APPLICATION NOTE

Routine Monitoring of Nanoscale Coatings using the benchtop MiniSIMS

Mini SIMS

MiniSIMS imaging & profiling rapidly analyse multi-layer coatings for continuity, composition and thickness.

- Large Area Imaging to Reveal Surface Defects
- Measurement of Nanoscale Coating Thickness
- Chemical Analysis of Surface and Sub-Surface Layers

Previous application notes have shown how the MiniSIMS can be used to detect and identify unwanted contamination on surfaces prior to coating or painting. However, this application note is concerned with analysis of the surface after the coating has been applied.

Surfaces may be coated to give them protection in a hostile environment, or to enhance their cosmetic appearance. For these purposes, the coatings are typically one micron to one millimetre in thickness to prevent minor abrasions exposing the surface underneath. Relatively thick coatings such as these can be examined by SIMS and other analysis techniques, including EDX and GDMS.

When coatings are used to impart specific properties to a surface, the desired effect can be achieved with a coating much less than one micron in thickness. Sometimes the coating is composed of several layers, each one between 10 nm and 100 nm in thickness.

The shallow information depth of the SIMS technique (1 nanometre) allows each layer to be analysed in isolation. This is not possible with EDX, where the information depth (~1 micron) means that all layers simultaneously contribute to the analysis.

This application note shows how the MiniSIMS provides this 3-dimensional information without the need for cross-sectioning of the sample prior to analysis. Large area SIMS imaging of the original surface is used to show the overall uniformity of the outermost coating layer. The primary ion gun in the MiniSIMS is then used for in-situ etching of the coating in a controlled manner, providing information on the thickness and composition of each layer in turn.

See overleaf for more detailed information.

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This example concerns a typical three layer metallic coating.

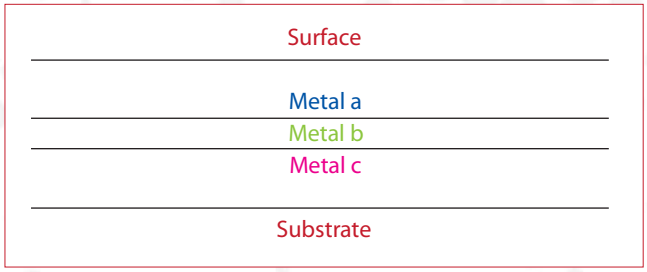

Figure (1) STRUCTURE OF THE MULTI-LAYER COATING

The first investigation is made by imaging the surface at low magnification. The fast imaging of the MiniSIMS means that each image can be acquired in times as short as 5 to 30 seconds. If the coating is continuous, the underlying layers will not be visible in a secondary ion image. However, in the 6mm² image (figure 2), small areas of Metal b are seen, indicating sub-millimetre defects in the integrity of the coating.

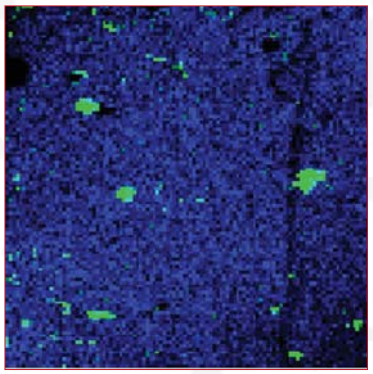

Figure (2) CHEMICAL IMAGE SHOWING DEFECTS (Mb IN GREEN) IN THE COATING (Ma IN BLUE)

The corresponding image for Metal c shows no intensity, immediately proving that the defects do not extend down to the third layer. The failure has therefore occurred at the first interface.

In areas where the coating is free of defects, profiling analysis is a simple method to monitor and measure the thickness of the coating layers. The primary ion beam is scanned over a small area 100 µm x 100 µm to etch through the coating layers. The mass spectrometer monitors the change in intensity of secondary ions characteristic of each layer. Since the etching and analysis are achieved simultaneously by the same primary ion beam, there is no delay cycling between these two functions. The MiniSIMS also features an electronic "gate" to reduce background signals from the walls of the etched crater.

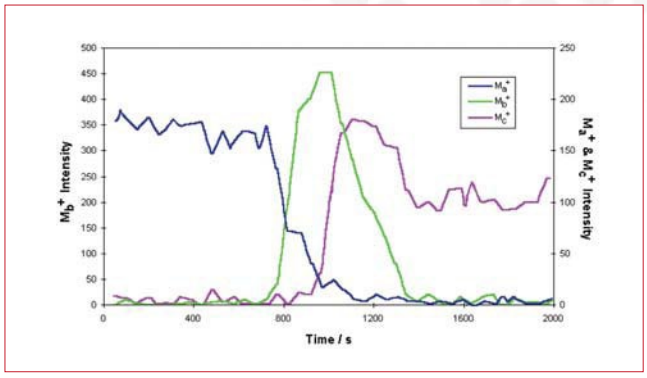

Figure (3) CHEMICAL PROFILE SHOWING THICKNESSES OF THE COATING LAYERS

The results of the profile are shown in the graph (figure 3) lower left. Each secondary ion peaks in turn, and the two interface points are clearly identified. The thickness of each layer is proportional to the time between interfaces.

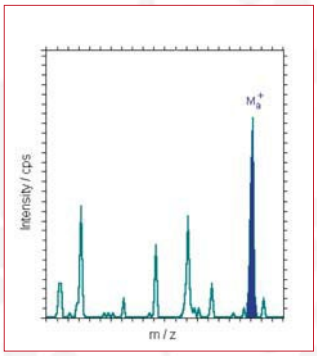

Figure (4a) MASS SPECTRUM FROM WITHIN FIRST COATING LAYER

The time axis can be converted toapproximate depth by making a theoretical estimate of the etch rate, in this case about 8 nm in 100 seconds. Alternatively, an exact value for the etch rate can be determined by physical measurements of the depth of the crater, and this calibration can then be used for all subsequent analyses of the same material.

If desired, the etching can be stopped at any depth and a full mass spectrum collected from the bottom of the crater. These spectra will show other components of the layer, or any contaminants presentin the layer, with no interference from the layers underneath.

For example, using the two spectra shown in figure 4a and 4b, the change in composition from one layer to the next can be confirmed.

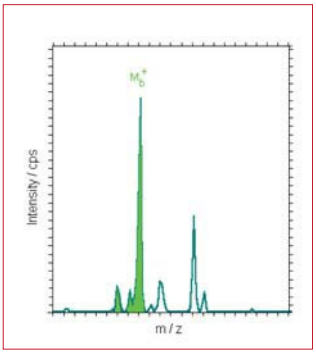

Figure (4b) MASS SPECTRUM FROM WITHIN SECOND COATING LAYER