

## Research on the development and measuring method of nanometre length standard reference material

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### Abstract

Nanometre length Standard Reference Material (SRM) is the important aspect of basic nano science research and the key technology for quality control in the manufacturing process of nano materials and devices. In the present work, a special series of nano gratings that are easy to be measured were designed, and large-scale metrological nanometre measuring machine was designed to measure the standard reference material of nano gratings, with laser interferometers in X/Y/Z axes, which can realize the direct traceability of nano length values to the SI unit. Homogeneity and stability of the standards were measured to evaluate the quality of gratings. One-dimension nanometre gratings Standard Reference Material and quantity transfer system of nanometre geometry based on large scale metrological nanometre measuring machine were established.

Keywords: Standard Reference Material; Nano metrology; Large-scale metrological nanometre measuring machine

### 1. Introduction

The development of semiconductor industrial follows Moore's Law that the number of transistors per square inch on integrated circuits has doubled every year since their invention, which means Critical Dimension (CD) of Integrated Circuits (IC) is getting smaller and smaller. In the last few years, the CD shrinks in 50% every six years. This development demands a new micro-nano precision measurement criterion about manufacture process parameters and permissible error of measurement and control instrument and apparatus. International manufacture of semiconductor devices requires comparability of measurement results. First, measurement values must trace to SI unit, which means CD of micro-nano structure must trace to wavelength of the meter definition in micrometre and nanometre scale. Second, evaluation methods of measurement uncertainty must be specified and accepted[1,2].

Nanometre length Standard Reference Materials (SRM) are the general conditions of basic nano science research and the necessary factors for quality control in the manufacturing process. SRM is the guarantee for accurately and traceable transferring of length values in nanoscale. It is different from traditional SRM in chemistry field, the type of physical SRM quantity is rare, the manufacturing process is complex, and it can be reused for many years with regular calibration. Developing physical quantity SRMs and realizing precise measurement depend not only on advanced nano manufacturing technology, but also on metrological technology with high accuracy and direct traceability.

### 2. Manufacture methods

The manufacture progress of one dimension nanometre gratings includes follow procedures, layout design, mask preparation, photoetching to make guidance graph and explanation text, electron-beam exposure to make key graphics and standard reference material numbers, cleaning and quality

evaluation with Scanning Electron Microscope (SEM) and Atomic Force Microscope (AFM).

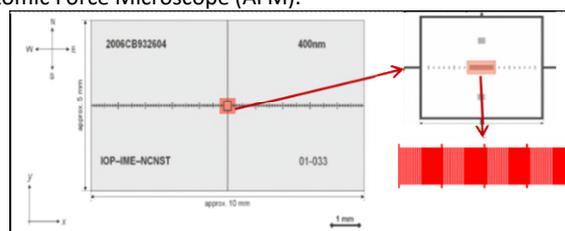


Figure 1. Diagram of nano grating SRM

First, L-Edit was used to design the layout, as shown in Fig 1. The SRM was built on a standard four inches silicon wafer, the layout has identification words, reference line and indication window, so the tested area will be easily found. Second, use GCA 3600F optical graphics generator to prepare the mask. Then, use photoetching technology and E-beam exposure to make the pitch structures. After that, with concentrated sulfuric acid to clean the organic contaminant, a standard nanometre grating was fabricated. SEM and AFM are used to test and evaluate the samples.

### 3. Measurement system

In the study, a large-scale metrological nanometre measuring machine, as shown in Fig 2, was utilized to measure the Standard Reference Material of nano gratings. This instrument can be used to measure nanometre geometry parameters such as line width, line separation, step height, surface texture in nano scale and roughness within millimetre range. The system is composed of four parts, multilevel and multi-DOF displacement system including macroscopic displacement system and nanoscale displacement system, three-dimensional orthogonal scanning atomic force measuring probe with traceability, multi-DOF interferometer system and control system[3].

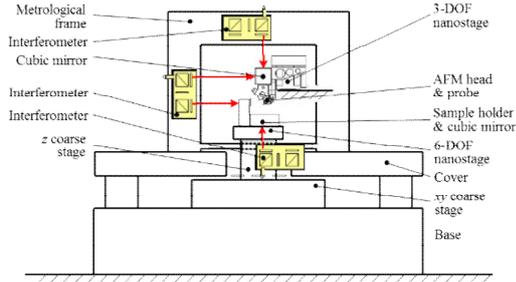


Figure 2. Measurement system

#### 4. SRM valued algorithm

In the measurement, the middle area was divided into several parts, and the measurement in each part was repeated 8 times to reduce random error. The average value of different position reflects the gratings pitch. Gravity center method was used to calculate the grating pitch, the procedure is shown as Fig 3.

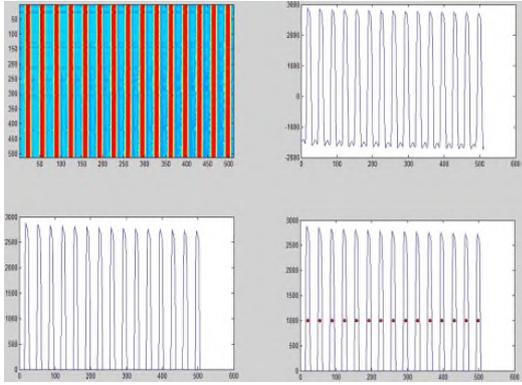


Figure 3. Schematic of algorithm process

The surface texture from nano measuring machine was shown above, including ten lattice periods. By setting a threshold value, the profile is divided into ten parts. The coordinate of gravity centre of each peak is given as

$$P_c = \frac{\sum x_i z_i}{\sum z_i} \quad (1)$$

where  $x_i$  represents horizontal coordinate of measurement points and  $z_i$  means height values.

The average lines' distance can be calculated as

$$\bar{D} = \frac{1}{M} (\sum_{m=1}^M \bar{D}_m) = \frac{1}{N} (\sum_{m=1}^M (\sum_{n=1}^N D_{mn})) \quad (2)$$

where  $N$  represents the point numbers on an profile,  $M$  represents the line numbers in a measured area,  $D_{mn}$  represents the distance between two points on the same line,  $\bar{D}_m$  represents the average distance of an outline and  $\bar{D}$  represents the average grating pitch in the measured area.

#### 5. Uncertainty evaluation

The uncertainty of measurement results is evaluated according to ISO uncertainty evaluation method. The uncertainty components are given as follows [6].

- 1) Uncertainty component caused by measurement repeatability, which is given as  $S_r$
- 2) Uncertainty component caused by sample inhomogeneity, which is given as  $S_h$
- 3) Uncertainty component caused by sample stability, which is given as  $S_t$
- 4) Uncertainty component caused by residual error of calibration device, which is given as  $S_c$
- 5) Uncertainty component caused by temperature change, which is given as  $S_T$

5) Uncertainty component caused by inclination angle of samples, which is given as  $\delta l_c$

Moreover, there are other uncertainty components such as surface roughness, surface friction, probe abrasion, Vander Waals force and so on, however, these influences are very hard to evaluate, and there is no experience to take an example, so all of this will be considered as system noise. The mathematical model of uncertainty is shown as

$$\Delta P = \delta S_r + \delta S_h + \delta S_t + \delta S_c + \delta T + \delta l_c + \delta N \quad (3)$$

where  $\delta N$  represents uncertainty caused by system noise.

#### 6. Measurement results and conclusion

The measurement results is give in Table 1.

Table 1. Calibration results (nm)

NO.	Nominal Value	Calibration Value	Uncertainty
070	400	400.3	2.7

The research and application status of nanometre grating Standard Reference Material shows that, this project can fill domestic blank and satisfy application need of nano science and technology development and the application requirement of semiconductor industry. The tested area of grating samples can be easily found with identification words, the reference line, and the indication window. Based on the mixture exposure technology combined with ultraviolet exposure and electron beam exposure, high efficiency and repeatable manufacture is easily realized. Large-scale metrological nanometer measuring machine designed to measure the standard reference material of nano gratings, can realize the direct traceability of nanometer length values to the SI unit.

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#### References

- [1] Richard A. Allen, Patrick Troccoli, James C. Owen III, James E. Potzick, and Loren W. Linholm. 1993. Comparisons of measured linewidths of sub-micrometer lines using optical, electrical, and SEM metrologies. *Proc. of SPIE*, Vol. **1926**:34-43.
- [2] Harald Bosse and Gunter Wilkening. 2005. Developments at PTB in nanometrology for support of the semiconductor industry. *Meas. Sci. Technol.* **16**: 2153-2166.
- [3] S. Gao, M. Lu, W. Li, Y. Shi, X. Tao and H. Du. 2012. A large range metrological atomic force microscope with nanometer uncertainty. XX IMEKO World Congress.
- [4] G. Dai, L. Koenders, F. Pohlenz, T. Dziomba and H.-U. Danzebrink. 2005. Accurate and traceable calibration of one-dimensional gratings, *Meas. Sci. Technol.*, Vol. **16**, pp. 1241-1249
- [5] G. Dai, F. Pohlenz, T. Dziomba, M. Xu, A. Diener, L. Koenders, H.-U. Danzebrink. 2007. Accurate and traceable calibration of two-dimensional gratings, *Meas. Sci. Technol.*, Vol. **18**, No. 2, pp. 415-421
- [6] BIPM IEC IFCC ISO IUPAC IUPAP OIML-1993. Guide to the Expression of Uncertainty in Measurement.