Application Note: 52126

XPS Analysis of Defects on Painted Surface

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Key Words

- K-Alpha
- Defect Analysis
- Painted Surfaces
- Retrospective Spectroscopy
- Snapshot Spectroscopy
- Surface Analysis
- XPS
- XPS Imaging

X-ray photoelectron spectroscopy (XPS) was used to map a crater defect on a painted surface. The center of the defect was found to be a small tin particle. This impurity was the likely initiator for the defect.

Introduction

There are a range of problems that can affect painted surface production. These include gross faults such as flaking or delamination of the paint film and more subtle effects such as mottling or crater defects. Failures can occur for a variety of reasons. For example, contamination on the surface prior to coating can result in poor adhesion of the paint film. Alternatively problems could occur in the coating itself, such as segregation of the components once applied. XPS is a very surface sensitive technique and the ideal analysis tool for identifying the cause of a particular failure. In this case study the defect under investigation is a crater defect, which appears as a shallow bowl in the film with a raised center.

Experimental

XPS is a rapid and non-destructive technique for characterizing the surface of both conducting and insulating materials. It is quantitive, responsive to changes in chemical state, and extremely surface sensitive.

An image from the Reflex optics system is shown in Figure 1 (crater center indicated with an arrow). XPS images of the defect were collected by rastering the stage under the X-ray beam and collecting 128-channel snapshot spectra at each point. The one-click charge compensation system was used as the painted film was electrically insulating.

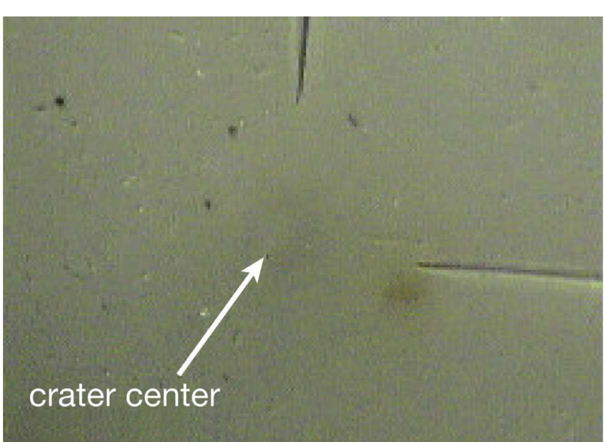

Figure 1: Optical view of the crater defect. The limits of the defect indicated by horizontal and vertical score lines.

Results

The defect was investigated by acquiring wide scan survey spectra from the center and edges to identify the elements present, and then mapped to generate XPS images for the chemistries present. The atomic concentration images are shown in Figure 2. It can be seen that there is tin present at the center of the crater but nowhere else on the surface. In contrast there is depletion of the *N1s* and *O1s* signals at the same point. By peak fitting the spectra associated with each pixel of the image, chemical state images such as those shown for the *C1s* chemical states can be obtained.

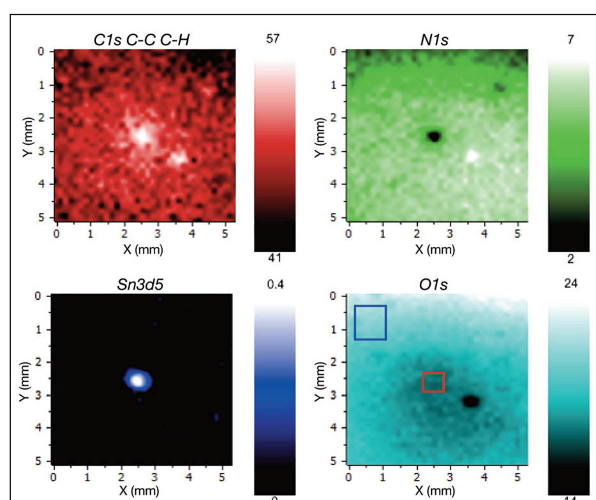

Figure 2: XPS atomic percent images of the crater for relevant chemical states. The indicated areas in the *O1s* image denote the pixels averaged to generate the spectra in Figure 3. A secondary defect can also be seen most noticeably in the *O1s* image to the lower right of the center, which appears to have a different cause.

Again, it can be seen that there is a difference in the carbon chemistry of the defect center compared with the rest of the crater. The *C-C/C-H* component is in greater concentration at the defect center.

Spectra can be retrospectively extracted from images. In Figure 3 the red spectra are created by averaging the area marked with a red box on the *O1s* image in Figure 2. The blue spectra were obtained by averaging the spectra in the pixels enclosed by the blue box. The differences between the individual areas can be clearly seen again. In particular the increase in intensity of the *C-C/C-H* peak in the *C1s* spectrum (indicated by an arrow) is very obvious. Reductions in the intensity of the N1s peaks and the low binding energy *O1s* component (again marked with an arrow) are also easily evidenced from this retrospective spectroscopic procedure.

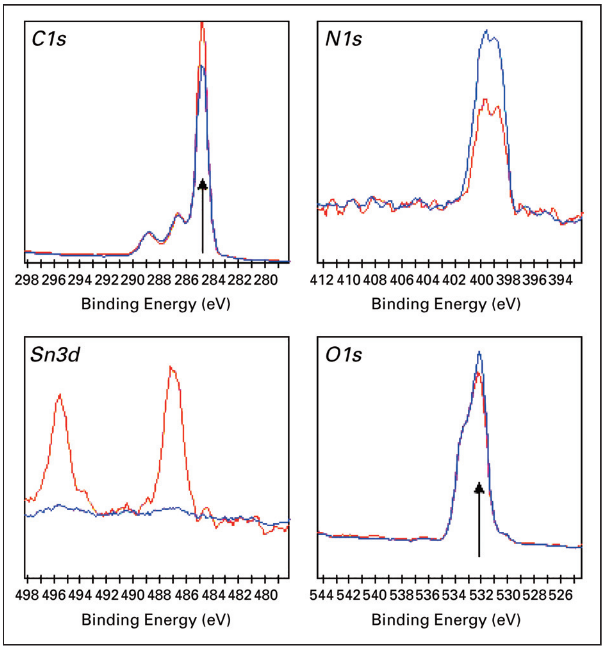

Figure 3: Average spectra generated from the center of the crater (red) the left side of the crater (blue)

This suggests that the presence of the tin particle not only causes the physical defects (*i.e.* the crater structure), but also affects the chemistry of the paint film at that location.

Summary

By using XPS analysis a tin particle was identified at the center of a crater defect. This surface contaminant had affected the film chemistry at the center as well as physically forming the bowl shaped defect in the film. This chemical change was evidenced by an increase in the *C-C/C-H* chemistry at the same point, and corresponding decreases in nitrogen and oxygen concentrations. An overlay of the *C1s(C-C)*, *Sn3d5* and *O1s* XPS images and the optical view is shown in Figure 4, which illustrates this conclusion.

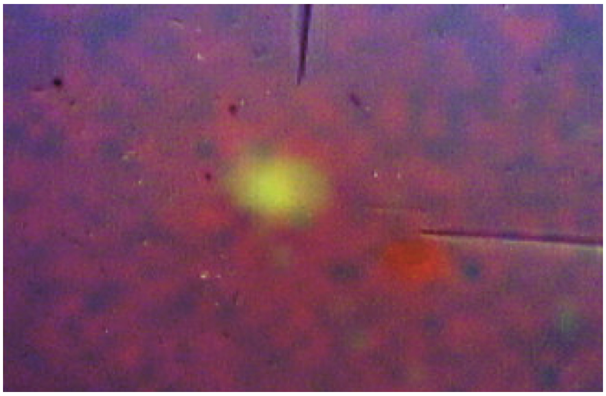

Figure 4: Overlay of the XPS images and the live optical view. *C1s(C-C)* is red, *Sn3d5* is green, and *O1s* is blue

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