INSTITUTE OF PHYSICS PUBLISHING MEASUREMENT SCIENCE AND TECHNOLOGY
Meas. Sci. Technol. **14** (2003) 2065–2074 PII: S0957-0233(03)66029-7

Submicrometre-pitch intercomparison between optical diffraction, scanning electron microscope and atomic force microscope

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Received 10 July 2003, in final form 27 August 2003, accepted for publication 2 September 2003
Published 6 October 2003
Online at stacks.iop.org/MST/14/2065

Abstract
Intercomparison of pitch measurements for one-dimensional-grating standards (240 nm pitch), one of the widely used reference standards for nanometric lateral scales, was performed by three different methods, optical diffraction, critical dimension scanning electron microscopy and nanometrological atomic force microscopy. Average pitch values obtained by the three methods deviated by a maximum of only 0.67 nm with expanded uncertainties ($k = 2$) of less than 1.2 nm. The calculated E_n number, the index of measurement quality, of less than 1 indicates consistency of the measured pitch values and subsequent uncertainty analyses performed by three methods.

Keywords: intercomparison, optical diffraction pitch calibration apparatus, CD-SEM, AFM, uncertainty, nanometrology, calibration, metrological equivalence

(Some figures in this article are in colour only in the electronic version)

1. Introduction

Due to the recent miniaturization of small surface features to the nanometric scale, nanometrology, the dimensional measurement of surface features of nanometre order, is becoming important. Several nanometrological instruments, for example the critical dimension scanning electron microscope (CD-SEM), the atomic force microscope (AFM), the optical diffractometer (OD) and the optical scatterometer, are used for the evaluation of small-size features of

submicrometre scale. In order to calibrate the scale of nanometrological instruments, high-reliability nanometrology standards are required. Furthermore, in order to account for the reliability of nanometrology standards, they must be calibrated using length-standard-traceable instruments. The National Metrology Institute of Japan, AIST (NMIJ/AIST) has developed a 'nanometrological AFM' system with an ultrahigh-resolution three-axis laser interferometer [1] and now provides a calibration service for one-dimensional (1D)-grating standards, one of the nanometrology standards, using the system [2].

In the field of nanometrology, National Metrology Institutes (NMIs) carry out or take part in intercomparison measurements in order to ensure their calibration results. These intercomparison measurements are roughly divided into three types.

(1) An intercomparison may be planned and operated by consultative committees of the Bureau International des Poids et Mesures. These include international key comparisons in which a few representative NMIs from one region participate, regional comparisons (for example EUROMET in the EU, APMP in the Asia and Pacific area) and bilateral comparisons between two NMIs. In these kinds of comparisons, the measurement capabilities of individual NMIs are assumed to be equal to each other and the reference value of the intercomparison is calculated from all participants' results after the removal of outlying ones. Supplementary key comparisons of 1D-gratings (CCL NANO4) carried out from 1999 to 2001 between NMIs can be given as an example of this kind of intercomparison [3]. In the supplementary key comparison, optical diffractometers (ODs) and scanning probe microscopes (SPMs) were mainly used. The results appear in the key comparison database as appendix B in [4]. NMIJ/AIST, formerly the National Research Laboratory of Metrology (NRLM), took part in the supplementary key comparison using a nanometrological AFM and the results of NMIJ/AIST were confirmed by this comparison.
(2) An intercomparison may be carried out by both NMIs and other participants, for example private companies, in domestic or regional areas. In this kind of intercomparison, the reference value of the comparison is decided from the results of NMIs. An intercomparison of linewidth using optical beam methods and electron beam methods between Japanese private companies and the former NRLM [5], and an intercomparison using SPMs between NMIs, private companies and universities in the EU are examples [6].
(3) An intercomparison may be carried out within one NMI. Such intercomparison is performed to ensure consistency between measurement results obtained by different methods in the same NMI. An intercomparison of linewidth using AFM, SEM and electrical critical dimension (ECD) carried out at NIST is an example of this kind of intercomparison [7]. However, an intercomparison using all optical instruments, CD-SEMs and SPMs has not yet been carried out. This requires the equality of measurement results for the same nanometrology standards obtained using several kinds of nanometrological instruments.

In this research, NMIJ/AIST, as the pilot laboratory, organized intercomparison measurements of 1D-grating standards. The Japan Quality Assurance Organization (JQA), Hitachi Science Systems (HSS) and NMIJ/AIST took part in the intercomparison using an optical diffraction pitch calibration apparatus (OD), a CD-SEM and a nanometrological AFM, respectively. The objective of this intercomparison is to establish whether or not the equality of average pitch values obtained from several nanometrological instruments could be confirmed or not. NMIJ/AIST, the pilot laboratory for this intercomparison, decided a technical protocol draft of the intercomparison, based on the technical protocol of the supplementary key comparison of 1D-gratings (CCL NANO4) and approved by the participants. In this paper, firstly the specifications of the 1D-grating standards and the measurement positions are shown. Next, measurement procedures using an OD, a CD-SEM and a nanometrological AFM are explained. Finally, the results and discussions of the intercomparison are shown.

2. Measurement procedures

2.1. 1D-grating standards and measurement positions

Standard samples for the intercomparison are required to satisfy the following conditions.

(1) Standard samples should be measurable using the following nanometrological instruments: ODs, CD-SEMs and SPMs.
(2) Standard samples should be conventional and commercially available. The advantage of this is that each participant can easily prepare the standard samples before the intercomparison and optimize the measurement procedures.
(3) Standard samples should be used or ready to be used in nanotechnology in practice.
(4) The safety of the standard samples should be clear.
(5) Standard samples should be fine enough that measurement is challenging.

According to the above conditions, three samples of standard microscales (nominal pitch value: 240 nm) for the calibration of SEM magnification made by Hitachi Science Systems were selected as the standard samples for the intercomparison [8, 9].

Grating patterns were fabricated over the entire surface of the (110)Si substrate (4 mm × 4 mm × 0.3 mm). The fabrication procedures for grating patterns were as follows. (1) A thin thermal oxide film was formed on the (110)Si substrate and then the resist film was coated on the oxide film. (2) The resist grating patterns were directly fabricated by laser interferometer lithography. The source was an Ar ion laser ($\lambda = 351$ nm). The pitch size of the grating p was obtained as follows:

$$p = \lambda/(2\sin\theta) \tag{1}$$

where θ is the angle of incidence. After that, a mask of thin thermal oxide film was made using a buffered HF solution. (3) The grating patterns were transferred into (110)Si by anisotropic chemical etching. The fabricated grating patterns have good edge roughness and the cross section of a rectangular wave. These standard samples are

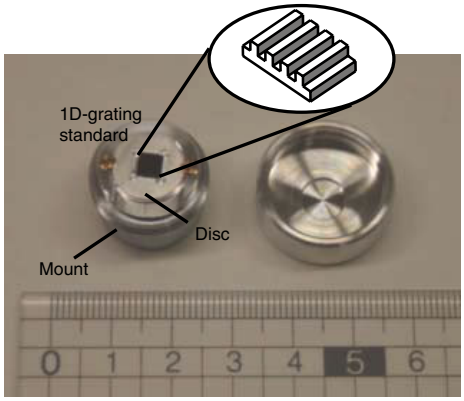

Figure 1. Photograph of a 1D-grating standard, a disc and a mount.

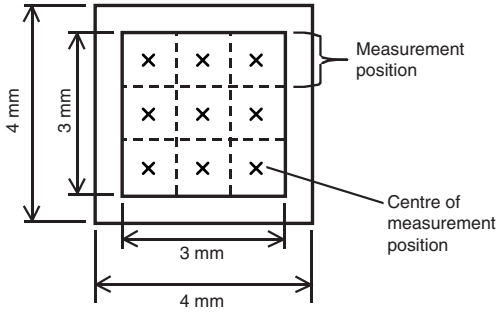

Figure 2. Schematic drawing of the measurement positions. Grating patterns are fabricated over the entire surface. Nine measurement positions are selected, as shown in the figure.

made of silicon single crystals and they are stable in terms of temperature change, humidity change and electron beam irradiation. The formation of a natural oxide layer and the influence of contamination derived from the irradiation of an electron beam are negligible. The contamination effects caused by electron beam are checked by 100% inspection. The inspection procedures are as follows. The sample is irradiated by a high magnification electron beam (magnification 100 000, probe current 10 pA and TV scanning) for 60 s. After that, the contamination is checked using a lower magnification of 60 000. If contamination cannot be found out, the product is accepted. This inspection condition is stricter than normal measurement conditions. Therefore, it can be said that the sample is stable to the electron beam irradiation.

As shown in figure 1, the sample in the intercomparison is mounted on an aluminium disc (the diameter is 12 mm and the thickness is 1.5 mm) for the AFM measurements. The sample is fixed on the aluminium disc using carbon paste with adhesive glue. When the sample is fixed on the disc, the sample is vacuumed through a small hole of the disc in order to be fixed horizontally. At this point, we would like to consider a bimorph effect for the sample and the disc. We assumed the combination of the sample and the disc acts as a composite beam, and calculated the change of pitch caused by a bimorph effect. The calculated length change was approximately 4.4 and 10.4 ppm for the pressure change 100 kPa and the temperature change 2 K. In this calculation, the Young's modulus of aluminium and silicon are 70 and 170 GPa. This aluminium disc is loaded at the top of an aluminium mount (the diameter is 15 mm and the height is approximately 10 mm) for the measurements using an OD and a CD-SEM. The thermal expansion coefficient of silicon [10] is 2.6×10^{-6} K^{-1}.

The nine measurement positions of the samples in the intercomparison have been chosen as shown in figure 2. The measurement area at each position, however, is defined according to the nanometrological instrument since the suitable measurement areas are dependent on the characteristics of the nanometrological instrument. Therefore, the numbers of pitches for the calculation of the average pitch values differ according to the nanometrological instrument. Uncertainty in measurements is expected to become smaller when an instrument capable of a larger numbers of pitches is used. In the intercomparison, it is important that uncertainty in measurements using each nanometrological instrument is estimated reasonably. Therefore, large or small uncertainty does not directly reflect whether a nanometrological instrument functions well or poorly.

2.2. Nanometrological instruments and measurement procedures

Table 1 shows the participants and the order of nanometrological instruments used in the intercomparison, as well as the date. NMIJ/AIST, the pilot laboratory for the intercomparison, performed the measurements at the start and the end of the intercomparison. The first measurement by NMIJ/AIST is denoted as NMIJ/AIST (1st) and the last measurement as NMIJ/AIST (2nd). The measurements in the intercomparison started in October 2000 and finished in December 2001. The intercomparison report was written by NMIJ/AIST and the report was submitted to the participants in March 2002. During the intercomparison, NMIJ/AIST took part in the supplementary key comparison of step height standards (CCL NANO2) using a nanometrological AFM. Therefore, the intercomparison was interrupted from May to September 2001.

2.2.1. Optical diffraction pitch calibration apparatus (OD). JQA carried out the pitch measurements using an optical diffraction pitch calibration apparatus (OD) made by KOHZU Precision Co, Ltd. Figure 3 shows the optical configuration and a photograph of the OD. The OD consists of a He–Cd laser ($\lambda = 325.0$ nm, IK3083R-D (0013), Kimmon Electric Co. Ltd), an XYZ-axis fine-motion table, a rotary table (θ-axis part of the $XYZ\theta$ table, PST-20 (900440 #09), KOHZU Precision Co, Ltd), a laser power monitor (PM-100 (116), Kimmon Electric Co. Ltd) and a controller for the fine-motion table. The apparatus is located in a clean booth in a clean room.

The beam of the He–Cd laser was bent at a mirror, passed through a slit and a half-mirror and irradiated the 1D-grating surface. The zeroth diffracted beam from the 1D-grating surface passed through a half-mirror and irradiated the laser power monitor via two mirrors. First, the position of the detector centre of the laser power monitor was aligned to the incident beam axis. Next, the rotary table was rotated and the reflected beam was directed to the detector of the laser power monitor. The angular position of the rotary table with maximum power of the reflected beam was found and recorded as Z_n. Then the rotary table was rotated clockwise (CW) or

Table 1. Order, participants, instruments and time schedule in the intercomparison. (The intercomparison was interrupted from May 2001 to September 2001 due to NMIJ/AIST's participation in other supplementary key comparisons of step height (CCL NANO2).)

Order	Participant	Instrument	Date
1	NMIJ/AIST(1st)	Nanometrological AFM	Oct.–Dec. 2000
2	JQA	OD	Jan.–Feb. 2001
3	HSS	CD-SEM	Mar.–Apr. 2001
4	NMIJ/AIST(2nd)	Nanometrological AFM	Oct.–Dec. 2001

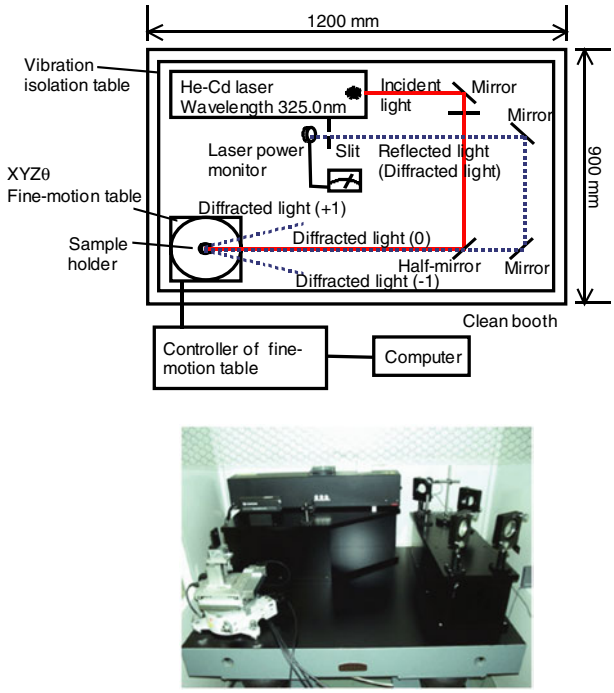

Figure 3. Optical diffraction pitch calibration apparatus JQA.

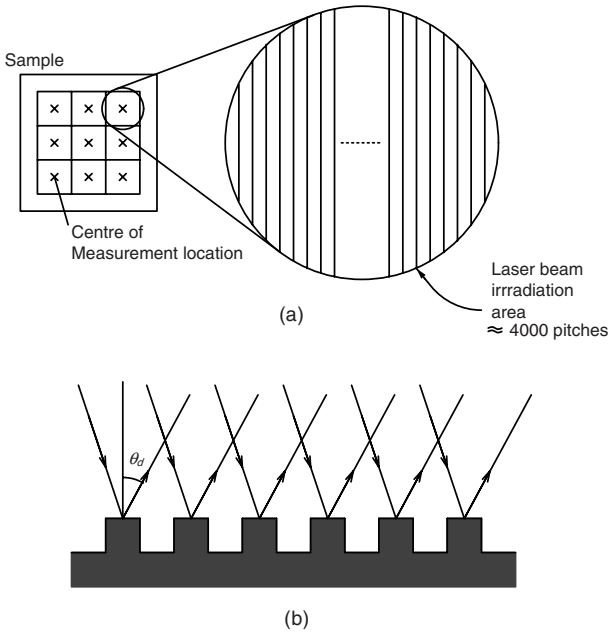

Figure 4. Schematic drawing of pitch measurement using an optical diffraction pitch calibration apparatus. (a) Laser beam irradiation area. Approximately 4000 pitches are included in the area. (b) Diffraction of laser beam. Average pitch values are calculated using diffraction angle and laser wavelength.

counterclockwise (CCW) so that the +1st or −1st diffracted beam was aligned with the detector, and the angle of the rotary table was recorded as P_n or N_n, respectively.

Measurements were repeated twice at each side of the +1st P_n (CW) and the −1st N_n (CCW) for nine measurement positions (figure 4) of the 1D-grating standards as follows:

$$Z_1 \to P_1 \to Z_2 \to P_2 \to Z_3 \to N_1 \to Z_4 \to N_2 \to Z_5.$$

At this point, the diffraction angle θ_d was simply calculated from Z_n–P_n. Furthermore, when the interval of measurements, the arrows in the above flow of measurement, is the same and the least-squares method can be applied to the calculation, the linear time drift derived with the passage of time can be eliminated and the diffraction angle θ_d can be obtained precisely. The difference between Z_n and P_n was taken as the partial regression coefficient and the diffraction angle was obtained using

$$\theta_d = \tfrac{1}{2}(\theta_p + \theta_N) = \tfrac{1}{8}(-Z_1 + 2P_1 - 2Z_2 + 2P_2 - 2N_1 + 2Z_4 - 2N_2 + Z_5). \quad (2)$$

In order to check the bias of results obtained at the +1st side (CW) and the −1st side (CCW), the sample was turned over vertically and the same set of measurements was carried out. Four sets of measurements were performed under different conditions, for example the date and setting of the sample.

The average pitch was calculated from the diffraction angles θ_d at the four set of measurements using

$$p = \frac{n\lambda}{2\sin\theta_d \cdot \cos\phi \cdot \cos\frac{\varphi}{2}}(1-\alpha(20-t))^{-1} \approx \frac{n\lambda}{2\sin\theta_d \cdot \cos\phi \cdot \cos\frac{\varphi}{2}}(1+\alpha(20-t)), \quad (3)$$

where p is the average pitch value, λ the laser wavelength, n the diffraction order, θ_d the rotation angle of the rotary table when the Bragg condition is satisfied, φ the horizontal compensation angle, ϕ the vertical compensation angle, α the thermal expansion coefficient and t the temperature of the 1D-grating standards during the measurements in degrees Celsius. The precise sizes of the beam irradiation areas were not determined. However, if the beam irradiation area is assumed to be 1 mm^2, as shown in figure 4, the number of grooves in the area would be approximately 4000, over which the pitch is averaged.

2.2.2. CD-SEM. Hitachi Science Systems (HSS) performed the measurement of pitch using a critical dimension scanning electron microscope (CD-SEM). Figure 5 shows a photograph

Figure 5. Hitachi CD-SEM (S-9220).

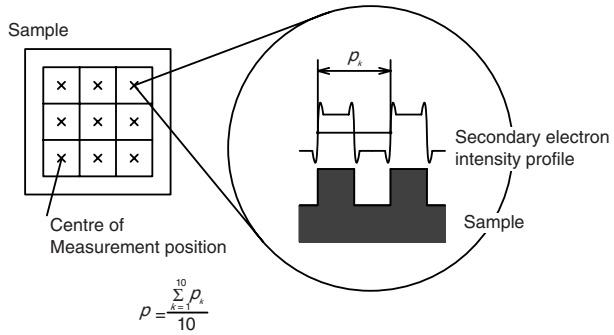

Figure 6. Schematic drawing of pitch measurement using a CD-SEM. Pitch is calculated from a secondary electron intensity profile.

of a CD-SEM[7]. The CD-SEM is a 200 mm diameter wafer-loaded type and its magnification range is 1000–300 000 times. The maximum measurable range is 2 μm.

The measurement procedures using a CD-SEM were as follows. First, the magnification of the electron beam was calibrated using a standard microscale. The microscale was calibrated by JQA, and the calibrated pitch and the expanded uncertainty were 240.2 and 1.0 nm, respectively. Twenty points on the 1D-grating standard for magnification calibration were measured. The average pitch was used to calculate the calibration parameter. The standard deviation was used to estimate the standard uncertainty of the magnification calibration. Next, a primary electron beam was scanned on the surface of the 1D-grating standard, the object of the measurement, and a secondary electron intensity image was obtained. Acceleration voltage was 800 V, probe current was 8 pA, magnification was 100 000 and the number of integrated frames in the TV scan mode was 16. Pitch was calculated using the obtained secondary electron intensity signal, as shown in figure 6. The total number of pitches at any measurement position was ten. Ten images were taken at each measurement position. The obtained average pitch represents the pitch at the measurement position.

2.2.3. Nanometrological AFM. NMIJ/AIST carried out pitch measurements of the 1D-grating standards using an AFM with laser interferometers, the nanometrological AFM. Precise information about the nanometrological AFM and the pitch measurements have been described in detail elsewhere [1, 2]. The laser interferometer unit is shown in figure 7. Interferometer units are aligned to each surface of a three-sided mirror that is used as both the target mirror of the interferometers and the sample scanner. The laser interferometer unit is a homodyne interferometer with four paths and the system resolution reaches approximately 0.04 nm by interpolating the fringe electrically. Laser sources are frequency-stabilized He–Ne lasers which were calibrated by an I_2-stabilized He–Ne laser. Therefore, the nanometrological AFM is traceable to the length standard of NMIJ/AIST. The scanning stroke of the stage is 17.5 μm (X) × 17.5 μm (Y) × 2.5 μm (Z). The atomic force is detected by an optical lever method in contact mode with a microcantilever probe spring constant of 0.01 N m^{-1} (Veeco Instruments Inc., formerly Thermomicroscopes, MSCT-AUNM).

[7] Hitachi CD-SEM catalogue.

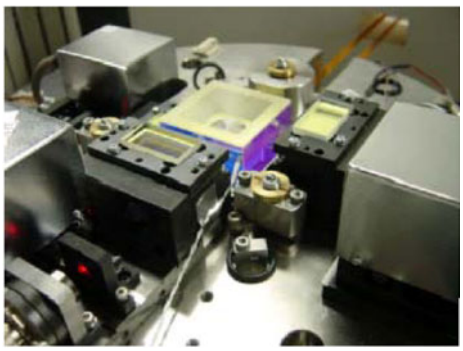

Figure 7. Laser interferometer unit of a nanometrological AFM.

The measurement procedures were as follows. The probe was moved to the centre of each of the nine measurement positions and scanned an area as large as 10 μm (X) × 10 μm (Y) for NMIJ/AIST (1st) and 5 μm (X) × 5 μm (Y) for NMIJ/AIST (2nd), as shown in figure 8. The captured image had 32 line profiles. Twenty out of 32 line profiles were used for the calculation of pitch values. The total number of peaks used for the calculation was approximately 880 for NMIJ/AIST (1st) and approximately 440 for NMIJ/AIST (2nd). The pitch in the measurement area was the calculated average pitch.

2.3. Analytical procedures

2.3.1. Uncertainty in measurements. All participants determined the average pitch of three samples and evaluated the expanded uncertainty based on GUM [11]. The listed components in an uncertainty budget are characteristic of each measurement method. Examples of uncertainty components are as follows: repeatability of measurements, calibration error of scale in the instrument, compensation error of the refractive index of air, Abbe error, cosine error and thermal expansion of the sample.

2.3.2. Statistical test of reference value in the intercomparison. Since NMIJ/AIST's method is directly traceable to the national standard of length, the pitch and the expanded uncertainty of each sample obtained by NMIJ/AIST were defined as the reference value x_{ref} and the expanded uncertainty of

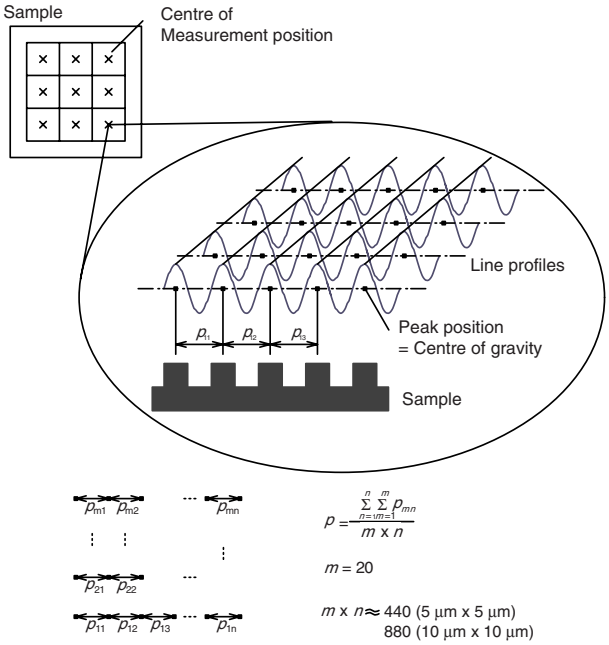

Figure 8. Schematic drawing of pitch measurement using a nanometrological AFM. Pitch is calculated from the distance between peak positions (centre of gravity).

the reference value $U(x_{\mathrm{ref}})$ in the intercomparison. During the intercomparison, NMIJ/AIST carried out two sets of measurements, one each at the start and the end of the intercomparison. Therefore, it is necessary to confirm that there is no statistically significant difference between the results of NMIJ/AIST (1st) and those of NMIJ/AIST (2nd). The test procedures are as follows [12]. (1) Using the variance ratio test (F-test), the similarity of the two population variances is examined. (2) The mean value difference test (t-test) reveals whether or not the difference between the mean values of two measurands with the same variance is statistically significant.

(1) *Variance ratio test* (*F-test*). The observation in NMIJ/AIST (1st) and that in NMIJ/AIST (2nd) are defined as x and y, respectively. It is assumed that the parent populations of observations x and y were normal distributions with variances σ_x^2 and σ_y^2, respectively. At that point, samples with the number n_x and n_y are obtained from the populations of observations x and y, and their sample variances are defined as s_x^2 and s_y^2. The equation,

$$\frac{n_x s_x^2}{(n_x-1)\sigma_x^2} \Big/ \frac{n_y s_y^2}{(n_y-1)\sigma_y^2}, \tag{4}$$

exhibits F distribution with $n_x - 1$ and $n_y - 1$ degrees of freedom. When the null hypothesis $\sigma_x^2 = \sigma_y^2$ is substituted into the equation (4), the variance ratio F,

$$F = \frac{n_x s_x^2}{n_x-1} \Big/ \frac{n_y s_y^2}{n_y-1} = \frac{\sum(x-\bar{x})^2}{n_x-1} \Big/ \frac{\sum(y-\bar{y})^2}{n_y-1} \tag{5}$$

becomes a statistical value. The statistical significance of the difference between s_x^2 and s_y^2 can be tested by the F distribution with measured values as the test statistics. When it is assumed that the null hypothesis is H_0: $\sigma_x^2 = \sigma_y^2$, the alternate hypothesis is H_1: $\sigma_x^2 \neq \sigma_y^2$, and the significant level is α, the rejection region of the F-test is obtained as follows:

$$\begin{aligned} &H_1\text{: If } \sigma_x^2 > \sigma_y^2, \qquad \text{then } F > F_{\alpha/2} \\ &\quad \text{(degrees of freedom } n_x - 1, n_y - 1) \\ &H_1\text{: If } \sigma_x^2 < \sigma_y^2, \qquad \text{then } 1/F > F_{\alpha/2} \\ &\quad \text{(degrees of freedom } n_x - 1, n_y - 1). \end{aligned} \tag{6}$$

Here the critical value F_{c} is obtained from the F table at the probability $\alpha/2$.

(2) *Mean value difference test* (*t-test*). The populations of observations x and y are assumed to have normal distributions with mean value and variance (μ_x, σ^2) and (μ_y, σ^2), respectively. The averages of observations x and y with sampling numbers n_x and n_y are defined as $\bar{x}$ and $\bar{y}$, respectively. Since variances of both x and y are the same, an unbiased estimate of variance is obtained as

$$\hat{\sigma}^2 = \frac{n_x s_x^2 + n_y s_y^2}{n_x + n_y - 2}, \tag{7}$$

and the following statistics value,

$$t = \frac{(\bar{x}-\bar{y}) - (\mu_x - \mu_y)}{\hat{\sigma}\sqrt{(1/n_x) + (1/n_y)}}, \tag{8}$$

has t distribution with $n_x + n_y - 2$ degrees of freedom. The significance test of the mean value difference can be used, based on the above relations. When it is assumed that the null hypothesis is H_0: $\mu_x = \mu_y$, the alternate hypothesis is H_1: $\mu_x^2 \neq \mu_y^2$, and the significant level is α, t can be tested as follows:

$$\begin{aligned} &\text{If } |t_0| > t_\alpha, \qquad \text{null hypothesis } H_0 \text{ is rejected} \\ &\quad \text{(the rejection region)} \\ &\text{If } |t_0| \leqslant t_\alpha, \qquad \text{null hypothesis } H_0 \text{ is accepted} \\ &\quad \text{(the acceptance region).} \end{aligned} \tag{9}$$

Here the critical value t_α is obtained from the t table at the probability $\alpha/2$.

2.3.3. Evaluation method for the consistency of pitch measurement results. According to ISO/IEC GUIDE 43-1 [13], the calculation of the E_n number is one of methods of checking whether a measured value x_i and its expanded uncertainty $U(x_i)$ are consistent with a reference value x_{ref} and its expanded uncertainty $U(x_{\mathrm{ref}})$. The E_n number is expressed as follows [13]:

$$E_n(x_i) = \left| \frac{x_i - x_{\mathrm{ref}}}{\sqrt{U^2(x_i) + U^2(x_{\mathrm{ref}})}} \right|. \tag{10}$$

If the E_n number is less than or equal to 1, the consistency of measurements is assured. If the expanded uncertainty $U(x_i)$ is too small and/or the difference between the measured value x_i and the reference value x_{ref} is too large, the E_n number exceeds 1 and the equality of measured values cannot be assured.

Table 2. Mean values $\bar{x}$, $\bar{y}$, standard deviations s_x, s_y and degrees of freedom $n_x - 1$, $n_y - 1$ obtained using the nanometrological AFM.

Sample	Observed value	Mean value $\bar{x}$, $\bar{y}$ (nm)	Standard deviation s_x, s_y (nm)	Degree of freedom $n_x - 1$, $n_y - 1$
T002	x	239.97	0.13	12.6
	y	240.03	0.16	53.0
T005	x	239.95	0.17	20.1
	y	239.90	0.14	90.4
T006	x	239.97	0.13	11.1
	y	239.94	0.15	54.9

Table 3. The values of F, $1/F$ and $F_{\alpha/2}$ used for the F-test.

Sample	F	$1/F$	$F_{\alpha/2}$
T002	1.400	0.714	2.200
T005	1.471	0.680	1.864
T006	1.436	0.696	2.242

Table 4. The values of $\hat{\sigma}^2$, t_0 and t_α used for the t-test.

Sample	$\hat{\sigma}^2$	t_0	t_α
T002	0.023	−1.314	2.000
T005	0.022	1.407	2.000
T006	0.022	0.644	2.000

3. Results

3.1. Reference value

The observations of NMIJ/AIST (1st) and NMIJ/AIST (2nd) are x and y, respectively. The obtained mean values $\bar{x}$ and $\bar{y}$, standard deviations s_x and s_y, and degrees of freedom $n_x - 1$ and $n_y - 1$ are shown in table 2. The values F, $1/F$ and $F_{\alpha/2}$ used in the F-test are shown in table 3 where α is set as 5%, which means that the reliability range is 95%. As shown in the table, the following conditions were satisfied for all samples:

$$F < F_{\alpha/2}, \qquad 1/F < F_{\alpha/2}. \tag{11}$$

According to the rejection region (6), the difference between the sample variances s_x^2 and s_y^2 is considered to be accidental and for the population variances σ_x^2 and σ_y^2, therefore,

$$\sigma_x^2 = \sigma_y^2. \tag{12}$$

Next, the results of the t-test for the mean value difference between the parent populations of observations x and y are explained. The values $\hat{\sigma}^2$, t_0 and t used in the t-test are shown in table 4 where $P = \alpha/2 = 0.025$ and $\upsilon = 60$. For samples T002, T005 and T006, the following equation was satisfied:

$$|t_0| \leqslant t_\alpha. \tag{13}$$

Therefore, the null hypothesis

$$H_0\colon \mu_x = \mu_y \tag{14}$$

was accepted. In other words, the mean values μ_x and μ_y of population variances of observations x and y are the same.

The results of the F-test and the t-test indicated that there were no significant differences in the variances and the mean values of observations x and y. In other words, it could be concluded that the results of NMIJ/AIST (1st) and those of NMIJ/AIST (2nd) were obtained from the same parent population. Thus, it is reasonable for the results of NMIJ/AIST (1st) or those of NMIJ/AIST (2nd) to be the reference values in the intercomparison. Here we set the results of NMIJ/AIST (2nd) as the reference values in the intercomparison, where these reference values were assured by the results of NMIJ/AIST (1st). Table 5 shows the reference values x_{ref} and the expanded uncertainties $U(x_{\mathrm{ref}})$ ($k = 2$).

Table 5. Reference value and expanded uncertainty ($k = 2$).

Sample	Reference value x_{ref} (nm)	Expanded uncertainty $U(x_{\mathrm{ref}})$ (nm) ($k = 2$)
T002	240.03	0.310
T005	239.90	0.282
T006	239.94	0.302

3.2. Results of intercomparison measurements

The measurement results reported by the participants are shown in table 6 and figure 9. In figure 9, the marker indicates the average pitch p, and the error bar shows the expanded uncertainty $U(x_i)$ ($k = 2$). The expanded uncertainties $U(x_i)$ ($k = 2$) were a maximum of 0.14 nm for the OD, 1.2 nm for the CD-SEM and 0.35 nm for the nanometrological AFM. The uncertainties of the three samples were almost the same when the same instrument was used. The differences between the maximum and the minimum pitch measured by the three methods for each sample were in the range of 0.38–0.67 nm, and were enough small compared with the nominal pitch of 240 nm.

Here, the pitch is the average pitch in one measurement position, and it can be predicted that the uncertainty caused by random errors would decrease if the measurement area were to increase. In one measurement position, the measurement area was approximately 1 mm^2 for the OD, less than 1 μm^2 for the CD-SEM and 25–100 μm^2 for the nanometrological AFM. In this intercomparison, the uncertainty for the CD-SEM became the maximum as predicted. On the other hand, the uncertainty of the OD was the minimum and the uncertainty of the nanometrological AFM was intermediate. Similar results were obtained in the supplementary key comparison (CCL NANO4) and the expanded uncertainty of the OD was smaller than the uncertainty of SPM [3].

Ratios of the expanded uncertainty to the pitch $U(x)/p$ were 0.06% for the OD, 0.50% for the CD-SEM and 0.15% for the nanometrological AFM, respectively, and all uncertainties were small. Furthermore, E_n numbers for the OD and CD-SEM were less than 1 and consistency with the reference value was confirmed.

3.3. Major uncertainty components

Table 7 shows the major uncertainty components in pitch measurements in the intercomparison. Major components for the OD were repeatability of the rotary table, detectability of the laser power monitor observations and vertical angle correction under the Bragg condition. Since the rotary table

Table 6. Pitch p, expanded uncertainty $U(x_i)$ and E_n number (samples T002, T005, T006).

Participant (instrument)	Pitch p (nm)	Expanded uncertainty $U(x_i)$ $(k = 2)$ (nm)	Ratio of expanded uncertainty to pitch $U(x_i)/p$ (%)	E_n number
		Sample T002		
NMIJ/AIST (1st) (nanometrological AFM)	239.97	0.262	0.109	—
JQA (OD)	239.92	0.132	0.055	0.33
HSS (CD-SEM)	240.3	1.0	0.416	0.26
NMIJ/AIST (2nd) (nanometrological AFM)	240.03	0.310	0.129	—
		Sample T005		
NMIJ/AIST (1st) (nanometrological AFM)	239.95	0.342	0.143	—
JQA (OD)	239.93	0.132	0.055	0.10
HSS (CD-SEM)	240.5	1.2	0.499	0.49
NMIJ/AIST (2nd) (nanometrological AFM)	239.90	0.282	0.118	—
		Sample T006		
NMIJ/AIST (1st) (nanometrological AFM)	239.97	0.252	0.105	—
JQA (OD)	239.93	0.132	0.055	0.03
HSS (CD-SEM)	239.3	1.0	0.418	0.61
NMIJ/AIST (2nd) (nanometrological AFM)	239.94	0.302	0.126	—

Table 7. Major uncertainty components in pitch measurements.

Participant	Instrument	Major uncertainty components	Standard uncertainty $U_c(x_i)$ (nm)
JQA	Diffractometer	Repeatability of rotary table	0.0470
		Detectability of the laser power monitor	0.0366
		Vertical angle correction under Bragg condition	0.0240
HSS	CD-SEM	Magnification calibration	0.5
		Standard sample for magnification calibration	0.3
		Repeatability of measurements	0.2
NMIJ/AIST (1st)	Nanometrological AFM	Laser interferometer nonlinearity	0.115
		Nonuniformity of sample	0.041
		Repeatability of measurements	0.032

has not been calibrated using an angle standard [14], the uncertainty of repeatability of the rotary table may increase. The expanded uncertainty for the OD is already quite small but it can be reduced still further by calibrating the rotary table and compensating the bias error.

The major uncertainty components in pitch measurements using the CD-SEM were magnification calibration, standard sample for magnification calibration and repeatability of measurements. The uncertainty of magnification calibration may decrease if the number of measurement positions increases.

The major uncertainty components in pitch measurements using the nanometrological AFM were laser interferometer nonlinearity, nonuniformity of sample and repeatability of measurements [2]. Standard uncertainty of repeatability of measurements means the standard deviation of average pitch values obtained in three measurements at the same position. Laser interferometer nonlinearity means cyclic error, when the interferometer signals are interpolated at a high resolution. The interpolation includes offset correction and gain adjustment of the original interferometer signals. However, the correction of phase shift, from 90° as the phase difference between the two interferometer signals, has not yet been carried out. This correction may reduce the interferometer nonlinearity.

The uncertainty of the nonuniformity of the sample was small (0.017%) compared to the pitch itself (240 nm). It indicates that the quality of samples used in the intercomparison is good enough to confirm the suitability of the samples as standards.

Through the participants' estimations of the uncertainty in measurements, the major uncertainty components were revealed to be as described above. Participants can therefore realize ways of improving their instruments and measurement techniques. It can be concluded that the estimation of uncertainty in measurements is useful for participants.

4. Discussion

The key issue of the intercomparison measurements is the differences in calculation methods of the average pitch in one

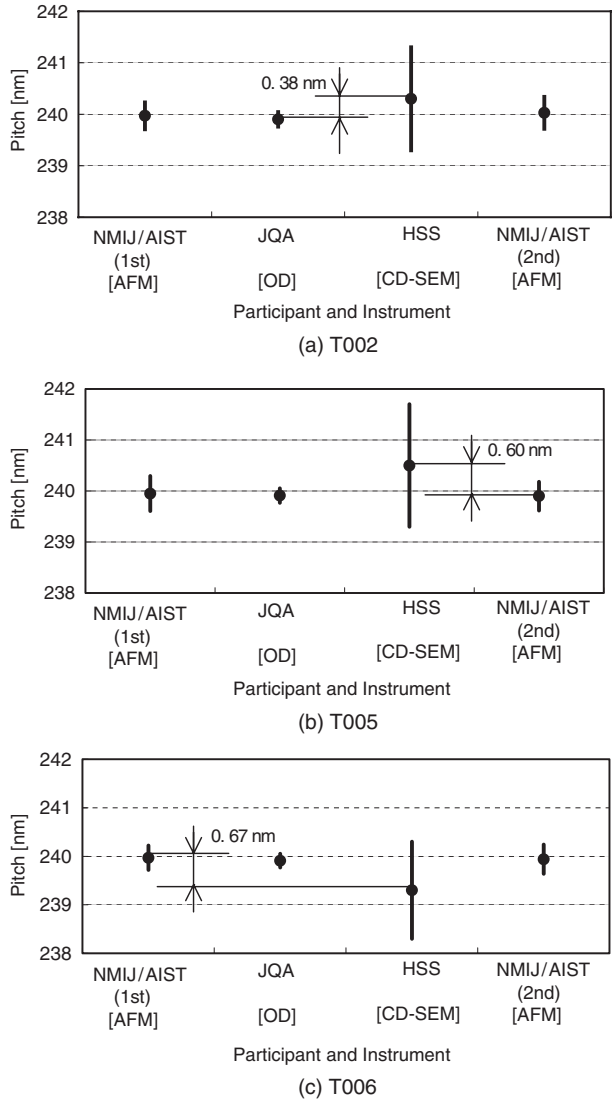

Figure 9. Pitch and expanded uncertainty ($k = 2$).

measurement position between participants. The instruments used in the intercomparison are different and the numbers of pitches in one measurement at one measurement position vary, from ten to several thousand. It is therefore considered that the expanded uncertainty of the average pitch decreases in inverse proportion to the number of pitches. In the actual estimation of uncertainty, the expanded uncertainty for the OD was the smallest of the three methods and the number of pitches included in the diameter of the beam spot was approximately 4000. The expanded uncertainty for the CD-SEM was the largest and the number of pitches in one measurement position was ten. However, the E_n numbers for the OD and CD-SEM were less than 1 and the equality of the measurements to the reference value was assured. It is worth noting that the consistency of the measurement results was assured despite the use of different instruments.

The optical diffraction pitch calibration apparatus (OD) can be constructed relatively easily. It has the merit that the average pitch can be directly obtained in a short time from measurements of the diffraction angle and laser wavelength. However, in the case of the OD used in this intercomparison, the laser wavelength and the angle measurement are not directly traceable to the length standard and the angle standard [14], respectively. Especially considering the traceability to the length standard, there are several calibration methods of the He–Cd laser by the I_2-stabilized He–Ne laser. One of calibration methods is as follows. An interferometer system using both the He–Cd laser and the I_2-stabilized He–Ne laser as light sources is constructed and a calibrated gauge block is measured using the interferometer. Therefore, the He–Cd laser can be calibrated by the I_2-stabilized He–Ne laser through the gauge block. The other calibration method can be considered as follows. A grating sample, more than 317 nm pitch, is measured using the OD system where both the He–Cd laser and the I_2-stabilized He–Ne laser are used. From the measurement results obtained by the I_2-stabilized He–Ne laser, the wavelength of the He–Cd laser can be calibrated. However, these two methods need new instruments and the standard uncertainty caused by the He–Cd laser calibration may not be small. Therefore, in order to realize the traceability of the calibrated pitch without installing new instruments, the OD should be calibrated using the 1D-grating standard calibrated by the nanometrological AFM.

The critical dimension scanning electron microscope (CD-SEM) is widely used for the control of semiconductor manufacturing processes, and the nonlinearity in the scanning range of the CD-SEM and the magnification calibration have been evaluated using the 1D-grating calibrated using the OD. In the intercomparison, it cannot be said that the measurement using the CD-SEM is strictly independent of that using the OD. However, similar situations arose in the supplementary key comparison [3] and the participants involved were not prevented from taking part in the intercomparison. The CD-SEM used in the intercomparison achieves a high accuracy level in practice. Furthermore, in order to realize direct traceability to the standard, a CD-SEM with a directly length-standard-traceable scale can be used [15].

The nanometrological AFM has the length-standard-traceable laser interferometer in the XYZ-axes. Furthermore, the equality of measurement to those at other NMIs was assured through the supplementary key comparison of the 1D-grating (CCL NANO4). Therefore, the measurement results obtained using the nanometrological AFM are suitable for use as reference values in the intercomparison. When the average pitch value in one measurement position was calculated, it was assumed that the approximately 880 (NMIJ/AIST (1st)) or 440 (NMIJ/AIST (2nd)) pitches in one measurement position were completely independent of each other. However, it can be considered that the pitches are not completely independent of each other under certain conditions of the distance between neighbouring line profiles or the spatial frequency of edge roughness. More precise analyses of pitch value calculation will be necessary in the future.

5. Conclusions

In this study, intercomparison measurements of 1D-grating standards were carried out in accordance with the technical protocol, using the optical diffraction pitch calibration apparatus (OD), the critical dimension scanning electron

microscope (CD-SEM) and the nanometrological AFM. The difference between the measured values obtained using fundamentally different instruments was less than 0.67 nm for three samples with a nominal pitch of 240 nm. The expanded uncertainties were less than 0.14 nm for the OD, 1.2 nm for the CD-SEM and 0.35 nm for the nanometrological AFM. E_n numbers, which indicate the degree of consistency in pitch measurements, were less than 0.33 for the OD and 0.61 for the CD-SEM in the case of reference values based on NMIJ/AIST's results. The results of measurements and the uncertainty estimation assured the consistency of obtained pitch values and expanded uncertainties despite the use of different instruments. Thus we confirmed metrological equivalence. Furthermore, it was revealed that the 1D-grating standards are suitable for use as the reference standards in the intercomparison using the above three nanometrological instruments.

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