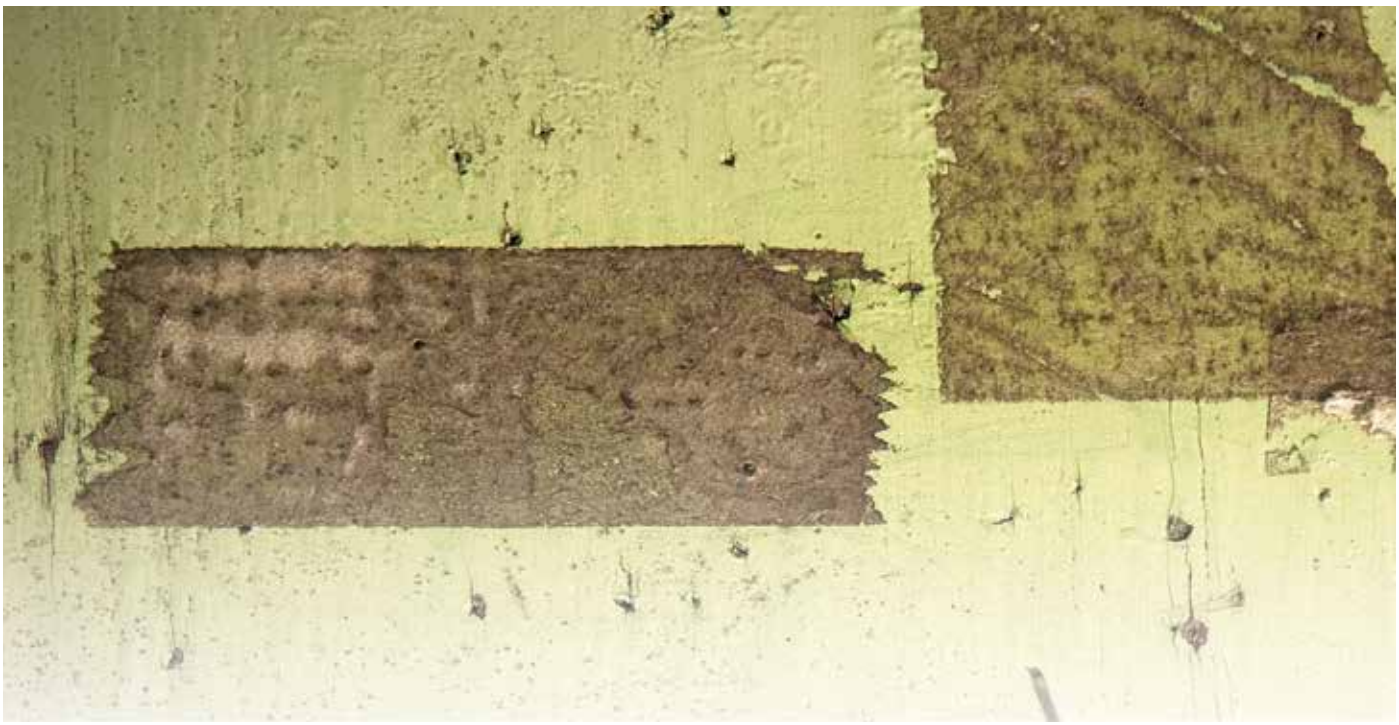
APPLICATION NOTE



Adhesion Failure Analysis

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The Thermo Scientific™ K-Alpha™ X-ray Photoelectron Spectrometer (XPS) System was used to study pressure sensitive adhesives where critical failures had occurred. K-Alpha was able to show that there was a siloxane (silicone) contamination on the failed substrate surface which caused adhesive detachment.

Thermo Scientific K-Alpha XPS

Experimental

A section of the surface where the adhesive had been applied was mapped to evaluate the chemistry across of the 14 mm × 5 mm sample surface. Maps of several elements were acquired to identify differences between the areas where the failure occurred and where adhesive had worked as expected. Figure 1 is an optical image of the sample where the adhesive had failed and left residue on the surface. The image was taken with K-Alpha's unique Reflex optics which allows a continuous live view of the analyzed sample from the same aspect as the analyzer and the detector. The adhesive residue can be clearly seen on the lower half of the image (indicated with the red line).

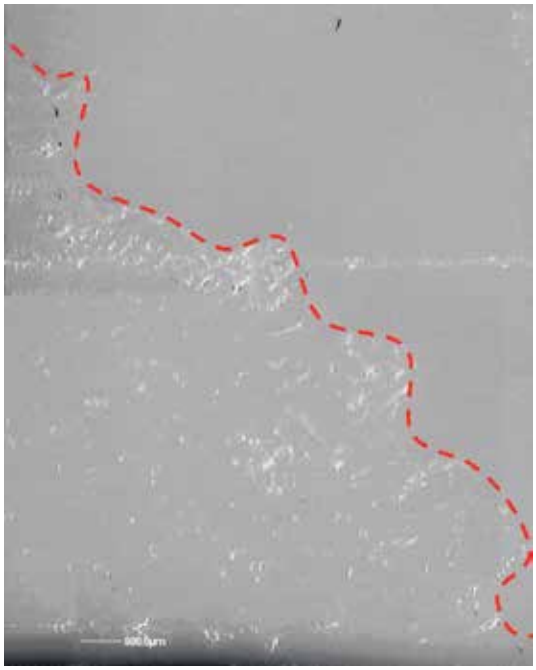

Figure 1: Live Reflex optics image of the sample in K-Alpha. The edge of the adhesive residue is indicated with red line in the lower part of the image.

To produce a map using K-Alpha the sample stage is scanned under the X-ray spot. This means that both very small and very large fields of view are possible (up to

60 mm × 60 mm). The 128-channel detector in K-Alpha makes it possible to collect spectra using rapid snapshot acquisition mode. This enables entire spectral regions to be collected in seconds whilst maintaining the chemical state information that is the principal benefit of XPS. Thus each pixel of the image contains a spectrum of each element under investigation.

As the samples are electrical insulators, the K-Alpha's turn-key charge compensation system was employed to aid the analysis. This system enables analysis of insulating samples with the same ease as conductors.

Results

Figure 2 is a schematic drawing of the problem. The pressure sensitive adhesive was applied between surfaces A and B. When the parts where peeled apart some of the PSA failed to detach from the surface B leaving residue to the surface.

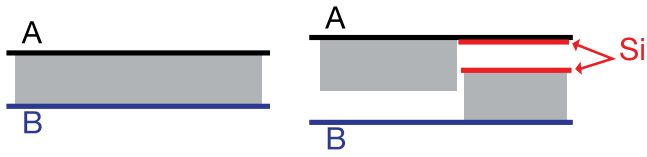

Figure 2: Schematic drawing of the analyzed parts

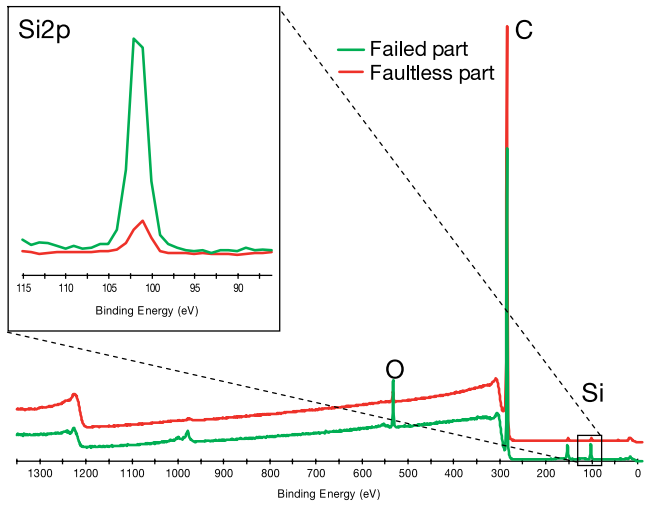

Figure 3: Survey spectra of the two parts of the sample. The main difference is seen in the silicon region. Spectra have been normalized and offset for clarity.

Figure 3 is the survey spectra of the two different parts of the sample. The clear difference in the amount of silicon can be seen between these parts. No other elements that could have caused the failure were observed on the surface.

Figure 4 shows an atomic percent image of silicon over the mapped area of 14 mm × 5 mm. The map shows very high concentrations of silicon in the region where the failure occurred compared to the rest of the sample. The graph beside the map shows the average spectra of Si2p from the indicated areas of the map. The position of the Si2p peak is characteristic of a siloxane chemistry. The likely cause of the problem is therefore silicone contami-nation on the surface A causing adhesion failure, allowing the adhesive to remain on the surface B.

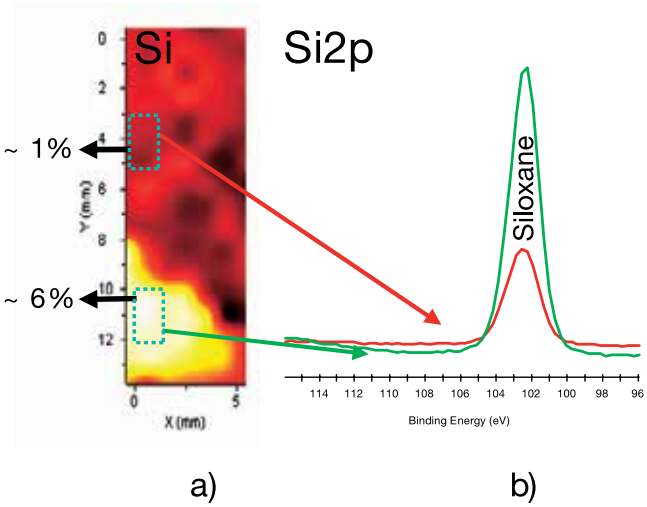

Figure 4: a) Atomic percent map of Si. b) Overlay of average spectra from marked areas.

Summary

The Thermo Scientific K-Alpha XPS was used to identify the cause of failure in pressure sensitive adhesives. Siloxane contamination was found in the failure regions. Further investigations were launched to identify the source of the contamination. Siloxane surface contaminants, even in low concentrations are common causes of adhesion failure, and a surface sensitive analysis technique such as XPS is an ideal method for identifying such contamination.

Keywords

K-Alpha, Adhesion, Contamination, Imaging, Mapping, Surface Analysis, XPS