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Fine ceramics (advanced ceramics, advanced technical ceramics) — Determination of content of coarse particles in ceramic powders by wet sieving method

Céramiques techniques — Détermination du contenu en particules grossières des poudres de céramique par la méthode de tamisage humide

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Foreword

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ISO 24369 was prepared by Technical Committee ISO/TC 206, *Fine ceramics*.

Fine ceramics (advanced ceramics, advanced technical ceramics) — Determination of content of coarse particles in ceramic powders by wet sieving method

1 Scope

This International Standard specifies the procedure to determine the content of coarse particles in a fine ceramic powder and/or in a fine ceramic suspension using an aqueous-based wet sieving method. The procedure is applicable to fine ceramic powders of both micrometre and submicrometre size ranges. It is valid when there are greater than 10 mg/kg coarse particles in the powders.

NOTE It is recommended that new operators of this test method become familiar with the procedure, using a reference powder on slurry with a known quantity of coarse particles present.

2 Terms and definitions

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

2.1
coarse particles
particles and/or aggregates that cannot pass a 500 mesh sieve with 25 µm mesh size

2.2
percentage of coarse particles
ratio of the mass of the coarse particles (remaining on the sieve) to the total mass of the powder sample analysed

2.3
suspension
ceramic powder suspended in an aqueous medium

2.4
solid content
amount of powder in a suspension, ratio of the mass of powder to the total mass of the suspension (powder + medium)

3 Apparatus

3.1 **Sieve**: 500 mesh (the aperture size is 25 µm).

A stainless-steel rimmed sieve is recommended.

3.2 **Analytical balance**, having a readability of at least 0,1 mg.

3.3 **Bottle**: a glass weighing bottle with known mass m_a.

3.4 **Glass beaker**, with a known mass m_e and a volume of at least 250 cm^3.

3.5 **Pipette**, 10 ml.

3.6 **Oven**: capable of controlling a temperature of 105 °C ± 5 °C.

3.7 **Stirring apparatus**: magnetic stirrer and polytetrafluoroethylene (PTFE)-coated stir bar.

3.8 **Ultrasonic bath**.

3.9 **Desiccator** or **vacuum chamber**.

4 Sampling

A sample of suspension is required. The amount of powder needed for the measurement is about 30 g ± 2 g. The solid content of the suspension may vary but must be known (in weight percent).

5 Measuring procedures

In the following measuring procedures, humidity is easily adsorbed either from hands or from the air. Therefore, be careful not to touch the sieve and glass weighing bottle with bare hands; use clean tongs or powder-free latex gloves.

5.1 Washing and drying of sieve

Submerse the entire sieve in a clean ultrasonic bath filled with distilled water, and ultrasonicate for 10 min. It is recommended that the sieve is upright in the ultrasonic bath to avoid the re-adhesion of undesirable particles. Remove the sieve and drain bath, refill with fresh distilled water, and repeat the treatment.

After washing, place the sieve in an oven, and dry it at 105 °C for 2 h. After drying, cool the sieve down to room temperature in either a desiccator or a vacuum chamber and let it equilibrate there at room temperature for at least 10 min.

5.2 Weighing of sieve

After equilibration, determine the mass m_s of the sieve, to within 0,1 mg, with a balance directly from the desiccator or vacuum chamber. Calculate the sieve mass m_{s*} by averaging the values of three measurements, each obtained after equilibration of the balance.

5.3 Determination of solid content

The following is the procedure for the determination of solid content.

a) Preliminary treatment of the suspension for sampling: mildly agitate the suspension by shaking or magnetic stirring long enough, so that any sedimentation is compensated for and homogeneity is attained. For a well-dispersed suspension, this might be in the range of 2 to 3 min; for a suspension that is partly deposited, the required time may be up to several hours. Do not use ultrasonic treatment, as this could influence the coarse particle fraction by rupturing existing agglomerates.

b) Take an amount of about 1 to 2 ml of suspension, by means of a pipette, and place it in a glass weighing bottle of known mass m_a (to within 0,1 mg). Determine the mass of suspension with a glass weighing bottle m_b to within 0,1 mg. Calculate the suspension plus bottle mass m_{b*} by averaging the values of three measurements, each obtained after equilibration of the balance.

c) Place the weighing bottle with the suspension sample in an oven to dry at 105 °C for at least 2 h. After drying, transfer the weighing bottle with the dried suspension to either a desiccator or a vacuum chamber for cooling to room temperature, and let it equilibrate there at room temperature for at least 10 min.

d) After equilibrating, determine the mass of the glass weighing bottle with the dried suspension m_c, to within 0,1 mg, directly from the desiccator or vacuum chamber. Calculate the sample plus bottle mass m_{c*} by averaging the values of three measurements, each obtained after equilibration of the balance.

e) Calculation of the solid content SC as a ratio of masses of powder and suspension:

$$m_{\text{suspension}} = m_{b*} - m_a, \text{ in grams} \quad (1)$$

$$m_{\text{powder}} = m_{c*} - m_a, \text{ in grams} \quad (2)$$

$$\text{SC} = \frac{m_{\text{powder}}}{m_{\text{suspension}}} \quad (3)$$

5.4 Sampling and determination of mass of suspended powder

The following is the procedure for sampling and determination of mass of suspended powder.

a) Preliminary treatment of the suspension for sampling: mildly agitate the suspension by shaking or magnetic stirring, for long enough so that any sedimentation is compensated for and homogeneity is attained. For a well-dispersed suspension, this might be in the range of 2 – 3 min; for a suspension that is partly deposited, the required time may be up to several hours. Do not use ultrasonic treatment, as this could influence the coarse particle fraction by rupturing existing agglomerates.

b) The absolute amount of powder needed is about 30 g. Therefore, the sample for measurement is obtained from the suspension with a now known SC by weighing. The amount used depends on the solid content, so that the amount of powder is about 30 g ± 2 g. The required mass of suspension can be calculated by

$$m_{\text{suspension}} = \frac{m_{\text{powder}}}{\text{SC}} \quad (4)$$

EXAMPLE When SC in a suspension is 0.3, 100 g of a suspension is required. Also, 37,5 g of a suspension is required when SC is 0,8.

c) Place the required amount of suspension into a beaker of known mass m_e (to within 0,1 mg). Determine the mass m_f of beaker with suspension to within 0,1 mg. Determine the suspension plus beaker mass m_{f*} by averaging the values of three measurements, each obtained after equilibration of the balance. The mass m_g of the suspension is given by Equation (5)

$$m_g = m_{f*} - m_e \quad (5)$$

According to Equation (4), the mass m_p of the powder in the suspension is given by

$$m_p = m_g \times \text{SC} \quad (6)$$

5.5 Separation of coarse particles

The following is the procedure for separation of coarse particles.

a) Wet the sieve and place it in the shallow washing bath. Adjust the level of the mesh so that it is just below the water surface of the bath. In this way, the mesh of the sieve will be covered with water.

b) Pour the sample suspension slowly, and little by little, into the sieve so that nothing of the suspension is lost. In case the sieve becomes clogged with material, dilute and disperse the clogged material by pouring distilled water into the sieve. Take care that the suspension is quantitatively transferred to the sieve by rinsing the beaker with distilled water at the end.

NOTE 1 Coarse particles tend to sediment during slow pouring, and therefore concentrate in the last 5 – 10 % of the slurry.

Coarse particles may adhere to a glass beaker. It is recommended to dry the beaker and inspect retained coarse particles. If these are found, they should be removed with water and the resultant suspension should be wet-sieved.

c) Rinse away any suspension that is attached to the sieve frame with distilled water, then pour a sufficient amount or volume of distilled water into the sieve.

NOTE 2 About 10 L might be a sufficient volume.

d) Take care that all particles are on the mesh of the sieve and nothing is left on the sieve frame.

e) If the slurry viscosity is excessively high, dilute the slurry with a proper liquid, for which the chemistry is the same as that of the supernatant of the slurry. Alternatively, take care that the mesh of the sieve be covered with water, with no air bubbles between the mesh and water surface, during sieving.

5.6 Drying and weighing of sieve with coarse particles

Follow the same procedures as detailed in 5.1 and 5.2 to measure the mass m_{scp*} of the sieve with coarse particles.

5.7 Washing the sieve

Wash the sieve upside down for 10 min in clean water in an ultrasonic bath. Remove the sieve and turn it over, so that the mesh is upright, and wash for an additional 10 min. Drain the bath and refill with fresh water. Repeat the washing treatment. Dry the sieve as in 5.1.

6 Calculation

The percentage X of coarse particles in the powder, in milligrams per kilogram, can now be determined according to Equation (7).

$$X = \frac{m_{scp*} - m_{s*}}{m_p} \times 10^6 \qquad (7)$$

where

m_{scp*} is the mass, in grams, of sieve plus coarse particle (5.5);

m_{s*} is the sieve mass, in grams, before the experiments (5.1);

m_p is the determined mass, in grams, of powder.

The values of m_{scp*} and m_{s*} are the average ones.

7 Report

For the report, fill in the attached data sheet.

Data Sheet

Table 1 — Experimental parameters

Laboratory or organization	
Analyst	
Date of analysis	
Powder	
Type	
Comments	
Dispersion	
Type	
Comments (pretreatment, pH, etc.)	
Sieve	
Inside diameter (mm)	

Table 2 — Determination of solid content, weighing results

m_a g	m_b g	m_c g	$m_{suspension}$ g	m_{powder} g	Solid content
	1	1	$m_{b*} - m_a$	$m_{c*} - m_a$	SC
	2	2			
	3	3			
	m_{b*}	m_{c*}			

Table 3 — Weighing-test results

m_e g	**Sieve mass** g		**Mass of beaker with suspension** m_f g	**Suspension mass** $m_{f*} - m_e$ g	**Powder mass** $m_g \cdot SC$ g	**Mass of coarse particles + sieve** g	**Content of coarse particles** mg/kg
	Before experiments m_{sb}	After experiments m_{sa}					
	1	1	1	m_g	m_p	1	X
	2	2	2			2	
	3	3	3			3	
	m_{sb*}	m_{sa*}	m_{f*}			m_{scp*}	