Lecture

Imaging with Auger Electrons

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Quantitative chemical mapping of the surface of a solid is commonly carried out using secondary ion mass spectroscopy (SIMS) or scanning Auger microscopy (SAM). Both methods use scanned, focussed beams of charged particles incident upon the sample. The charged particle optics for focussing and deflecting the ion or electron beams are available with sub-100nm beam diameters and adequate beam currents for acceptable statistics of the scattered ions or electrons. The depth resolutions of both methods are comparable. The SIMS method is in widespread use for the determination of the concentration depth profiles of dopants in semiconductors. It is an extremely sensitive technique for this purpose, being capable of depth resolutions of about 1 nm and sensitivities of the order of 10^{14} atoms cm^{-3} for ion beam diameters greater than about 100 nm. However, because the sample is consumed in the production of secondary ions the sensitivity falls as the diameter of the incident ion beam. Below beam diameters (and hence spatial resolutions) of about 100 nm scanned electron beam methods become more sensitive. This is because, to a large extent, the analyst is free to choose the time that an electron beam is allowed to strike a particular small area of a surface and generate scattered electrons. The signal to noise ratio will scale as the square root of this dwell time and can be increased provided that the sample is not damaged or contaminated by the beam and the sample does not move during the acquisition time. It is for this reason that SAM has become an important method of generating chemically specific maps of the concentration of materials in the surface of a solid.

One important objective for SAM is to be able to produce maps of the variations of the concentrations of each element in a surface – quantitative chemical surface mapping. This is often essential if the set of one map for each element present that is produced by a SAM experiment is to be more meaningful than an intuitive indication of the variation of the concentrations from place to place. This is a complex requirement. The fact that the analyst requires to study surface chemical maps usually means that the sample under investigation is heterogeneous and rough. Therefore methods need to be found to compensate for or eliminate various effects known to cause variations in the yield of Auger electrons even when the surface the composition is perfectly homogeneous.

These effects include:

- changes in the Auger yield due to variations in the local angle of incidence and the local take off angle into the electron energy analyser. This is the effect of surface topogrphy.
- Changes in the Auger yield due to variations in the composition of the material beneath the surface. These variations cause spatial variations in the backscattered electron yield and so the number of Auger electrons generated in the surface region.
- Changes in the orientation of grains in the sample. This can alter the yield for an Auger process because of channelling of the incident electron beam in particular crystal directions and also because of

diffraction of the Auger electrons on their route out of the solid.

The approaches to the design of SAM instruments have varied considerably in their main objectives. Most commercial instruments have designs that attempt to minimise the analyst's time in studying a sample – an important consideration in an industrial laboratory or quality control situation. Venables *et al* [1] have aimed at modifications of STEM instruments in order to obtain the best possible spatial resolution – 4 nm at 100 keV has been achieved here. Prutton *et al* [2] have placed less emphasis on spatial resolution and have developed an instrument and associated computer software for quantitative surface mapping.

This latter instrument – the York multi-spectral Auger Microscope (MULSAM) – is a UHV, energy analysing, multi-imaging, scanning electron microscope. The idea underlying the design of MULSAM is that the incident electron beam excites a wide variety of processes in the solid. Each process has a particular information depth that carries different information about the properties of the place where the beam impinges. A variety of detectors can be placed around the sample to pick up these scattered photon and electron signals. If the signals are collected synchronously, then a set of images can be obtained which may be combined, using physical models, so as to separate the topographical, sub-surface and diffraction effects mentioned above.

This instrument has been applied to a number of different problems in imaging methodology and applied materials science.

The developments in methodology have involved a search for the best means of removing topographical contrast from Auger images so as to leave only chemical variations. This has been achieved by collecting energy analysed images on each Auger peak and on the background with kinetic energies above the peak. The peak height can be estimated by extrapolating, at each pixel, the background to the peak energy and subtracting the background height from the peak height. If this difference is divided by the extrapolated peak background height then the resulting ratio is roughly independent of topography. However, the effects of topography have been overcorrected with the use of this ratio for the pixel values. This overcorrection can be compensated by estimating the atomic number of the sample material with a backscattered electron (BSE) detector that has been appropriately calibrated. The results of this approach are very good – the result being near to the limits of precision set by the signal to noise ratios from the detectors involved.

The same BSE detector can be used to estimate the Auger backscattering factor at each place. This is done by using the Z contrast provided by this detector together with Shimizu's Monte Carlo simulations[3] of the Z dependence of the Auger backscattering factor. By combining the spatially registered Auger and Z contrast images with the relationships derived by Shimizu the effects upon the contrast in the Auger maps due to sub-surface concentration variations can be removed.

The removal of diffraction effects remains an aspiration. Fortunately, many samples studied using SAM are cleaned *in-situ* with inert ion bombardment. This disorders the surface region of the solid and reduces the channelling and diffraction effects to acceptably low levels.

A powerful analytical tool set is available for sets of spatially registered images in multi-variate statistics (MSA). The tools used for surface imaging have included multi-dimensional scatter plots, image segmentation, principal component analysis (PCA) and target factor analysis (TFA). Scatter diagrams are a particularly useful tool for the production of a *phase map* of a surface from a set of Auger maps. As well as being a useful reduction in the

quantity of data to be handled and interpreted, these maps are a very helpful means for identifying the number and areas of statistically distinct types of region in the surface. Subsequent spectroscopy at the centres of such regions provides an objective method for the characterisation of each different kind of region in the surface. This method has been applied to the study of the structure of magnetic multilayers and the interfaces in a variety of different metal to semi - conductor contacts.

These MSA methods can be applied to a wide variety of analytical techniques. Energy dispersive X-ray detection is particularly suited to this set of tools because Si(Li) detectors reveal the entire X-ray spectrum simultaneously. They are a good example of parallel detection. Other methods which can exploit MSA include time of flight SIMS and parallel electron loss spectroscopy in STEMs.

The method of PCA is also used to identifying the number of statistically uncorrelated regions in a sample. It too can be used to condense the raw data set obtained from the microscope to a smaller image set that is accompanied by some improvement in the signal to noise ratios. It helps the analyst to decide how many different types of region are in the surface and, by examining the fractions of the raw images making up each principal component image, to input some physical insight to the processes at work in determining the image contrast. An example of this approach has been in the study of used PtRh catalysts. These materials develop very extreme topography associated with re-crystallisation when they are operated at the temperatures needed for effective catalysis. A combination of the methods of scatter diagram plotting and PCA has allowed the isolation of those regions of the surface that cannot possibly be analysed. Such regions are those not struck by the incident beam and those not having a line of sight into the electron energy analyser because of the surface roughness. Having identified these inaccessible regions the remaining surface can be analysed quite effectively using the methods outlined above.

One important use of Auger spectroscopy and microscopy is in the determination of the composition depth profiles (CDPs) of a solid. SAM methods have been powerful in this area when combined with ion beam machining. A computer controlled ion beam is used to cut a bevel at a small angle to the original surface (often in the range 10^{-1} –10 mrad). The CDP is then revealed on the surface of the bevel and can be analysed by collecting and interpreting Auger line-scans or images. This method has the advantage that the data acquisition time can be chosen to obtain satisfactory counting statistics for the elements present in the CDP. Also, the analyst is not required to predict the elements present in a CDP in order to set up the depth profiling in a pre-determined set of energy ranges spanning the expected peaks. Rather, the entire spectrum can be acquired at each point in a linescan – a spectrum linescan – and unexpected (and interesting) peaks due to precipitated or segregated material are observable and can be quantified.

In semiconducting device studies it is often of interest to characterise the buried interface at a metal-semiconductor junction. This may reveal unexpected chemical reactions or interdiffusion effects that may modify the electrical properties of the junction. Sputtering material away for several microns to reach this interface starting at the free surface can be very unsatisfactory because of the roughening effects of differential sputtering yields and the mixing effects of the inert gas ion bombardment. One approach to the characterisation of such a buried interface is to polish the underlying substrate away, maintaining a small angle

to the plane of the junction. The CDP at the interface of interest then appears at some position along the inclined polished and cleaned surface.

Another powerful method of sample preparation is micromachining by fast ion bombardment (FIB). Here, for instance, a 30 keV Ga^+ beam can be focussed onto the sample and scanned to cut a slice standing up perpendicular to the surface. By tilting the sample the CDP can be studied on the sides of this slice either by Auger or SIMS or by transmission electron microscopy.

References.

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