International standardization of scanning probe microscopy in VAMAS/TWA 2 and ISO/TC 201 (Surface Chemical Analysis)

Daisuke Fujita [1*]

[1] Research Center for Advanced Measurement and Characterization (RCAMC),
National Institute for Materials Science (NIMS)

The recommended process for international standardization consists of two stages: VAMAS (Versailles project on Advanced Materials And Standards) responsible for pre-standardization and ISO (International Organization for Standardization) responsible for international standardization. Since VAMAS/TWA 2 was established in 1985 as the technical work field (TWA) of VAMAS, it has promoted international joint testing and research in the pre-standardization stage of the surface chemical analysis field. ISO/TC 201 was established in 1991 as one of ISO's technical committees (TC) and is responsible for establishing international standards for surface chemical analysis. ISO/TC 201 has a history of being born from the international collaboration of VAMAS/TWA 2, and has a close liaison relationship with each other. Technological development in the field of surface chemical analysis over the past 30 years has been remarkable, and various new measurement methods have appeared on the global market. In particular, the scanning probe microscope (SPM) method has grown significantly as a major method of surface chemical analysis, along with surface electron spectroscopy. The trend of international standardization of SPM in both VAMAS/TWA 2 and ISO/TC 201 and the recent trend are introduced.

1.Introduction

ISO/TC 201 (Surface Chemical Analysis) and ISO/TC 202 (Microbeam analysis) were established in 1991. The establishment of both TCs was led by members of JSPS's 141st Committee for Microbeam Analysis. JSPS stands for the Japan Society for the Promotion of Science. For this reason, the "International Standardization Committee for Surface Chemistry Analysis Technology (JSCA)" was established in 1992 as a domestic deliberative body corresponding to both TCs under the umbrella of the Japan Industrial Standards Committee (JISC). Approximately 80 researchers and engineers in the field of analysis and measurement representing Japan from national research institutions, universities, and companies participate in JSCA. The surface chemical analysis under the jurisdiction of TC 201 is an analytical method for investigating the chemical state and composition at the surface and interface of a material with atomic layer level resolution. Auger electron spectroscopy, X-ray photoelectron spectroscopy, secondary ion mass spectrometry, glow discharge spectroscopy, scanning probe microscopy, X-ray reflectometry and X-ray fluorescence analysis, biomaterial surface analysis, etc. are included. The author is the representative director of JSCA and the JISC representative for ISO/TC 201.

At the G7+EU Summit held in Versailles, France, in 1982, it was agreed to promote the Versailles Project on Advanced Materials and Standards (VAMAS). Since then, VAMAS is the only Versailles project that has continued for nearly 40 years. VAMAS membership was expanded in 2008, and emerging areas such as Brazil, Mexico, Chinese Taipei, South Africa, Australia, Korea, and India joined. In 2013 China also participated. Figure 1 is a group photo of the 44th VAMAS Steering Committee (SC44) held at the National Institute of Standards and Technology (NIST) in Boulder, USA in 2019. I am participating in the VAMAS Steering Committee as one of the representative members of Japan, and I would like to contribute positively to the operation of VAMAS as a whole. The 2020 VAMAS Steering Committee (SC45) was planned to be held in Japan but postponed to 2021 due to the global Covid-19 infection.

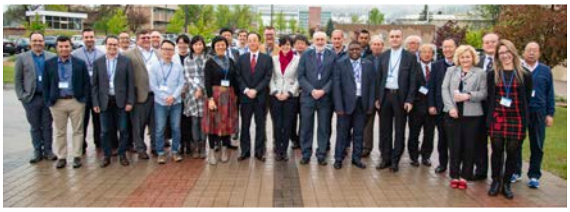

Figure 1. Group photo of the 44th VAMAS Steering Committee

---

*E-mail: FUJITA.daisuke@nims.go.jp

VAMAS/TWA 2 is one of the four working groups (TWA) that were initially approved for installation, and surface chemistry analysis is their area of responsibility. VAMAS/TWA 2 is the Technical Working Area that has been most actively promoting pre-standardization research at VAMAS since its establishment in 1985. Figure 2 shows its organizational structure and its relationship with ISO/TC 201. Dr. I. Gilmore of the National Physical Laboratory (NPL), UK is the chair and Dr. C. Clifford (NPL) is the vice-chair. VAMAS/TWA 2 conducts an international joint test for pre-standardization of quantification, sensitivity, resolution, etc. related to surface chemical analysis methods through a round-robin test (RRT) between laboratories in each country. Due to the wide variety of surface chemical analysis methods, four program themes have been set up: (1) Mass Spectrometry, (2) Scanning Probe Microscopy, (3) Electron & Optical Spectroscopy, and (4) Data Workflow, Methods, and Best Practices. For each theme, Dr. J. Bunch (NPL), the author (NIMS), Dr. A. Shard (NPL), the author and Dr. J. Lau (NIST, USA) are managing the RRT projects as the theme chairs. NIST The fourth data-driven metrology field was established in 2018 and its official name is“Data workflow, methods, and best-practices for multimodal spectroscopy and hyperspectral imaging”. This is a cross-cutting field of surface chemistry analysis methods utilizing data science. It corresponds to the multidimensional spectrum imaging type surface chemical analysis methods that are dramatically becoming big data.

This paper introduces the trends of international standardization of scanning probe microscopy (SPM), which I have been in charge of, focusing mainly on activities in VAMAS/TWA 2/SPM and ISO/TC 201/SC 9 (SPM) and recent topics.

Figure 2. VAMAS/TWA 2 Structure: Four major fields, their coordinators, and liaison with ISO/TC 201

2. Background of SPM standardization

The scanning tunneling microscope (STM) was invented in 1981 by Binnig and Rohrer *et al.* 1). They received the Nobel Prize in Physics in 1986 for their achievements in the design and development of STM, a microscope that visualized surface atoms. It should be noted that the pioneering development was done by Young *et al.* of NIST before the invention of STM. In 1971, Young *et al.* demonstrated that the surface topography can be microscopically measured by using an electric current between the surface and the probe using a device called a Topografiner 2). At that time, atomic resolution could not be realized, but it was a pioneering measurement device equipped with the main components of STM. In 1986, the atomic force microscope (AFM) was invented by Binnig, Quate, and Gerber as a microscope applicable not only to conductive samples but also to insulators 3). After that, various types of SPM were developed, and nanoscale measurement of various surface physical quantities was realized. The SPM measuring head is easy to downsize and has excellent environmental compatibility. It can be used in various environments and extreme fields such as in liquid, ultra-high vacuum, extremely low temperature, high temperature, high magnetic field, stress field, and so on 4). It is also applied as a nanotechnology processing tool such as atom manipulation, nanodot creation, nanolithography, periodic structure control, etc. by utilizing the close interaction between the probe and surface atoms 5).

Among the SPMs, AFM has been successfully applied to insulators and has succeeded in forming a globally growing market as a general-purpose nanoscale surface measurement method. It is used not only for research and development but also as an inspection device in the industrial field. The STM can measure the electronic state of the surface of a conductive sample with atomic resolution, and it has formed a market next to AFM as a high-end ultra-high vacuum STM system.

The United States is the first to respond to SPM standardization, and standardization as a guide document has been developed in ASTM since the 1990s 6). The SPM pre-standardization project at VAMAS started around 2002. First, TWA 29 (Materials Properties at Nanoscale) was established under the initiative of NIST, and an international RRT such as a method for calibrating the spring constant of the cantilever probe was conducted. On the other hand, TWA 2, which oversees surface chemical analysis, started a project related to SPM standardization around 2011.

Attempts to quantify AFM as a dimensional measurement method from the standpoint of metrological standards have been carried out since the latter half of the 1990s, mainly by researchers at the National Metrology Institute (NMI) around the world. International joint research on length measurement such as nanoscale step measurement and pitch measurement by AFM has been started 7). It was implemented from the standpoint of measurement standards as a project of the Consultative Committee for Length of CIPM (Comité international des poids et mesures).

3. ISO/TC 201 SPM international standardization

With the spread of SPM as a general-purpose analysis tool and its various derivations, the need for standardization of terminology increased. At that time, the technical terms used for SPM were not unified among vendors, and different names were used for the same method. As the SPM market expands worldwide, the unification of terminology with industrial application in mind has become a top priority, and early development of international standards has become desirable. From this perspective, SC9 was established in 2004 as a Sub-Committee in ISO/TC 201 that oversees SPM. In TC201, SPM is positioned to promote international standardization as one of the surface chemical analysis methods.

In the process chart for SPM international standardization proposed by the authors in 2006, the highest priority work item was standardization of SPM terms 8). Standardization was promoted with Dr. Seah (NPL) as the project leader in SC1, which oversees standardizing terms in TC201, and was published in 2010 as ISO 18115-2. This standard contains abbreviations related to SPM, definitions of SPM methods, definitions of terms related to SPM and contact mechanics, and acronyms. On the other hand, technological development of SPM is extremely active, and new measurement modes and probes are being developed one after another. The already established ISO 18115 was revised as early as 2013 to incorporate technological progress. In addition, in order to improve the development of the SPM industry and the convenience of users in Japan, JIS for ISO 18115 was formulated mainly by JSCA SPM-WG members. It was published in 2017 as JIS K 0147-2: 2017.

As shown in the process chart of Fig. 3, international standardization is considered to develop into two direction vectors starting from the term standard. One is the direction regarding data management such as data format and image processing. The other is the direction regarding quantification of calibration methods and reference materials. In TC201, SC3 oversees data management. Since each SPM vendor uses its own data format, there is no data compatibility, making it difficult to compare data. Therefore, the author proposed the standardization of the SPM data transfer format from JISC as a project leader. The international standard was published in 2011 as ISO 28600:2011. It is expected that it facilitates the development of a data processing program that is compatible with data interchangeability and contributes to the improvement of quantitative analysis. In the next stage of data management, standardization of data processing methods such as probe shape evaluation and image correction will be promoted 9). The goal is to build an SPM data platform integrated with a common data processing environment.

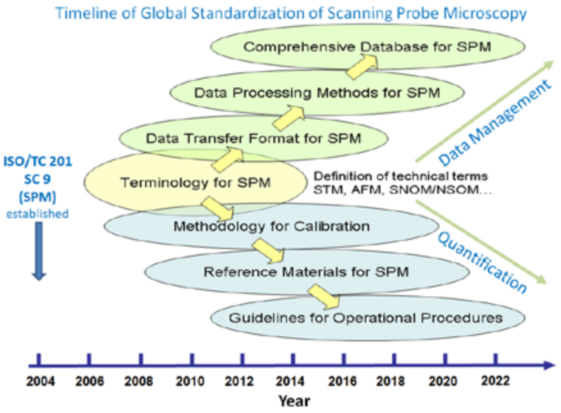

Figure 3. International Standardization Timeline in SPM

4. Recent trends in SPM standardization

An essential artifact in surface topography measurement by AFM is caused by the finite size of the probe tip. AFM topography imaging can be expressed by "dilation" operation in mathematical morphology 10). The AFM topography image z(x, y) corresponds to the surface image s(x, y) dilated by the probe shape function (PSF) t(x, y). An AFM topography image containing such artifacts can be partially reconstructed by "erosion" operation in mathematical morphology. As a result, it is possible to extract the approximate image r(x, y) of the true surface topography. However, it should be noted that the erosion process is accompanied by an unreproducible area unlike deconvolution. On the other hand, it is also possible to extract the tip shape of the probe from AFM topography imaging of a reference material (RM) with a known nanoscale shape [11)]. By promoting standardization of the probe evaluation protocol and RMs, it is possible to obtain a probe characteristic function (PCF) and PSF that quantitatively evaluate the tip shape of the probe [12)]. PCF is a quantitative index of the sharpness of the probe tip along with the other probe characteristics such as radius of curvature and cone angle. By performing correction processing using PSF, it is possible to reconstruct a topography image close to the true one. Measurement of the probe shape function and reconstruction processing are indispensable for the advancement of CD measurement that quantitatively evaluates the dimensions and shape of nanodevices.

From the above viewpoint, “Guideline for Restoration Procedure for AFM Images Dilated by Finite Probe Size” was proposed by the author to ISO/TC 201/SC9 in 2019. It was voted and adopted by the majority. This new work item (NP 23729) was proposed by JISC after validating the protocol based on RRT (A15: Reproducible Restoration Methodology for AFM Topography Images using Probe Shape Function) in VAMAS/TWA 2. In the future, the author will promote the formulation of the International Standard document with the experts from each country as the project leader.

Figure 4. Group photo at the 28th ISO TC201 General Assembly in 2019 (Tsukuba Conference, Japan)

5. Summary

In October 2019, the 28th ISO/TC 201 Plenary Meeting, Subcommittee Meeting, and the VAMAS/TWA 2 Annual Meeting were held at the Tsukuba International Congress Center in Tsukuba City, Ibaraki Prefecture, Japan. As shown in Figure 4, about 70 delegates from Europe, North America and Northeast Asia participated. JSCA-JISC sent a delegation of 30 people to the TC 201 plenary meeting and satellite meetings (10/30 to 11/2). In hosting the ISO/TC 201 plenary meeting, JSCA-JISC prepared beforehand and operated it on the day. We received great support from the NIMS International Standardization Committee and the RCAMC of NIMS. In international standardization activities in the SPM field, most proposals from Japan on new work items were made. Japan's role in leading the world in advanced measurement and analysis is becoming increasingly important.

Seven experts from JSCA-JISC participated in the SC9 meeting in charge of SPM standardization. SPM is in the stage of maturing from qualitative analysis to quantitative analysis, and the participation of experts who deal with various methods is required more and more. In the future, standardization needs will be directed toward quantitative measurement of mechanical and electromagnetic properties of soft materials and devices. As part of the international collaboration conducted by industry, academia, and government, we would like to ask for your further cooperation in international standardization activities to evolve SPM into a quantitative nano-property analysis method.

References

1) G. Binnig and H. Rohrer, *Helv. Phys. Acta* **55**, 726 (1982).
2) R. Young, J. Ward and F. Scire, *Rev. Sci. Instrum*. **43**, 999 (1972).
3) G. Binnig, C.F. Quate and Ch.Gerber, *Phys. Rev. Lett*. **56**, 930 (1986).
4) H. X. Guo and D. Fujita, *Rev. Sci. Instrum*. **82**, 123706 (2011).
5) D. Fujita and K. Sagisaka, *Sci. Technol. Adv. Mater*. **9**, 013003 (2008).
6) ASTM E1813-96, “Standard Practice for Measuring and Reporting Probe Tip Shape in Scanning Probe Microscopy”, (1996).
7) T. Kurosawa, J. Surf. Anal. 11, 178 (2004).
8) D. Fujita, H. Itoh, S. Ichimura and T. Kurosawa, *Nanotechnology* **18**, 084002 (2007).
9) D. Fujita, K. Onishi and M. Xu, *J. Phys. Conf. Ser.* **159**, 012002 (2009).
10) M. Xu, D. Fujita and K. Onishi, *Rev. Sci. Instrum.* **80**, 043703 (2009).
11) C.M. Wang, H. Itoh, J.L. Sun, J. Hu, D.H. Shen, and S. Ichimura, *J. Nanosci. Nanotechnol*. **9**, 803 (2009).
12) K. Onishi and D. Fujita, *Anal. Sci*., **27**, 157 (2011).