Effectiveness of an Aqueous Degreasing Process Evaluated and Improved by XPS

Production Process Problem

A small nickel-chromium based metallic component is routinely cleaned using an aqueous degreasing process following a cutting process using an organic lubricant. If this oil is left in significant quantities on the component surface, it not only interferes with the ease of laser welding to another subcomponent, but also renders the product unfit for functional use.

The component appears optically a different colour if it is not cleaned, but if partially clean can appear similar to a good clean component.

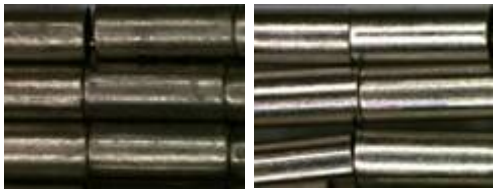

Fig.1. Optical images of as-cut tubes (left) and after aqueous degreasing (right).

Analytical Technique Capabilities

XPS (X-ray Photoelectron Spectroscopy) is a technique which chemically analyses the top few atomic layers of a material. It not only permits the surface elements to be determined, but also the chemical state and atomic concentration (directly from the areas under the peaks).

XPS was carried out on the Ni-Cr metallic components before and after an aqueous degreasing process to evaluate the effectiveness of the cleaning process.

XPS Surface Analysis Results

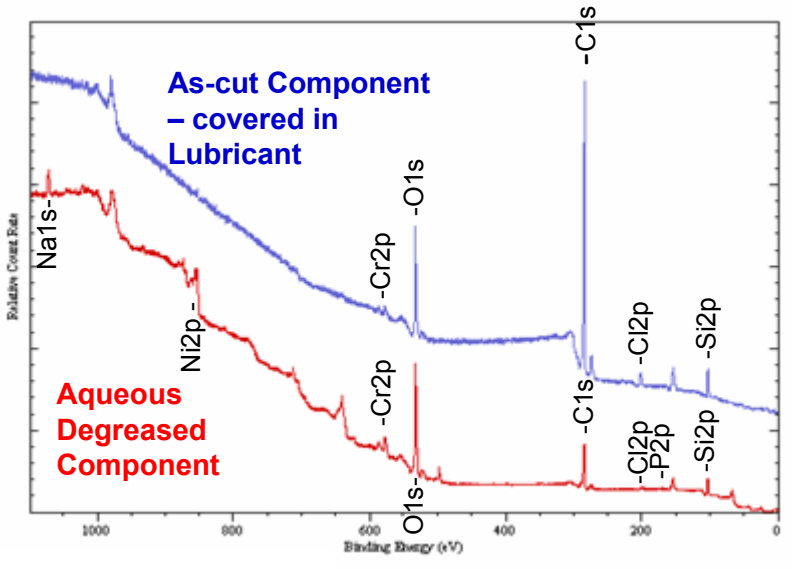

Fig .2. Comparison of the XPS survey spectra from the surfaces from the as-cut tubes and similar components after aqueous degreasing.

Aqueous degreasing of the cut tubes had removed most of the surface carbon (hydrocarbon) and surface chlorine. This cleaning process had revealed the underlying nickel and chromium.

The fact that the surface chromium could be seen in the dirty sample suggests the lubricant was less than a few nm thick. Since there was only subtle evidence of nickel, this suggested the lubricant was a fairly continuous overlayer rather than localised blobs.

(* denotes UKAS accredited test)

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Sodium residue was evident on the surface of the degreased components. There were also trace levels of calcium, phosphorous (present as a phosphate) and nitrogen (present as a cyano or nitride species).

These results indicated the rinsing off of degreasing solution was incomplete and the presence of calcium indicated a poor purity of water was used to either make up the degreaser solution or rinse off the aqueous degreaser.

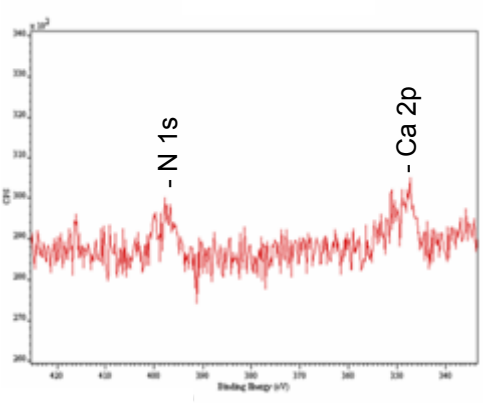
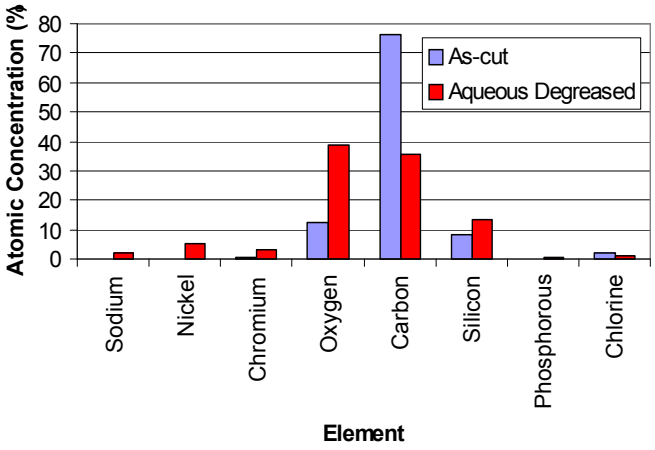


Fig.3. Zoomed survey scan from the aqueous degreased tubes showing the presence of trace calcium and nitrogen as a cyano or nitride species.



	Quantification - Atomic Concentrations (%)	
	As-cut	Aqueous Degreased
Sodium	0.01	2.24
Nickel	0.13	5.25
Chromium	0.49	3.00
Oxygen	12.48	38.96
Carbon	76.27	35.47
Silicon	8.43	13.47
Phosphorous	0.04	0.64
Chlorine	2.16	0.96

Fig.4. Bar chart and corresponding table quantified elemental comparison of the main elements present on the as-cut and aqueous degreased tube surfaces.

Process Problem Solution

The aqueous degreasing process had removed the vast majority of the lubricant exposing the underlying metal, but had left sodium compounds behind on the production component surface which could cause functional problems.

The extent of the cleaning residuals was reduced by more extensive post-cleaning rinsing processes. As XPS was far more sensitive than optical examination, it was used to tune the cleaning and rinsing processes minimising harmful residuals and improve welding process yields and eliminating downstream functional issues.

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