Current Activities of SC4 Depth Profiling in ISO/TC201 Surface Chemical Analysis

Mineharu Suzuki
ULVAC-PHI, Inc., 370 Enzo, Chigasaki, Kanagawa 253-8522
mineharu_suzuki@ulvac.com

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ISO / TC201 (Surface Chemical Analysis) / SC4 is the subcommittee to work for standardization in depth profiling. The standard drafts of ISO 14606, TR 15969, and TR 22335 have been published in order to achieve a good depth resolution, to estimate a sputtered depth, and to convert a sputtering time to the sputtered depth by a sputtering rate. ISO/TC201/SC4 is also working to prepare new work items on “ion beam alignment” and “utilization of standard multiple layers”.

1. Resume of ISO/TC201

TC201 is the technical committee to discuss standardization on Surface Chemical Analysis, including the analysis techniques such as AES (Auger electron spectroscopy), XPS (x-ray photoelectron spectroscopy), SIMS (secondary ion mass spectrometry), SPM (scanning probe microscopy), GDS (grow discharge spectrometry), TXRF (total reflection x-ray fluorescence spectroscopy), and XRR (x-ray reflectivity). It also consists of sub-committees, corresponding to general issues, terminology, general procedures, data management, and depth profiling that is commonly needed for multiple techniques. The titles of SC’s (sub-committees) in TC201 are shown in Table 1.

The scope of TC201 is described as “Standardization in the field of surface chemical analysis. Surface chemical analysis includes analytical techniques in which beams of electrons, ions, neutral atoms or molecules, or photons are incident on the specimen material and scattered or emitted electrons, ions, neutral atoms or molecules, or photons are detected. It also includes techniques in which probes are scanned over the surface and surface-related signals are detected. Excluded: Scanning electron microscopy which is within the scope of TC 202. Note: With current techniques of surface chemical analysis, analytical information is obtained for regions close to a surface (generally within 20 nm) and analytical information-versus-depth data are obtained with surface analytical techniques over greater depths.”

Table 1 Titles of TC201 and its SC’s.

TC/SC/WG	Title
TC 201	Surface Chemical Analysis
SC 1	Terminology
SC 2	General Procedures
SC 3	Data Management and Treatment
SC 4	Depth Profiling
SC 5	Auger Electron Spectroscopy
SC 6	Secondary Ion Mass Spectrometry
SC 7	X-ray Photoelectron Spectroscopy
SC 8	Glow Discharge Spectroscopy
SC 9	Scanning Probe Microscopy
WG 2	Total Reflection X-ray Fluorescence Spectroscopy
WG3*	X-ray Reflectivity

*The establishment of WG3 was resolved at the 17th TC201 plenary meeting in Vevey, Switzerland, September 2008.

2. Structure of the SC4, depth profiling

The scope of SC4 is defined as “Standardization of methods for instrument specification, instrument calibration, instrument operation, data acquisition, and data processing used to determine composition versus depth with surface analytical techniques.”

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As of January 1, 2009, the membership of ISO/TC 201/SC 4 is 8 P-members (China, Hungary, Japan, Republic of Korea, Philippines, Russian Federation, UK, and USA), 12 O-members (Australia, Austria, Belgium, Brazil, Finland, France, Germany, India, Italy, Norway, Sweden, and Switzerland), and 3 Liaison organizations (IUPAC, IUVSTA, and VAMAS). Here P-member and O-member mean Participation member and Observer member, respectively, and P-members take an active part in a project with the right to vote in standardizing processes.

The secretariat of SC4 has been in Japan from the beginning and the successive chairmen have been Prof. Siegfried Hofmann (1992-1999, Max Plank Institute / National Research Institute for Metals), Dr. Kazuo Kajiwara (2000-2002, SONY Corp.), and Dr. Mineharu Suzuki (2003-now, NTT / ULVAC-PHI). SC4 consists of the two WG's (working groups): WG1 is working for "Definitions and Procedures" and WG2 is "Reference Materials" in that the convenors are MS and Dr. Dae Won Moon (KRISS, Korea), respectively.

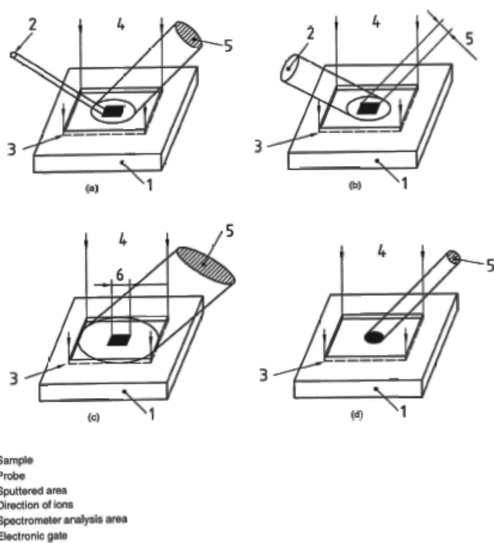

Fig. 1 Illustrated principle of alignment of primary beam, detected signal, and ion beam foe sputtering. (after ISO 14606)

3. Published drafts in SC4

Since 1992 this sub-committee has published the three ISO drafts.

The first one is "ISO 14606:2000, Surface chemical analysis -- Sputter depth profiling -- Optimization using layered systems as reference materials." This standard describes the important procedure to align the ion beam with primary electron/x-ray/ion beam to achieve a good depth resolution. Typical recommendations of alignment are shown in Fig. 1.

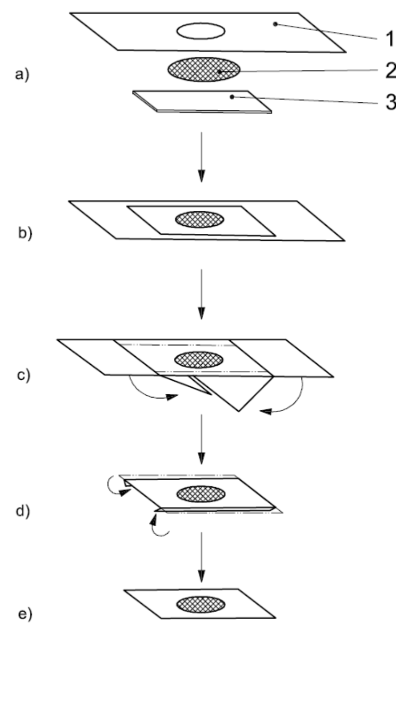

Fig. 2 Typical preparation procedure for mesh-replica method. (after ISO TR 22335)

The second is "ISO/TR 15969:2001, Surface chemical analysis -- Depth profiling -- Measurement of sputtered depth." This draft introduces how to estimate sputtered depths by many kinds of techniques including stylus profilometer, electron microscopes, ellipsometry, chemical analysis, and so on. It does not mention detailed measuring procedures for these techniques, however it cites 38 bibliographies and it is very useful as a reference book. The SC4 community has developed key technologies introduced in this draft. One of them is the following draft.

The third is "ISO/TR 22335:2007, Surface chemical analysis -- Depth profiling -- Measurement of sputtering rate: mesh-replica method using a mechanical stylus profilometer." This draft describes how to estimate the sputtering rate of the material of interest by measuring the replica pattern formed by sputtering through a metallic mesh. It is useful to apply the

sputtering rate of an actual measured material in data analysis, while the sputtering rate of a reference material such as SiO_2 is usually adopted. It was prepared as a IS (international standard) because its effectiveness was already shown by several researches for practical use, but published as a TR (technical report) due to judgment that the technique is still premature. Then the draft is described as the style of IS. One of the sampling procedures is shown in Fig. 2 and the example of traces of replica pattern is shown in Fig. 3.

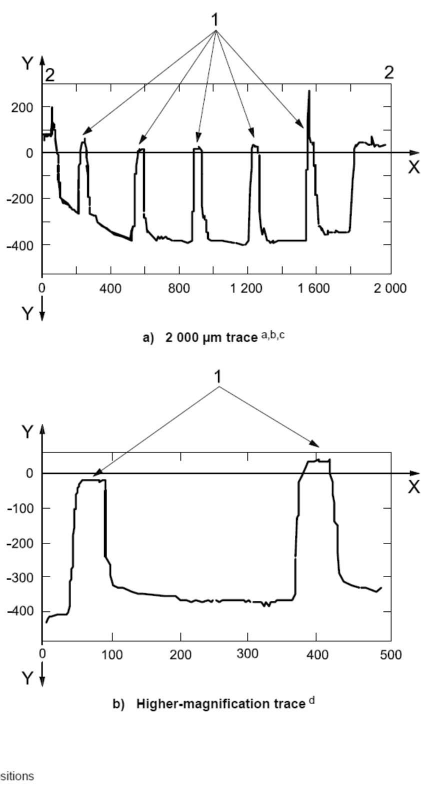

Fig. 3 Examples of morphology tracing of sputtered replica. (after ISO TR 22335)

4. Issues under discussions

Now SC4 is working for new work items in WG1 and WG2. One is "Surface Chemical Analysis – Depth Profiling - Guidelines for ion beam alignment" and the project leader is Dr. Isao Kojima (AIST) and the other is "A method for sputter rate determination in XPS and AES sputter depth profiling using multiple delta-layers" and the project leader is DWM. The former is the stage to prepare the working draft submission by the end of May 2009 and it will focus on the optimization of depth resolution. In the latter study group the RRT (round robin test) was performed to estimate layer thicknesses of an unknown specimen from the reference specimen that are Si/Ge multilayer materials. Measurement techniques applied are AES, XPS, and D-SIMS and analysis of reported results is being carried out.

5. Remarks

This report introduces the activity in ISO / TC201 (Surface Chemical Analysis) / SC4 (Depth Profiling). SC4 has published three ISO drafts and is working for new work item development. Depth profiling is one of the important analysis techniques to evaluate materials and devices under developing as well as troubleshooting and quality control in fabrication processes. SC4 will welcome any comments for standardization of depth profiling from worldwide scientists, engineers, and instrument operators.