Surface analysis — current capabilities

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Oberflächenanalyse — gegenwärtige Möglichkeiten


Die Arbeit beschreibt einige Fortschritte in diesen verschiedenen Richtungen und gibt Beispiele für Anwendungen.

Summary. Surface analytical techniques are now used so routinely and frequently in technological problems that technology is itself dictating some of the directions in which the techniques should develop; in particular, the directions of improved spatial resolution, of combining surface analytical with bulk analytical techniques in the same system, and of the analysis of materials, either in difficult physical states or in difficult ambients. This paper describes some of the recent advances in these various directions, with examples given of how they have been applied.

1. Introduction

The principal surface analytical techniques AES, XPS, SIMS and ISS are now very widely used on a great variety of problems in almost every area of technology. As a result of attempts to push the application of the techniques beyond their limits of development at any point in time, technologists have been putting pressure on instrument manufacturers to concentrate their development efforts in certain particular directions. Because micro-electronic circuitry is tending towards ever smaller dimensions, and because, for example, Auger Spectroscopy (AES) is used in the electronics industry probably more than in any other, there is a continual demand for the ability to analyse ever smaller surface areas. Similarly in those product areas where an incident electron beam should not be used, e.g. in catalysis, there is also pressure for reduction in area analysed, this time on X-ray Photoelectron Spectroscopy (XPS). In all technological fields, too, there is a growing demand for the capability in a single instrument of combining surface and bulk analyses, the obvious advantage being that the specimen or product does not have to be removed from the vacuum environment to undergo the separate analyses, thus avoiding the risk of interfering contamination. Yet another strong thrust from the technological side comes from those who have to try to analyse materials in a difficult form, that is to say, where the material itself is perhaps a liquid, or where it is required to study its surface composition following treatment at high temperatures in higher pressures of a reacting gas mixture than could be tolerated inside the spectrometer.

In this paper the ways in which instrument manufacturers and individual workers have been tackling the developments necessary to meet the demands and pressures will be described, and examples given of the capabilities of current surface analytical instrumentation.

2. Spatial resolution

2.1 Auger spectroscopy

Improved analysis capability of small areas has taken two directions in AES. The first and most obvious one is that of further reduction in the spot size of the incident electron beam, the other is the computer processing of scanning Auger micrographs, or maps, either to enhance the contrast or to improve the quantification of them.

To be able to achieve an electron spot size of less than 0.5 μm involves the use of primary electron energies in the range 15–30 keV, i.e. much higher than those used in conventional AES. Electrostatic focussing at such relatively high energies in the type of compact electron gun suitable for AES systems is not easy, and the desired small spot sizes probably impossible to achieve. Instead electromagnetic focussing must be used, and since the entire gun must of course be UHV compatible and therefore bakeable, difficulties arise in the design and fabrication. The various electromagnetic coils must be capable of withstanding the bakeout temperature, and the mechanical alignment of all the electrodes in the gun must not suffer as a result of bakeout. In the most recent models of electromagnetic guns these problems have been largely overcome by careful engineering and choice of materials. One of these is shown in Fig. 1; it is capable of being focussed to a spot size of 35 nm at 25 keV, but is still a compact device, in fact so compact that it actually fits co-axially inside a Cylindrical Mirror Analyser (CMA).

It should be remembered, of course, that a quoted electron beam spot size is not the same as the spatial resol-
tion in AES. Although much of the ionisation of near-surface atoms is caused by the primary beam itself, there is a substantial contribution to ionisation from inelastically scattered electrons diffusing back to the surface, and these will cause ionisation over an area significantly larger than that of the spot size. Thus, for example, a spot size of \( \sim 35 \text{ nm} \) might lead to an Auger resolution of \( 100 - 200 \text{ nm} \), the precise figure depending on the nature of the specimen. For this reason the spatial resolution in AES is unlikely ever to approach that available in a good Secondary Electron Microscope (SEM).

Having said that, recent applications of high resolution AES have shown distinct advances in analytical capability, and a few examples will be shown here. In Fig. 2 is described [1] the investigation of a multi-layer film on an electrical contact. The contacting surface material is required to be Au, and this is plated as a thin film on a Cu substrate, but to prevent interdiffusion of Au and Cu at high temperatures, an intermediate layer of Ni is laid down before the Au. Sometimes, however, the Ni itself diffuses through the Au, causing malfunction of the contact. It is necessary to know in quality control not only if there is Ni on the surface, but how much there is on average, and how it is distributed in detail. The upper photograph is an SEM image of the Au/Ni/Cu specimen, showing micro-roughness after high temperature treatment. The next picture is a Scanning Auger Microscopy (SAM) image, using the principal Ni Auger peak to modulate the brightness, thus producing a map of the Ni distribution across the surface. It can be seen that the surface composition contains a large amount of Ni, and that the Ni is distributed in micro-areas of average dimensions 0.5 - 3.0 \( \mu \text{m} \). The high spatial resolution capability can be further applied by carrying out point analyses, which has been done at the points marked 1 and 2, combined with simultaneous depth profiling by ion bombardment. The profiles shown in the lower two diagrams reveal that at point 2 the correct Au/Ni/Cu multi-layer structure has been maintained during heating, but that at point 1 there is almost pure Ni at the surface and that it is oxidised. The analyses at points 1 and 2 were representative of areas of diameter about 250 nm.

Improved spatial resolution by reducing the incident beam spot size means not only that the ultimate imaging capability is improved, but also by definition that the sharpness of images acquired at lower magnification is also improved. In Fig. 3 the sharpness of the Auger maps using