



MILLBROOK

Monitoring Monolayer Coatings

Spectral examination of surfaces after application of a coating overlayer is used to monitor the coverage of the substrate by the coating.

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.

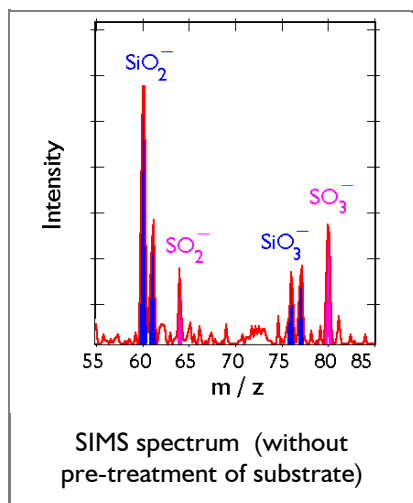
This application note shows how the MiniSIMS is used to monitor the continuity of a monolayer coating. If the static SIMS spectrum obtained from the material after coating still shows peaks characteristic of the substrate, then the coating cannot be continuous. The disappearance of the substrate peaks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

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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.

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.

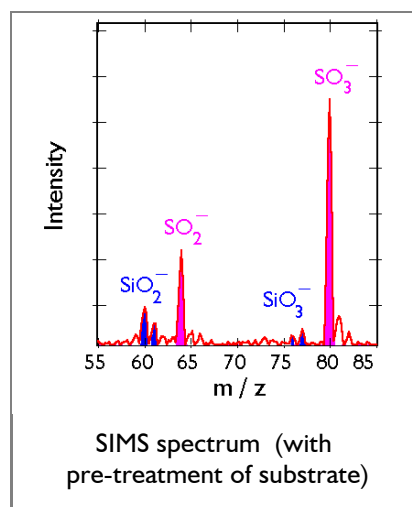


Analysis of the composite material prepared under standard conditions gave the negative SIMS spectrum shown above. The peaks characteristic of sulphur-containing secondary ions indicate the presence of the polymer on the surface of the particles.

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.

A fresh batch of silica-gel was then prepared, and was treated with a silane coupling agent to improve adhesion of the polymer. This pre-treated 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 peaks due to silicon-containing secondary ions (arising from the treated substrate) are much less intense relative to the peaks due

to sulphur containing peaks (arising from the polymer coating) than before. This confirms that the expected improvement in coverage by the polymer has been achieved.

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 (see table below). In this case, the information obtained about the chemical composition of the surface can be confirmed by physical measurements of conductivity, also shown in the table below. 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.

Acknowledgment

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

Correlation of improved conductivity of composite material with changes observed in SIMS spectrum