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PROFILE SURFACE ROUGHNESS MEASUREMENT USING METROLOGICAL ATOMIC FORCE MICROSCOPE AND UNCERTAINTY EVALUATION

Ichiko MISUMI, Kazuya NAOI, Kentaro SUGAWARA and Satoshi GONDA

National Metrology Institute of Japan, National Institute of Advanced Industrial Science and Technology (AIST),
Tsukuba Central 3, Umezono 1-1-1, Tsukuba, Ibaraki 305-8563, Japan, misumi.i@aist.go.jp

Abstract:
Surface roughness measurements are sometimes performed using an atomic force microscope (AFM) in order to evaluate conditions of thin film fabrication and of 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 Japan Industrial Standard (JIS) R 1683: 2007 "Test method for surface roughness of ceramic thin films by atomic force microscopy". In this study, surface roughness measurements were performed based on the JIS R 1683: 2007 and the measurement results are reported.

Keywords: surface roughness, metrological AFM, calibration, traceability

1. INTRODUCTION

Surface roughness measurements in nanometer order are widely performed, by using such a methodology as an atomic force microscope (AFM) in order to control quality of thin-film fabrications and material surface treatments. Recently more and 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, user 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 - 8 μm) [8-9] and step height (calibration range: 10 nm - 2.5 μm) [10] using the 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 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. As cross sectional shape of the probe examination sample is almost same as that of knife edge shape, the type B3 sample specified in the 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 the JIS R 1683 and the procedure of a 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. INSTRUMENT, SAMPLES, MEASUREMENT METHOD AND UNCERTAINTY EVALUATION

2.1 Instrument

A metrological AFM, an AFM with differential laser interferometer (DLI-AFM) was used for surface roughness measurements. More detailed information about this instrument is described elsewhere [7]. The DLI-AFM consists of an interferometer unit, a stage unit and a probe unit. The interferometer unit has the two-path differential homodyne laser interferometers on the X and Y axes and a four-path homodyne laser interferometer on the Z axis. Its theoretical resolution is approximately 0.04 nm, which is realized by optical and electrical division. A box-shaped five-sided moving mirror is used for the XYZ-axes interferometer. Sample is placed in this five-sided mirror. At maximum, a sample of 30 mm x 30 mm x 4 mm can be measured with the metrological AFM. The laser source is an offset locked laser which constantly tracks frequency of iodine stabilized He- Ne laser calibrated by an optical frequency comb system, the national length standard. Therefore, the DLI-AFM is traceable to the national length standard. The stage unit consists of a leaf-spring-type XY stage driven by piezo actuators and a piezo-driven tube-type Z scanner. The scanning range is 100 μm (X) x 100 μm (Y) x 12 μm (Z). The AFM cantilever is detected by a conventional optical lever system. The cantilever is driven in the intermittent contact mode (AC mode). The air temperature and humidity in the calibration room are approximately 20 ℃ ± 1 ℃ and 50 % ± 10 %, respectively.

2.2 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 x 30 mm x 4 mm or preferably smaller than 20 mm x 20 mm x 4 mm, provided that several measurement points 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]. As 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.

Table1: Specifications of samples

Sample No.	Sample	Manufacturer	Product number	Substrate material	Remark
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	-

2.3 Measurement method

This chapter 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 deliberated by Japanese Industrial Standards Committee, JIS R 1683 was established in 2007. Unfortunately, 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 nm to 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 above.

(2) Preliminary roughness measurement of a material sample : The preliminary roughness measurement of a sample is performed for the purpose of obtaining estimates of *Ra* and *RSm*, both of which are used in next step (3). The scanning range is 2 μm and 10 μm and the number of pixels is 256 or 512. The scanning range of the roughness measurement needs to be determined in a way to allow an evaluation length to cover twenty to fifty cycles of *RSm*.

(3) Tip test: Fig. 1(a) shows a schematic drawing of the measurement of a probe examination sample. *R* is curvature radius of an AFM tip. *D* is cross-sectional diameter of a tip. *D’* is measurement value of the cross-sectional diameter at 10 nm below the top of a probe examination sample. TGT1, a probe examination sample made by NT-MDT Co., Ltd., was selected [18]. TGT1 features patterns fabricated at 3 μm intervals, conical angle of 50 ± 10 degrees, curvature radius of are 10 nm or less and pattern height of 300 nm to 700 nm. JIS R 1683: 2007 specifies that the scanning area for the probe examination sample measurement is 1280 nm x 1280 nm and the number of pixels is 512 x 512. In the case of the metrological AFM, however, when its scanning area is adjusted, the AFM tip automatically contacts with a sample surface at the center of the scanning area before starting of scanning. The AFM tip and a probe examination sample will be broken if the center of scanning area is set at the peak of a probe examination sample as specified in the JIS standard. In order to prevent the tip from being broken, the center of scanning area is set at the point of 320 nm away from a peak of a probe examination sample, and the scanning area and the number of pixels are set at 1280 nm x 640 nm and 512 x 254, respectively. According to Appendix A of JIS R 1683:

2007, *D*' gets closer to *D* as *R* becomes larger, and when *R* exceeds 10 nm, the difference between *D* and *D*' will become less than measurement error and therefore *D*' can be considered as *D*. In Appendix B of JIS R 1683: 2007, the AFM tip evaluation criteria are specified. Desirable value of AFM tip's cross-sectional diameter *D* to measure surface roughness with sufficient precision is expressed below by using the model shown in Fig. 1(b). In this model, the surface profile is assumed to be a sine curve with the cycle of *RSm* given by Equation (1),

$$f(x) = \frac{\pi Ra}{2} \cdot \cos\left(\frac{2\pi x}{RSm}\right) \quad (1)$$

In this case, the maximum height of roughness profile *Rz* is expressed as follows:

$$Ra = \frac{Rz}{\pi} \quad (2)$$

Equation (2) gives a value between a triangle wave surface and a rectangular wave surface, and the value is often close to a real sample value. Next, it is assumed that the tip shape is sphere with its curvature radius *r* and that the tip is considered desirable when it can be inscribed at the bottom of a roughness profile. A tip with radius larger than the maximum radius ($r = r_{\max}$) of circle to be inscribed, therefore, is not suitable for the roughness measurement as the tip cannot contact with the bottom. In other words, a tip with *r* of equal or smaller than $r_{\max}$ is considered acceptable. Based on the above model, $r_{\max}$ is given as follows:

$$r_{\max} = \frac{RSm^2}{2\pi^3 Ra} \quad (3)$$

Then, *D* value at the $r_{\max}$, $D_{\max}$ is expressed as follows:

$$D_{\max} = 2\sqrt{20 r_{\max} - 100} \quad (4)$$

In the case of a sharp tip with *r* of less than 10 nm, however, $D_{\max} = 2r_{\max}$ should be used. By using *Ra* obtained in Step (2) as a constant, $D_{\max}$ is calculated in Equation (4) and the tip test curves of $D_{\max}$ and $D_{\max}/2$ are plotted in a graph. A point determined by *RSm* obtained in Step (2) and *D* obtained in Step (3) is plotted on the same graph. It is expected, based on the previous study [13], that *Ra* can be measured within the error of 5 % if *D* is equal or less than $D_{\max}/2$, and that *Ra* can be measured within the error of 20 % if the *D* is equal or less than $D_{\max}$. In the other cases, the tested tip is not acceptable for the *Ra* measurements.

(4) The surface roughness measurements are performed by using those tips found acceptable in the above-described tip test when errors of *Ra* measurements are allowed to be within 5 % or 20 %. Five points are measured for each material sample: four corners and a center of a 10 mm x 10 mm area located around the middle of the sample. If a material sample is smaller than 10 mm x 10 mm, five measurement points are selected from a large area as much as possible. Measurements are repeated twice at each measurement. Though JIS R 1683: 2007 specifies the number of pixels of (256 ~ 512) x (10 ~ 512), the number of pixels is set at 512 x 16 in this study to decrease tip wear. An image (number of pixels: 512 x 512), however, is captured at the final measurement location for obtaining reference surface information. *Ra*, *RSm* and *D* are calculated by using the roughness analysis module and particle analysis module of the Scanning Probe Image Processor (SPIP) software (made by Image Metrology A/S) [19]. Some of the new commercially-available AFMs have JIS R 1683-complient software pre-installed including a function of tip test [20]. As the measurand in this study is *Ra* which can be obtained by the sharpest tip in a real world, convoluted component of tip shape is not eliminated from obtained measurement curves.

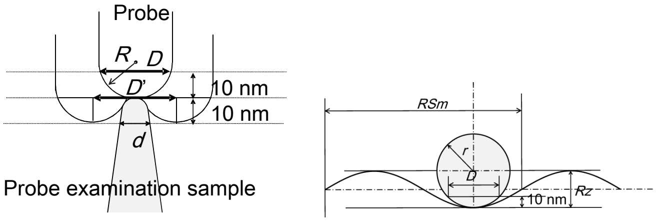


Fig. 1: (a)Schematic drawing of the measurement of a tip test sample, (b) Assumed model.

2.4 Uncertainty evaluation

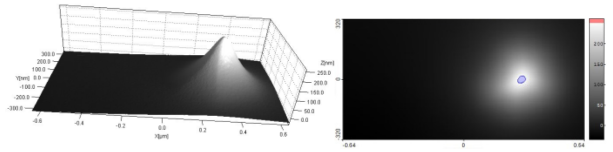
Sources of uncertainty in surface roughness measurements using a metrological AFM include, just like those of pitch [8] and step height [10] measurements, nonuniformity of a sample, repeatability of measurements, nonlinearity and resolution of interferometer, Abbe error, compensation of an index of refraction in air, change of optical path difference in interferometer and stability of laser frequency. Additionally, the tip test also constitutes a major source of uncertainty in surface roughness measurements. The uncertainty derived from tip test is estimated to be 5 % of *Ra* in the case of $D \le D_{\max}/2$ and 20 % of *Ra* in the case of $D \le D_{\max}/2$. It is also necessary to apply the analysis of variance [9] as there is a correlation between nonuniformity of a material sample and repeatability of measurements. The uncertainty of measurement is evaluated based on the ISO/IEC Guide 98-3, "Guide to the expression in of uncertainty in measurement (GUM)", 2008 [21].

3. RESULTS AND DISCUSSIONS

3.1 Results of AFM tip tests

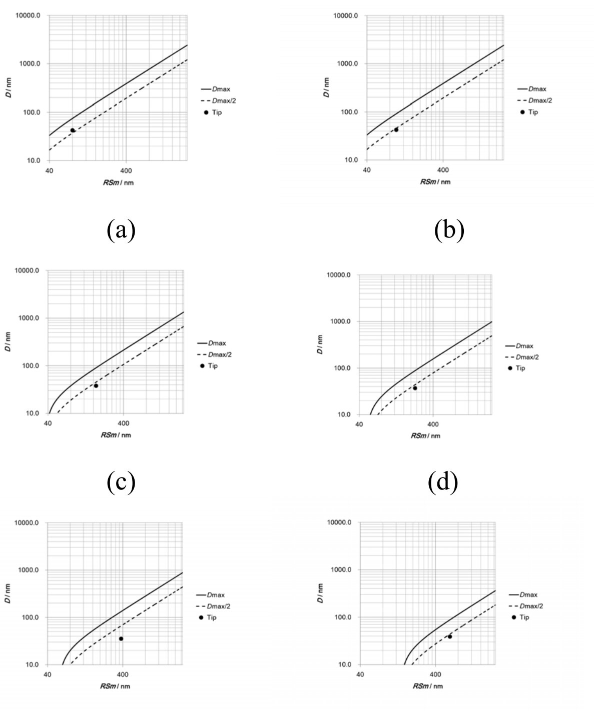
Two types of AFM cantilevers were used in the AFM tip test measurements. One is a standard silicon cantilever (OMCL-AC160TS, Olympus Co. Ltd.) featuring spring constant of approximately 42 N/m and resonance frequency of approximately 300 kHz and the other is a cantilever with carbon nanotube (CNT) tip (SI-DF40C, Hitachi High-Tech Science Corporation) featuring spring constant of approximately 42 N/m and resonance frequency of approximately 300 kHz. The diameters *D* obtained by the standard silicon cantilever and the cantilever with CNT tip were almost the same at approximately 35 nm - 40 nm. Since there was no difference observed in the calculated diameters between two types of cantilevers, the standard silicon cantilevers were mainly used in this study because of their lower costs. Fig. 2(a) is a three-dimensional image of a probe examination sample obtained by the standard silicon cantilever. Fig. 2(b) is a top view of (a) indicating a cross-sectional area at 10 nm below the top of a probe examination sample.

Fig. 3 shows results of the tip tests. The solid lines represent $D_{\max}$ and the dashed lines represent $D_{\max}/2$. The curves were calculated by using *Ra* obtained from the preliminary measurements of samples. The solid circles are determined by *D* obtained from the tip tests and *RSm* obtained from the preliminary measurements of the samples. Fig. 3(a), (b), (c), (d), (e) and (f) show the results of Samples 1, 2, 3, 4, 5 and 6, respectively. In Fig. 3(a), as the solid circle is located between the solid line and the dashed line, the tip is found acceptable for the *Ra* measurements within the error of 20 %. In Fig. 3(b) – 3(f), as the solid circles are located below the dashed lines, the tips are found acceptable for the *Ra* measurements within the error of 5 %. Based on the above AFM tip test results, measurement conditions were defined as shown in Table 2.



(a) (b)

Fig. 2: Examples of measurements of a probe examination sample. (a) a three-dimensional image of a probe examination sample obtained by the standard silicon cantilever. (b) a top view of (a) indicating a cross-sectional area at 10 nm below the top of a probe examination sample.



(a) (b)

(c) (d)

(e) (f)

Fig. 3: Results of AFM tip tests of Samples 1 – 6.

Table 2: Measurement conditions of each sample

Sample No.	Scanning area	Scanning speed	Number of pixels	Number of measurement points	Repetition number at each measurement point
1	2 μm x 2 μ m	400 nm · s^{-1}	512 x 16	5	2
2	2 μm x 2 μm	400 nm · s^{-1}			
3	5 μm x 5 μm	500 nm · s^{-1}			
4	5 μm x 5 μm	500 nm · s^{-1}			
5	10 μm x 10 μm	1 μm · s^{-1}			
6	20 μm x 20 μm	1.2 μm · s^{-1}			

3.2 Results of surface roughness measurements and their uncertainty evaluation results

Fig. 4 shows measurement curvature images. Fig. 4(a) shows measurement curvature images of Sample 3 at each measurement point used for *Ra* calculations. The number of pixels is 512 x 16 for the five measurement points and 512 x 512 for the fifth measurement point. The images with pixel count of 512 x 16 are sufficient enough for *Ra* calculation. In order to precisely investigate the sample surfaces, however, an image with higher resolution (the number of pixels: 512 x 512) is captured at the fifth measurement point for each material sample. Fig. 4(b) - 4(g) show the measurement curvature images at the fifth measurement point of Samples 1 - 6. Surfaces of Samples 1, 2, 3, 4 and 6 are found almost isotropic, and therefore scanning can be performed in any directions. On the surface of Sample 5, there are grooves almost perpendicular to the scanning direction, and therefore this scanning direction is appropriate based on the JIS B 0633: 2001 (ISO 4288: 1996)

Table 3 shows the major sources of uncertainty in *Ra* measurements. Nonlinearity and resolution of interferometers are constant, not depending on *Ra*. Therefore, relative uncertainties derived from these two sources tend to become larger when *Ra* is small. On the other hand, uncertainty derived from AFM tip test is proportional to *Ra*, and therefore becomes larger as *Ra* increases. Uncertainties derived from the other sources were sufficiently small and negligible. Table 4 shows the measurement results *Ra* and the expanded uncertainty U (k = 2).

For Samples 2 and 4, the entire series of measurement steps was repeated ten times by using the same AFM cantilever in order to study tip wear and its impacts on the *Ra* measurement results. Tip test was performed at every series of measurement steps. Fig. 5(a) and (b) show results of the ten series of measurements for Samples 2 and 4, respectively. The square symbols represent measurement values of *Ra*. The error bars are the expanded uncertainties U (k = 2) of *Ra* and the dashed line is approximation line of *Ra*. The circle symbols represent the measurement results of *D* and the straight line is approximation line of *D*. Within the error of 5 %, $D_{\max}/2$ is approximately 44.0 nm for Sample 2 and approximately 72.9 nm for Sample 4. For Sample 2, based on the tip test results, uncertainties of the first to third measurements were evaluated within the error of 5 % and uncertainties of the fourth to tenth measurements were evaluated within the error of 20 %. The *Ra* measurement results became smaller after each measurement as tip wear increased. It is, therefore, considered appropriate to change the error in evaluation of uncertainties based on the tip test results. By contrast, for Sample 4, the measurement results of *Ra* and expanded uncertainties remained almost unchanged although tip wear increased. It was decided, therefore, to apply the error of 5 % to evaluation of uncertainties of all the measurements.

4. DISCUSSION

Surface roughness measurements of several types of materials using a metrological AFM were performed and their uncertainties of measurements were evaluated. It is verified whether or not the instrument, samples and

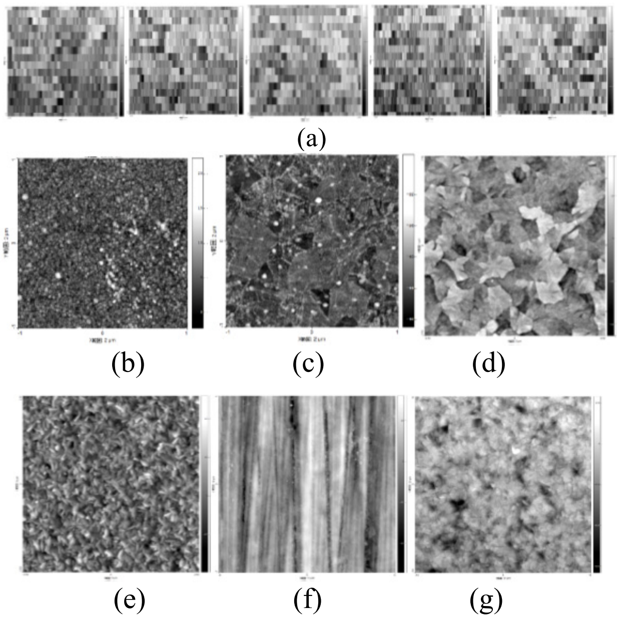


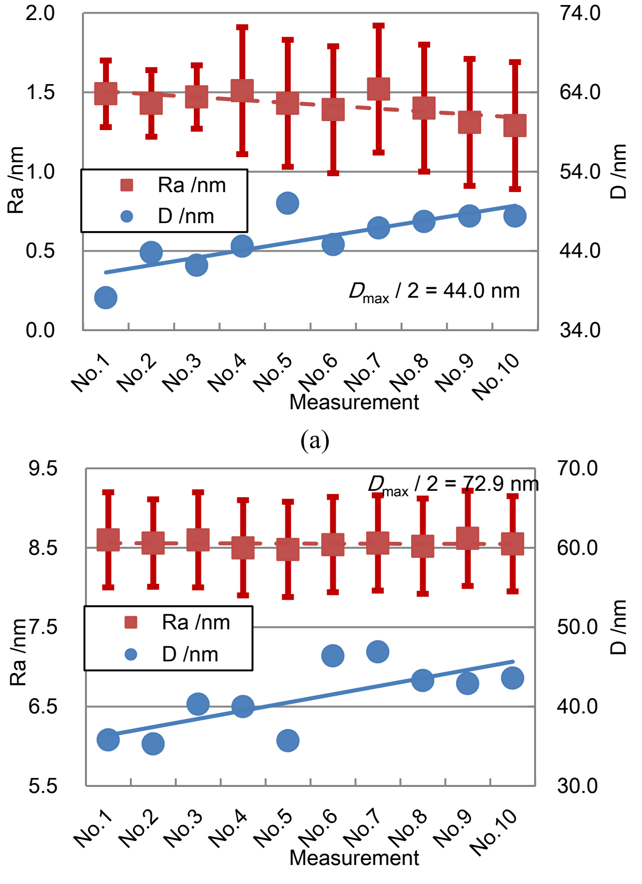
Fig. 4: Measurement curvature images. (a) Sample 3 at each measurement point used for *Ra* calculations. The number of pixels is 512 x 16 and the scanning area is 5 µm x 5 µm. (b) Sample 1. The number of pixels is 512 x 512 (c) Sample 2. (d) Sample 3. (e) Sample 4. (f) Sample 5. (g) Sample 6.

Table 3. Major sources of uncertainty in *Ra* measurements and contributions to the combined standard uncertainty

Major source of uncertainty	Contribution to the combined standard uncertainty					
	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6
Interferometer nonlinearity	0.07 nm	0.07 nm	0.07 nm	0.07 nm	0.07 nm	0.07 nm
Tip test	0.16 nm	0.04 nm	0.20 nm	0.24 nm	0.30 nm	1.8 nm
Non-uniformity of a sample	0.03 nm	0.03 nm	0.14 nm	0.09 nm	0.74 nm	1.5 nm
Interferometer resolution	0.02 nm	0.02 nm	0.02 nm	0.02 nm	0.02 nm	0.02 nm
Repeatability of measurement	0.01 nm	0.01 nm	negligible	negligible	negligible	0.06 nm

Table 4. *Ra* measurement results of each sample and expanded uncertainties ($k = 2$).

Sample No.	Measurement result, *Ra*	Expanded uncertainty, U ($k = 2$)
1	1.4 nm	0.4 nm
2	1.4 nm	0.2 nm
3	4.5 nm	0.5 nm
4	8.3 nm	0.6 nm
5	10.4 nm	1.6 nm
6	61.5 nm	4.6 nm



(b)

Fig. 5: The results of ten sets of measurement using the same AFM cantilever. The square symbols represent measurement values of *Ra*. The error bars are the expanded uncertainties U ($k = 2$) of *Ra* and the dashed line is approximation line of *Ra*. The circle symbols represent the measurement results of D and the straight line is approximation line of D. Fig. 5(a) and (b) show the results of Samples 2 and 4, respectively.

measurement procedure adopted in this study are appropriate. The roughness measurement results demonstrate that the metrological AFM is useful not only for pitch and step height measurements but also for surface roughness measurements. Several types of materials were selected as samples in this study. In general, users find it useful when they are provided with a set of roughness standards featuring about three different roughness levels. In this study, two samples, namely Samples 2 and 3, were compared with each other. They were composed of the same thin film materials but featured different film thickness. It was found that *Ra* measurement results were different between Samples 2 and 3. This finding may suggest the possibility that a standard of desired roughness could be developed by precisely controlling thickness of thin film. Sample 4 was used in this study, aiming essentially at studying AFM tip wear. Shape of Sample 4 is basically suitable for AFM measurements and it was easy to measure its surface roughness with the metrological AFM. Fabrication method of Sample 4 is not made public at present, but if the Sample 4 fabrication method can be used to achieve different roughness levels, it may become possible to develop a set of easy-to-use roughness standards. In this study, the measurement procedure compliant with JIS R 1683: 2007 was adopted. The procedure was found applicable to the surface roughness measurements using a metrological AFM and their uncertainty evaluations. Based on the above, the instrument, samples and measurement procedure adopted in this study are found to be one of the bests available at present.

As a result of the roughness measurements and uncertainty evaluations, however, the following challenges were identified:

(1) One of the challenges is related to AFM tip test. In this study, the tip tests were performed twice for each material sample, before and after a series of roughness measurements. In most cases, *D* did not change between the first tip test and the final tip test, or *D* got larger due to tip wear. In several cases, however, *D* in the first tip test was larger than that in the final tip test. This is attributed to failure to use the same peak of a probe examination sample between the first and final tip tests and to variance of curvature radius among peaks. It will become possible to measure average *D* with higher reliability if several peaks can be selected to be scanned several times. This solution, however, is not realistic because tip wear will increase as more peaks are measured in tip tests. It is necessary to keep it in mind that *D* measurement results are always accompanied with some uncertainty.

(2) The second challenge is related to filtering. JIS R 1683: 2007 stipulates that the surface profile may be used as roughness profile because it has no short-wavelength component due to the size of tips and because it has no long-wavelength component due to short evaluation length in AFM measurement. This study has adopted what is stated in JIS R 1683: 2007. For those who use AFMs alone in surface roughness evaluation, JIS R 1683: 2007 is very useful because of its simplicity. However, if users want to compare roughness measurement results between AFMs and other methods, it is necessary to use some suitable filters to separate roughness profiles from waviness profiles [1]. This is a challenge to be addressed in the future. In less than 1 nm *Ra* measurements, in spite of the outside of the scope of the JIS R 1683: 2007, there is a possibility that some noises caused by an AFM apparatus cannot be negligible. In order to eliminate short-wavelength components in raw profiles, it is possible to filter the raw profiles with the cut off wavelength value λs closed to the AFM tip size.

(3) The third problem is uncertainty derived from AFM tip test. AFM tip test is one of major sources of uncertainty and uncertainty derived from this test becomes larger in proportion to *Ra*. In this study, based on the criteria of tip test, the errors of 5 % and 20 % were directly used and its uncertainties were evaluated by assuming a rectangular distribution. As shown in Fig. 3(a), the solid circle is located between the curve of $D_{\max}$ and the curve of $D_{\max}/2$, but the circle is closer to the curve of $D_{\max}/2$ and therefore the uncertainty can be estimated to be smaller than 20 %. As mentioned in the above (1), however, an estimated value of *D* has some uncertainties, which are considered to be included in the uncertainty derived from AFM tip test. This is why the errors of 5 % and 20 % were directly used in this study.

This study focused on *Ra* alone for roughness measurements and uncertainty evaluations. The reasons are twofold: 1) *Ra* measurement is sufficient enough for daily quality control of thin film fabrication and 2) this study aims at evaluating uncertainties derived from nonuniformity of a sample by repeating measurements at several measurement points while suppressing tip wear. In reality, however, there is an increasing demand to use AFMs not only for *Ra* measurement but also *Sa* (arithmetical mean height of the scale limited surface) measurement since AFMs can acquire measurement curvatures of samples as shown in Fig. 4 [3]. The calibration instrument, samples and a calibration procedure proposed in this study will be able to contribute to progress of surface texture measurement by using areal methods.

5. CONCLUSIONS

Surface roughness measurements of several types of materials were performed using a metrological AFM and their uncertainties were evaluated. AFM tip test is a key technology in roughness measurements and the procedure specified in JIS R 1683: 2007 was adopted in this study. The following three findings have been revealed from the results of roughness measurements and uncertainty evaluations: (1) the metrological AFM can be used for surface roughness measurement in nanometer order, (2) a set of thin film samples with their thickness varied and a sample to study tip wear will serve as candidates for future surface roughness standards and (3) measurement procedure specified in JIS B 1683: 2007 can be applied to surface roughness measurements using AFMs. The instrument, samples and the measurement procedure proposed in this study are found to be one of the bests available at present for surface roughness measurement using an AFM. Meanwhile a few challenges to be solved are identified, including filtering and uncertainty derived from tip test. It is essential to develop new calibration instruments and surface roughness standards for AFMs, and standardize the measurement procedure.

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