers are also determined by special spindles such as Krebs or vane types. Some single cylinder viscometers do not have a demarcation point between the body/shaft and spindle to define the amount of rotating surface submerged, so the shear rate is not fully determined in that case.

### 5.3.2 Spindle

Do not use spindles that are corroded or show eccentric rotation. Choose the spindle taking into account the value of viscosity to be measured, based on the model of viscometer and angular velocity. It is preferable to use the same spindle to compare samples, even when the measured value is outside the spindle's optimum range, rather than change spindles and rely on instrument accuracy. For an unknown sample without any prior information, choose the proper exchangeable spindle that gives a proper magnitude of torque when tested through the full range of angular velocity.

## 5.4 Temperature-control device.

### 5.4.1 Thermostat

The circulation temperature of a constant temperature bath or electric heated wall temperature of a constant temperature bath shall be maintained within  $\pm 0,2$  °C for temperatures between 0 and 50 °C. Wider tolerances (e.g.  $\pm 0,4$  °C) are often sufficient outside this temperature range. Closer tolerances (e.g.  $\pm 0,1$  °C) may be necessary for precise measurements.

### 5.4.2 Thermometer

The accuracy of the thermometer shall be  $\pm 0,2$  °C or the apparatus shall contain a device to measure temperature with equivalent accuracy.

## 6 Calibration of the rotational viscometer

Viscometers shall be calibrated periodically, e.g. by measuring the torque characteristics or by using reference liquids of known viscosity (Newtonian fluids). Standard pre-mixed liquids are preferable to the use of distilled water and diluted standard solutions. If the best-fit straight line drawn through the measured points for the reference liquid does not pass through the origin of the coordinate system, within the limits of the accuracy of the method, the procedure and the apparatus shall be checked more

extensively in accordance with the manufacturer's instructions. The viscosity of a standard liquid used for calibration should be in the same range as the viscosity of samples to be measured.

## 7 Measurement condition

### 7.1 Temperature

Generally, because of the temperature dependence of viscosity, measurements for comparison purposes shall be carried out at the same temperature. If measurements are required to be made at ambient temperature, a measurement temperature of  $23,0\text{ °C} \pm 0,2\text{ °C}$  is preferred.

### 7.2 Selection of geometry and/or spindle (shear rate or angular velocity)

Choose the suitable viscometer, spindle and geometry to measure the viscosity of the sample. Measure the viscosity at defined shear rates (or rotational frequencies) through the full possible measurement range. Viscosity should be measured at more than four defined shear rates to determine a flow curve or viscosity curve, preferably covering a wide range of shear rates (or rotational speeds). In measuring a non-Newtonian sample, it is necessary to consider the possible range of viscosity when choosing shear rate or rotational speed.

NOTE The occurrence of Taylor flow poses a limit to the highest shear rate used for low-viscosity ceramic slurry, if using a rotational viscometer with inner cylinder rotation.

## 8 Pretreatment of the sample

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### 8.1 General

Pretreatment of the sample shall include tempering of the sample to the desired measurement temperature using a constant temperature bath. Any additional pretreatment shall be performed under controlled temperature conditions.

If any settling or sedimentation of solid particles has occurred, it is necessary to disperse the sample constituents by stirring, mixing or shaking. For some samples a simple manual shaking could be sufficient to homogenize the slurry, but this procedure is hardly reproducible. Therefore, it is advisable to perform this step using equipment such as mechanical stirrers or ultrasonic baths. Ceramic slurry may generate bubbles during mixing, changing the viscosity, so care should be taken during this step.

### 8.2 Degassing

Although it is effective to degas by decompression if bubbling has occurred, it is not recommended because this may change the after-preparation properties of the sample. It has to be reported if performed.

### 8.3 Mixing and dispersion by ultrasonication

Dispersion by rapidly stirring the sample for 5 min before measurement or ultrasonication may increase the temperature of ceramic slurry. This is not a recommended treatment since it may change the slurry viscosity, a result of irreversible changes in the polymer dispersant or binder additives. Moreover, it has to be reported if it is unavoidable for dispersion.

## 9 Measurement procedure

### 9.1 Sampling

Take the sample carefully without pretreatment when there is no settling or sedimentation of solid particles is observed. Transfer the sample into the viscosity measurement container, taking care not to create bubbles. The sample in the container of the viscometer needs sufficient time to recover its structure prior to measurements; this varies with the slurry. However, it has to be reported if it is unavoidable for dispersion. Single cylinder viscometers such as the Brookfield generally cannot fully break down the thixotropic structure of many ceramic slips and would give anomalously high and unstable viscosity readings. Slips need to be in a stable agitated condition before testing. A recommended preparation procedure is to mechanically stir slip samples for 5 min before measurement unless the sample has been kept agitated.

### 9.2 Measurement at fixed shear rate or angular velocity

Allow enough time in the viscosity measurement for stabilization with the selected measurement system and the fixed shear rate (or angular velocity) to cover the various samples being compared. In the case of a flow curve, the measurement shall be started with increasing shear rate, followed by decreasing rate, until the maximum and minimum shear rates are reached, respectively. In this case, it should be measured in as short a period as possible.

NOTE If the stabilization time is short at the defined shear rate (or angular velocity), the viscosity is not the value in equilibrium at the shear rate (or angular velocity).

It is desirable to seal the container to prevent drying if there is concern about drying during measurement.

### 9.3 Repeat

Make at least three determinations each with a new portion of the sample.

## 10 Expression of the results

Record the final viscosity point after stabilization, corresponding to the fixed shear rate (or angular velocity) which was selected. In the case of fluctuations of viscosity during measurements, i.e. without stabilization, take several points to make an average. When there is a discrepancy between the viscosity obtained while increasing the shear rate and the viscosity obtained while decreasing it, make an average for that shear rate. In the case of a thixotropic slurry, record two values each of viscosity obtained while increasing and decreasing shear rate. Record two or more determinations of viscosity, and their average, along with measurement temperature and shear rate (or angular velocity if the shear rate is not determined). The results are rounded off with three effective significant figures.

When the shear rate and shear stress are measured, the flow curve is plotted. If the flow curve passes through the origin and is linear, viscosity is given by the slope (Newtonian). If the flow curve is not proportional, viscosity depends on the shear rate or shear stress (non-Newtonian). In this case, a viscosity curve, of viscosity ( $\eta$ ) against shear rate, is plotted.

## 11 Test report

The test report shall be in accordance with the provisions of ISO/IEC 17025 and shall include the following information.

- a) the name of the testing establishment, the date of the test, report identification, number, operator and signatory;
- b) a reference to this document, i.e. ISO 19613;