ABSTRACT
Nanometrology, which is dimensional metrology on the nanometer scale, is rapidly gaining importance. In Nanometrology, length standard traceable measurements are required to certify the values obtained from measurements. Standard Reference Material (SRM) is a sample for calibrating the magnification scale of a Scanning Electron Microscope (SEM). The standard are produced by recording a precision, laser-generated, interference pattern using photosensitive materials and other processing steps. This fabrication technique provides an accurate measure of the period of the line-space. In this paper, we describe initial characterization of 1D grating standard with nominal 300 nm and 700 nm using SEM and its measurement uncertainty. The measurements were performed at 10 different measurement position at each nominal. Total number of pitch at all measurement position in each nominal was 180. An image was taken at each measurement position. The average pitch of them represents the pitch value of the specimens. The results show measured pitch values for nominal 300 nm and 700 nm are (293.6±4.4) nm and (700.3±5.5), respectively.

Keywords: calibration; characterization; nanometrology; pitch measurement; SEM; uncertainty; 1D grating.

Kata Kunci: kalibrasi; karakterisasi; nanometrologi; pengukuran pitch; SEM; ketidakpastian; kisi 1D.

I. INTRODUCTION

The Increase in the number of nanotechnology products are getting more and more in the commercial market. According to statistics on Nanotechnology Products Database (NPD), the USA and China are the largest producers especially in carbon nanotubes (CNT). Around 131 of 6432 nanotechnology products have used CNT in their structures to create or to enhance specific properties. Japanese companies are the major consumers (44%) of CNTs in commercial products. UK, China, the USA, and Poland follow the Japanese by having a share of 25%, 11%, 11%, and 4%, respectively. (Database, 2017)

Based on the demand of nanotechnology products, establishment of a quality assurance system in this field has become an important task. To fulfil this task, several kinds of research activities should be carried out. First, development of measuring instruments, for a past decade a number of national metrology institutes (NMIs) have developed measuring instruments for calibration of nanometrological standards such as a calibrated atomic force microscope (CAFM) at National Institute of Standards and Technology (NIST) (Schneir, McWaid, Alexander, & Wilfley, 1994), a long-range AFM profiler at Federal Institute of Metrology (METAS) (Meli & Thalmann, 1998), a metrological scanning force microscope at Physikalisch-Technische
Bundesanstalt (PTB) (Bienias, Gao, Hasche, Seemann, & Thiele, 1998), an atomic force microscope and a surface texture measuring instrument with traceable metrology at National Physical Laboratory (NPL) (Leach et al., 2001), and ‘nanometrological AFM’ system with a ultra-high resolution three-axis laser interferometer at National Metrology Institute of Japan (NMIJ) (Gonda et al., 1999a, 1999b). Second, development and investigation of calibration standards, the development of one-dimensional (1D) grating standard has become essential to guarantee the traceability of nanometrological instruments. Several kinds of method in fabrication of nanometric lateral scales using different technique, materials and also the investigation of its performance have been done by NMIJ (I Misumi et al., 2006; Ichiko Misumi et al., 2008; Ichiko Misumi, Gonda, Sato, Sugawara, et al., 2007; Ichiko Misumi, Gonda, Sato, Yasutake, et al., 2007). Third, development of data evaluation methods and the uncertainty of measurement. PTB has reported the measurement strategies using two methods, namely a gravity centre method and a fourier transform method. Both of them can be applicable in 1D grating measurements (Dai, Koenders, Pohlenz, Dziomba, & Danzebrink, 2005). Furthermore, the uncertainty in pitch measurements of 1D grating standard and step height has been reported by NMIJ using a nanometrological AFM (Ichiko Misumi, Gonda, Kurosawa, & Azuma, 2006; Ichiko Misumi, Gonda, Kurosawa, & Takamasu, 2003).

In order to check metrological equivalence between NMIs, various international key comparisons have been performed in different classes. They started in different times. The last one, supplementary comparison APML.L-K5 nano particles measurements, which started in 2012 is still on progress (BIPM, CMS-ITRI, & NMIJ, 2017). The developed economies in Asia, Europe and America use measuring instruments such as scanning electron microscope (SEM), interference microscope, optical diffraction technique, scanning probe microscope, atomic force microscope and other stylus instruments (Bosse & Wilkening, 2005; Danzebrink et al., 2010; Decker et al., 2009; Garnaes & Dirscherl, 2008; Koenders et al., 2003; Koenders, Klapetek, Meli, & Picotto, 2006; Ichiko Misumi, Dai, & Peng, 2007).

In Indonesia, the most measuring instrument used for inspection of fabricated nanotechnology products is
SEM. However, the main issue in nanotechnology instruments in Indonesia is the traceability of measurement that has not been fulfilled. In order to solve this problem, Research Center for Metrology – Indonesian Institute of Sciences (RCM – LIPI) purchased two specimens of 1D grating standards with the nominal values of 300 nm and 700 nm which are traceable to the SI through Center of Measurement Standard – Industrial Technology Research Institute of Taiwan (CMS – ITRI) against a laser diffractometer standard. These specimens were used as the artifact for comparison of national laboratories to measure pitch value of 1D grating standards using SEM. There were six nanotechnology laboratories participated i.e., Research Center for Physics – Indonesian Institute of Sciences, Laboratory for Polymer Technology – Agency for The Assessment and Application of Technology (BTP – BPPT), Center for Science and Advanced Material Technology – National Nuclear Energy Agency of Indonesia (PSTBM – BATAN), Surya Institute and Faculty of Mathematics and Natural Sciences – State University of Jakarta (FMIPA – UNJ). Before the activity was held, RCM – LIPI in collaboration with Research Center for Physics – LIPI were carried out the initial characterization of 1D grating standards with nominal values of 300 nm and 700 nm by using SEM. In this study, firstly the specifications of the 1D grating standard, the measurement positions and the suitability of the artifact are discussed. And lastly, the measurement result and the evaluation of uncertainty based on JCGM 100:2008 “Evaluation of measurement data — Guide to the expression of uncertainty in measurement” are described in detail (JCGM, 2008).

II. THEORITICAL BACKGROUND

A. 1D Grating Standards

Nanoscale 1D grating standards have been developed by Electron Microscopy Sciences (EMS) with nominal values of 300 nm and 700 nm. The specimens were mounted with conductive carbon tabs onto steel disks ∅25 mm and thickness 5.9 mm. The specimen with the nominal pitch of 300 nm consists of silicon wafer substrate with titanium ridge (line-space pattern) on the surface. The dimensions of this specimen is 4 mm × 3 mm with a height of ridge about 30 to 40 nm. The pattern on this specimen consists of a number of lines perpendicular to the long axis (x-axis) as shown in Fig. 1.
Fig. 1. Photograph of a set of two specimens (nominal values of 300 nm and 700 nm) and schematic pattern of lines on the specimens

The other specimen with the nominal pitch of 700 nm is fabricated also using silicon wafer substrate but overcoated with a tungsten materials. The dimension of this specimen is also 3 mm × 4 mm. However, unlike the 301BE one, the pattern on the 701CE specimen is parallel to the long axis (y-axis) as shown in Fig. 1. Details of technical specifications of each specimen is shown in Table 1.

The pitch value \((a_i)\) is taken to be the distance between two neighboring peak positions. Approximately 180 pitch values \((a_1...a_{180})\) are obtained and the average of them \((\bar{p})\) represents the pitch value of the specimens. The mathematical model of pitch measurement is expressed as follows.

\[
\bar{p} = \frac{\sum_{i=1}^{180} a_i}{180}
\]

(1)

The uncertainty in the pitch measurements was evaluated based on JCGM 100:2008 “Evaluation of measurement data — Guide to the expression of uncertainty in measurement” (JCGM, 2008). The combined standard uncertainty \(u_c(\bar{p})\) is the square root of the sum of squares of each standard uncertainty \(u_j\) and sensitivity coefficient \(c_j\). It is expressed as

\[
u_c(\bar{p}) = \sqrt{\sum_{j=1}^{10} c_j^2 \times u_j^2}
\]

(2)
Details of the methods for evaluating source of uncertainty measurement are provided as follows:

1) Methods

a) Targeting error \((u_1)\). The targeting error was strongly influenced by the ability of the personnel and the methods used. It has been estimated by taking the standard deviation of pitch values between two neighboring peak positions in the y-axis. The distribution of this standard uncertainty is normal.

b) Cosine error \((u_2)\). Source of uncertainty caused by the angular error of SEM image acquisition, where the structure of line pattern is not ideally perpendicular to the x-axis. Cosine error can be measured by equation \(E = L(\cos \theta - 1)\) with rectangular distribution.

2) Instruments

a) Resolution \((u_3)\). The resolution of the instrument indicated in the manufacturer’s specifications. For Hitachi SEM, the resolution in Secondary Electron (SE) method is 3 nm at 30kV high vacuum mode and Back Scattering Electron (BSE) method is 4 nm at 30kV high vacuum mode.

b) Magnification level \((u_4)\). Magnification level is a source of uncertainty resulting from the influence of different magnification of SEM used to obtain the pitch value. The measurement was performed at two different magnifications, i.e. 9,000 \((x9k)\) and 19,000 \((x19k)\) with 20kV of acceleration voltage. Measurement at each magnification took 180 pitch data. The standard uncertainty of magnification level was obtained from standard deviation of 360 pitch values with rectangular distribution.

c) Calibration error of scale in the instrument \((u_5)\). Standard uncertainty from dimensional calibration certificate of SEM.

d) Repeatability \((u_6)\). The repetitiveness of the measurements is one of the major sources of uncertainty. The standard uncertainty of repeatability derived from standard deviation of measuring pitch in the same position for 10 times.

3) Environments

a) Fluctuation of temperature \((u_7)\). The source of uncertainty derived from the change of temperature is estimated as being more than 0.1 nm with thermal expansion of silicon at \(2.6 \times 10^{-6} \text{ K}^{-1}\) as measured by NMIJ/AIST (Ichiko Misumi et al., 2003).
b) **Noise (u₈).** Noise is a source of uncertainty comes from other sources such as local charging variation that affects the level of contrast of the image generated by SEM. This effect can be estimated as being not more than 0.1 nm with rectangular distribution.

4) Specimens

a) **Non-uniformity (u₉).** The non-uniformity of specimen is a source of uncertainty caused by the manufacturing process, where the value of pitch varies in whole areas on the specimen. The standard uncertainty was obtained from the standard deviation of mean of pitch values in 10 different areas of the specimen.

b) **Sample tilt (u₁₀).** Error of tilt due to misalignment during setting up the measurement will darken the image or make it not clearly visible. This effect can be estimated as being not more than 0.1 nm with rectangular distribution.

**B. Line Edge Roughness (LER)**

The term ‘LER’ is generally understood to mean the fluctuation of edges along patterned lines, such as gate lines, or nanowire channel lines. The fluctuations in both edges will cause variation in the width of narrow lines. LER mainly originates from the fabrication process, such as lithography and etching (Jiang, Wang, Yu, Chen, & Huang, 2013). In semiconductor industry, LER needs to be measured and controlled because it has been observed to be detrimental to IC performance (Bunday et al., 2004). In dimensional measurement, especially in 1D pitch measurement, LER is one of the key quality attribute to the variation in the average pitch of the lines. According to study by M. Tortonese et al. “the accuracy of the pitch measurement are depends on the line qualities. If the lines were rough, pitch measurement should be done in large number of sample locations to obtain an accurate pitch values. Otherwise, if the lines are uniform, the same accuracy can be achieved with fewer measurements and can be done much faster” (Tortonese, Prochazka, Konicek, Schneir, & Smith, 2002).

In recent review of the literature by C. Shin (Shin, 2016) found that the scanning electron microscopes with high resolution critical dimension (CD-SEMs) is one of common tool that can characterize the LER. In order to characterize the LER, firstly, the regular intervals (i.e., Λ) must be defined. It is defined by the different between distance point along the line where the edge is measured (Λ = xᵢ₊₁ − xᵢ) as seen in
Fig. 2. Definition of LER (Shin, 2016)

Secondly, the average edge ($\bar{x}$) and the standard deviation ($\sigma_{LER}$) of the LER calculated by using equations expressed as:

$$\bar{x} = \frac{1}{N} \sum_{i=1}^{N} x_i$$  \hspace{1cm} (3)

$$\sigma_{LER} = \sqrt{\frac{1}{N} \sum_{i=1}^{N} (\delta x_i)^2} = \sqrt{\frac{1}{N} \sum_{i=1}^{N} (x_i - \bar{x})^2}$$  \hspace{1cm} (4)

where $x_i$ is the local position measured at its point of the line edge, $N$ is a number of measurements, and $L$ is the length of measured line.

III. METHOD

In this research, the measurement of pitch was performed using a SEM SU3500 manufactured by Hitachi as seen in Fig. 3. The Hitachi SEM is a 200 mm diameter wafer-loaded type and its magnification range is 5 ~ 300,000 times on photo and 7 ~ 800,000 times on display. The resolution for SE method is 3 nm at 30 kV high vacuum mode and the BSE method is 4 nm at 30 kV high vacuum mode. The maximum image shift is ± 50 μm (WD=10). It has 5-Axis motorized stage (X, Y, Z, R, & T) to align the specimen.

For the whole experiments to obtain SEM images, the following conditions were commonly applied in SEM equipment, i.e. the aperture size of 50 nm, the mode of a SE method, the acceleration voltage of 20 kV, the emission current of 113.6 pA and 112.9 pA for 300 nm and 700 nm gratings, respectively. The SEM images were taken at magnification of 45,000 (x45k) and 19,000 (x19k) for 300 nm and 700 nm gratings, respectively. In this magnification, we will able to see around 20 line profiles to be measured as pitch value.

Fig. 4 shows the pitch measurement conditions. The number 1 to 10 represents a sampling area for the measurement of sample uniformity. The location of each area should be different,
so there is no line profiles are taken twice. Because of the SEM image contains 20 line profiles, we able to measure of 18 times of pitch value for every image. The measurement of pitch should be measured both of left and right side of pitch to compensate roughness line profile. The room condition needs to be maintained during the measurement in the range of temperature of (20±4) °C and relative humidity of (55±10) %.

In order to identify the contribution of LER, the LER was measured in the single line profile using Image J program macro designed to automatically calculation the parameters such as: the width, line edge roughness, and orientation angle of line profile (Bickford, 2013). The value of LER will be compared with targeting error from same sample used.

Fig. 4. The layout of sampling strategy is used for the measurement of sample uniformity. Each black square represents an area of calibration.

IV. RESULT AND DISCUSSION

Two measurement steps were carried out to obtain the necessary data. First measurement was performed to learn about general condition of the specimen, alignment and marking the calibration areas. In macroscopic size, we had to shift the image in the whole area of specimen due to limitation of maximum image shifting of SEM. There are several defects spotted on the surface of the specimen, such as pits and scratches. These defects are normal in production of this class of specimen and do not affect the accuracy of the pattern elsewhere because measurements can be carried out on many other parts which are free from defects. Most defects can be seen by using SEM at a magnification range 50-1000x and acceleration voltage 20kV. They are on the edge of specimens that may be resulted from the use of tweezers. Fortunately, defects can be used to obtain the best focus on the specimen surface.

The second measurement was performed to measure pitch values from both specimens. The technique to obtained pitch value was described in chapter 3. Simple random sampling is used to get the data. Each data has same probability of being chosen at any
position in the same line during the sampling process. The assumption that is used in this scheme is that every data represents the pitch value of a particular line. In order to assess the suitability of the specimens as transfer standard, the measurement was performed on the specimen model 301BE and 701CE. It was done using same technique to get sample uniformity as shown in Fig. 4. The results showed that the standard deviation of the mean of pitch values in 10 different areas of the specimens are 2.4 nm and 5.6 nm for specimens 301BE and 701CE, respectively. These tests revealed that there is no significant difference between pitch values in whole areas, in other words the specimens are suitable for comparison. However, these values have been taken into account as standard uncertainty of non-uniformity.

Fig. 5 shows the SEM images of the specimens model 301BE and 701CE, the size of which are (6.66×4.99) µm or (1290×960; RGB). The image from specimen model 701CE has better contrast than the other, making it easier to be measured. All images should be aligned to eliminate cosine error when the structure of line pattern is not perpendicular to the x-axis. This error can be ignored if the angle formed by the horizontal plane (e.g., monitor) is not more than 1°. In order to measure pitch values, the scale on the SEM needs to spatially calibrated, i.e. the distance in pixel against the known distance. The later is located at the corner of the image with same magnification and real units. After the scale has been spatially calibrated, the software automatically changes the distance in pixel to the real units. All pitch value reported in this measurement have been calibrated based on the known distance for every images.

Fig. 6 shows the frequencies in histogram and the distribution of pitch measurement from both specimens. Total number of pitch measurement is 180 data from 10 areas in each specimen. The distribution of all data in both specimens is normal. The highest frequency on the specimen 701CE are 50 data in the range of 700 – 710 nm.
and 42 data in the range of 296 – 298 nm on the specimen 301BE. The averages of pitch value are 293.6 nm and 700.3 nm for specimen model 301BE and 701CE, respectively.

The cumulative counts of pitch value recorded 50% of data less than the mean value of both the specimens. The standard deviation of specimen model 701CE is larger than of specimen model 301BE. It is because the quality of the lines edge in some locations of specimen model 701CE are very rough and affected to the variation of the pitch values at such locations.

![Fig. 6. The histogram of 180 pitch values from 10 different areas in specimen model 301BE and 701CE, respectively. All figures in units of nm.](image)

The LER measurement was performed on the selected area (yellow box) where size of which is $(0.7 \times 2.5) \mu m$ as shown in Fig. 7. The software automatically detects the edge of line profile and calculates the parameters. The result obtained from selected area shows that line width is 386.78 nm, root mean square (RMS) of both side of line edge roughness is 10.97 nm and the average angle is 89.9694 degrees. The line profile was slightly tilted about 0.0306° or ~111 arcsec. This angle value will be taken into account in cosine error.

Targeting error measurement was performed on specimen 701CE by using the same specimen in LER measurement. As shown in Fig. 8, the average value of targeting error is 701.4 nm with minimum value of 688.74 nm and maximum value of 719.13 nm.
The total number of targeting error is 22 data with standard deviation of 8.23 nm. The standard deviation of targeting error is slightly close to LER measurement. It reveals that for SEM has no capability to measure LER, this method can be applicable to estimate the contribution of targeting error or LER in pitch measurement. This value will take into account of targeting error.

The uncertainty evaluation was evaluated based on JCGM 100:2008. The combine standard uncertainty is given by (2) where all input quantities are independent. The result shows that the expanded uncertainty of specimen 701CE is 5.5 nm and the major source of uncertainty comes from non-uniformity of 40.89% and targeting error of 40.15%. In the specimen 301BE, the expanded uncertainty is 4.4 nm where 42.52% was comes from non-uniformity, 21.02% from targeting error and 36.46% from the other sources accordingly. The contribution of non-uniformity and targeting error requires further analysis to decrease uncertainty measurements.

V. CONCLUSION

The initial characterization of 1D grating standards have been done by using SEM. The results showed that measured pitch values for nominal 300 nm and 700 nm are (293.6±4.4) nm and (700.3±5.5) nm, respectively. The measurements to test the suitability of specimens as transfer standard revealed that the specimens are suitable for comparison. The multi strategy also was performed for the uncertainty evaluation to obtain cosine error, non-uniformity and targeting error. The metrological characteristics of each input variable were taken into account in the uncertainty. The major source of uncertainty for specimen 301BE and 701CE are come from non-uniformity and targeting error. The contribution of those values requires further analysis to decrease uncertainty measurements.
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VII. REFERENCES


Misumi, I., Dai, G., & Peng, G.-S. (2007). Final report on Supplementary Comparison APMP.L-S2: Bilateral comparison on pitch measurements of nanometric lateral scales (50 nm and 100 nm) between NMIJ/AIST (Japan) and PTB (Germany). *Metrologia, 44*(1A), 4006. Retrieved from http://stacks.iop.org/0026-1394/44/i=1A/a=40006


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