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Surface Analysis of Insulators: a New Charge Neutralisation System

by Ian Gilmore and Martin Seah

Surface chemical analysis is today of primary importance in the development and quality control of high technology materials and products. In particular, static secondary ion mass spectrometry, S-SIMS, is the fastest developing of the relevant techniques and is especially important worldwide in the polymer and coatings industries.

In the S-SIMS technique, medium energy primary ions impact the sample causing the emission of less than 1% of the surface monolayer as ionised atomic and molecular fragments, which are subsequently mass analysed. The mass spectrum provides a highly sensitive and chemically very detailed analysis of the surface. A considerable proportion of practical analyses of solid surfaces using S-SIMS is of insulating samples. This poses a particular problem because the build-up of surface charge on insulators due to the

impacting primary ions results in an uncontrolled surface potential. This affects the measured fragment energies and leads to unreproducible intensities.

Many methods have been used to stabilise the surface potential of insulators in SIMS. Commonly, high energy electrons are made to strike the surface, compensating for the accumulated charge. Such methods cause poor spectral repeatability and can produce



considerable surface damage. At NPL a new procedure of charge stabilisation has been developed, allowing the acquisition of highly repeatable data from insulators for the first time.

This procedure, using an emission plate placed between the sample and the mass spectrometer, gives an even flux of low energy electrons that strike the sample from many incident angles. Electrons, emitted from the plate by the action of an electron beam, bathe the sample and stabilise its surface potential very close to those of the plate and sample holder. Most of the emitted electrons have very low energies and so cause little surface damage. The narrow energy acceptance of the mass analyser is now broadened by applying a triangular waveform of 32 volts peak to peak to the sample environment. This removes the sensitivity of the system to the precise sample surface potential.

The repeatability of the emission plate neutralisation was tested by analysing a set of PTFE samples. Variations between the spectra were found to be less than 2%, representing an improvement of several orders of magnitude, and paving the way to valid analytical measurements in this particularly difficult sector. ■

