
**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

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).

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For an explanation on 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.

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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.

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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, R_a , in the range of about 1 nm to 30 nm and a mean width of roughness profile elements, RS_m , 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: $1 \times 10^{-3} \text{ m/s}^2$ (<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

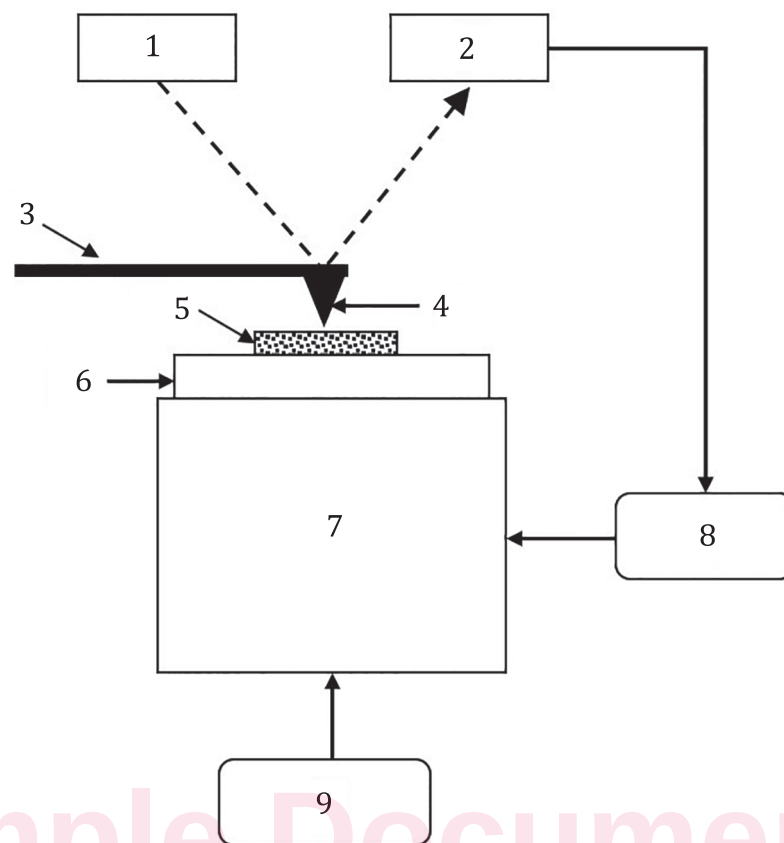
6.1 Cantilever

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 $10 \mu\text{m} \times 10 \mu\text{m}$ 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 | 6 | specimen stage |
| 2 | light detector | 7 | X, Y and Z scanner |
| 3 | cantilever | 8 | Z-position control circuit |
| 4 | probe tip | 9 | X-Y scan circuit |
| 5 | specimen | | |

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.