BS ISO 19606:2024

BSI Standards Publication

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

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# National foreword

This British Standard is the UK implementation of ISO 19606:2024. It supersedes BS ISO 19606:2017, which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee RPI/13, Advanced technical ceramics.

A list of organizations represented on this committee can be obtained on request to its committee manager.

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International Standard

# ISO 19606

Second edition
2024-11

# 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 document 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 of 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 www.iso.org/iso/foreword.html.

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

This second edition cancels and replaces the first edition (ISO 19606:2017), which has been technically revised.

The main changes are as follows:

— in Clause 3, ISO 4287 and ISO 4288 have been replaced with ISO 21920-1–3;

— revision of 6.2 and Clause 3;

— symbols have been revised throughout by moving text to subscript;

— Annex A changed from normative to informative.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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# Introduction

Surface roughness measurements of fine ceramic thin films at the nanometre scale by atomic force microscopy is widely applied to quality control and assurance in industries.

One frequently occurring issue in roughness measurements by atomic force microscopy resulting from its scale dependency is the deviation of roughness due to wear of the probe tip or the deviation in the curvature of commercially available probe tips. This 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 21920-2. 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 specifies a method to evaluate the adequateness of a probe tip for fine-ceramic thin-film surface roughness measurements by atomic force microscopy. This method applies to 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, $R_{sm}$, 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 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 21920-1, *Geometrical product specifications (GPS) — Surface texture: Profile — Part 1: Indication of surface texture*

ISO 21920-2, *Geometrical product specifications (GPS) — Surface texture: Profile — Part 2: Terms, definitions and surface texture parameters*

ISO 21920-3, *Geometrical product specifications (GPS) — Surface texture: Profile — Part 3: Specification operators*

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 21920-1, ISO 21920-2, ISO 21920-3, ISO 18115-2, ISO 11039, ISO 11952 and ISO 25178-2 and the following apply.

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

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

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

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

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**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 sufficiently low 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: $1 \times 10^{-3}$ 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 Atomic force microscopy system

The atomic force microscopy (AFM) system consists of a cantilever, the detecting system for the cantilever deflection, and the sample stage with *XYZ* motion to displace the sample, including a piezoelectric positioning system. The AFM system should be put on an anti-vibration table inside an acoustic enclosure in order to fulfil the requirements on noise level and mechanical vibration given in Clause 4.

### 6.2 Cantilever

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

### 6.3 Scanner

The scanner shall be capable of scanning the 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. The surface profile is measured by scanning the specimen stage in the *XY* plane.

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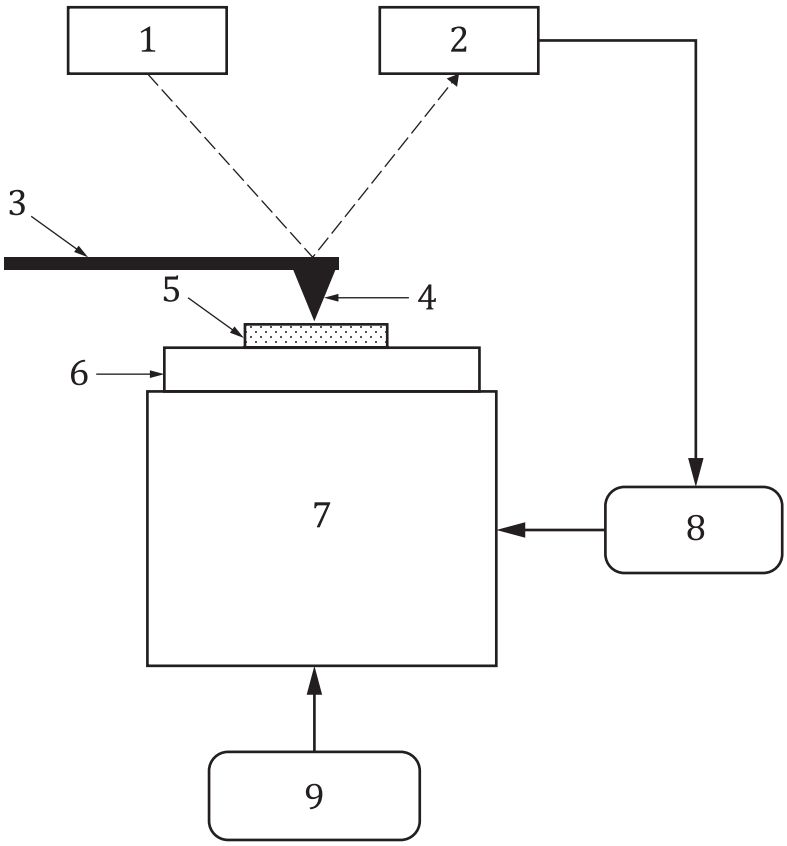


**Key**

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 an AFM system**

### 6.4 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 21920-1, ISO 21920-2, ISO 11039, ISO 11775, and ISO 11952.

## 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;

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