Importance of VAMAS and ISO in Developing Reference Standards and Documentary Standards for Practical Surface Analysis

C. J. Powell and R. Shimizu*

Surface and Microanalysis Science Division, National Institute of Standards and Technology, Gaithersburg, MD 20899, USA
**Department of Applied Physics, Faculty of Engineering, Osaka University, Suita, Osaka 565, Japan*

(Received October 7 1998; accepted December 22 1998)

An overview is given of the development of reference standards by the Surface Chemical Analysis group of the Versailles Project on Advanced Materials and Standards (VAMAS) and of the development of documentary standards by Technical Committee 201 on Surface Chemical Analysis of the International Organization for Standardization (ISO).

1. Introduction

Surface analysis is being successfully used for the solution of a great variety of scientific and technological problems. Nevertheless, analysts currently face two challenges. First, there is increasing pressure to cut costs and to provide timely services. While the analytical techniques in common use are rather simple in concept, the instrumentation is often complex and there are many choices to be made in modes of instrument operation and in methods of data analysis. Tradeoffs have to be made between sensitivity, spatial resolution, data-acquisition time, and possible damage to specimens during analysis. Specimen materials may have complex morphologies. As a result, it can be difficult to perform surface analyses efficiently and reliably. A second challenge is that many surface-analysis laboratories are finding it desirable or necessary to seek registration under the ISO 9000 quality-management system or some other form of accreditation. Such laboratories need to document their laboratory procedures, the calibration of their equipment, and their measurement uncertainty. It is now not often possible or easy to document the accuracy of surface analyses due to the lack of accepted calibration procedures and the limitations of current methodology and data [1].

It is convenient to consider two types of standards here [2]. The first type, reference or measurement standards, includes reference data, reference materials, and reference procedures which may be provided by individual scientists, groups of scientists, national laboratories, and companies. The second type, documentary standards, are developed usually by national or international standardization bodies. We will consider here the work of two complementary international groups that have been formed to develop reference and documentary standards for surface analysis. These groups are Technical Working Area 2 on Surface Chemical Analysis of the Versailles Project on Advanced Materials and Standards (VAMAS) and Technical Committee (TC) 201 on Surface Chemical Analysis of the International Organization for Standardization (ISO). A brief summary will be given of the reference standards work by the VAMAS group and of the documentary standards activities of ISO/TC 201.

2. Development of Reference Standards by the VAMAS Surface Chemical Analysis Group

VAMAS, a cooperative program among seven member states (Canada, France, Germany, Italy, Japan, United Kingdom, and

the United States of America) and the European Economic Community (EEC), was established in 1982 to support trade in high-technology products by providing the technical basis for drafting codes or practice and specifications for advanced materials. Cooperative projects usually involve scientists from two or more of the member states and the EEC although scientists from other countries may be permitted to participate.

The VAMAS surface analysis group began work in 1984 with the objective to produce, by coordinated effort, the reference procedures, reference data, and reference materials necessary to establish standards for surface chemical analysis. Table 1 shows the current list of 36 projects [3]. These projects can be classified into five main sectors [4]: development or validation of the basic theory for the physical processes involved in the commonly used analytical techniques; understanding and calibrating the measuring instruments; development or validation of software for data reduction and communication; development of reference materials; and development of reference data.

Further information on the VAMAS projects and project leaders can be obtained from the chairman of the group [5].

3. Development of Documentary Standards by ISO/TC 201 on Surface Chemical Analysis

The ISO committee (ISO/TC 201) was formed in 1991 and began meeting on an annual basis in 1992. The scope of this committee is: standardization in the field of surface chemical analysis 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 [6].

ISO/TC 201 has eight subcommittees: Terminology (SC1); General Procedures (SC2); Data Management and Treatment (SC3); Depth Profiling (SC4); Auger Electron Spectroscopy (SC5); Secondary Ion Mass Spectrometry (SC6); X-Ray Photoelectron Spectroscopy (SC7); and Glow Discharge Spectroscopy (SC8). Currently, a total of fifteen working groups have been established to work on specific tasks [6].

According to ISO Directives, all national standards organizations have the right to participate in the work of ISO technical committees and subcommittees as either participators (P-members) or observers (O-members). P-members have the right to nominate experts to working groups and to vote on proposed standards. ISO/TC 201 and its subcommittees have also established liaisons with the VAMAS surface chemical analysis group and two other international organizations (the Applied Surface Science Division of the International Union of Vacuum Science, Technique and Applications and Commission I.6 on Colloid and Surface Chemistry including Catalysis of the International Union of Pure and Applied Chemistry). These liaisons provide a convenient mechanism for participation in working groups by experts in countries that are not P-members of ISO/TC 201.

Table 2 shows the titles of one documentary standard developed by ISO/TC 201 and the titles of 10 other standards under development. Each potential standard is referenced by a unique number and its stage of development (final draft international standard (FDIS), draft international standard (DIS), committee draft (CD), or working draft (WD)). The first standard approved by ISO/TC 201 (ISO 14976) is a data transfer format that had been initially examined as a VAMAS project (project 10 in Table 1).

Table 3 shows the titles of 19 proposed documentary standards that are currently under consideration by ISO/TC 201. Many of the potential and proposed documentary standards listed in Tables 2 and 3 are closely related to the VAMAS projects listed in Table 1. Some of the documentary standards have been based on VAMAS projects while, in other cases, VAMAS projects have been initiated to provide the needed reference standards.

Further information on ISO/TC 201 documentary standards and on the project leaders for the standards can be obtained from the ISO/TC 201 Secretariat [7].

4. Summary

A synergistic relationship has grown between the VAMAS Surface Chemical Analysis group and ISO/TC 201. The VAMAS group has been active in the development of reference standards, and the resulting reference data, reference materials, and reference procedures have naturally been incorporated into ISO/TC 201 documentary standards. As needs for new documentary standards have been identified, it has been convenient and appropriate for the VAMAS group to evaluate proposed procedures (e.g., by interlaboratory comparisons), to provide needed reference data, and to evaluate needed reference materials.

5. References

[1] C. J. Powell and M. P. Seah, J. Vac. Sci. Tech. A 8, 735 (1990).
[2] M. P. Seah, Phil. Trans. Roy. Soc. (London) A354, 2765 (1996).
[3] M. P. Seah (private communication).
[4] M. P. Seah, Surf. Interface Anal. 19, 247 (1992).
[5] M. P. Seah, National Physical Laboratory, Teddington, Middlesex TW11 0LW, UK.
[6] C. J. Powell and R. Shimizu, Surf. Interface Anal. 25, 860 (1997).
[7] T. Ito, Japanese Standards Association, 4-9-22 Akasaka, Minato-ku, Tokyo 107-0052, Japan.

Table 1. Projects of the VAMAS Surface Chemical Analysis Technical Working Area 2 on Surface Chemical Analysis [3].

1.	Development of thin oxide films as reference materials
2.	Development of calibration data for the energy scales of Auger-electron spectrometers
3.	Procedures for quantitative x-ray photoelectron spectroscopy (XPS)
4.	Measurement of spatial resolution in Auger-electron spectroscopy (AES)
5.	Development of reference materials prepared by ion implantation
6.	XPS intensity calibration and stabilization with polymeric reference materials
7.	Correction methods for backscattering in AES
8.	Reference data for sputtering rates in oxides
9.	Intercomparison of Auger-electron energy and intensity measurements
10.	Development of a standard data transfer format
11.	Multitechnique characterization of vacancies in alumina
12.	Calibration of surface layers by nuclear reaction analysis
13.	Tests of algorithms for data processing in AES – factor analysis and intensity
14.	Tests of algorithms for background subtraction in XPS and AES and for quantitative XPS by peak and background shape analysis

15. Evaluation of sensitivity factors for sputtered neutral mass spectrometry

16. Intercomparison of surface analyses of thin aluminum oxide films

17. Quantitative AES of Au-Cu alloys

18. Evaluation of LOGIT, an algorithm for fitting sputter-depth profile data, for the measurement of interface widths of a NIST thin-film reference material

19. Round-robin measurements of impurities in GaAs crystals measured by secondary ion mass spectrometry (SIMS)

20. Round-robin AES measurements of Co-Ni alloys

21. Tests of algorithms for the analysis of multicomponent spectra in XPS

22. Test of the calibration stability of channel electron multiplier detection efficiencies

23. Absolute calibration of XPS-instrument intensity scales

24. Conventions for spectral data bases

25. Quantitative XPS of Au-Cu alloys

26. Theoretical assessment of electron escape depths

27. Multiline reference material for differential AES intensity calibration

28. Quantitative XPS of Au-Cu alloys

29. Development of a file format translation system

30. Development of a common data processing system for AES and XPS

31. Evaluation of the accuracy of measurement of thicknesses of thin oxide films by XPS

A1. Evaluation of the infinite velocity method for SIMS quantification

A2. Evaluation of static charge stabilization and determination methods for XPS measurements on nonconducting samples

A3. Interlaboratory study of static SIMS repeatability and reproducibility

A4. Evaluation of multilayer reference coatings for quantitative depth profiling by glow-discharge optical emission spectrometry

A5. Interlaboratory study of the degradation of organic materials by X rays in XPS

Table 2. Documentary standards developed or under development by ISO/TC 201 (as of December, 1998). Information is given in parentheses on the particular subcommittee (SC) and working group (WG) of the subcommittee that is responsible for the standard.

ISO 14976	Surface Chemical Analysis – Data Transfer Format (SC3/WG1)
FDIS 14237	Surface Chemical Analysis – Secondary Ion Mass Spectrometry – Determination of Boron Content in Silicon Using Uniformly Doped Materials (SC6/WG1)
DIS 14606	Surface Chemical Analysis – Sputter Depth Profiling – Optimization Using Layered Systems as Reference Materials (SC4/WG2)
DIS 15472	Surface Chemical Analysis – X-Ray Photoelectron Spectrometers – Calibration of Energy Scales (SC7/WG2)
CD 14975	Surface Chemical Analysis – Data Information Formats (SC3/WG1)
CD 15969	Surface Chemical Analysis – Sputter Depth Profiling - Measurement of Sputtered Depth (SC4/WG1)
CD 15470	Surface Chemical Analysis – X-Ray Photoelectron Spectroscopy – Description of Selected Instrumental Performance Parameters (SC7/WG1)
CD 15471	Surface Chemical Analysis – Auger Electron Spectroscopy – Description of Selected Instrumental Performance Parameters (SC7/WG1)
CD 14707	Glow Discharge Optical Emission Spectrometry (GDOES)– Introduction for Use (SC8)
CD 14706	Surface Chemical Analysis – Test Method of Surface Elemental Contamination on Silicon Wafers by Total Reflection X-Ray Fluorescence Spectroscopy (TC 201/WG2)
WD 15760	Surface Chemical Analysis – Data Management and Treatment – Data Dictionary for Surface Science Spectral Databases (SC3/WG2)

Table 3. Titles of proposed standards under consideration by ISO/TC 201 (as of December, 1998).

Surface Chemical Analysis – Vocabulary (SC1/WG2)

Surface Chemical Analysis – Handling of Specimens Prior to Analysis (SC2/WG1)

Surface Chemical Analysis – Preparation and Mounting of Specimens for Analysis (SC2/WG1)

Surface Chemical Analysis – Ion-Implanted Surface-Analytical Reference Materials – Procedure for Standardizing the Retained Areic Dose in a Working Reference Material (SC2/WG2)

Surface Chemical Analysis – Auger-Electron Spectroscopy – Reporting of Surface Analytical Data - Spectra (SC2/WG3)

Surface Chemical Analysis – X-Ray Photoelectron Spectroscopy – Reporting of Surface Analytical Data - Spectra (SC2/WG3)

Surface Chemical Analysis – Auger Electron Spectroscopy and X-Ray Photoelectron Spectroscopy – Guidelines on the Use of Experimental Relative Sensitivity Factors for the Quantitative Analysis of Homogeneous Materials (SC5/WG1)

Surface Chemical Analysis – Auger Electron Spectroscopy and X-Ray Photoelectron Spectroscopy – Peak Intensity Determination (SC5/WG1)

Surface Chemical Analysis – Auger Electron Spectroscopy and X-Ray Photoelectron Spectroscopy – Guide to Smoothing Data (SC5/WG1)

Surface Chemical Analysis – Secondary Ion Mass Spectrometry – Method for Depth Profiling of Boron in Silicon (SC6/WG1)

Surface Chemical Analysis – Secondary Ion Mass Spectrometry – Determining Relative Sensitivity Factors from Ion-Implanted Reference Materials (SC6/WG2)

Reporting Sputter Depth Profile Data from Secondary Ion Mass Spectrometry (SC6/WG2)

Reporting Mass Spectral Data from Secondary Ion Mass Spectrometry (SC6/WG2)

Reporting Imaging Data in Secondary Ion Mass Spectrometry (SC6/WG2)

Surface Chemical Analysis – Secondary Ion Mass Spectrometry – Procedure for Evaluating SIMS Depth Resolutions with Delta Multi-layer Reference Materials (SC6)

Surface Chemical Analysis – Medium Resolution Auger Electron Spectrometers –Calibration of Energy Scales for Elemental Analysis (SC7/WG2)

Surface Chemical Analysis – High Resolution Auger Electron Spectrometers –Calibration of Energy Scales for Elemental and Chemical State Analysis (SC7/WG2)

Surface Chemical Analysis – X-Ray Photoelectron Spectroscopy - Guidelines for Estimating Unintended Degradation in a Material (SC7/WG3)

Determination of Thickness and Chemical Composition of Zinc-Based Metallic Coatings by Glow Discharge Optical Emission Spectrometry (SC8)
