Metrology and standard which underpin the development of nano technology

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Abstract – No one doubts both metrology and standard are essential in the development of nano technology. There are two standards, i.e. industrial standard and measurement standard, and they are in a complimentary relationship thus referring to the other. In addition, two kinds of standards are useless unless appropriate measurement techniques and their traceability are established. In this article, the relationship among the industrial and measurement standards, measurement techniques and traceability is explained following their necessity for the development of nano technology. We can conclude metrologists can play an important role in the development of nano technology.

***Key Words*:** Nano metrology, traceability, measurement standard, industrial standard

1. Importance of standard in nano technology

It is beyond discussion that nano technology underpins current society and no one questions that metrology supports nano technology.

We can easily point out typical examples. Metrology makes an important role for the quality control of production process and the evaluation of functions of nano products. When we assess the influence of nano materials for human bodies and environment, size measurement of nano products is essential. In addition improvement of functions of nano products directly leads energy saving.

2. Industrial standard and measurement standard

Before starting the consideration of nano metrology, we are going to review industrial standard, measurement standard, and their differences. They are often confused with each other.

Concisely speaking industrial standards are documents such as ISO and JIS, so that it is also called document standards. Common agreements which stipulate the specifications of products or testing procedures are documented.

On the other hand, measurement standards are the definitions of units and their realization. Measurement standards are needed to secure the correctness of the results of measurements. For this purpose, in addition to the definition of units the traceability system is indispensable.

These two standards are completely different, but in practical use they are closely related and refereed each other.

2.1. Measurement standards

Measuring instruments which are used in many occasions in industries should be calibrated by higher level measuring instruments. These higher level measuring instruments also should be calibrated by much higher level measuring instruments. The system in which tracing this calibration chain leads to national or international standards is called traceability (system).

In Japan there is JCSS (Japan Calibration Service System) regulated by the Measurement Law. JCSS is not a sole accreditation system and there are many other accreditation systems by which traceability is secured. JCSS, however, is one of the most reliable systems; therefore its effective use is quite profitable.

In the traceability system defined by the JCSS, the combination of the optical frequency comb and the atomic clock synchronized with UTC (Universal Time, Coordinated) owned by NMIJ is located at the top level, i.e. designated as a primary standard, and all other instruments are linked below in hierarchy system like a pyramid.

Let us introduce one useful topic for length metrologists. The frequencies of stabilized lasers whose frequencies are locked with the absorption lines of atoms are intrinsically fixed and hence known without calibrations. Such measurement standards which take advantage of physical phenomena and do not need calibrations are called intrinsic standards.

Intrinsic laser frequency standards are approved by Committee of International Weights and Measures (CIPM) and listed in the table by the name of MeP (Mise en pratique).1) An unstabilized He-Ne laser at 633 nm has been registered in MeP, which is the most commonly used for length measurements.2) It means the He-Ne laser at 633 nm is intrinsically traceable without calibration as far as they are emitting light. (As far as JCSS is concerned, the He-Ne laser at 633 nm still needs calibration because the frequency comb owned by NMIJ is only one designated primary standard and all length measuring instrument in JCSS system shall be calibrated and traceable to the primary standard.)

2.2. Industrial standards

There are two categories of industrial standards. One is a product standard which defines specifications of industrial products, such as size, shape, characteristics, and so on. The other category is a procedure which defines testing or measuring method of industrial products.

The most famous industrial standard is ISO standard. ISO standards are being discussed in Technical Committees (TC), each of which is responsible for a specific technical area. Technical committees which are relevant to nano technology have been existed, such as TC 201 (Surface Chemical Analysis) and TC 202 (Microbeam Analysis). In addition to them, TC 229 (Nanotechnologies) has been founded which is responsible for nano technology and has received worldwide attention. TC 229 will be explained later.

3. Problems raised in nano metrology

There are a lot of varieties of length measuring instruments for different sizes of measuring objects. The most high resolution instruments are transmission electron microscopes (TEM) and scanning probe microscopes (SPM). These instruments enable to observe objects with sub-nanometer resolution, even atoms can be observed.

By the way, the practical length standard is a laser wavelength. It was supposed to be very short for a long time, however, compared to the size treated in the current nano technology it is not so long any more. To keep the traceability of nano metrology from the primary standard, i.e. laser wavelength, the wavelength must be subdivided. It means the phases of light waves must be determined. Normally this task is performed by using interferometers, but the accuracy of phase detection is not so high due to several reasons.

Although I wrote atoms can be observed, only ‘observations’ can be possible but ‘measurements’ are impossible. In fact, accuracy of nano metrology is not always so high than it is thought to be (correctly speaking, the term ‘uncertainty’ should be used instead of ‘accuracy’, but two words are used as almost the same meaning hereafter). Only the resolution is extremely high. It is often the case with nano metrology that the reliable digits of measurement results are only two and in extreme cases only one.

When nano metrology is actually performed, one can see the instability of measurement results. Several reasons for this can be considered.

1) Low quality of measuring objects

   As the measuring objects are extremely small, fabricating the objects as they have been designed is difficult. Consequently different measurement results can be obtained even if the measuring position is slightly shifted.

2) Environmental effect

   Air turbulence causes unstable measurement results for interferometric measurements, for example. Dusts may contaminate the surface of measuring objects thus resulting in unstable measurement results.

3) Difference of physical phenomena occurs between the measuring object and the probe

   Measurement results can be different for different measuring methods since the physical interactions or phenomena occurred between the probe and the surfaces of the measuring objects are different. This fact has been considered the largest problem in nano metrology. Examples will be shown in the next section.

4) Ambiguous definition of the measurand

   Even if the measurands (quantity to be measured) are called the same for different measuring methods, definitions of the measurands are not always the same. There is a posssiblity to observe different physical properties.

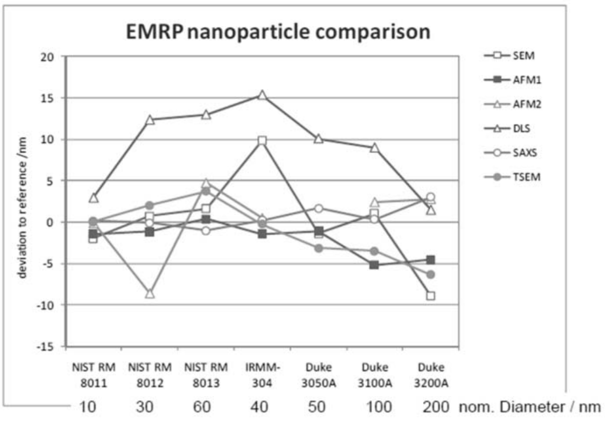

Fig. 1 Result of the nano particle comparison (copied from the presentation by Dr. Thalmann in CCL Nano Symposium 2009 in A*Star Singapore)

4. Necessity of advanced measuring technology

The fact that different results are obtained by different measuring methods is inevitable in most cases nowadays.

Let us see one example. Nano particles are one of the most typical and commonly used nano products which are very versatile in many industrial areas. There are many measuring principles and instruments to measure the particle size, such as SEM (Scanning Electron Microscopy), TEM (Transmission Electron Microscopy), SPM (Scanning Probe Microscopy), DLS (Dynamic Light Scattering), SAXS (Small Angle X-ray Scattering), DMA (Dynamic Mobility Analysis), and so on. However different instruments are not always bring the same result. Figure 1 shows the result of an international comparison initiated by European Metrology Research Program (EMRP).

Another example is the line width measurement of nano scales. There are three common methods for measuring the line width, SEM, AFM, and an optical probe microscope. In SEM measurement, secondary electrons scatter at the edges of the lines. When the electron beam scans perpendicular to the line and the beam crosses the edge, the signal is observed larger by the scattering effect. In AFM measurement, the convolution of the tip form of the probe and the object is observed. To retrieve the true form of the object, a de-convolution calculation is necessary. In optical probe measurement, the spot size of the optical probe is far larger than the size of the object; hence a simple geometrical optics theory cannot be applied. Figure 2 explains the bias observed between SEM and AFM measurements.

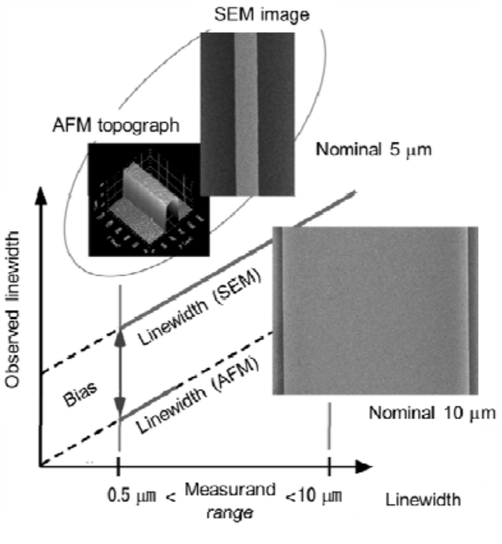

Fig. 2 Line width bias correction between AFM and SEM

To overcome this difficulty, development of accurate and stable measurement techniques is essential and this task is required for the scientists researching on precision engineering primarily metrology. In the process to pursue this task, we may go back to basic researches to investigate on fundamental physical phenomena.

In the discussion of the Consultative Committee for Length (CCL) of International Committee of Weight and Measures (CIPM), a suggestive comment was given which impressed me deeply. ‘Currently, different measurement results can be obtained by different measurement methods in nano metrology. It is caused by the lack of our knowledge for natural phenomena. To diminish this difference is the mission given for metrologists.’

5. Necessity of measurement standard

Development of advanced measurement technology is not a goal. It is necessary to confirm if the measuring technology developed is valid and the measurement that users are actually performing is correct.

If calibration services or certified reference materials (CRM) are available, users can check the correctness of their own measurements easily. In case there is a bias between the measurement value and the calibrated value, users can compensate this bias by themselves; the usefulness of the calibration services and CRM are obvious.

In response to these requirements, AIST is providing various calibration services and selling CRM. In the next section, a few examples will be shown. A strategic plan to expand the scope of the calibration services and CRMs has been made. Since it is a mission for AIST to satisfy the demands from industrial users, any requests and opinions for the measurement standard maintenance plan are always welcome.

Another tool to encourage various stakeholders to collaborate is the Strategic Technology Map published by the Ministry of Economy, Trade and Industry (METI).[3)] NMIJ is acting as a secretariat to compile the Measurement System section of this roadmap and many NMIJ researchers are involved in. This roadmap lists all technologies needed for future and shows the directions of technology development in order to promote the collaboration of different sectors, such as industrial, academic and public sectors. This roadmap is updated every year. Any inputs which will be included in the next version are welcome.

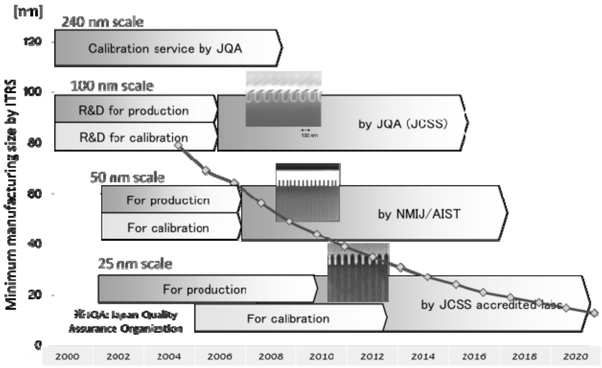

Fig. 3 Nano scales and calibration services which underpin the development of semiconductors. The red line indicates the minimum manufacturing size expected in the International Technology Roadmap for Semiconductors (ITRS).

6. Measurement standards provided by NMIJ

Two calibration services in nano metrology developed and supplied by NMIJ are shown here.

The first one is one dimensional nano scale (1-D gratings). In the production process of semiconductors, critical dimension scanning electron microscopes (CD-SEM) are often used. The accuracy of length measurement is critical to improve the quality and the yield rate of semiconductors. CD-SEMs, however, are not able to secure the traceability of length measurements by itself. For this purpose, CD-SEMs are equipped with calibrated 1-D gratings on the side of the specimen tables and the nano scales are routinely measured so that the traceability can be secured.

The world market share of CD-SEM by Japanese manufacturers is approximately 70 %. This fact implies the 1-D gratings are of crucial importance. A grating of 240 nm pitch has been used for many years for this purpose and recently 100 nm pitch grating is used. As the process rule of semiconductors is becoming smaller, 100 nm grating is no more sufficient. NMIJ has completed the development of a 25 nm grating which will be used for the production of next generation semiconductors. The history and roadmap of nano scale development is shown in Fig. 3. The nano scales have been prepared well in advance than expected by International Technology Roadmap for Semiconductors (ITRS).

The 25 nm scale is so small to make even by the electron beam lithography. We have invented a tricky manufacturing process to make nano scales very easily as follows.[4)] First, fabricate supperlattice structure by layer-by-layer deposition by molecular beam epitaxy (MBE) (for GaAs/InGaP suparlattice) or sputtering deposition with alternative target change (for Si/SiO$_2$ super lattice) so that two different materials are layered several ten times. Second, cleave the supperlattice. Finally, etch the cleaved face to remove one material selectively and a comb shape nano scale can be fabricated. This process is illustrated in Fig. 4.

The second calibration service is for the line width of a photo mask drawn on a glass substrate with chromium. The calibration is done by using a SEM, but as explained before, the measurement results by SEM has a systematic bias in the edge detection. The bias is compensated by comparing the measurement results of the traceable AFM. Nevertheless the AFM also has a systematic bias; therefore its probe shape is characterized by measuring a sharp needle. To resolve this bias compensation for different measuring methods, the traceability chart for the line width calibration became complicated as shown in Fig. 5.

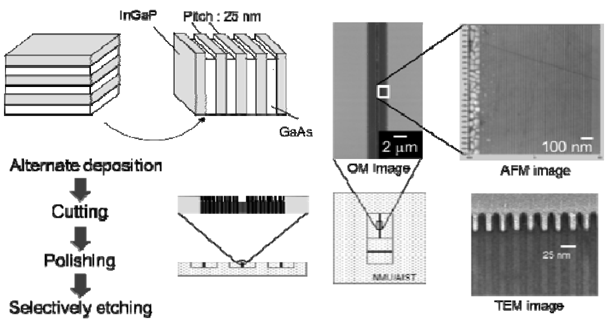

Fig. 4 Fabrication of ultrafine 1-D grating

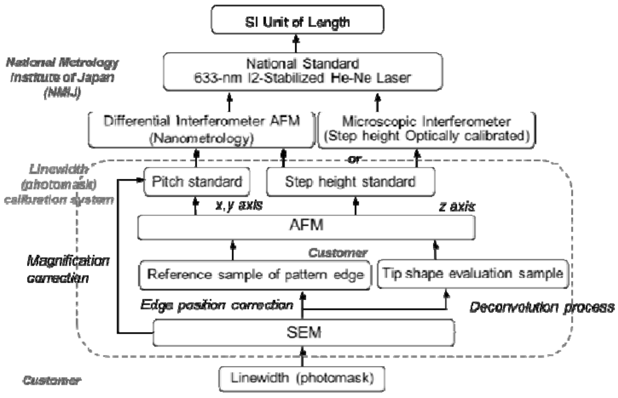

Fig. 5 Traceability chart of line width (photomask)

7. Necessity of industrial standard

As explained many times, in nano metrology, measurement results are method dependent and therefore most nano technology users are in inconvenient situation.

One of the peculiarities of nano technology is that it is used in many industrial fields. Let us imagine conventional precision engineering. It has been primarily used in machining industry. On the contrary, nano technology is used not only in semiconductor industry but in many others such as chemical, medical, food, cosmetics, and environmental industries. It means ‘interchangeability’ and ‘interoperability’ are important. If the measurement procedures and conditions are commonly agreed and documented; i.e. industrial standards are established, interchangeability and interoperability will effectively work.

While the original purposes of making industrial standards are to improve interchangeability of industrial products and promote development of industries, industrial standards have been utilized as a strategic tool of international competition. Since nano metrology is a newly emerging filed, this strategy is emphasized more and international competitions are more intense.

ISO/TC 229 introduced above is a newly established technical committee in May 2005. Since then two meetings are held every year. One technical report (TR) and one technical specification (TS) have been published and 6 TR, 22 TS and 6 international standards (IS) are being discussed in TC 229.

I have been involved in standardization activities for many years and understand the importance of industrial standards including TC 229. However, I found a notable sentence in the minute of TC 229 held recently that ‘the importance of the committee’s strategic plan regarding the needs of the users were emphasized, as it appeared that some NWIPs were opportunistic and not aligned with the strategic plan (from ISO/TC 229 N 658, Draft report of the 9th meeting).’

Concerning this expression I don’t know whether the strategic plan or the experts participating in the TC is bad. I, however, am afraid that the importance of industrial standards is emphasized too much so that too rapid and rough discussions and proposals have been made in the TC.

Possible factors for this is

- Some members are misunderstanding the objective of industrial standards,
- Some members are impatient as they are thinking ‘First come, first served.’
- Some members are not accustomed with standardization well.
- As the TC is new, it is still in chaos and rules are not fixed yet.

I believe the most important thing to avoid this confusion is having discussions based on technical background, tangible evidence and the scientific consideration of metrology and traceability. Only metrologists and National Metrology Institutes can settle this controversial situation and play important roles to underpin the development of nano technology.

References

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