APPLICATION NOTE

Analysis of Self-Assembled Monolayers using the high throughput MiniSIMS

Mini SIMS

The shallow information depth available from MiniSIMS analysis allows detailed characterisation of monolayer films.

- Investigation of Surface Coverage and Layer Density
- Determination of Molecular Orientation
- Confirmation of Molecular Structure

Various techniques are used for the production of thin films, and most rely on careful external control to achieve films of the required uniformity. In contrast, certain molecules have the ability to deposit on selected surfaces utilising internal forces.

These self-assembled monolayers (SAMs) provide a fast and easy way to produce highly ordered films, which can then be further modified to introduce the desired functionality. This offers a potential route for mass production of surface engineered films at low cost.

SAM research brings with it a need for analysis to examine the resulting monolayer structures. SIMS is an ideal technique to do this but until now has been too complex and expensive to use routinely.

The MiniSIMS however provides a relatively low cost and high throughput solution for chemical characterisation of the film, showing its orientation on the surface and providing further information about the density of the layer.

The provision of samples by and discussion of results with Dr F. Chérioux and Dr L. Gauthier-Manuel (Laboratoire FEMTO-ST, Besançon) is gratefully acknowledged.

See overleaf for more detailed information.

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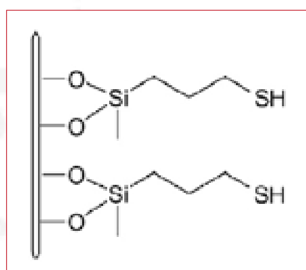

Figure (1) STRUCTURE OF THE SELF ASSEMBLED MONOLAYER

The SAM analysis concerns the thiol-terminated molecule HS-C_3H_6-Si(CH_3)O_2 (Mol. Wt. 150) on a silica substrate (Figure 1).

STRUCTURE OF THE SELF ASSEMBLED MONOLAYER

In the negative SIMS spectrum (Figure 2), the intact molecular ion (M-H)$^-$ is seen at m/z 149-. At low mass, there is a peak at m/z 25- corresponding to C_2H from the aliphatic chain. There is another strong peak at m/z 32-, a combination of both S and O_2^-. However, the similarly intense peak due to SH at m/z 33- confirms the presence of sulphur. The peak at m/z 60- is SiO_2^-.

The negative spectrum also shows other peaks assigned to stable fragment ions, for example:

59	CH_3SiO	75	CH_3SiO_2 and HSC_3H_6
89	$CH_2(CH_3)SiO_2$	101	$C_3H_6(CH_3)SiO$
119	HSC_3H_6SiO		

For a series of samples, the changing intensity of the SH and (M-H)$^-$ peaks can be used as a simple monitor of the surface coverage achieved in the SAM preparation.

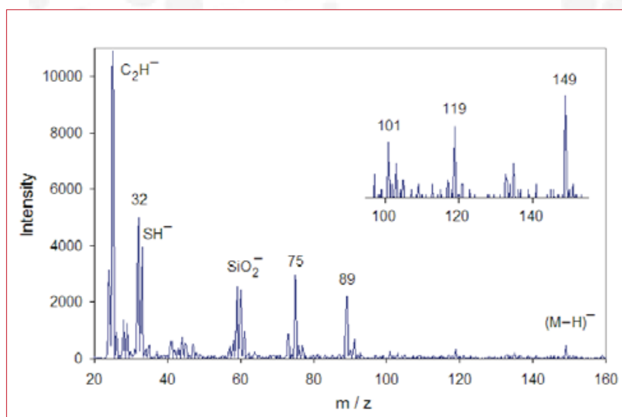

Figure (2) STATIC SIMS SPECTRUM OF THE THIOL MONOLAYER

The spectrum in Figure 2 was obtained with a low primary ion dose from the incident beam, leaving the surface virtually unchanged. However, prolonged bombardment of the surface can be used to sputter away the molecular layer. The ratios of selected secondary ions can be monitored during this process (Figure 3).

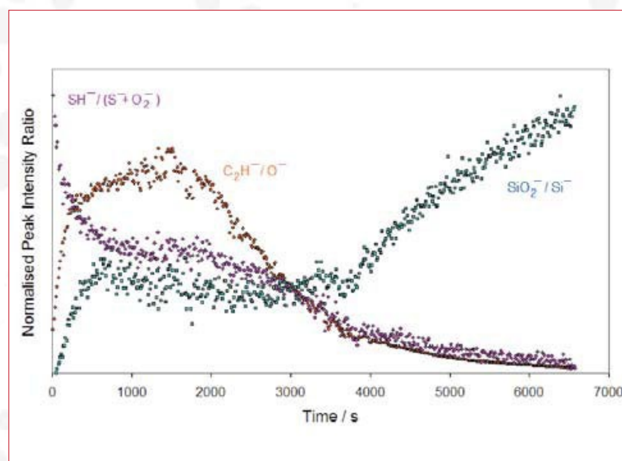

Figure (3) CHANGES IN SECONDARY ION INTENSITY RATIOS AS THE MONOLAYER IS SPUTTERED AWAY

The fragment secondary ion (SH) corresponding to the thiol group is the first to disappear. This confirms the orientation of the molecule on the substrate, with the sulphur containing groups uppermost. The carbon signal (C_2H) remains high as the aliphatic chain is removed, then as the substrate is exposed the SiO_2 silica signal increases to reach a steady state.

The elapsed time for this process to occur is a measure of the amount of material being removed, which reflects in particular the length of the molecular chain.

Subsequent examination of the analysis region by secondary ion imaging confirms these findings. Figure 4 shows SiO_2 peak intensity (red) is the dominant ion where the SAM has been fully removed in the analysis area. At the outer boundaries, where the sample has undergone only minor sputtering, there is a rim of enhanced C_2H signal (green) where the thiol groups have been preferentially sputtered.

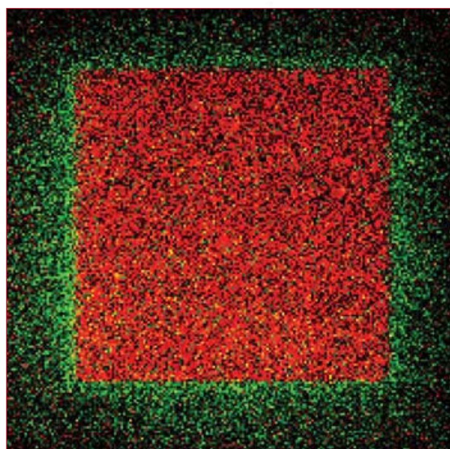

Figure (4) SECONDARY ION IMAGE OF THE 1 MM ^{2} SPUTTERED AREA (SILICA IN RED, CARBON IN GREEN)