

Rapid Profiling of Glass Coatings using the benchtop MiniSIMS

Mini SIMS



Three-dimensional images obtained with the MiniSIMS ToF show the full composition of complex multi-layer surface coatings.

- Thickness Measurement of Multi-Layer Coatings
- Chemical Analysis of Thin Films and Interfaces
- Detection of Sub-Surface Defects

Flat glass is used in both buildings and automotive construction. Increasingly, layer structures are used to enhance performance whilst maintaining an aesthetically pleasing appearance. Surface coatings, for example low emissivity coatings, reflective coatings, solar control coatings and self-cleaning coatings, are of particular importance.

Physical vapour deposition (PVD), chemical vapour deposition (CVD) and magnetron sputtering are the main technologies used for glass coating. These processes are compatible with the properties of glass, and can form uniform films over large areas with accurate variation of the film thickness. The coatings typically consist of stacked layers of metals, metal oxides, metal nitrides and metal alloys. The thickness of each layer is critical to performance, with dimensions ranging between one nanometre and one micron in thickness.

The SAI MiniSIMS can not only monitor the composition and thickness of these multi-layer coatings with excellent resolution, but also, especially important for production environments, with high speed. In the MiniSIMS, the fact that the analysis beam is also profiling obviates the need to continually switch between analyse and etch cycles. Furthermore, data of adequate signal to noise ratio is obtained by SIMS much more quickly than by most other techniques due to the superior sensitivity. In comparison with other ToF-SIMS instruments, the MiniSIMS ToF also operates with a continuous rather than a pulsed primary ion beam, further increasing the achievable profiling speed.

See overleaf for more detailed information.

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A MiniSIMS depth analysis is achieved by in-situ etching of the multi-layer coating with a focused beam of high-energy gallium ions. The material being sputtered therefore changes as each layer is sequentially exposed, and the corresponding secondary ions are detected using a mass spectrometer. The shallow information depth of the technique means that layers just a few nanometres in thickness can be analysed without interference from deeper layers.

More accurate depth calibration can be performed by physical measurement of trial craters etched under the same conditions in each of the different materials. After this calibration work has been performed once, the same parameters can be used for all subsequent analyses.

Figure 2 is an example of a conventional depth profile plot but in reality it has been extracted from a larger set of data, namely an array of voxels (or pixels in 3 dimensions). The data set was acquired by collecting secondary ion intensity data at each point in sequential images of the bottom of the etch craters. The bottom of the crater becomes deeper as material is removed by the ion beam and so each image relates to a different depth. The 3D array is therefore the complete set of images from the acquisition. The main difference between the MiniSIMS alpha and the more powerful MiniSIMS ToF is that the number of secondary ions that can be monitored per voxel is restricted in the alpha and these have to be chosen before the profile starts. In the MiniSIMS ToF this restriction is lifted allowing a complete mass spectrum to be stored at every voxel. The practical benefit of this feature is to allow a retrospective analysis of unknown species that might not have been predicted at the start of the experiment. This aspect of the MiniSIMS ToF makes it ideal for failure analysis.

For example, Figure 3 shows the MiniSIMS ToF 3-D positive ion image of another glass coating of similar structure. For clarity, the distribution of only three secondary ions is shown in the 3-D image. The sodium ion (Na^+) intensity is shown in red. This is high at the surface and in the glass substrate, but also it is seen in an isolated inclusion part-way through the profile. A full mass spectrum can again be generated retrospectively from this localised area to allow a full identification of the inclusion.

Figure (1) SCHEMATIC STRUCTURE OF A TYPICAL GLASS COATING



For example, Figure 1 shows the structure of a typical multi-layer coating on glass, incorporating a single silver layer. Figure 2 shows part of the profile through this coating performed with the MiniSIMS. Positive and negative secondary ions have been monitored by two sequential analyses from adjacent areas. The metal nitride layer is characterised by emission of the MN^- ion, followed by the metal oxide layer (monitored by the major M^+ isotope). After the alloy layer (monitored by the major isotope M^+ of each elemental component), the silver layer appears (Ag^+). To etch through the entire multi-layer coating typically takes less than one hour.

The depth scale of Figure 2 is based on a constant average erosion rate. For quality monitoring, this produces a profile that can simply be compared to a standard profile measurement to check they are identical. In practice, the etch rate will change in the different materials of each layer.

Figure (2) CHEMICAL DEPTH PROFILE THROUGH SUCCESSIVE LAYERS OF THE COATING

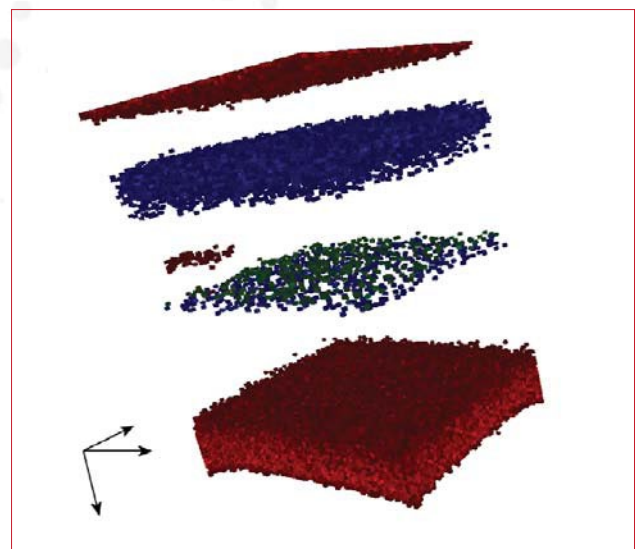
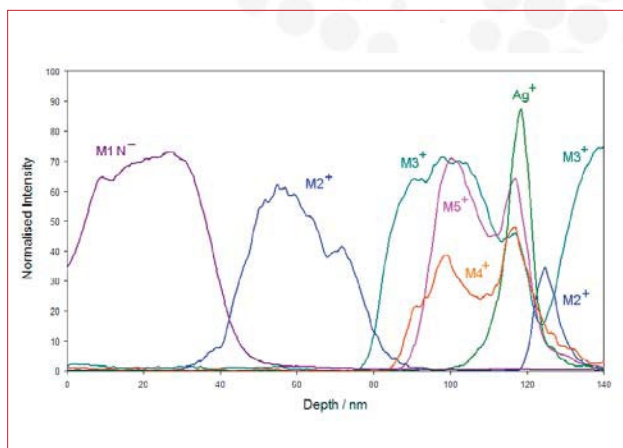


Figure (3) 3-DIMENSIONAL RECONSTRUCTION OF SELECTED COATING LAYERS