Nanometric Lateral Scales as CRM Candidates for AFM, SEM and Optical Diffractometer

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Abstract. National Metrology Institute of Japan, National Institute of Advanced Industrial Science and Technology (NMIJ/AIST) have designed nanometric lateral scales with a pitch of less than 100 nm for atomic force microscope (AFM), scanning electron microscope (SEM) and optical diffractometer. The pitches of the scales were calibrated by nanometrological AFM with differential laser interferometer (DLI-AFM) and the uncertainty in the pitch measurements was evaluated. The average pitches were quite close to the designed pitches and the expanded uncertainty ($k = 2$) was less than 0.5 % of the nominal pitch. It became clear that proposed nanometric lateral scales had sufficiently high quality as candidate certified reference materials (CRMs). A domestic intercomparison of the nanometric lateral scales is underway.

1. Introduction
Standards in nanometrology have become prominent. National metrology institutes (NMIs) have developed nanometrological instruments, which are traceable to the unit of length, and have been calibrating standard samples. Some of these institutes have been concentrating on the development of traceable atomic force microscopes (AFMs) which are promising instruments for nanometrology [1-4].

National Metrology Institute of Japan, National Institute of Advanced Industrial Science and Technology (NMIJ/AIST) has developed an atomic force microscope with high-resolution-XYZ-interferometers (nanometrological AFM)[5] and an atomic force microscope with differential laser interferometer (DLI-AFM), and is supplying a calibration service for pitch of one dimensional (1D) grating standards [6, 7] (calibration range: 200 nm – 8 μm ) using the metrological AFMs. Furthermore, test measurements of prototype 1D-gratings with 50 – 100 nm pitches were carried out in order to expand the pitch calibration range of 1D-gratings [8, 9]. However, not only the pitch calibration service of 1D-gratings, but also the distribution of 1D-gratings as usable certified reference materials (CRMs) is strongly required.

In this study, nanometric lateral scales as CRM candidates for AFM, scanning electron microscope (SEM) and optical diffractometer (OD) were designed and fabricated. The pitch measurements of the fabricated lateral scales were carried out and the uncertainty in pitch measurements was evaluated.

2. Samples, instruments and measurement conditions
2.1. Nanometric lateral scales
2.1.1 Necessary conditions for 1D-grating standards as CRMs. Reference material (RM) is defined that material or substance one or more of whose property values are sufficiently homogeneous, stable, and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials in ISO Guide 30 (1992). And the definition of certified reference material (CRM) is that Reference material, accompanied by a certificate, one or more of whose property values are certified by a procedure which establishes its traceability to an accurate realization of the unit in which the property values are expressed, and for which each certified value is accompanied by an uncertainty at a stated level of confidence. Based on the above definitions, 1D-grating standards as CRMs are required to satisfy the following conditions.

1. 1D-grating standards are available for the calibration of several types of measurement instruments, for example AFM, SEM and OD. Then 1D-grating standards have to be suitable size and shape for several different instruments.

2. 1D-grating standards are sufficiently high quality. In this study, it is determined that the goal of expanded uncertainty \((k = 2)\) in pitch measurements is 0.67 % of nominal pitch. Since the required accuracy for inspection in the International Technology Roadmap for Semiconductors (ITRS) is 2 % of the technology node in \(3\sigma\), the required accuracy of standard samples is assumed 1 % \((3\sigma)\) and the accuracy is calculated from \(3\sigma\) to \(2\sigma\) and the goal of expanded uncertainty \((k = 2)\) is obtained.

3. 1D-grating standards can be purchased. A number of 1D-grating samples with pitches of more than 100 nm as RMs are available commercially [10-12]. It is demanded that 1D-grating standard samples with smaller pitches as CRMs are distributed since miniaturization in nanometrology is undergoing rapid progress.

2.1.2 Constraint fabrication conditions of 1D-grating standard samples with less than 100 nm pitch. Nanometrological standard samples, for example 1D-gratings, are used to calibrate several types of measurement instruments for the measurement of the critical dimension of small features as mentioned above. However, suitable specifications of nanometrological standards are slightly different depending on the instrument types, as shown in table 1. AFM and SEM are mainly used for measurements in relatively narrow area, approximately a few micrometer order. On the other hand, wide area, approximately several hundreds micrometer or millimeter order, is necessary for measurements by OD since the incident light beam cannot be collimated thinly and sufficient intensity of diffracted light is obtained. A relatively large aspect ratio, more than 1, of grating patterns is required for measurements by SEM and OD while a small aspect ratio, less than 1, is sufficient for measurements by AFM.

The size of the fabricated pattern area of 1D-grating standards is different depending on pitches and fabrication methods as shown in table 2. 1D-grating patterns of relatively large pitch, for example more than 100 nm, are mainly fabricated by an optical lithography method and the size of the fabricated pattern area is also large, approximately several millimeter order. On the other hand, 1D-grating patterns of small pitch, less than 100 nm, are mainly produced by electron beam (EB) lithography method and the pattern area is small.

In this study, nanometric lateral scale with less than 100 nm pitch was designed considering several constraint conditions as mentioned above.

| Instrument | Area of 1D-gratings | Aspect ratio |
|------------|---------------------|--------------|
| AFM        | Narrow              | Low          |
| SEM        | Narrow              | High         |
| OD         | Wide                | High         |

Table 1. Specifications of nanometric one-dimensional grating standards for a few types of nanometrological instruments.
Table 2. Pitch sizes, fabrication methods and pattern areas.

| Pitch Size     | Typical Fabrication Method | Pattern Area |
|----------------|-----------------------------|--------------|
| more than 100 nm | optical lithography         | Wide         |
| less than 100 nm  | EB lithography              | Narrow       |

2.1.3 Nanometric lateral scale. Figure 1 shows a schematic drawing of a nanometric lateral scale. The scale was made of silicon substrate (10 mm × 10 mm × 0.525 mm) and consisted of a scale pattern, a reference pattern and a guide pattern. The scale pattern area was fabricated in the center of the substrate and the pattern area was approximately 200 μm square. The wide area of scale pattern with approximately 200 μm square was realized by a combination of small cells 8 μm square, which were able to be fabricated by electron beam (EB) lithography. The small cell 8 μm square had a grating pattern area 7.2 μm square and a gap. If the pitch of the scale was the common denominator of 8 μm and 7.2 μm, the positions of gaps in all cells were the same. On the other hand, if the pitch was not the common denominator, the positions of gaps in all small cells should be justified to align with the phases of spatial frequency of 1D-grating patterns in order to eliminate the decrease in the intensity of the diffracted light. The reference pattern was located approximately 1 mm above the 1D-grating pattern area and approximately 200 μm square also. The reference pattern was used for the optimization of measurement conditions before the measurements of the scale pattern and the damage to the scale pattern was prevented. The guide pattern was located to support the easy positioning of the AFM cantilever probe on the scale pattern. The aspect ratio of the patterns was more than 1 for SEM and OD measurements. The nanometric lateral scales were fabricated by Nippon Telegraph and Telephone Advanced Technology Corporation (NTT AT) based on the design.

Figure 2 shows a photograph of the nanometric lateral scales. Two scales with 100 nm pitch, two with 60 nm pitch and three with 50 nm pitch were fabricated. Measurements of one scale with 100 nm pitch and one with 60 nm pitch were carried out and the uncertainties in the measurements were evaluated.

2.2. Atomic force microscope with differential laser interferometer (DLI-AFM)

Figure 3 shows a schematic drawing of an atomic force microscope with differential laser interferometer (DLI-AFM). The DLI-AFM consisted of an interferometer unit, a stage unit and a probe unit, and was installed on a vibration isolation table. The DLI-AFM was loaded in the acoustic protection shield and realized measurements in a well-controlled environment. The interferometer unit was composed of two-path differential laser interferometer units in the XY-axes (figure 4) and a four-path homodyne-type laser interferometer unit in the Z-axis. Interferometer resolution in the XYZ-axes reached approximately 40 pm using optical and electrical multiplication factors. The optical path differences between the measurement and reference arms of the laser interferometers in XYZ-axes were nearly zero. The arrangement of the interferometers in the XY-axes was symmetrical and eventually the optical path differences maintained cancelled out even if the base of the DLI-AFM was thermally expanded. The laser source of the interferometers was a slave laser in an offset-locked He-Ne laser system. The frequency of the slave laser was operated to trace the frequency of an I2-stabilized He-Ne laser, a length standard, with an offset frequency. Therefore, the DLI-AFM was always traceable to the length standard. The stage unit consisted of a piezo actuator-driven leaf spring stage in XY-axes and a piezo-tube scanner in the Z-axis. The scanning range of the stage unit was approximately 100 μm(X) × 100 μm(Y) × 12 μm(Z). The tip of a cantilever probe was loaded the origin of the coordinate system of the laser interferometers every time by a kinematical mount of a probe unit and an Abbe error was reduced. The atomic force between the tip and the sample surface was detected by a conventional optical lever system. A laser diode (LD), a laser source of the optical lever system, was located outside of the shield and a laser beam from the LD was introduced by an
optical fiber since the thermal source generated by the LD was eliminated. The measurement mode of AFM was the AC mode. Furthermore, the probe unit and the base plate of the interferometer unit were made from low-thermal-expansion material (super invar) and the drift caused by temperature change was reduced.

A schematic drawing of the differential laser interferometer in the X or Y axis is shown in figure 4. A five-sided moving mirror (MM) was used as the target mirror of the XYZ-axes laser interferometer.
Figure 3. Schematic drawing of main unit of atomic force microscope with differential laser interferometer (DLI-AFM), which consists of a stage unit, an AFM probe unit and interferometer units. The laser source is the slave laser of an offset-locked He-Ne laser system.
Figure 4. Optical path arrangement of one-axis interferometer unit (X or Y axis).

unit and the sample container. Two mirrors facing each other were used in the X-axis interferometer, the other two mirrors were used in the Y-axis interferometer and the remaining one mirror in the bottom side was used in the Z-axis. The laser beam from the optical fiber coupler was divided into two beams at the plate polarizing beam splitter (PBS). The two beams travelled back-and-forth twice between the MM, the reference mirror (RM) and the corner cube prism (CCP). After that, the two beams were reflected by the other CCP and passed under the MM. At that time, the inner beam and the outer beam switched positions with each other. The two beams were combined at the other plate PBS. Lastly, the beam was divided into four and the four laser interferometer signals were detected at the phases of 0°, 90°, 180° and 270° using the four photodetectors (PDs).

2.3. Measurement conditions and evaluation uncertainty

Table 3 shows the pitch measurement conditions. Scanning lines, oscillation frequency and cantilevers were common. The cantilevers used in the pitch measurements were fabricated by Olympus Co., Ltd. For 100 nm pitch measurements, the scanning area and speed were 5 μm(X) x 5 μm(Y) and 1 μm/s respectively. For 60 nm pitch measurements, the scanning area and speed were 1.8 μm(X) x 1.8 μm(Y) and 0.36 μm/s respectively. The definition of pitch is described elsewhere [6].

Uncertainties in pitch measurements were evaluated the basis of the “Guide to the expression of uncertainty in measurement (GUM)” [13]. The sources of uncertainty in pitch measurements are shown in table 4. The standard uncertainties derived from all sources were evaluated, and expanded uncertainty was calculated from the standard uncertainties. The evaluation method in pitch measurements is described elsewhere [6].
### Table 3. Pitch measurement conditions for DLI-AFM.

| Nominal pitch (nm) | Scanning area (μm) | Scanning speed (μm/s) | Scanning lines | Oscillation frequency (kHz) | Cantilever |
|--------------------|--------------------|----------------------|----------------|-----------------------------|------------|
| 100                | 5.0 x 5.0          | 1                    | 32             | approximately 300           | OMCL-AC160TS |
| 60                 | 1.8 x 1.8          | 0.36                 |                |                             |            |

### Table 4. Source of uncertainty in pitch measurements using DLI-AFM.

| Source of uncertainty |
|-----------------------|
| I. Measurement         |
| (1) Repeatability     |
| (2) Nonuniformity     |
| II. Slope correction  |
| (1) Cosine error (vertical inclination) |
| (2) Cosine error (lateral inclination) |
| III. Laser interferometer |
| (1) Frequency variation of the laser |
| (2) Frequency stability of the laser |
| (3) Change in the dead path (temperature) |
| (4) Change in the dead path (thermal expansion) |
| (5) Interferometer resolution |
| (6) Cosine error in the optical alignment |
| (7) Abbe error         |
| (8) Change in the optical path |
| (9) Interferometer nonlinearity (cyclic error) |
| IV. Refractive index of air |
| (1) Refractive index of air (temperature) |
| (2) Refractive index of air (humidity) |
| (3) Refractive index of air (pressure) |
| (4) Refractive index of air (CO₂ density) |
| V. Sample temperature  |
| (1) Difference in the sample temperature |
| (2) Thermal expansion  |

### 3. Results and discussions

Figure 5 shows an optical microscope image of the combination of a nanometric lateral scale and a cantilever close to the scale pattern in the lateral scale. The guide pattern of the lateral scale supported to find out the scale pattern using a conventional optical microscope installed in the DLI-AFM. A DLI-AFM image of the lateral scale is shown in figure 6.

Table 5 shows the major sources of uncertainty and their standard uncertainties. The first major source was interferometer nonlinearity and the standard uncertainty was approximately $9.6 \times 10^{-2}$ nm. Other major sources were the nonuniformity of samples, interferometer resolution, cosine error and
repeatability as shown in table 5. The major sources of uncertainty were common with those in other measurements [6] and the elimination method of the standard uncertainty caused by major sources has been investigated elsewhere [14].

Figure 7 shows the average pitches and expanded uncertainties \((k = 2)\) obtained by the DLI-AFM. The average pitches were quite close to the nominal pitches \((100, 60\ \text{nm})\) and the expanded uncertainties were sufficiently small, less than \(0.5\%\) of the nominal pitches. The goal of the expanded uncertainty was achieved and it became clear that the designed and fabricated nanometric lateral scales satisfied one of the necessary conditions for realizing usable CRMs as mentioned in 2.1.1.

| Source of uncertainty                  | Standard uncertainty (nm) |
|----------------------------------------|---------------------------|
|                                        | pitch: 100 nm             | pitch: 60 nm               |
| Interferometer nonlinearity             | \(9.5 \times 10^{-2}\)    | \(9.5 \times 10^{-2}\)    |
| Nonuniformity                          | \(5.7 \times 10^{-2}\)    | \(4.2 \times 10^{-2}\)    |
| Interferometer resolution              | \(2.2 \times 10^{-2}\)    | \(2.2 \times 10^{-2}\)    |
| Cosine error (lateral inclination)     | \(9.5 \times 10^{-3}\)    | \(2.3 \times 10^{-2}\)    |
| Repeatability                          | \(1.8 \times 10^{-3}\)    | \(1.6 \times 10^{-3}\)    |
4. Summary

Nanometric lateral scales (nominal pitch: 100 and 60 nm) as usable certified reference materials (CRMs) for AFM, SEM and OD were designed and fabricated. The pitch of the lateral scales was measured by DLI-AFM and the uncertainty in pitch measurements was evaluated. The obtained average pitches were quite close to the designed pitches and the expanded uncertainties were less than 0.5 % of the nominal pitches. It became clear that the fabricated nanometric lateral scales satisfied condition (2) of the candidates of the CRMs as mentioned in 2.1.2. The nanometric lateral scales were fabricated by NTT AT Co., one of the leading makers of nanometric standards, and the scales will be available commercially after the conditions have been met. It can be said that condition (3) has been satisfied.

Recently, National Metrology Institute of Japan, AIST (NMIJ/AIST) have conducted a domestic intercomparison of the fabricated and calibrated nanometric lateral scales as a pilot laboratory to check whether the scales are suitable standards for conventional AFM, SEM and OD. Figure 8 shows a
photograph of a set of two lateral scales, a data logger and a pin set for intercomparison. Participants mainly use their developing or commercial AFMs but a few participants measure the scales by an OD with a deep ultraviolet laser and critical dimension SEM (CD-SEM). Condition (1) mentioned in 2.1.1 will be satisfied when the intercomparison is successful. The results of the intercomparison will be reported by the pilot.

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