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M. P. Seah

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Standards for surface analysis: a clash between bureaucracy and science?

BY M. P. SEAH

*Division of Materials Metrology, National Physical Laboratory, Teddington,
Middlesex TW11 0LW, UK*

Scientists at the frontiers of research are often disturbed by the threat that they themselves may have to conform to standards or that they may have to use standards which curtail their freedom of action. Hence the perceived 'clash' of the title. It is important to recognize that there are two main classes of standards: (i) reference standards, with traceability to the SI system of measurement; and (ii) documentary standards, established through approved bodies with due consultation with relevant experts. In the first class of standard, it is clear that very few measurements can be meaningfully transferred from laboratory to laboratory without a degree of traceability. Furthermore, as the traceability increases, the value of networking increases and cooperative activity becomes more constructive. The value of traceability will be illustrated by developments in electron spectroscopy which make data-basing activities very powerful and which allow crucial tests of theory. In the second class, standards are now being established through ISO. Properly constituted, two aspects deserve discussion. Firstly, documentary standards may embody a distillation of our expertise which may reach a far greater body of users than most other written articles and, secondly, these standards enable us to do our own research more effectively.

1. Introduction

This paper is an attempt to place in perspective the above title, seen as an important concern by the conference organizers.

Pure research in universities usually involves intense work by one or a small group of scientists who know more about their own topic area than anyone else. Applied research involves scientists interacting both upstream and downstream from their own activities. Development and product definition involves similar interactions together with interactions across to engineers and people in other disciplines. As we move along this series, we recognize a need to interact more and more with other professionals whose primary expertise is more and more disparate. These interactions require communication and this is best achieved with a common terminology and common measurement system. These two aspects form the mainstay of two archetypal forms of standards: documentary standards and reference standards. We shall later consider both and their current status for surface chemical analysis. The more one has to be involved with more disciplines and other research laboratories, the more useful standard interfaces, ways of working, terms and definitions of algorithms are found. Despite this, the fear expressed in the title selected for this

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presentation is a very valid fear that individuals will be required to work in conformance with a documentary standard with which they do not agree. This same fear was the theme behind the recent Institute of Physics symposium entitled 'Total quality management—imposition or opportunity' (IOP 1995).

Reference or measurement standards, called *étalons* in French, have a wholly scientific basis. Documentary standards, called *normes* in French, are 'documents established by consensus and approved by a recognized body, that provides, for common and repeated use, rules, guidelines or characteristics for activities or their results, aimed at the achievement of the optimum degree of order in a given context' (BSI 1991). Consensus is defined as 'general agreement, characterized by the absence of sustained opposition to substantial issues by any important part of the concerned interests and by a process that involves seeking to take into account the views of all parties concerned and to reconcile any conflicting arguments. Consensus need not imply unanimity'. Already we can see some grounds for concern that may be expressed by individuals since a lack of unanimity means that someone may not be satisfied. However, even so there should still be optimum community benefits.

More serious grounds for concern occur when documentary standards are pushed strongly by experts wishing to establish a standard incorporating the frontier knowledge that they have just developed in the last week! This will generally date rapidly and be of interest to a very limited group. Also worrying is the standard which is devised for a particular purpose and which is generalized by the promulgator to have a much wider scope than is warranted. It is not uncommon to read research papers on a particular widget in which the conclusions and abstract are written as if the results apply to *all* widgets. An otherwise good standard with a poorly worded scope may thus be misapplied to the detriment of all. The mis-application of standards can also inhibit development since a worthwhile new product or concept may fall outside the framework of a given standard used for specification in commercial transactions, whilst actually being more effective for the required end use.

Part of the reason for concern is that it is recognized that standards are very powerful precisely because they are part of our everyday life and that when they are used we expect them to work perfectly. The plugs and sockets, the ultra-high vacuum fittings, hardware and software in computing may all be purchased from different vendors in the knowledge that compatibility exists. Without such standards progress would be severely impeded.

In order that standards are well founded, particularly at the International Standards Organisation (ISO) level, many checks and consultations occur. However, this process becomes unproductive unless the standard is launched with certain considerations, admirably listed by Spanner (1995) as follows.

Standardization is appropriate only when the industrial need is sufficient to justify the effort.

A standard should not be written until an adequate technology and experience base is available.

Standards should only be developed by groups with a balanced representation of users and suppliers.

Industrial standards should emphasize the interests of the user because the supplier intrinsically possesses a greater knowledge of the technology.

In the second item, the technology and experience base should fully cover the content of the proposed standard and not just be associated with it. For this precise reason the general programmes of the VAMAS technical working activities (TWAs)

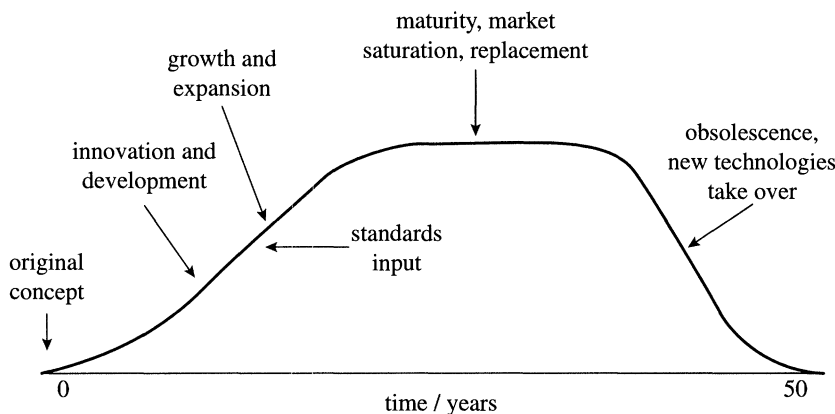


Figure 1. Typical schematic of the development of a trade or product cycle.

were launched. Details of the activities in the surface chemical analysis TWA are given by Seah (1990a).

Timeliness is critical. A standard developed too early will embody obsolete concepts. A standard developed too late will have to unite the *ad hoc* standards that individuals or groups have had to develop for their own purposes. In all product fields there is a maturing process (Kondratieff 1935) which runs from innovation through development to product launch and refinement with market growth. Next, as shown in figure 1, comes market saturation with falling profit margins and replacement products at lower price and eventually decline as other sectors take over. Such cycles may vary in duration typically from 30 to 100 years but, in general, the correct time for initiating standards development is during the stages of market growth where trends are clear but *ad hoc* standards have not crystallized. Surface analysis started for all of the major techniques around 1970 (Seah & Briggs 1990) and so, after 25 years of development, we are well into the period where standards should have been established.

At the present time, as we shall discuss in the next sections, in surface analysis a considerable number of measurement standards and documentary standards (ASTM 1994) do exist and are used on a voluntary basis. To the author's knowledge, the only public standards to which surface analysts currently conform, are the important quality systems such as ISO 9000 (1987), the National Measurement Accreditation System (NAMAS), etc. These systems, which will involve relevant surface chemical analysis standards as they develop, are growing strongly. In the UK the number of ISO 9000 certifications grew from 18 577 in January 1993 to 28 096 by September 1993 and 36 823 by June 1994 (Verhoeven 1995). This growth will continue.

2. Measurement standards for surface analysis

Measurement standards involve reference data, reference methods and reference materials. The values of measurands, either in the data or for the reference materials, all need traceability to the SI system. This means that there is an unbroken chain of links between the measurand and the units of the SI system allowing the full uncertainty of the value of the measurand to be correctly deduced. In analysis, however, we do not always need the full traceability and this may be exemplified by the mass scale in mass spectrometers where the relative masses in amu are known

Table 1. *Absolute values of calibration XPS binding energies, in electronvolts, for core levels using Al or Mg K α unmonochromated radiation (after Seah 1989)*

	Al K α ^a	Mg K α
Cu 3p	75.14 \pm 0.02	75.13 \pm 0.02
Au 4f _{7/2} ^b	83.98 \pm 0.02	84.00 \pm 0.01
Ag 3d _{5/2} ^c	368.26 \pm 0.02	368.27 \pm 0.01
Cu L ₃ MM	567.96 \pm 0.02	334.94 \pm 0.01
Cu 2p _{3/2}	932.67 \pm 0.02	932.66 \pm 0.02
Ag M ₄ NN	1128.78 \pm 0.02	895.75 \pm 0.02

^aAl K α – Mg K α = 233.02 eV.^bAu 4f_{7/2} Al K α BE lowered by Au 4f_{5/2} tail.^cAg 3d_{5/2} Mg K α BE raised by Ag 3d_{3/2} X-ray satellite.

to 1 in 10⁹, and could be used at this accuracy (if required), whilst the value of the amu in kg is only known to 5 parts in 10⁷ (Cohen & Taylor 1990), i.e. we can often perform meaningful work at higher accuracy than the accuracy of the traceability to the SI system!

Below, we assess a necessarily small example selection of the measurement standards for the basic surface analysis techniques. In all spectroscopic techniques we need clear, validated physical laws by which we convert measurands into compositions (or whatever). Where those laws are incomplete we may need tables of parameters or data banks of spectra to assist interpretation. If so, we need to be sure that these data may be transferred from laboratory to laboratory with accuracy and, if non-trivial data processing is involved, that the software involved is properly validated.

(a) *X-ray photoelectron spectroscopy (XPS)*

X-ray photoelectron spectroscopy is used firstly in the analysis of surfaces to determine the chemical state of the species present, secondly to determine the average chemical composition in the region emitting the photoelectrons and thirdly to determine the variations of composition within that region. In the first topic, the chemical state is defined by measurements of the positions of the XPS peaks on the energy scale. These chemical state shifts range over some 5 eV but true assignment needs an energy scale accuracy of ± 0.1 to ± 0.2 eV. If we have a data bank of states and a spectrometer, both of which have a scale standard uncertainty of ± 0.4 eV, the state assignment cannot be made as there are too many chemical state choices, whereas at ± 0.1 eV, assignment of the state is usually unique. For this reason NPL developed a traceable binding energy calibration scheme, shown in table 1, with an accuracy of ± 0.02 eV (Anthony & Seah 1984a; Seah 1989), which reduced typical errors from ± 0.40 to ± 0.04 eV (Anthony & Seah 1984b). These energy calibrations have been implemented, some retrospectively, in the NIST XPS database, version 1.0, which contains 13000 line positions. This database is now in use world-wide and would not be either feasible or useful without an agreed and traceable method of calibrating the energy scales.

The second aspect needing attention is the intensity scale. It is well known that different instruments have different sensitivities but that does not matter if the data reduction involves normalization. What does matter are the changes to the relative

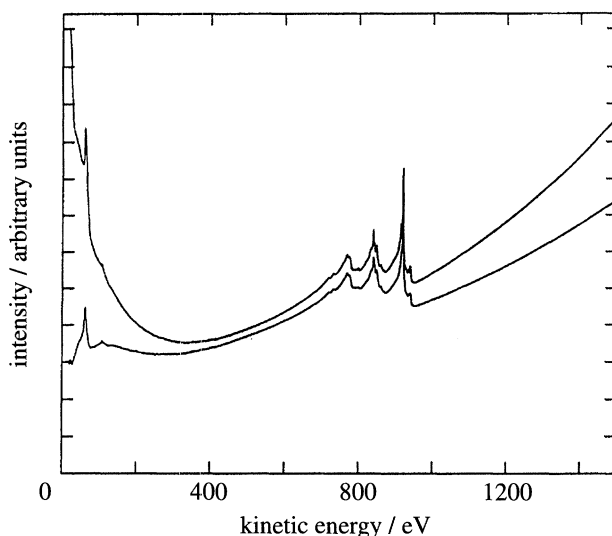


Figure 2. AES spectra from copper using two different instruments in the constant $\Delta E/E$ mode.

intensities of peaks at different energies. The different electron optical designs of spectrometers make this inevitable but it was not until 1993 that Seah (1993) provided a method based on detailed calibrations of the *metrology spectrometer* at NPL (Seah & Smith 1990) to calibrate the response functions and show that the different instruments varied by more than an order to magnitude over the energy range 200 to 1500 eV. For the use of relative sensitivity factors (Seah 1980; Moulder *et al.* 1992) or basic parameters such as photoionization cross sections (Scofield 1976; Band *et al.* 1979), asymmetry parameters (Reilman *et al.* 1976; Band *et al.* 1979) and electron inelastic mean free paths (Tanuma *et al.* 1994), these response functions are required. It is hoped that a service to provide these response functions routinely can soon be provided from NPL to accommodate this.

(b) Auger electron spectroscopy (AES)

The problems of the energy scale calibration in AES were not as urgent as in XPS, since AES has only recently begun to be used for chemical state identification. Here, by developing a second traceable calibration (Seah *et al.* 1990), Seah & Smith (1990a) were able to reduce the scatters in peak positions from ± 2.0 to ± 0.1 eV. The problems for the intensity scale, as in XPS, involved variations in the relative intensities of peaks by up to an order to magnitude, as shown in figure 2. Using a prototype calibration system, these variations could be reduced to a few percent (Seah & Smith 1991). This calibration system has now been refined to provide a fully traceable response function of Auger electron spectrometers in units of sr eV, as shown in figure 3, so that proper correlations may be made with theory.

At the present time, few analytical techniques can provide an analysis without recourse to calibration using reference samples. One of the few techniques is Rutherford backscattering spectrometry (Chu *et al.* 1978), where the intensities depend mainly on the scattering cross section (which may be calculated) and the detector geometry (which may be measured). Recent work for AES shows that a similar result is now possible in surface analysis. Auger electron intensities, I_A , for homogeneous

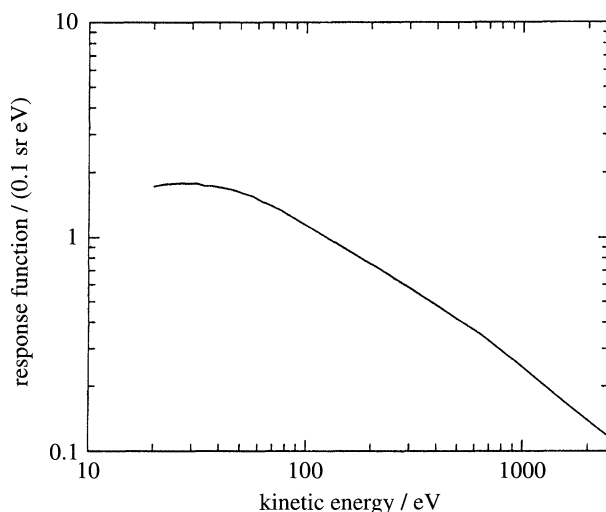


Figure 3. Calibration of a spectrometer response function for the constant ΔE mode at a resolution of 1 eV in absolute units, after Seah (1995).

pure solids are given by the relation (Seah 1990*b*)

$$I_A = I_0 \gamma_A \sigma_A \sec \alpha (1 + r_A) N_A Q_A \lambda_A \cos \theta \Omega / 4\pi, \quad (2.1)$$

where I_0 is the electron beam current, γ_A is the probability that the ionized core level decays by an Auger process (Burhop 1952), σ_A is the cross section for ionizing the core level (Gryzinski 1965), α is the angle of incidence of the electron beam, r_A is the backscattering enhancement (Shimizu 1983), N_A is the atomic density, Q_A is an elastic scattering correction term of approximately 0.9 (Jablonski 1989), λ_A is the inelastic mean free path (Tanuma *et al.* 1994) and θ is the angle of emission of the detected electrons from the surface normal into the effective solid angle Ω of the spectrometer. In practice, to allow for Coster–Kronig transitions, the intensities in equation (2.1) should be summed for all levels with the same principal quantum number. Figure 4*a* shows recent experimental measurements for all of the K, L or M transitions in our digital Auger data bank and figure 4*b* the equivalent predictions. The agreement is excellent despite the absence of any fitting or adjustable parameters. In our first attempts to compare theory and experiment a problem occurred in the region from the rare earth metals to the refractory metals, i.e. as the atomic number Z increased from 55 to 75. The original predictions shown by the solid dots in the lower frame in figure 5 show a step between Lu and Hf not seen in the experimental data of the upper frame. By inspection, it would not be clear if the series Ce to Lu was correct and Hf to Re in error or vice versa. However, comparison of the absolute values in the upper and lower frames indicates that the error arose from a progressive underestimate of the theoretical intensity through the series Ce to Lu. This is precisely the range over which the 4f electrons had been included in the calculations for λ_A . These electrons have too great an angular momentum to be readily excited and should be excluded from these calculations, as shown in figure 4*b*. Thus, the use of fully traceable calibrations allows a clearer validation of the detailed aspects of theory so that our understanding may proceed with firm foundations. The reader will find many correlations of limited data sets of parameters related to I_A with the product $\sigma_A(1+r_A)\lambda_A$ in the literature, where σ_A , r_A and λ_A are taken from

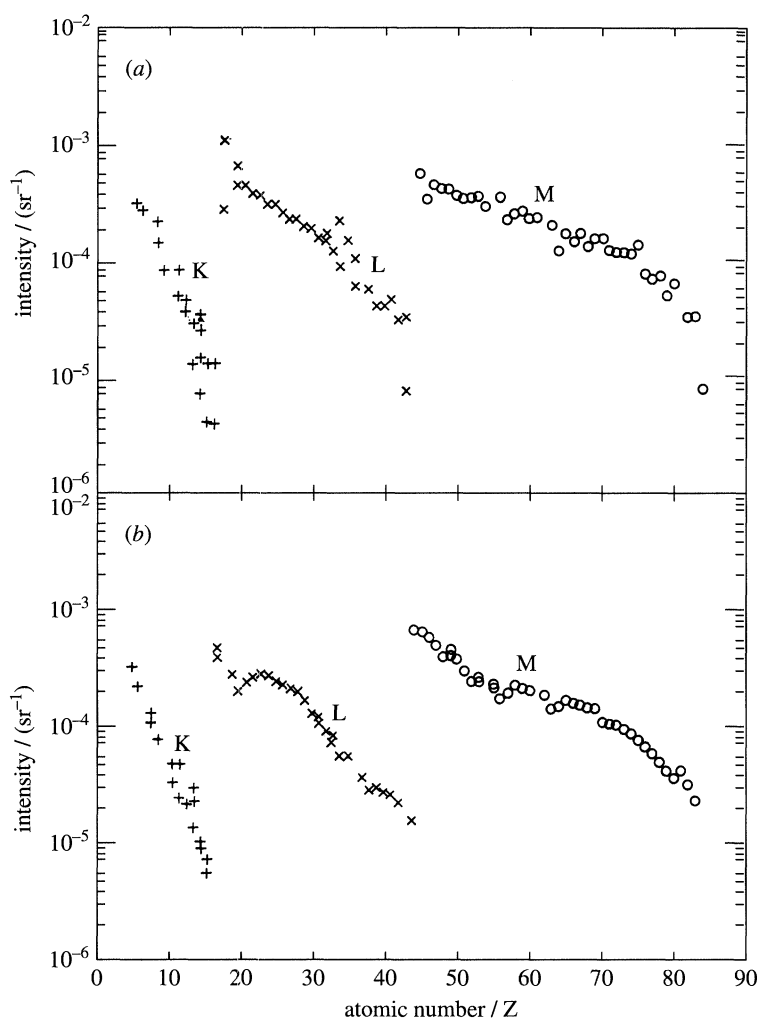


Figure 4. Auger electron peak areas for elements and compounds using a 5 keV electron incident at 30° to the surface normal as a function of atomic number: (a) experimental data; (b) theoretical predictions, for peaks at energies above 150 eV.

different sources but with sufficient undetermined fitting parameters, that there is little confidence that the fit would be valid outside the set of data that had been used in the fitting.

(c) Secondary ion mass spectrometry (SIMS)

The problems in both static and dynamic SIMS are very different from those in AES and XPS. Here, in static SIMS, the abscissa is mass and the masses of possible fragments have been determined with sufficient accuracy (Benninghoven *et al.* 1987) for all present needs from work in other fields. However, the intensity scale of the mass spectrometers has not been calibrated but it is often assumed that both magnetic sector and time-of-flight instruments have a full and constant transmission as a function of mass whereas the older quadrupole-based systems have poorer efficiency, especially at high masses. This may or may not be true but, in addition, the detectors used have an efficiency which is both dependent on the precise specie measured

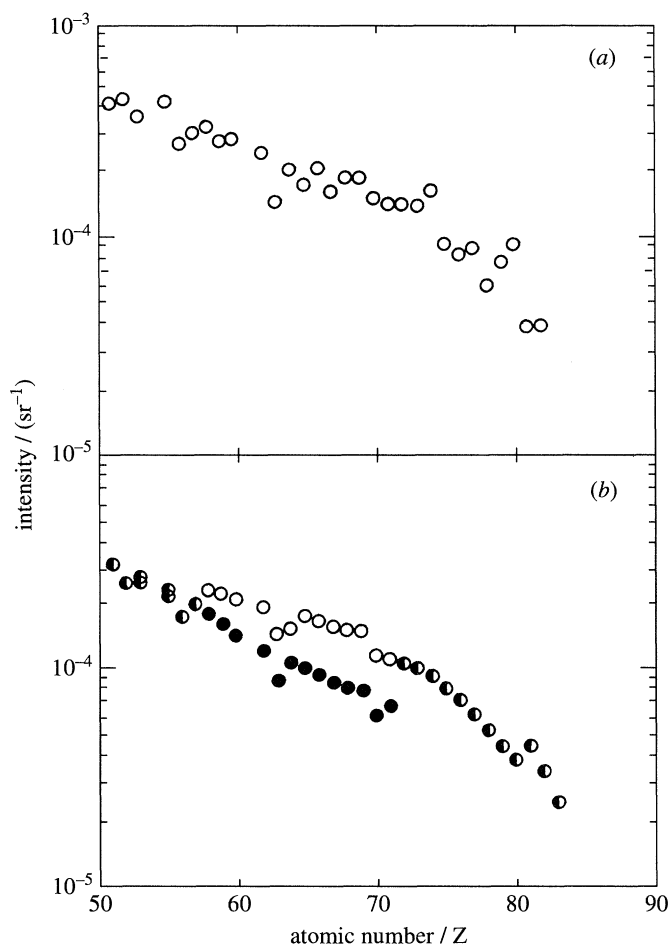


Figure 5. Detail of figure 4a showing the experimental data (upper) with the predictions using Tanuma *et al.*'s (1994) general equation as in figure 4b (lower). The solid points in the lower plot show the predictions using Tanuma *et al.*'s (1994) equation as presented without modification. The open circles show the exclusion of the 4f electrons from Ce to Lu and the half-filled points the unaltered predictions outside this range.

(Rudat & Morrison 1978) and sometimes the precise tuning of the spectrometer. This is an area where considerable future work is needed. At the present time, static SIMS is used to evaluate the major contaminants present on a surface by studying fingerprint spectra and comparing the results with handbooks (Briggs *et al.* 1989; Newman *et al.* 1991) or in-house standards. However, we do not know, at the present time, exactly how transferable the handbook data are. An interlaboratory exercise is urgently needed.

These early handbooks are much like the early AES and XPS handbooks which were ideal for helping in qualitative analysis but had strong limitations for quantitative work. These paper-based handbooks will shortly be complemented by a static SIMS digital database (Vickerman *et al.* 1995) in which the acquisition conditions have been more carefully standardized. This general field is particularly powerful for studying delicate materials in a wide range of developing areas and it is expected that the future contribution of standards will be particularly strong.

With dynamic SIMS the abscissa is usually sputtering time, to be converted, after the measurement, to depth. In its simplest approach, one would use the known current density of the ion beam and the sputtering yield from tables of data to convert the fluence to depth. Whilst this is possible, the errors in sputtering yield data (Matsunami *et al.* 1984) leave this as a method to be used only if all else fails. More commonly, the sputtering rate will be measured from the crater depth in the sample itself, particularly as much of the dynamic SIMS work is for depth profiling on semiconductor materials which have been polished to a high quality of flatness and smoothness. Whilst this is reasonably accurate for single phase samples, serious errors occur in multilayer materials where the sputtering rate changes from layer to layer (Barlow *et al.* 1992). For full accuracy in all materials many further corrections are needed. For rough samples of arbitrary matrix, the first simple and rather inaccurate approach, may however, be the only economically practical method.

Quantitative analysis for static or dynamic SIMS using theoretical equations without close reference material standards is likely to involve errors of a factor of 3 or more. Normally in dynamic SIMS, the quantification is achieved by comparing an unknown with a (certified) reference material with the same analyte/matrix combination, where the analyte in both the sample and the reference material is at concentrations well below 1%. This comparison removes much of the need to calibrate instrumental parameters. Reference materials of this type are available for ion-implanted doses of very many elements in Si, GaAs and InP (Newbury 1992). If accurate crater depth measurements are not available, the depth may be obtained from a match of the measured profile shape for the implant and that expected on the basis of transport of ions in matter or other simulation codes. These codes contain considerable data of the ion solid interactions and are used by many researchers. Some are regularly updated and are well maintained (Webb 1992), however, even careful researchers can find that they waste considerable effort using versions of these programs which have not been validated and which give different results to other versions (Vandervoort 1994, personal communication). This would not, of course, occur if the laboratories generating software were under full quality control, as required for ISO 9000. Software would then be under revision control and all changes to programs would be clearly documented within the revision control. One should be clear that quality systems such as ISO 9000 cannot comment on the quality of science involved in, say, a software program but it will make mistakes and errors less likely and, when they do occur, easier to track. The more complex the software and the more programmers involved, the more the need for a quality system.

In the above, through AES, XPS and SIMS, we have demonstrated how examples of measurement standards, in the form of reference data, reference procedures and reference materials, together with basic science, are the essential catalysts to the development and use of surface analytical techniques. These by themselves are inadequate without standard ways of doing the analyses, etc. We treat these aspects in the next section.

3. Documentary standards for surface analysis

Documentary standards range from the standard procedures that are written by analysts as in-house procedures to conform to the requirements of NAMAS accreditation, ISO 9000 certification, VAM (1995) or one of the other quality systems, to internationally agreed procedures established as ISO standards. In surface analysis,

published standards are currently available from ASTM (ASTM 1994), the International Union of Pure and Applied Chemistry (IUPAC 1972, 1987) and the surface chemical analysis technical working activity of the Versailles project on advanced materials and standards (VAMAS TWA 2). Activity to establish standards is now also strong in the ISO technical committee 201 on surface chemical analysis (Powell & Shimizu 1994).

The IUPAC standards mainly concern terminology. A clear set of definitions for the terms in common use is clearly vital for the accurate communication of ideas. ASTM E-42 committee also provides definitions for terms.

Before studying what documentary standards are available in surface chemical analysis, it is useful to see what types of activity occur in other areas. General quality systems such as NAMAS require, *inter alia* and where necessary, calibration of all equipment traceable to national standards using approved methods at appropriate intervals for the required analysis, the use of documented methods of working which must be unambiguous, the use of certified reference materials and, where available, involvement in proficiency testing. The ASTM E-42 standards (ASTM 1994) shown in table 2 provide a useful support to such requirements.

Before considering the ASTM standards, it is useful also to look at one standard from a parallel field, the ISO standard for measuring surface roughness (ISO 4287-1 1984). This standard presents, very clearly, several parameters, all clearly defined using diagrams and equations. These parameters, to a scientist, may seem somewhat arbitrary but, to an engineer, are extremely valuable. Surface scientists would require parameters such as the frequency distributions in Fourier space, fractal dimensions, populations of heights or of local slope. These are not mentioned in the standard but are clearly established in the scientific literature. Research scientists do not need standards for these parameters since effective standards already exist to the extent that research has progressed. The engineer, however, who is not researching surface form, needs one or two well-established parameters that provide the necessary characterization for his product to be deemed fit for its intended purpose (either by him or someone else, maybe in a different country). Before the establishment of ISO 4287-1:1984, different countries used the same symbols for different parameters which led to considerable confusion. The point here is that this standard is clearly very effective and useful but, if surface scientists had been involved, they may well have taken exception to the terms and expressions used (as being arbitrary) and have been concerned that these concepts could be imposed on them, in some way, to the clear detriment to their basic science. For instance, it could be that under ISO 9000, or some other system, a user was required to conform to available ISO standards. ISO 4287-1 (1984), in its scope, notes that it contains, *inter alia*, terms used in scientific publications. Thus, if the analyst was dealing with an excessively bureaucratic organization in which the requirement had already been included into many procedures of a quality system, he may find that genuine improvements were not accepted as they would lead to costly revision, checks for internal consistency, authorization, reissue, etc. Thus, the system set up to improve quality could inhibit the development of that quality (Feynman 1988).

Where events are reasonably clear, standards remove confusion. However, even where ISO documents do exist, as in the statements of uncertainties (ISO 1993), different laboratories interpret the contents differently so that they can manipulate the value of their best measurement capability, even to values below those of the

Table 2. *ASTM documentary standards for surface analysis (ASTM 1994)*

guides for:	
E983-89	electron beam effects in AES
E984-89	identifying chemical and matrix effects in AES
E995-89	background subtraction techniques in AES
E1078-90	specimen handling in AES, XPS and SIMS
E1127-91	depth profiling in AES
E1438-91	widths of interfaces in SIMS sputter depth profiling (SDP)
E1505-92	determining SIMS sensitivity factors from ion implanted standards
E1523-93	charge control and referencing techniques in XPS
E1577-93	guide for reporting ion beam parameters in surface analysis
practices for:	
E684-93	approximate current density of large-diameter ion beams for SDP
E827-93	elemental identification by AES
E902-93	checking the operating characteristics of XPS instruments
E996-89	data in AES
E1015-90	spectra in XPS
E1016-90	specifying the properties of electrostatic electron spectrometers
E1162-87	sputter depth profile data in SIMS
E1217-87	determination of the specimen area detected in XPS and AES
E1504-92	reporting mass spectral data in SIMS
terminology relating to:	
E673-93	surface analysis

national measurement laboratory from which they derive their measurement, in order to undercut the competition!

Thus, use of a standard may be advantageous in trade and marketing but purchasers of the appropriate goods or services need to assure themselves that the products do conform. Accreditation bodies such as NAMAS assist in this procedure sometimes rather more positively than journal referees. Those who do make false claims regarding accreditation can attract an unwelcome early morning visit from a member of the Fraud Squad. This may lead to a custodial sentence (NAMAS 1995).

Conformance to a standard can therefore be very useful when purchasing an item from a field in which you may have knowledge but not detailed expertise. It is not a guarantee of quality—there can never be such a guarantee—but at the present time it is an essential safeguard. Other safeguards also exist, such as purchasing an analysis from a recognized expert, however, the purchaser may find this route sometimes expensive, slow and of a higher quality than necessary for the purpose. Standards organizations emphasize many of these aspects and, in addition to the

four points given earlier by Spanner (1995), that standards should contain the best available practice for the use intended, including the cost to the user.

We would expect, therefore, that standards would develop most strongly around the most common and repetitive types of analysis made at surfaces. Standards would concern instrument calibration, basic sample handling and analytical procedures, data management and treatment, etc., for AES, XPS, SIMS and sputter-depth profiling. Even though these standards need consideration at the basic level, one needs the full backing of all of the experts so that one knows which items should be included to guarantee even a simple result. The above structure encompasses the work programme for the subcommittees for ISO TC/201 (Powell & Shimizu 1994), namely: instrument specifications, instrument operations, specimen preparation, data acquisition, data processing for qualitative analysis, data processing for quantitative analyses, reporting results, terminology and reference materials.

Before considering the ISO programme, it is worth returning to the ASTM standards listed in table 2, which have developed in a much more *ad hoc* way than is planned for the ISO work. The ASTM programme began the work on documentary standards for surface analysis in the late 1970s. It is interesting that ASTM have two further categories of documentary standard in addition to the three shown in table 2; namely *specifications* and *test methods*. Specifications are precise statements of requirements to be satisfied, whereas test methods are definitive procedures producing a test result. Practices are definitive procedures *not* producing a test result. We see in the *Annual book of ASTM standards for 1994*, vol. 03.06 (ASTM 1994) that in the analysis of metals, ores, etc., the test methods outnumber practices by an order of magnitude, i.e. practitioners in that area are interested in specific analyses, often by a specific method, of an analyte in a certain compositional range in a given matrix. These are needed as products are specified using them. Although such specific requirements regularly occur in surface analysis, e.g. the detection of specific silicone-based contaminants on wafers by static SIMS or certain trace impurities in silicon, these are usually the regular requirements of one customer who will develop with the analyst an in-house standard method; i.e. the problem has not been general enough to warrant an ASTM test method.

If surface analysis is not used in the above manner, one may ask, why is there a strong move for documentary standards? The reason is two-fold. As shown in figure 1, surface analysis is becoming more mature, and hence more routine, with analysis being conducted more and more by people who have not grown up with the field. Secondly, surface analysis is rather complex as each technique usually involves the two aspects of analysis and sputtering (for depth profiling), with data provided which could be three dimensional maps with each voxel associated with spectra containing chemical state information. These are not simple spectral techniques. This complexity makes it necessary for surface analysts to pool data. For this reason, the VAMAS activity developed a standard for data transfer (Dench *et al.* 1988). This allows data recorded at one laboratory to be processed on routines at another and, additionally, has opened up the transferability of data between commercial software packages. At NPL this has allowed us to do work which otherwise would simply have been beyond our resources. Elsewhere it has allowed effective interlaboratory networking in a way previously impossible (Yoshihara *et al.* 1992).

It is not the purpose here to analyse ASTM, or any other standards, but to consider their benefit. In the ASTM terminology standard E673-93, there will be found useful definitions of relevant terms such as shake-up satellites in XPS or cascade mixing in

sputtering. Some terms are rather closer to the edge of science than one would expect in standards, however, the rigor of defining terms has clarified the ideas in the field and has reduced the confusion that would otherwise have prevailed in the research literature. From the point of view of setting up instruments, electron spectrometers are represented in E1016-90 and XPS in E902-93. E1016-90 is an interesting and detailed introduction to the electron optics of spectrometers and was first produced in 1984. Despite its good intentions, no manufacturer has, to the author's knowledge, ever described or specified the properties of electron spectrometers with reference to this standard. Thus, despite the hard work, the correctness of any equations, etc., the standard is not used since it is not helpful to the user. It is best for the manufacturer to describe his instrument not in terms of its design as emphasized in that standard but in terms of its performance. It is only the minority of spectrometer experts who would want the details of design.

The standard for XPS, E902-93, on the other hand, is a simple way of checking that the spectrometer is maintaining a consistent behaviour. This is easy to implement and something of this type should be done by analysts on a regular basis. Thus, it is unlikely that much would be found that is critically wrong with published standards. The more pertinent questions are whether a developing standard is appropriate, practical and useful or is it over-complicated, inadequate, diversionary and likely to retard development?

For purchasers of analytical time from vendors of services it is important to know several simple things such as:

- that the samples were correctly handled after receipt, stored before analysis and mounted on the specimen holder;
- that effective charge neutralization was used;
- that the instrument abscissa scale was within calibration; and
- that the instrument intensity scale was repeatable.

This set at least lets us compare last week's data with this weeks! NAMAS laboratories all have to write their own procedures to do this but, of course, it would be much easier for them, and everyone, if documentary standards already existed to which they could add their own local requirements.

The programme for ISO TC/201 gives us a framework for future work but where should efforts be concentrated? This is clearly up to the voting members in ISO TC/201 and those who are willing to contribute effort. Terminology must be considered and so must instrument calibration. For XPS, it may even be worth defining calibration limits of uncertainty for effective chemical shift studies. A simple XPS energy calibration procedure could use the Au $4f_{7/2}$ and Cu $2p_{3/2}$ peaks shown in table 1 but, if a user regularly studies other materials with suitable peaks, it is clear that he could develop effective secondary standards for himself which allow him to track the calibration routinely in his own work without the extra effort of regularly loading and cleaning Cu and Au. It would be up to the analyst to choose his own route for regular calibration, providing he could *demonstrate* the relevant accuracy.

Instrument calibration covers the ordinate (usually intensity or count rate) and abscissa (mass or energy), the calibration accuracy and intervals between calibration. It also covers magnification for imaging, angles for sample orientations, aperture sizes for solid angles and parameters of ion beams for sputtering, including constancy of current. Any analyst purchasing sputter profiles would like to relate one profile to another and would need the sputtering rate to be constant with time, i.e. they would like the ion beam current density to be constant. If the sputtering gas pressure is

maintained from a vessel at one atmosphere pressure, leaking gas into an ion gun with an electron beam ionizer, differentially pumped at 10^{-7} mbar by a pump of 100 l s^{-1} , it is easy to see that the pressure in a reservoir vessel of a few cc would fall significantly in one working day so that the ion beam current could fall equivalently. For sputtering systems, an instrument specification could thus usefully cover the ion beam current stability over a defined period and this would, in turn, catalyse appropriate measures to reduce any variability (e.g. if a passive reservoir vessel is used, it should be larger than a certain size or, if a servo on some relevant measurement sensor is used, its behaviour should be understood).

A large programme of work covering the definitions of terms, the clarification of algorithms, of instrument calibration and of the needs for reference materials has been launched within ISO TC/201 (Powell & Shimizu 1994). Each project, on the whole, is led by an expert with interest in that activity. If this were not the case progress would necessarily be slow. It is inappropriate here to comment on any project in detail but it would be worthwhile for each participating or observing member body balloting all users in their country to identify those who would actually use each standard, when developed, in their laboratory.

4. Conclusions

It is clear that measurement standards within a logical framework are essential underpinning for the development of any measurement or analytical technique. For these standards to be effective they need a simple measurement procedure for their realization in the analytical laboratory.

It is clear that, if carefully approached, many useful documentary standards covering procedures could be established and that these would raise the quality of instruments and of analytical work with no additional cost to the instrument vendor or the analyst. It is also clear that most of the effective standards will be for aspects which are already regular working practice in the better laboratories. In general, those developing documentary standards should:

- make sure of the true need by consulting users;
- involve users in the drafting procedure;
- keep it simple and direct;
- avoid their own latest ideas; and
- ensure all the relevant experts have been consulted.

A failure to do this could lead to inappropriate standards being developed. Where conflict may evolve if these are not followed will be in areas where there is no clear problem, or perhaps a solution exists for which a broad problem is invented. The consultative nature of the standards framework is intended to ensure that whatever is developed is genuinely useful.

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