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Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for surface roughness of fine ceramic films by atomic force microscopy

Céramiques techniques — Méthode d'essai pour la rugosité de surface des films céramique fins par microscopie à force atomique

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Foreword

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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).

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

Introduction

Surface roughness measurements of fine ceramic thin films in nanometer scale by atomic force microscopy have become one of the techniques widely applied to quality control and assurance in industries.

One of the problems most frequently occurring in roughness measurements by atomic force microscopy resulting from its scale dependency is the deviation of roughness due to the wear of the probe tip or the deviation in the curvature of commercially available probe tips. This problem makes it difficult to obtain a reliable and reproducible result of the roughness measurement. Therefore, it is highly desirable to standardize a method to evaluate probe tip diameter or curvature radius.

This document covers the evaluation of probe-tip diameter and provides a method to judge the adequateness of a probe tip for use in day-to-day roughness measurements of fine ceramic thin films with a certain arithmetical mean roughness in the range needing the use of atomic force microscopy in production lines or quality assurance processes.

It should be noted that because surface roughness is a scale-dependent metrology parameter, it is unavoidable that the probe-tip evaluation process contains some contradictory procedures, namely the adequateness of the probe tip for a roughness measurement depends on unmeasurable true roughness in a scale of interest.

In this document, the parameters based on roughness profiles are used. The roughness profile is obtained by using a low-pass filter according to ISO 16610-21. The process to obtain the sampling length, which is identical to cut-off wavelength, is given in ISO 4288. Some different sampling lengths to process a primary profile can be applied to obtain appropriate values of arithmetic mean deviation of a roughness profile, if necessary.

Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for surface roughness of fine ceramic films by atomic force microscopy

1 Scope

This document describes a method to evaluate the adequateness of a probe tip for fine-ceramic thin-film surface roughness measurements by atomic force microscopy, of surfaces with an arithmetical mean roughness, *Ra*, in the range of about 1 nm to 30 nm and a mean width of roughness profile elements, *RSm*, in the range of about 0,04 µm to 2,5 µm.

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 4287, *Geometrical Product Specifications (GPS) — Surface texture: Profile method — Terms, definitions and surface texture parameters*

ISO 4288, *Geometrical Product Specifications (GPS) — Surface texture: Profile method — Rules and procedures for the assessment of surface texture*

ISO 11039, *Surface chemical analysis — Scanning-probe microscopy — Measurement of drift rate*

ISO 11952, *Surface chemical analysis — Scanning-probe microscopy — Determination of geometric quantities using SPM: Calibration of measuring systems*

ISO 18115-2, *Surface chemical analysis — Vocabulary — Part 2: Terms used in scanning-probe microscopy*

ISO 25178-2, *Geometrical product specifications (GPS) — Surface texture: Areal — Part 2: Terms, definitions and surface texture parameters*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 4287, ISO 4288, ISO 18115-2, ISO 11039, ISO 11952 and ISO 25178-2 and the following apply.

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

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at http://www.iso.org/obp

3.1
evaluation length
ln(X), *ln(Y)*
length of surface profile in the *X* or *Y* direction

3.2
probe-tip diameter evaluation standard plate
plate on which needle-shaped spikes are formed

Note 1 to entry: The plate is used to evaluate the *probe-tip diameter* (3.3).

3.3
probe-tip diameter
D
diameter of a probe tip at a distance of 10 nm from the tip end

4 Test environment

Testing shall be carried out only where temperature change, sound noise and mechanical vibration of the floor or walls are small enough to perform the measurements. The following installation environment is recommended:

a) temperature: 18 °C to 25 °C;

b) humidity: 70 % or less;

c) noise level: 60 dB or less;

d) mechanical vibration of the floor or the wall: 1×10^{-3} m/s^2 (<100 Hz) or less.

5 Roughness measurement specimens

Specimens for roughness measurements are ceramic thin films on a substrate. Any kinds of substrate material can be used, such as metal, glass, polymer, etc. The specimen shall be no larger than the specimen stage of the instrument being used.

6 Test apparatus

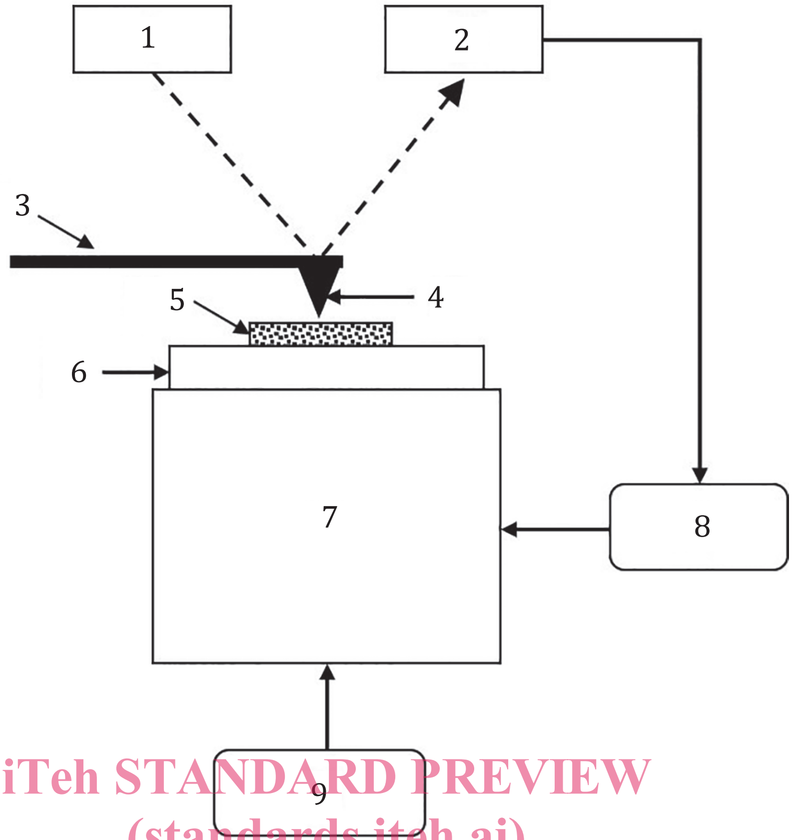
6.1 Cantilever

The cantilever shall be exclusively dedicated for a dynamic mode and commercially available. The resonant frequency should be higher than 100 kHz.

6.2 Scanner

The scanner shall be capable of scanning cantilever or specimen stage by shifting the *XYZ* position. The scanning area should be larger than 10 µm × 10 µm in the *XY* plane.

Figure 1 shows an example of a measurement system having a specimen stage scan mechanism. Position in the *Z* direction is controlled using a *Z*-position control circuit that keeps a constant separation between the probe and the specimen surface. For this purpose, a light beam from a laser diode illuminates the cantilever and the reflected beam position is monitored by a light detector. Surface profile is measured by scanning the specimen stage in the *XY* plane.



Key

1 laser diode
2 light detector
3 cantilever
4 probe tip
5 specimen
6 specimen stage
7 *X*, *Y* and *Z* scanner
8 *Z*-position control circuit
9 *X*-*Y* scan circuit

Figure 1 — Schematic of AFM system

6.3 Specimen stage

The specimen stage shall be capable of supporting a specimen horizontally. The test area of the specimen should be in the centre of the specimen stage. Scanning should be performed near the centre of the *X*, *Y* and *Z* axes of the scanner used.

7 Test apparatus calibration

This document only describes a method to evaluate the adequateness of a probe tip for fine-ceramic thin-film surface roughness measurements by atomic force microscopy. If the apparatus needs to be calibrated, refer to standards describing calibration criteria and methods for a scanning probe microscope; see ISO 4288, ISO 11039 and ISO 11775.

8 Probe-tip diameter evaluation standard plate

A standard for probe-tip diameter evaluation is a plate on which a number of needle-shaped spikes, arranged in a square matrix, are formed. The needle-shaped spikes are typically as follows:

— tip curvature radius: 10 nm;

— tip aperture angle: 20° or 50°;

— tip height: 300 nm to 600 nm;

— distance between any two nearest neighbour tips: 2,12 µm;

— plate size: 5 mm × 5 mm.

9 Calibration of *X*-*Y* and *Z* scan axes

The calibration of the *X*-*Y* scanner and *Z* scanner should be carried out by measuring the *X*, *Y* and *Z* profile of a certified calibration standard. The standard should have a grating with a certain pitch and a step with a certain height.

The standard sample is a specimen with calibrated height and pitch, which are certified and traceable with uncertainty data attached. It is recommended that the grating pitch is less than 2 µm and that the step height is less than 20 nm.

The calibration standard should be stored in a clean and dry box and be handled with care.

The calibration shall be carried out in the following sequence using the dynamic mode.

a) Mount the calibration standard in such a way that the grating is oriented to the *X*-*Y* axes of the scanner and that its plane lies nearly parallel to the *X*-*Y* plane of the scanner.

b) Set the number of picture elements at 512 × 512 or 256 × 256.

c) Scan an area of about 10 µm square of the calibration standard and store the surface profile data. An example is shown in Figure 2 a).

d) From the surface profile data, draw a surface profile along the *X* direction at a selected *Y* position where the surface profile contains several steps on the calibration standard and level off the one-dimensional profile along the *X* direction. An example is shown in Figure 2 b).

e) Measure profile peak height at the centre of top and bottom sections of the profile. Calculate mean height for at least five successive profile elements in the profile along the *X* direction.

f) Measure a pitch along the *X* direction by measuring the distance between the mid-points of two successive rising or falling parts of the profile.

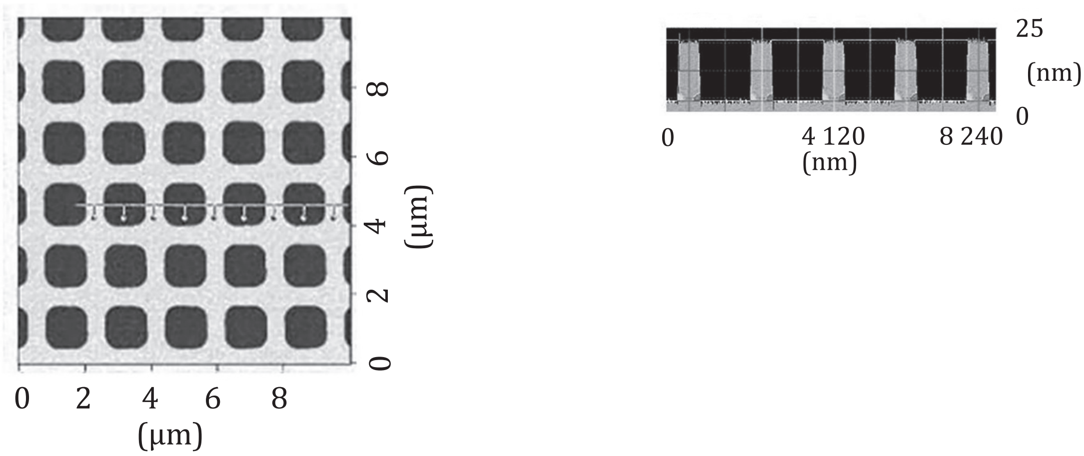
g) From the surface profile data, draw a surface profile along the *Y* direction at a selected *X* position where the surface profile contains several steps and level off the one-dimensional profile along the *Y* direction.

h) Measure profile peak heights at the centre of top and bottom sections of the profile. Calculate mean height for at least five successive profile elements in the profile along the *Y* direction.

i) Measure a pitch along the *Y* direction by measuring the distance between the mid-points of two successive rising or falling parts of the profile.

j) If an *X*- or *Y*-pitch measured is out of the range of uncertainty needed for roughness measurements, correct *X* or *Y* values by obtaining an *X*- or *Y*-axis calibration factor.

k) If the mean height obtained is out of the range of uncertainty needed for roughness measurements, correct *Z* values by obtaining a *Z*-axis calibration factor.



a) Surface profile

b) Surface profile along the *X* direction

Figure 2 — Examples of surface profile data for the calibration of the *X*-*Y* scanner and *Z* scanner

10 Probe-tip error evaluation

10.1 Outline of probe-tip error evaluation

The error for roughness measurements using a probe tip with a certain probe-tip diameter shall be evaluated by using a probe-tip error evaluation template, providing the error from the relationship between the probe-tip diameter, preliminary *Ra* and preliminary *RSm*. If a probe tip is judged to be unsuitable for a roughness measurement with a certain error for a specimen to be measured, it should be rejected and replaced with a new one.

Probe-tip evaluation shall be carried out using the probe-tip evaluation sequence shown in Figure 3.

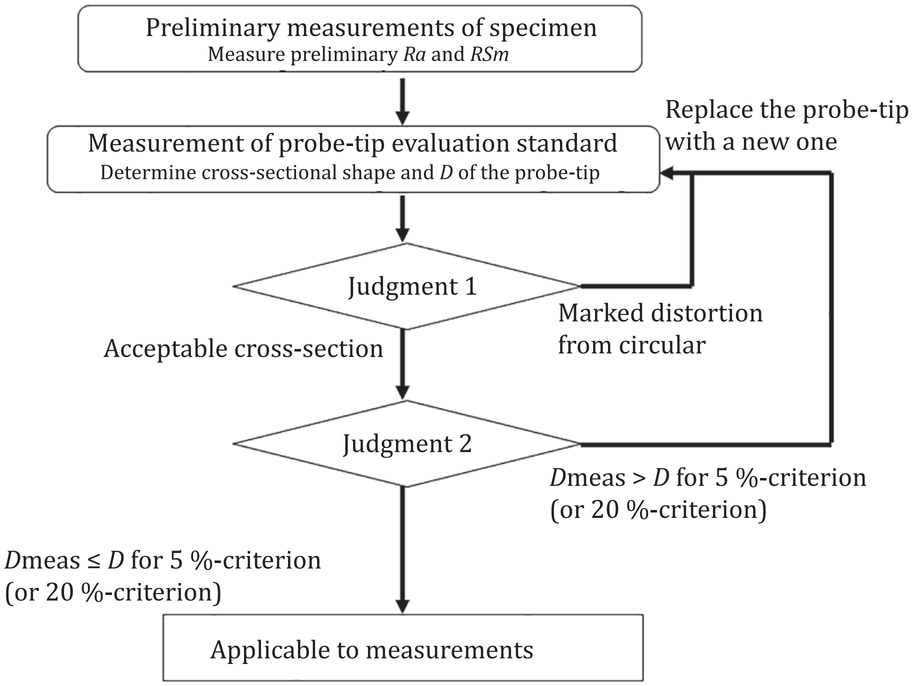


Figure 3 — Probe-tip evaluation sequence

10.2 Measurements of preliminary *Ra* and *RSm*

Preliminary roughness measurements shall be carried out to obtain preliminary *Ra* and *RSm*, the values to be used in the probe-tip judgement procedure.

a) Place the specimen to be measured on the specimen stage without any surface treatment or without using any adhesive.

b) Choose arbitrarily a measuring position on the specimen.

c) Set the number of picture elements at 256 pixels or 512 pixels in the *X* direction and the scanning speed within 0,5 Hz to 1 Hz.

d) Scan a 2 µm line in the *X* direction at a fixed *Y* position using the dynamic mode. Again scan a 10 µm line in the *X* direction at the same *Y* position. Apply an appropriate cut-off length.

e) Select one of the two surface profiles along the *X* direction, such that the number of profile elements in the evaluation length *ln*(*X*) is between 20 and 50.

f) Calculate preliminary *Ra* and *RSm* values from the selected surface profile.

10.3 Evaluation of probe-tip diameter

The evaluation of probe-tip diameter should be carried out using the following sequence. A schematic drawing of probe-tip diameter determination is shown in Figure 4.

a) Measure a part of the probe-tip diameter evaluation standard plate by scanning a 2 µm × 2 µm to 3 µm × 3 µm squared area with the number of picture elements ranging from 128 pixels × 128 pixels

