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# Profile surface roughness measurement using metrological atomic force microscope and uncertainty evaluation

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ABSTRACT

Surface roughness measurements are sometimes performed using an atomic force microscope (AFM) in order to evaluate conditions of thin film fabrication and material surface treatment. Recently precise and reliable surface roughness measurement has been required in order to further improve quality of both thin films and material surfaces. Evaluation method of AFM tip shape is a key technology in the surface roughness measurement using an AFM. An evaluation method of AFM tip shape using a probe examination sample and its evaluation criteria are stipulated in the Japanese Industrial Standard (JIS) R 1683: 2007 "Test method for surface roughness of ceramic thin films by atomic force microscopy". In this study, profile surface roughness measurements were performed based on JIS R 1683: 2007 and the measurement results are reported.



## 1. Introduction

Surface roughness measurements in nanometer order are widely performed, using methodology such as an atomic force microscope (AFM) in order to control quality of thin-film fabrications and material surface treatments. Recently more precise and reliable surface roughness measurements have been required [1–3]. In some cases, AFMs are calibrated by using calibrated pitch and height standards to further enhance reliability of surface roughness measurements. Alternatively if calibrated surface roughness standards of nanometer order are available as check standards, users will be able to extend calibration interval of AFMs while maintaining their acceptable uncertainties. For commercial AFMs, however, no calibrated surface roughness standards have been available yet, which feature *Ra* (the arithmetical mean deviation of the roughness profile) of less than 30 nm, and against which uncertainties are estimated [4,5].

Three elements are essential for developing and disseminating calibrated surface roughness standards for AFMs: (1) calibration instrument, (2) surface roughness standard and (3) calibration procedure. These elements cannot be developed separately but need to be developed together simultaneously.

We have developed atomic force microscopes with high-resolution laser interferometers mounted on their *x*-, *y*- and *z*-axes (metrological AFMs) [6,7] and provided calibration services of pitch (calibration range: 23 nm to 8 μm) [8,9] and step height (calibration range: 10 nm to 2.5 μm) [10] using metrological AFMs. In this study, this metrological AFM is used as calibration instrument.

Stylus roughness meters are mainly used to measure roughness of machined surfaces. Therefore, surface roughness standards for stylus roughness meters are designed in a way to fit machined surfaces [5]. On the other hand, AFMs are mainly used for analysis of thin film surfaces made in deposition process. Therefore, it is anticipated that AFM users will find it useful if surface roughness standards for AFMs are designed by giving considerations to deposited thin film. In this study, several types of material

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samples including thin films deposited on substrates are selected to measure their surface roughness while taking into account possible future development of surface roughness standards for AFMs.

The evaluation method of AFM tip shape is a key in the surface roughness measurement using an AFM. Several types of characterizers including line-and-space samples are proposed [11,12]. In this study, a probe examination sample is selected as a characterizer and the Japanese Industrial Standard (JIS) R 1683: 2007 "Test method for surface roughness of ceramic thin films by atomic force microscopy" is adopted. Since the cross sectional shape of the probe examination sample is almost the same as that of the knife edge shape, the type B3 sample specified in ISO 5436-1 "Geometrical Product Specifications (GPS) – Surface texture: Profile method; Measurement standards – Part 1: Material measures" it is easy for users to understand the tip evaluation principle of the method using probe examination sample and to use it. In this paper, the tip test method based on JIS R 1683: 2007 and the procedure of surface roughness measurement are described first [13] and then results of the surface roughness measurement using a metrological AFM and their uncertainty evaluation results are reported.

## 2. Samples, measurement method and uncertainty evaluation

### 2.1. Samples

Table 1 shows specifications of the six material samples used in this study. The material sample selection criteria are fourfold: (1) samples are commercially available, (2) their substrates are considered flat, for instance, optics, (3) *Ra* is expected to be between 1 nm and 30 nm and (4) their external dimensions are smaller than 30 mm × 30 mm × 4 mm or preferably smaller than 20 mm × 20 mm × 4 mm, provided that several measurement areas of interest (AOI) can be selected on each sample. Samples 1, 2 and 3 have thin films deposited on their substrates while Samples 4, 5 and 6 have no thin films on the surface. The two types of indium tin oxide (ITO) thin film samples are selected (Samples 2 and 3) partly because there is much demand for AFM measurement of their surface roughness and partly because JIS R 1683: 2007 refers to ITO thin film as a measurement example. As an optional sample, a roughness standard is added although its *Ra* is larger than 30 nm because its roughness was measured with AFM in previous studies [3,4]. The roughness standard of RnS 20 used in the previous studies is not commercially available now, however, RnS 40 is used in this study which features the smallest *Ra* among currently available roughness standards of the same type.

### 2.2. Measurement method

This subchapter provides a brief explanation of JIS R 1683: 2007 "Test method for surface roughness of ceramic thin films by atomic force microscopy" adopted in this study as a nanometer-order surface roughness measurement method. More detailed information is given elsewhere [13]. The draft of JIS R 1683 was proposed by Japan Fine Ceramics Association and Japan Fine Ceramics Center in an attempt to standardize methods to control AFM tip diameter in the surface roughness measurement using an AFM. After being considered by Japanese Industrial Standards Committee, JIS R 1683 was established in 2007. The corresponding international standard has not yet been established by the International Organization for Standardization (ISO). JIS R 1683: 2007 refers to JIS B 0601, 0633 and 0651, "Geometrical Product Specifications (GPS) – Surface texture: Profile method". JIS B 0601, 0633 and 0651 correspond to ISO 4287, 4288 and 3274, respectively. The measurand in this study is *Ra*, arithmetical mean deviation of roughness profile. The application scope of JIS R 1683: 2007 is sine waves featuring *Ra* of 1–30 nm and the mean length of the roughness profile elements, *RSm*, of 40 nm to 2.5 µm. The roughness measurement is performed in the following steps: (1) calibration of an instrument, (2) preliminary roughness measurement of a sample, (3) tip test and (4) roughness measurement.

(1) *Calibration of an instrument:* Usually an AFM is calibrated by the calibrated pitch and step height standards. In this study, however, this calibration is not necessary as the metrological AFM is traceable to the length standard as mentioned in chapter 1.

**Table 1**
Specifications of samples.

| Sample No. | Sample | Manufacturer | Product number | Substrate material | Remarks |
|---|---|---|---|---|---|
| 1 | Flat aluminum total reflection mirror | SURUGA SEIKI CO., Ltd. [14] | S01-20-3T | Float glass | Coated with dielectric protection film |
| 2 | Indium tin oxide (ITO) thin film | GEOMATEC CO., Ltd. [15] | 0007 | Soda lime glass | Thickness: approximately 90 nm |
| 3 | Indium tin oxide (ITO) thin film | GEOMATEC CO., Ltd. [15] | 0005 | Soda lime glass | Thickness: approximately 330 nm |
| 4 | TipCheck sample | Aurora NanoDevices Inc. [16] | TIP001 | Silicon | – |
| 5 | Gauge block | Mitutoyo Corporation [17] | 613614-02 | Ceramics | Length: 4 mm |
| 6 | Roughness standard | SiMETRICS GmbH [4] | RnS 40 | Silicon | *Ra*: 35–45 nm (by a stylus instrument) *Sa*: 70 nm (by a confocal optical microscope) |