APPLICATION NOTE

Rapid Assessment of Monolayer Coatings using the benchtop MiniSIMS

Mini SIMS

MiniSIMS mass spectral analysis of surfaces can be used to monitor surface treatments with sub-monolayer sensitivity.

- Monitoring the Effectiveness of Pre-Treatments
- Assessment of Coating Continuity
- Correlation of Physical Properties with Chemical Changes

Previous application notes have shown how the MiniSIMS can be used to detect and identify unwanted contamination on surfaces prior to coating or painting. Similarly, the SIMS analysis technique can be used to investigate or confirm the chemical changes that have occurred when surfaces have been deliberately pre-treated (e.g. to improve adhesion of the subsequent coating). This application note is concerned with the next stage of the process, the analysis of the surface after the coating has been applied.

When a coating is used to impart specific properties to a surface, the desired effect can usually be achieved with a coating much less than one micron in thickness. In the extreme, the coating may be only a monolayer in thickness, and surface sensitive techniques are essential for the analysis.

SIMS offers an information depth of just one nanometre, and high sensitivity to allow fast analysis. Even if both the coating and the substrate are organic, the SIMS mass spectrum will contain distinct peaks to allow the two materials to be easily distinguished.

This application note shows how the MiniSIMS is used to monitor the continuity of a monolayer coating. If the static SIMS spectrum obtained from the material after coating still shows peaks characteristic of the substrate, then the coating in this case cannot be continuous. The disappearance of the substrate peaks from the spectrum is a simple test to show when full monolayer coverage has been achieved.

See overleaf for more detailed information.

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This example concerns the development of a new class of inherently conducting polymeric materials.

The conductive polymer used was polypyrrole, doped with a sulphur containing anion. The resulting polymer has good conductivity, but is difficult to process. A composite material is therefore created, consisting of a layer of the polymer supported on silica-gel particles.

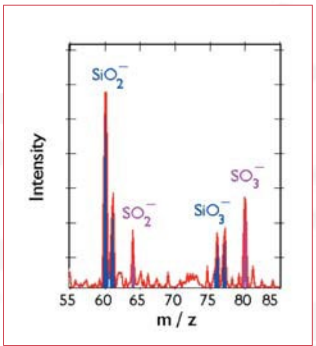

Figure (1) SPECTRUM OF MATERIAL WITHOUT PRE-TREATMENT OF SUBSTRATE

The objective is to achieve a continuous coating of the polymer using the minimum amount of material. The composite material would then exhibit the maximum possible surface conductivity without wastage of the polymer.

Analysis of the composite material prepared under standard conditions gave the negative SIMS spectrum shown in figure 1. The peaks characteristic of sulphur containing secondary ions indicate the presence of the polymer on the surface of the particles.

If a continuous coating had been achieved, the shallow information depth of SIMS (approximately 1 nm) means that peaks characteristic of the silica substrate would be absent from the spectrum. The presence of the relatively strong silicon-containing secondary ions in the spectrum clearly indicates that there are significant areas of exposed substrate.

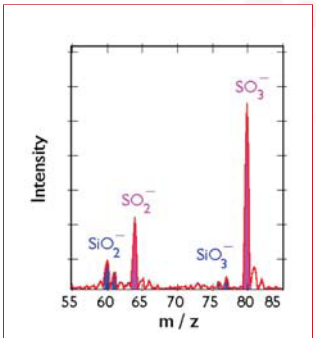

Figure (2) SPECTRUM OF MATERIAL WITH PRE-TREATMENT OF SUBSTRATE

A fresh batch of silica-gel was then prepared, and was treated with a silane coupling agent to improve adhesion of the polymer. This pretreated substrate material was then coated with the conductive polymer using the same conditions as before. After coating with the polymer, the composite material gave the negative SIMS spectrum shown above. In this spectrum, the ratio of sulphur rich to silicon rich secondary ions is much greater than that obtained from the spectrum in figure 1. This confirms that the expected improvement in coverage by the polymer has indeed been achieved by using the silane coupling agent.

The desktop MiniSIMS allows measurements of this type to be completed in just a few minutes and at much lower cost per sample than is possible with conventional surface analysis instrumentation.

The ratio of the polymer related peaks to the substrate related peaks was used as a monitor of the coverage of the substrate by the polymer as the silane pre-treatment conditions were varied (figure 3). In this case, the information obtained about the chemical composition of the surface can be confirmed by physical measurements of conductivity, also shown in figure 3. As the silicon related peaks disappear from the SIMS spectrum, the increasingly continuous coverage is reflected in a dramatic improvement of the conductivity of the composite material.

Pretreatment	Peak Ratio	Conductivity
0% γ-APS	0.6	10^{-6} S cm^{-1}
0.5% γ-APS	2.5	10^{-5} S cm^{-1}
1% γ-APS	3.6	10^{-2} S cm^{-1}
3% γ-APS	5.8	10^{-1} S cm^{-1}

Figure (3) CORRELATION OF THE IMPROVED CONDUCTIVITY OF COMPOSITE MATERIAL WITH THE CHEMICAL CHANGES OBSERVED IN SIMS SPECTRUM

Acknowledgment

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