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

Céramiques techniques (céramiques avancées, céramiques techniques avancées) — Mesurage du comportement thixotropique d'une barbotine de céramique à l'aide d'un viscosimètre rotatif

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Foreword

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This document was prepared by Technical Committee ISO/TC 206, *Fine ceramics*.

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

1 Scope

This document specifies a method for measurement of thixotropic behaviour of ceramic slurry with rotational viscometer. The slurry with high-solid loading, which is used in ceramic manufacturing, has a generally non-Newtonian property. This method is limited to measurement of thixotropic behaviour of high-solid loaded ceramic slurry with "coaxial double cylinder viscometer", "cone and plate viscometer" and "a parallel plate viscometer" as rotational viscometers.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions 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
force developed on the plane which is perpendicular to the shear force when there is shear applied to the fluid

3.2
shear rate
proportion of laminar flow rate change perpendicular to the fluid flow

3.3
Newtonian property
slurry property of which the shear stress is proportional to the shear rate

3.4
non-Newtonian property
slurry property of which the shear stress is not proportional to the shear rate

3.5
viscosity
proportion of shear stress and shear rate

Note 1 to entry: The proportion is a representation of resistance to flow.

3.6
flow curve
curve showing the relationship between shear rate and shear stress

3.7
thixotropic behaviour
behaviour of fluid which has time-dependence of flow behaviour so that the apparent viscosity decreases with time at controlled shear and recovers gradually with removal of the shear force

3.8
ceramic slurry
suspension of grinding ceramic powders mixed or dispersed in water or other liquid

Note 1 to entry: The suspension is used in the ceramic process, for example screen printing, slip casting, tape casting, granule production.

4 Principle

4.1 Evaluation of thixotropic behaviour by hysteresis measurement of flow curve

Measurement of flow curve of ceramic slurry is performed using a rotational viscometer, which can monitor shear rate and shear stress at the same time. The thixotropic behaviour is evaluated from the magnitude of hysteresis that is indicated by the process of the flow curve.

4.2 Evaluation of thixotropic behaviour by time-dependence of shear stress at constant low shear rate

Measurement of the flow curve of ceramic slurry is performed using a rotational viscometer, which can monitor shear rate and shear stress at the same time. After the viscosity of ceramic slurry is decreased by destruction of aggregated or flocculated structure in ceramic slurry at high shear rate, the thixotropic behaviour is evaluated by measuring the recovery process of viscosity at low shear rate. The relationship between fabrication of re-flocculated structure and time can be detected at low shear rate. Therefore, the thixotropic behaviour is evaluated by using data from low shear rate.

5 Apparatus

A rotational viscometer has various measuring geometrics. In this document, the rotational viscometers with defined shear rate are a coaxial double cylinder system, a cone and plate system and a parallel plate system.

5.1 Measuring system

The measuring system consists of two surfaces with symmetrical and coaxial geometrics, between which is the slurry whose viscosity is measured. One of these surfaces rotates at a constant angular velocity while the other remains at rest. A device for torque measurement is connected to one of the surfaces, thus permitting determination of the torque required to overcome the viscous resistance of the slurry.

A measuring system of a coaxial double cylinder system and a cone and plate system, which can decide shear rate at every measurement, is desirable. The measurement is possible by a parallel plate system, which is suitable for ceramic slurry with high viscosity.

5.2 Basic capacity of the instrument

The instrument for measurement shall be designed to permit alternative rotors and stators to be fitted for generation of a range of defined rotation frequency. In addition, it is necessary to use apparatus which can measure the torque. The apparatus has torque-measurement accuracy within 2 % of the full-scale reading. At the working range of measurement, the accuracy of rotational frequency measurement is within 2 % of the measured value. Furthermore, the repeatability of viscosity measurement has an accuracy of ±2 %.

5.3 Calibration of rotational viscometer

The viscometer shall be calibrated periodically by using the standard solution for which the viscosity is defined. If the best-fit straight line drawn through the measured points for the standard solution does not pass through the origin of coordinate system within the accuracy limit of the measuring method, the procedure and the apparatus should be checked more extensively. The viscosity of the standard solution shall have the same range as that of the measurement slurry sample. The calibration temperature shall be identical to the measurement temperature of the sample.

5.4 Temperature control device

The circulation temperature of the thermostat, which is between 0 °C and 50 °C, shall be maintained within ±0,2 °C. Outside of this temperature, the maintained temperatures are permitted within ±0,5 °C. The accuracy of the thermometer shall be ±0,2 °C.

NOTE In cases of precise measurement, closer tolerances may be necessary up to ±0,1 °C.

6 Pretreatment of the slurry samples

6.1 General

In the measurement of ceramic slurry there are many points that require attention. It is necessary to disperse the sample by stirring, mixing and shaking if the ceramic slurry has sedimentation property. Care should be taken not to generate bubbles during the process of stirring, mixing and shaking the ceramic slurry, as this may change the thixotropic behaviour.

6.2 Degassing

If bubbles have formed in the slurry after stirring, mixing and shaking, degassing by decompression can be effective. However, the properties of the slurry after decompression may differ from those of the slurry for ceramic manufacturing. This degassing process is not recommended unless the degassing process is performed for the ceramic forming after slurry preparation, because the influence of slurry viscosity may be large. If decompression is performed to degas a slurry, it shall be reported.

6.3 Dispersion by stirring, mixing and ultrasonication

The temperature of ceramic slurry is increased when using high-speed stirring, mixing or ultrasonication as a dispersion process. This is not recommended, because it may change the thixotropic behaviour of the slurry due to the polymer dispersant or binder in the slurry being changed irreversibly by the increasing temperature.

7 Measurement condition

7.1 Setting of ceramic slurry to rotational viscometer

Put the ceramic slurry carefully into the measuring system of the viscometer so that bubbles are not generated.

7.2 Temperature

Measurement for comparison purposes should be performed at the same temperature, because the viscosity of slurry depends on temperature. The measurement temperature of 23 ± 0,2 °C is preferred if it is not necessary to prescribe a process temperature.

8 Measurement procedure

8.1 Measurement of thixotropic behaviour by hysteresis measurement of flow curve

8.1.1 Condition of shear rate and setting of measurement range

The setting of the shear rate shall be performed under the permitted conditions of the instrument. Based on the measurement programme, the shear rate is increased continuously or stepwise. In order to allow sufficient destruction of the aggregated or flocculated structure in the slurry during the increase in the shear rate, a maximum shear rate of 300 s^{-1} or more is recommended. However, this does not apply if it reaches the measurement limit in the case of high viscosity. When the measurement time of the increase and decrease of the shear rate is short, it is not possible to observe a sufficient hysteresis. In addition, if the measurement time is long, sedimentation may occur. Therefore, a measurement time of 90 s each is recommended for the increase and decrease operation of the shear rate.

For the stepwise method, it is recommended that measurements of the increase or the decrease of the shear rate are performed at the shear rate as much as possible (10 stepwise).

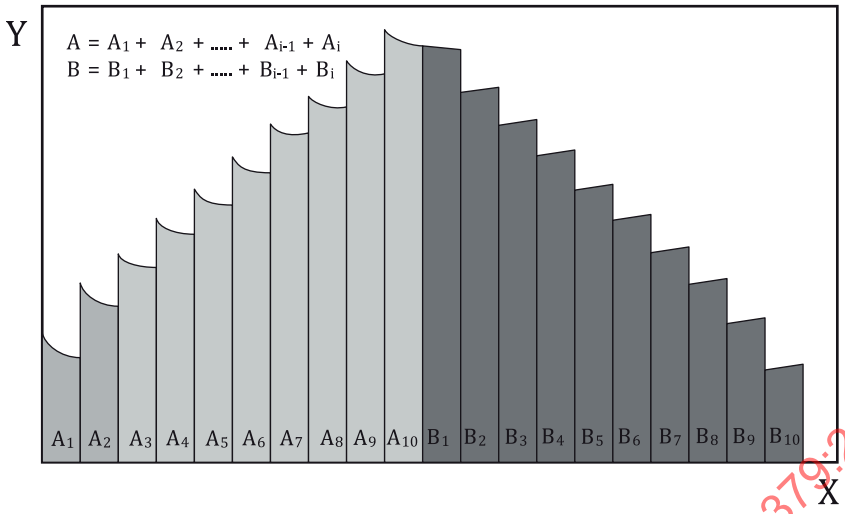
8.1.2 Measurement procedure

After putting the slurry into the viscometer, enough time shall be allowed to reach the setting temperature for measurement. If the slurry has sedimentation property, it shall be rotated at a constant shear rate until it reaches a constant temperature in order to prevent sedimentation. Under the conditions described in 8.1.1, the measurement of the flow curve shall be performed as a function of the increase of the shear rate. After reaching the maximum shear rate, the shear rate is stopped. After resting the measured slurry for 3 min to 5 min in order to recover the thixotropic properties of the slurry, the flow curve is measured again using the same procedure. The operations, i.e. the increase/decrease of the shear rate and the static state of the slurry, shall be carried out at least three times.

In order to minimize the influence of the flow curve due to particle sedimentation or drying out of the slurry, prompt measurement and sealing of the sample container are recommended.

8.1.3 Analysis method of measurement results

The hysteresis area of the flow curve tends to become larger as the slurry viscosity increases. Therefore, the evaluation of hysteresis is dimensionless, as follows. The relationship between the measurement time and the measured shear stress is shown in Figure 1. The area of the increase and decrease processes of shear rate are expressed as A and B, respectively. Owing to the non-dimension of hysteresis, the area is calculated as (A − B)/(A + B). This analysis is performed for each measurement, and the calculated value of (A − B)/(A + B) is plotted as a function of the number of measurements. Moreover, in the case of the stepwise increase and decrease of the shear rate, the evaluation area is calculated by the area integration of the trapezoidal approximation between the measured data curve and a horizontal axis, as shown in Figure 1. Since the first and second measurement data may vary, the evaluation data are used to analyse the data of the third, or subsequent, measurements. Because the measurement points just after changing the shear rate may vary widely, the removal of these points from the calculation is desirable.



Key

X measurement time

Y shear stress

NOTE The area of the increase and decrease processes of shear rate are expressed as A and B, respectively.

Figure 1 — Relationship between measurement time and shear stress

8.2 Measurement of thixotropic behaviour by time-dependence of shear stress at constant low shear rate

8.2.1 Setting of measurement programme

It is necessary for the selection of shear rate conditions to destroy the sufficiently aggregated or flocculated structure of the slurry at a high shear rate, and to recover the destroyed structure of the slurry at a low shear rate. In this measurement, it is recommended that the high shear rate and the low shear rate are 300 s^{-1} and 30 s^{-1}, respectively. The measurement time for both high and low shear rates is recommended as 10 min. The time interval shall be as short as possible when performing repeated measurements.

8.2.2 Measurement procedure

After putting the slurry into the viscometer, enough time shall be allowed to reach the set temperature for measurement. If the slurry has sedimentation property, it shall be rotated at a constant shear rate until it reaches a constant temperature in order to prevent sedimentation. The measurement programme described in 8.2.1 shall be performed at least twice to confirm the reproducibility of the measured shear stress. If the reproducibility is poor, additional measurements shall be carried out.

Because a slurry sample with a strong sedimentation property cannot be measured for a long time, the first measurement shall be performed again by introducing new slurry into the viscometer.

8.2.3 Analysis method of measurement results

After sufficient destruction of the aggregated or flocculated structure of the ceramic slurry at a high shear rate, the thixotropic property can be evaluated by the time-dependence measurement of the viscosity recovery process at a low shear rate. The construction of the aggregated or flocculated structure is relatively slow. In the case of measurement at a high shear rate, it is difficult to evaluate the time-dependence because the aggregated or flocculated structure is destroyed quickly. Therefore, the measurement data of the low shear rate is mainly used in the analysis. The time-dependence of shear stress or viscosity is estimated as zero time point when switching to a low shear rate from a

high shear rate. Thixotropic behaviour is evaluated by the estimation of this time-dependence. The data obtained by this method is consistent with Formula (1). The measurement results are evaluated using Formula (1).

$$\log(\eta/\eta^*) = (t/t_0)^n \quad (1)$$

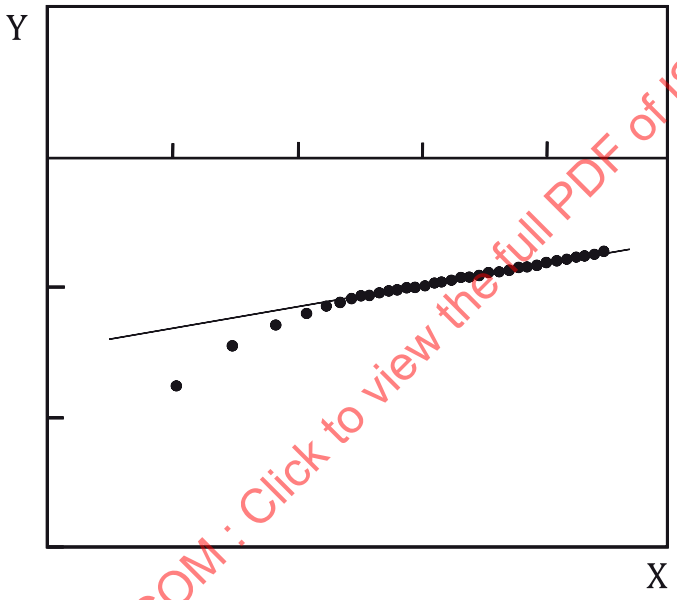
where

η^*, t_0 and n are parameters;

t is time;

η is viscosity.

If $n < 0$ and $t \to \infty$, η converges with η^*. If $n > 0$ and $t \to \infty$, η diverges from ∞. Figure 2 gives an example of an approximation of the time-dependence of viscosity at a steady shear rate.



Key

X $\log(t/t_0)$

Y $\log[\log(\eta/\eta^*)]$

● Measurement values when changing the steady shear rate from high shear rate to low shear rate.

Figure 2 — Example of an approximation of the time-dependence of viscosity at a steady shear rate

9 Test report

The test report shall be in accordance with the reporting provisions of ISO/IEC 17025 and shall contain the following:

a) the number and year of publication of this document;

b) all details necessary for identification of the measuring slurry;

c) the data of sampling;

d) the temperature in degrees Celsius;

e) the preparation method of the slurry;

f) the system for measurement of thixotropic behaviour;

g) the measurement conditions (hysteresis measurement: for example the maximum shear rate, stationary time, time interval between the increase and the decrease of the shear rate, number of steps and each shear rate; time-dependence of shear stress: high and low shear rate values);

h) the plot of the relationship between shear stress and measuring time (for hysteresis measurement) or measuring time and viscosity (or shear stress) (for the time-dependence of shear stress);

i) the value of (A − B)/(A + B) (for hysteresis measurement) or the function for data fitting, the figure of the plot results and the parameter values for calculation (for time-dependence of shear stress);

j) the date of measurement (YYYY-MM-DD).