Uncertainty in measurements of micro-patterned thin film thickness using Nanometrological AFM

- Reliability of parameters for base straight line -

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Abstract

Precision measurements of thin film thickness (10, 7, 5 and 3 nm) were carried out using an atomic force microscope with three-axis laser interferometer (Nanometrological AFM). Uncertainty in measurements of thin film thickness was estimated. In estimation of uncertainty, the reliability of parameters for base straight line was considered. From the results of uncertainty estimation, less than 0.4 nm expanded uncertainty ($k = 2$) was obtained.

Keywords

Precision Measurement, Laser Interferometer, AFM, Calibration, Traceability, Nanometrology

1. Introduction

Nanometrology, dimensional metrology in nanometer-scale, is becoming increasingly important. In nanometrology, length-standard-traceable measurements are required in order to certify obtained values in measurements. National Metrology Institutes (NMIs) have developed length-standard-traceable nanometrology instruments corresponding to this requirement. National Metrology Institute of Japan, AIST (NMIJ/AIST) has developed an atomic force microscope with XYZ-axis laser interferometer (Nanometrological AFM)[1] and has measured nanometrological standards precisely using a Nanometrological AFM. The Nanometrological AFM is servo-controlled by laser interferometer signals and can realize length-standard-traceable calibration of nanometrological standards in real time. In precision measurements of one-dimensional (1D) grating standards (nominal pitch 240 nm) using the Nanometrological AFM, expanded uncertainty ($k = 2$) of approximately 0.3 nm could be obtained[2].

In this study, precision measurements of thin film thickness (Nominal thickness values: 10, 7, 5 and 3 nm) using the Nanometrological AFM were carried out. Thin films were micro-patterned for AFM measurements. Furthermore, uncertainty in thin film thickness measurements was estimated. However, the micro-patterned thin films do not have enough measured data for base straight line approximation compared with stepheight nanometrology standards. Therefore, in the estimation of measurement uncertainty, the reliability of parameters for the Gaussian associated base straight line using least squares method is considered.

2. Experimental methods

2.1 Atomic force microscope with three-axis laser interferometer (Nanometrological AFM)

Figure 1 is a photograph of an atomic force microscope system with three-axis laser interferometer (Nanometrological AFM). The Nanometrological AFM system is a stage scanning type and is operated in contact AFM mode. The Nanometrological AFM system consists of a stage unit, an AFM probe unit and an interferometer unit. The probe unit is removed in Figure 1. The stage unit is comprised of a piezo-driven XY-axis leaf spring stage and a Z-axis scanner tube-type piezo actuator. Scanning area of this stage unit is approximately 17.5 µm (X)× 17.5 µm(Y) × 2.5 µm(Z). A three-sided moving mirror for the XYZ interferometer unit is set at the top of the Z-axis scanner. The interferometer has 4 optical paths in each axis and the total resolution of the interferometer unit is approximately 0.04 nm. Atomic force applied to a cantilever under a contact-mode is detected using a conventional optical lever method. Laser sources of the interferometer unit are practical frequency-stabilized He-Ne lasers with a wavelength of 633 nm (model 117A Spectra-Physics. Ltd). The laser frequency is calibrated by an I_2 stabilized He-Ne laser before calibration of nanometrological standards. The stage position is servo-controlled using the interferometer signals in real-time and the Nanometrological AFM can realize the direct-length-standard-traceable calibration of nanometrological standards. Main uncertainty components in measurements of one-dimensional (1D) gratings (nominal pitch value: 240 nm) using the Nanometrological AFM are shown in Figure 2. The maximum uncertainty component was cyclic error of laser interferometer and its value was 0.115 nm.

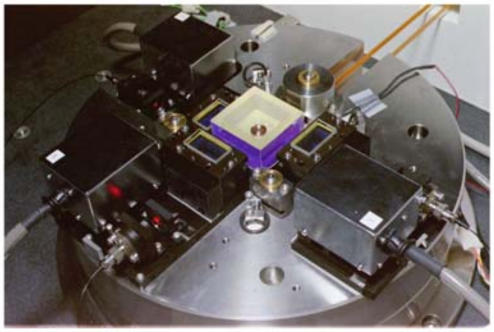

Figure 1 Atomic force microscope with three-axis laser interferometer (Nanometrological AFM)

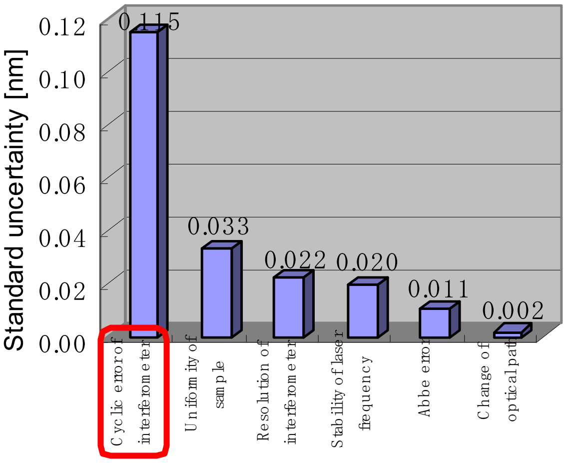

Figure 2 Main uncertainty components in pitch measurements using Nanometrological AFM.

2.2 Micro-patterned thin film

Thermal oxidized films of silicon substrates were wet etched precisely by buffered hydrofluoric acid and the thickness were controlled at 10, 7, 5 and 3 nm, respectively[3]. The thickness of thin films was checked using X-ray reflectivity (XRR). Furthermore, two-dimensional (2D) gratings (pitch value: approximately 3 µm) were made of thin films for AFM measurements. The size of silicon substrate was 15 mm(X)×15 mm(Y).

2.3 Measurement conditions and thickness calculation

Measurements of thin film thickness were operated in a temperature and humidity controlled room at 293 K and 50 %. The nanometrological AFM system was set on a vibration isolation system and covered with a shielding for stable measurements. Air temperature, neighborhood temperature of a sample, air humidity and air pressure were recorded during measurements of thin film thickness and the recorded data were used in order to compensate the refractive index of air and thermal expansion of samples.

Figure 3 is a nanometrological AFM image of micro-patterned thin film before slope correction. Scanning range and scanning lines were 5 μm(X) × 5 μm(Y) and 128 lines, respectively. Only line profiles suitable for thin film thickness calculation were selected from 128 lines obtained. Sampling interval of laser interferometer signals was approximately 6 nm. Figure 4 shows a schematic drawing of a sectional view of micro-patterned thin film. Calculation procedures of thin film thickness are as follows; (1) Slope of a line profile is calculated from the approximated straight line using measured data of Left part and Right part. (2) Slope of a line profile is corrected (3) Slope-corrected line profile is offset and base line derived from Left part and Right part becomes zero. (4) Average of measured data in Top part as a film thickness value is calculated. (5) Average and standard deviation of film thickness values of the selected line profiles are calculated as a representative value and standard deviation in the measured area.

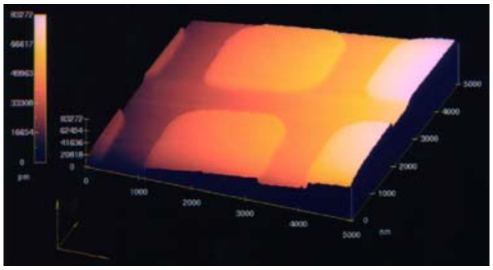

Figure 3 AFM image of micro-patterned thin film (thickness: 10 nm, measured area: 5 μm (X) x 5 μm(Y))

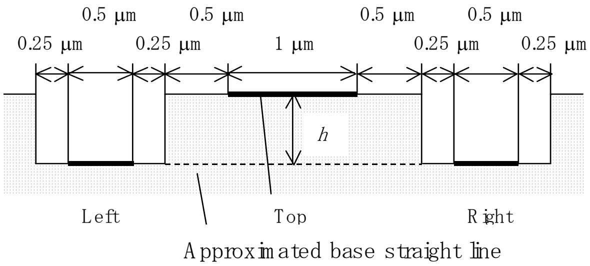

Figure 4 Schematic drawing of a sectional view of micro-patterned thin film

3. Reliability of base straight line parameter

Figure 5 shows a criterion for evaluation of stepheight standards in ISO 5436-1. Three times the line-width is required as a scanning range for the evaluation. However, the micro-patterned thin films do not have enough scanning range for base straight line approximation compared with the criterion. Therefore, in the estimation of measurement uncertainty, the reliability of parameters for the Gaussian associated base straight line using least squares method is considered. The calculation procedures of reliability of parameters for base straight line are as follows[4]; (1) the relation between error of measured data and error of parameters for straight line is calculated. (2) Reliability range at the position of least squares line is calculated. Jacobian matrix, **A** of straight line is expressed in eq(1);

$$\mathbf{A} = \begin{pmatrix} 1 & x_1 \\ \vdots & \vdots \\ 1 & x_n \end{pmatrix} \qquad (1)$$

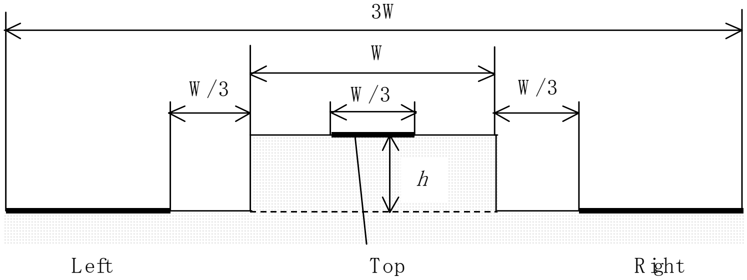

Figure 5 Schematic drawing of a criteria for evaluation of stepheight standards (ISO 5436-1).

Error matrix of parameters $\mathbf{S}_p$ is expressed in eq(2),

$$\mathbf{S}_p = \begin{pmatrix} {\sigma_{p_1}}^2 & \\ & {\sigma_{p_2}}^2 \end{pmatrix} = \left(\tilde{\mathbf{A}}\mathbf{S}^{-1}\mathbf{A}\right)^{-1} = {\sigma_0}^2 \begin{pmatrix} \frac{1}{n} & 0 \\ 0 & \frac{1}{\sum {x_i}^2} \end{pmatrix} \tag{2}$$

where $\mathbf{S}$ is the error matrix of measured data and $\tilde{\mathbf{A}}$ is the transposed matrix of $\mathbf{A}$. Error matrix $\mathbf{S}_m$, the reliability range at any position of associated straight line, is expressed in eq(3).

$$\mathbf{S}_m = \mathbf{A}\mathbf{S}_p\tilde{\mathbf{A}} \tag{3}$$

Measured points (number: $2n$) for base straight line are located as shown in Figure 6. In this case, Jacobian matrix of straight line, $\mathbf{A}$ can be expressed simply in (4)

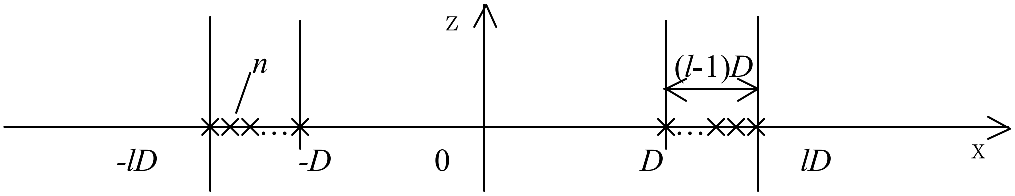

Figure 6 Measured points for base straight line.

$$\mathbf{A} = \begin{pmatrix} 1 & x \end{pmatrix} \tag{4}$$

Therefore, the reliability range at any position of associated straight line, $\mathbf{S}_m=\sigma_m$ is expressed in eq(5)

$$\sigma_m = \sqrt{\frac{1}{2n} + \frac{1}{n+(l-1)(n+1)+\frac{1}{6n}(n+1)(2n+1)(l-1)^2} \cdot \frac{x^2}{D^2}} \cdot \sigma_0 \tag{5}$$

where σ_0 is error of measured data.

Figure 7 shows the relationship between the normalized reliability range σ_m/σ_0 and the normalized horizontal position x/D (number of measured points $2n = 100$). The solid line shows the normalized reliability range in measured data parts for base line, $l = 1.4$ (micro-patterned thin films) and the dashed line shows the normalized reliability range in l = 1.8 (ISO). The normalized reliability range σ_m/σ_0 in l = 1.4 is slightly larger than the normalized reliability range in l = 1.8. Thickness of micro-patterned thin films is calculated in the range of $|x/D| < 1$. The maximum reliability range in l = 1.4 and $|x/D| < 1$, σ_m/σ_0 is approximately 0.15. The maximum reliability range, $\sigma_m/\sigma_0 = 0.15$ and the measured data variation for base straight line σ_0 are added as uncertainty components to the estimation of uncertainty in measurement.

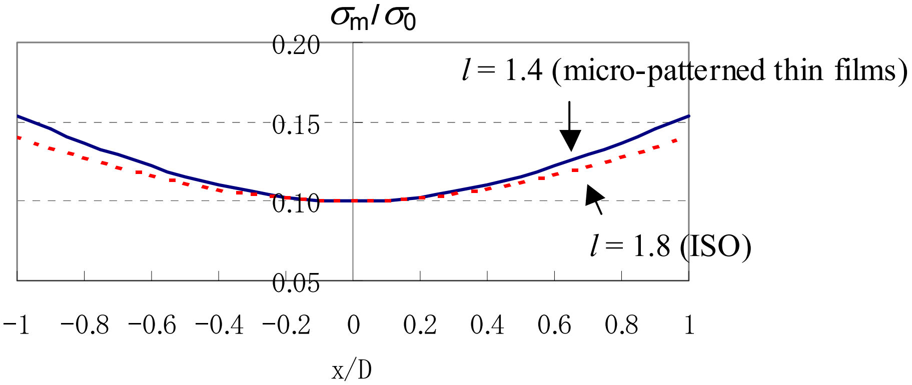

Figure 7 Reliability range versus position of approximated base straight line at number measured points $2n = 100$.

4. Uncertainty in measurements

In estimation of measurement uncertainty, seventeen uncertainty components were considered. Figure 8 shows six major uncertainty components in 10 nm thickness measurement. The maximum uncertainty component is cyclic error of laser interferometer, 0.115 nm. The uncertainty of reliability range of parameters for base straight line is 0.003 nm and the sixth major uncertainty component. On the other hand, figure 9 shows major uncertainty components in 5 nm thickness measurement. The uncertainty of measured data variation for base straight line is approximately equal to the uncertainty of cyclic error of laser interferometer. The uncertainty of the reliability range of parameters for base straight line increases in proportion to the uncertainty of measured data variation for base straight line compared with the uncertainty in 10 nm thickness measurement. It becomes clear that the uncertainty of reliability range of parameters is not a negligible uncertainty component when the measured data variation for base straight line is large compared with other uncertainty components, as in the case of 5 nm thickness measurement.

Table 1 shows measured values and expanded uncertainty ($k = 2$) in measurements of thin film thickness. Expanded uncertainty in 10, 7 and 3 nm thickness measurements is approximately 0.28 nm. On the other hand, expanded uncertainty in 5 nm thickness measurement is 0.35 nm. This is derived from the large measured data variation and the reliability range of parameters, as shown in Figure 9.

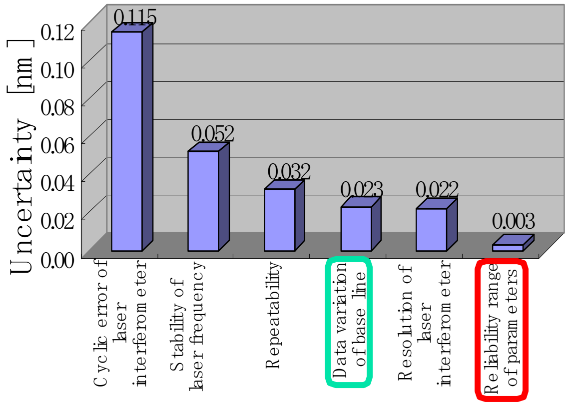

Figure 8 Major uncertainty components in 10 nm thickness measurements

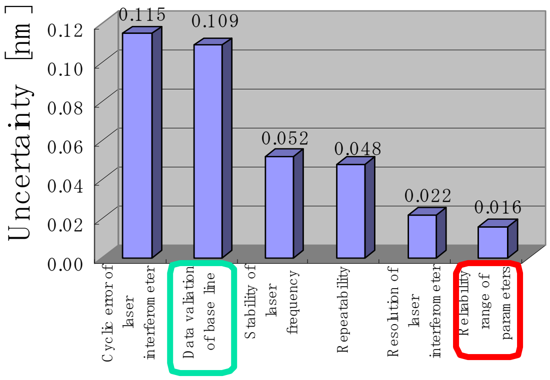

Figure 9 Major uncertainty components in 10 nm thickness measurements

Table 1 Measured values and uncertainty in measurements of thin film thickness

Nominal thickness, [nm]	Measured value, *h* [nm]	Expanded uncertainty, $U_{95}(p)$ [nm]
10	10.46	0.27
7	7.72	0.28
5	5.47	0.35
3	3.63	0.28

5. Conclusions

Precision measurements of thin film thickness (nominal value 10, 7, 5 and 3 nm) were carried out using nanometrological AFM. Expanded uncertainty (k = 2) was less than 0.4 nm. In estimation of uncertainty, the reliability of parameters for base straight line was considered. Uncertainty of the reliability of parameters for base straight line was the sixth major uncertainty component in seventeen uncertainty components. In estimation of measurement uncertainty, it is helpful to consider the reliability of parameters for associated features.

References

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