APPLICATION NOTE

Using the MiniSIMS with XPS to Investigate a Degreasing Process

Mini SIMS

Superior sensitivity and more direct molecular signatures by MiniSIMS complement the information from XPS analysis.

- Clarification of XPS Data by MiniSIMS Analysis
- Extra Information on Organic Structure by MiniSIMS Spectra
- Proof of Contamination Method by MiniSIMS Imaging

As new environmental regulations come into force, changes to production processes are often required. One example is the move from organic solvents to aqueous (surfactant) based degreasing agents. Aqueous solutions pose fewer hazards during use and in subsequent disposal, but treatment parameters must be completely re-optimized for the new solutions. Surface analysis is required to monitor the effectiveness of the cleaning agent, and to detect any residues that could lead to later problems such as corrosion or poor adhesion of coatings.

Monitoring of the surface condition requires an analysis technique with high surface specificity and sensitivity. Both Secondary Ion Mass Spectrometry (SIMS) and X-ray Photoelectron Spectroscopy (XPS) are well-established methods, which give complementary information about the chemical composition of a surface.

Each technique has its strengths; in particular, SIMS offers more information about molecular structure and a rapid imaging capability that is not available with lower cost XPS instrumentation. The extra information gained by visualization of the distribution of a contaminant can often give important clues about its origin.

This analysis examines a metallic component before and after cleaning with a proprietary aqueous degreasing agent. Residues are detected by XPS, but SIMS provides much more detailed information about their chemical identity and reveals the non-uniform distribution of some contaminants.

See overleaf for more detailed information.

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The component was first examined using an entry level (non-imaging) XPS instrument. As might be expected, the XPS spectrum (figure 1) of the uncleaned (blue) component shows a strong carbon peak.

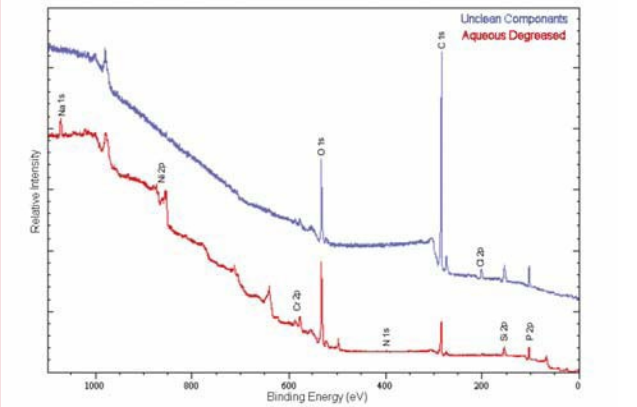


Figure (1) XPS SPECTRUM OF COMPONENT BEFORE AND AFTER CLEANING

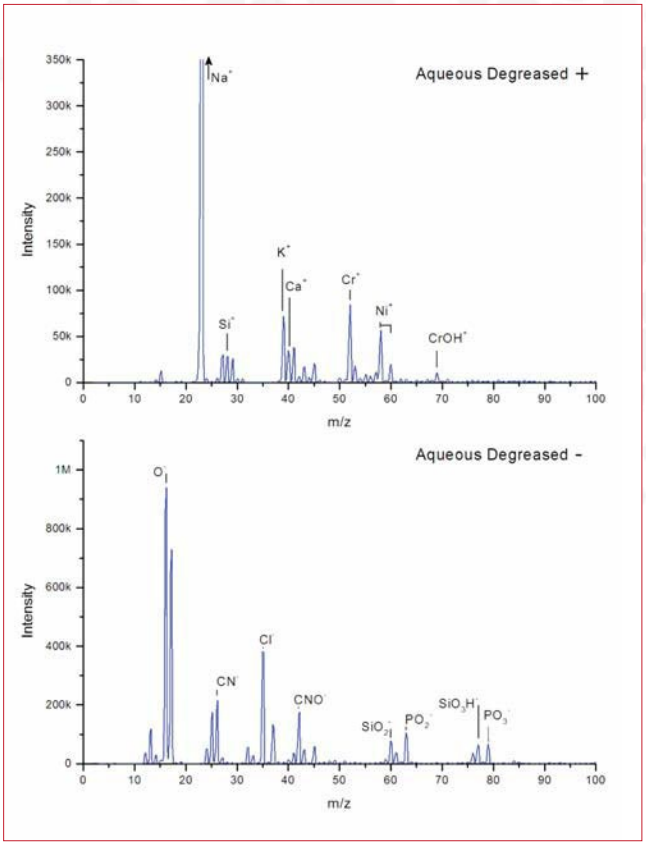
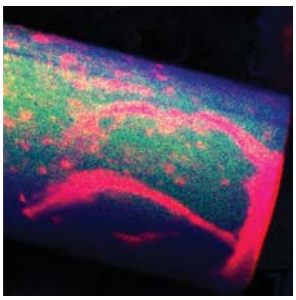


Figure (2) POSITIVE AND NEGATIVE SIMS SPECTRA OF CLEANED COMPONENT

There are weak peaks corresponding to nickel and chromium, which are components of the metal alloy. This suggests the carbon layer is not continuous, and quantification of the XPS data shows carbon constitutes 76 atomic % of the surface layer.

The XPS spectrum after cleaning (red) shows more intense nickel and chromium peaks and a much diminished carbon signal. This indicates the cleaning process has been partially successful. However, sodium, phosphorus and silicon signals are clearly visible in the spectrum, indicating the presence of these elements at the surface. There is trace nitrogen signal due to cyano or nitride species.

The cleaned component was then examined using the MiniSIMSin surface spectrum mode (figure 2). This confirmed the presence of a high level of sodium on the nickel / chromium surface. The chemical species containing silicon and phosphorus were revealed to be silicates and phosphates. The spectrum also confirms the nitrogen signal arises from a CN- species at the surface. Other peaks in the spectra are due to the residual hydrocarbon species.



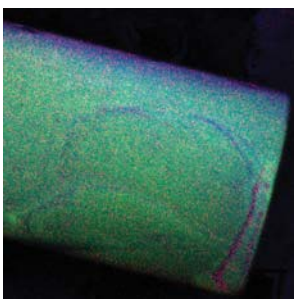
Na^+ (Sodium)
Cr^+ (Chromium)
O^- (Oxygen)

Figure (3a) CHEMICAL IMAGE OF CLEANED COMPONENT (SODIUM IN RED, CHROMIUM IN GREEN, OXYGEN IN BLUE)

The major advantage of the MiniSIMS is that the distribution of these species can be mapped (figure 3), an image being acquired in as little as 30 seconds. Most are seen to be uniform across the surface of the component. However the sodium is clearly present as non-uniform contamination.

The shape suggests that this is a drying stain, implying that the sodium originates from the degreasing agent rather than arising from surface segregation in the metal itself.

Samples and XPS analysis courtesy of Dr. S Jenkins, LPD Labservices, LG Philips +44(0)1254 507377



CH^- (Carbon)
PO_3^- (Phosphorous)
O^- (Oxygen)

Figure (3b) CHEMICAL IMAGE OF CLEANED COMPONENT (CARBON IN RED, PHOSPHORUS IN GREEN, OXYGEN IN BLUE)