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Microtomy Advantages for Cross-sectioning Materials

Discussion

Characterizing the structure of buried defects or layered materials requires a method designed to expose interior surfaces while protecting the three dimensional morphology of the sample. Moreover, when considering subsequent analytical measurements it is imperative to preserve the chemical integrity of the sample. In this note, we compare three methods used to expose the layer structure of a multi-component polymer system (automobile paint sample). The practicality of each method is detailed using scanning electron microscopy (SEM) to define surface morphology and time of flight secondary ion mass spectrometry (TOF-SIMS) to establish surface chemistry relationships on a microtomed surface.

The first technique used to reveal the inner layers of the paint sample utilizes a scribe (e.g. diamond or carbide tip) which helps establish a fracture plane. Unfortunately, the resulting fractured surface often contains a large number of topography related features resulting from the stress associated with tearing through the material. Figure 1a shows a SEM image that clearly resolves the topography associated with the fracturing process. Surface analytical measurements on this surface would be complicated since the information would arise from multiple depths within the respective layers.

In contrast, a smoother surface may be created using a 'sharp' edge to cut the sample. However, the word sharp is relative since the average stainless steel knife/blade possesses a rather wide cutting edge and may still introduce artifacts on the surface. Figure 1b shows a SEM image from the paint chip that has been sectioned using a clean stainless steel razor blade. Although surface roughness has been dramatically reduced, small grooves and debris from the cutting process are noticeable. The forces associated with dragging this 'sharp' blade through the sample are high enough to facilitate material transfer between regions. This phenomenon is called chemical smearing and may severely hinder surface analytical measurements designed to chemically identify specific layers or structures within the cross-sectioned sample.

SEM Images of Fractured, Razor Sliced, and Microtomed Paint Chip

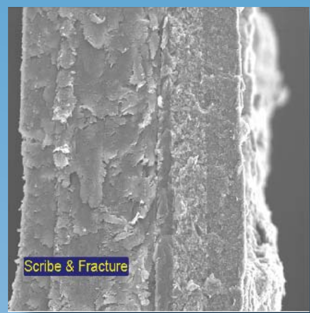

Figure 1a. Sample scribed and fractured with carbide tip

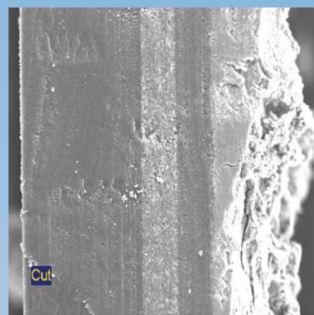

Figure 1b. Sample sliced with stainless steel razor blade

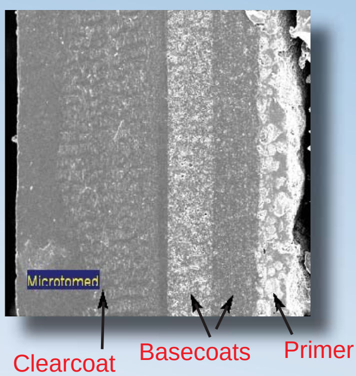

Figure 1c. Sample microtomed at RT using diamond blade

A dramatic difference is observed in the relative smoothness of the surface when using a diamond knife within an ultramicrotome. Figure 1c shows a SEM image where the layers have been preserved and the topography related features have been eliminated. The precision cutting mechanism associated with the microtome also allows the chemical integrity of the sample to be preserved.

Figures 2a-2c are TOF-SIMS images of the individual layers. These chemical images show the lateral distribution of species on the freshly cut surface. Clearly, we can see how the Fe, Al/Ti/Ba/Mg and Cl are localized within the primer, basecoat and clearcoat layers respectively. Consequently, by exposing the internal surfaces of an organic matrix, we are able to utilize surface analytical methods to explore the depth distribution of species within solid organic matrices. This methodology also extends to FTIR, RAMAN and XPS (ESCA) measurements.

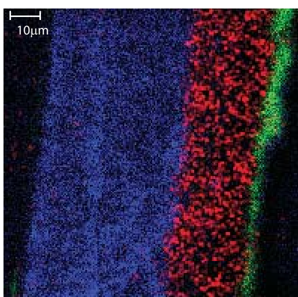

Figure 2b. Chemical Image-Overlay
Green: Fe from primer corrosion
Red: Al & Ti from basecoats
Blue: Cl from Clearcoat

TOF-SIMS Chemical Images of Microtomed Paint Chip

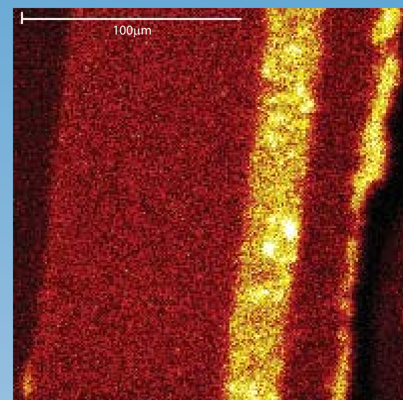

Figure 2a. Total Ion Image showing four layers

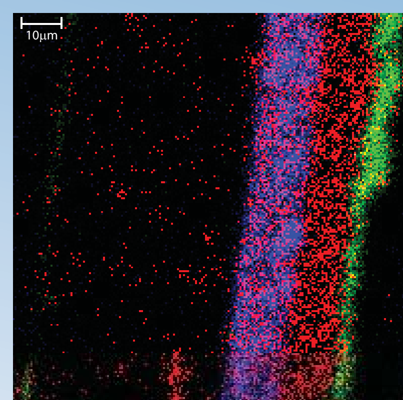

Figure 2c. Chemical Image-Overlay
Green: Fe from primer corrosion
Red: Al & Ti from basecoat
Blue: Ba & Mg from basecoat

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