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APPLICATION NOTE

XPS Identification of Stains on Split Steel Bearings

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Thermo Scientific™ K-Alpha™ X-ray Photoelectron Spectrometer (XPS) System was used to identify the elemental and chemical composition of stained areas on split steel bearings.

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Introduction

Steel bearings are typically coated to increase wear resistance, hardness and anti-friction properties. The coating also gives the bearing improved cosmetic appearance and corrosion protection. These characteristics lead to better performance and lifetime of the component.

Thermo Scientific K-Alpha XPS was used to analyze bearings with a steel base and lead/tin eutectic coating (thickness > 100 nm). These bearings were becoming stained after shipment from the factory. The stains did not significantly affect the bearing performance but resulted in customer dissatisfaction due to their appearance.

XPS is a rapid and non-destructive technique for surface characterization. It is quantitative, responsive to changes in chemical state and extremely surface sensitive. In this case XPS was used to identify the chemical changes between stained and normal areas of the bearing.

Experimental

The point-and-shoot capability of K-Alpha was used to identify elemental and chemical differences between stained and unstained parts of the sample. Initially, wide scan survey spectra were acquired on three points of sample which appeared optically different. The images of the sample in the Figure 1 were taken with K-Alpha's unique Reflex Optics system.

The points were subsequently analyzed for chemical differences by acquiring narrow region spectra. Finally, the sample area was mapped by rastering the sample stage under the X-ray beam and collecting 128-channel snapshot spectra at each imaging pixel.

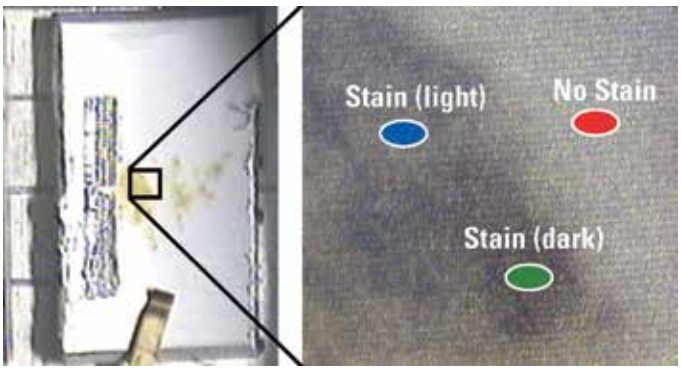

Figure 1: CCD images of the sample taken using K-Alpha's Reflex Optics system

Results

The survey spectra (Figure 2) revealed significant differences in the relative amounts of lead and tin in the unstained and stained areas.

The quantification table (Table 1) shows that there is more tin on the unstained area than on the stained areas. Different compositions of tin/lead eutectics can have subtly different appearances and so the stained appearance may be due to the varying composition of the eutectic coating of the bearing. The stained area also has significantly more carbon, which could affect the appearance of the surface.

Thermo Scientific K-Alpha XPS

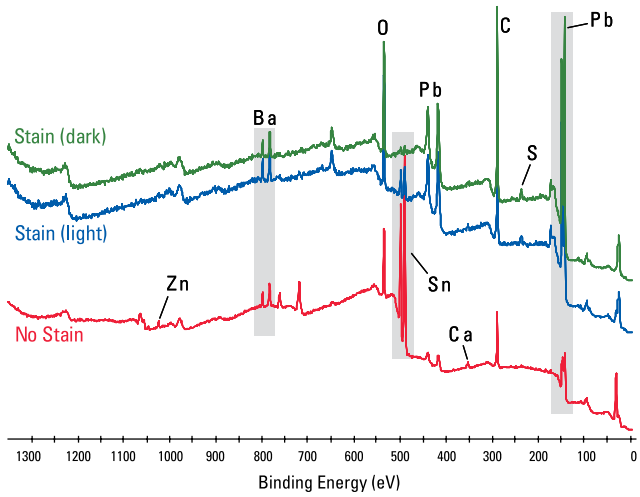

Figure 2: Elemental identification at the three analysis points

	Atomic Concentration / %		
Element	No Stain	Stain (light)	Stain (dark)
C	52.0	70.5	72.4
O	30.0	21.2	19.7
Pb	4.0	6.6	6.9
Sn	12.4	1.0	0.2
Ba	1.1	0.7	0.5
Zn	0.5	(tr)	(tr)

Table 1: Quantification table of the elements identified from the survey spectra

Chemical state analysis using high resolution Pb and Sn spectra of the unstained area revealed a mixture of lead metal and lead and tin oxides as can be seen in Figure 3 and 4. Significant differences in the relative amounts of chemical states were found. In the stained areas, which have very low tin concentrations, the lead was almost entirely oxydized. The formation of a tin oxide coating in the unstained area may protect underlying lead from being fully oxydized within the XPS sampling depth. The stained regions do not have this protection.

XPS maps of the sample show the distribution of the elemental and chemical states across the bearing surface. Quantified imaging of the bearing surface confirmed the higher levels of carbon and lead oxide on the stained areas. The K-Alpha's data system, Avantage, allows the user to create concentration ratio maps. The map of tin to total lead ratio (Figure 5) shows the correlated variation in concentration of these two elements.

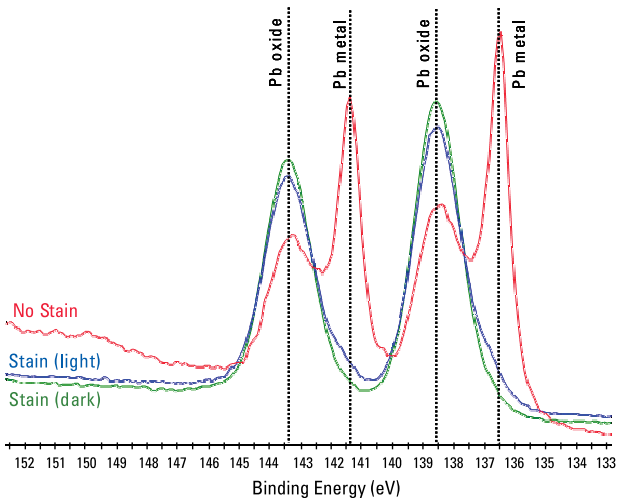

Figure 3: Chemical state analysis of Pb from the three analysis points

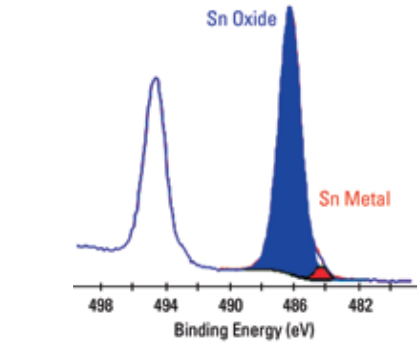

Figure 4: Tin spectrum from unstained area

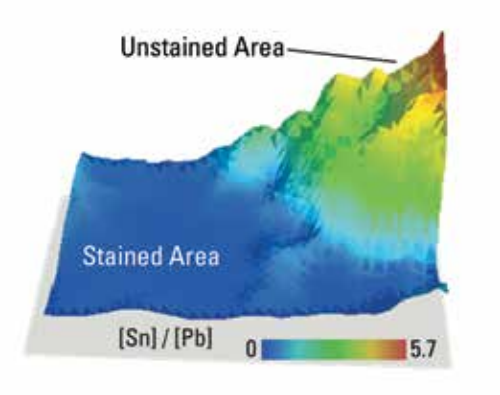

Figure 5: Map of ratio of tin to total lead

Summary

By using Thermo Scientific K-Alpha XPS to analyze the stains on steel-based bearings it was straightforward to detect the differences between unstained and stained areas. The stained areas were found to have low tin, high carbon and high lead oxide concentrations. The XPS results are consistent with a number of possible causes for the staining, such as problems with the eutectic prior to deposition or the way the eutectic was deposited, deposition of a carbon-based contaminant, or loss of a protective layer that allowed "corrosion" to take place in that area causing the stain.

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Notes

Keywords
K-Alpha, Bearing, Mapping, Metal, Stain, Steel, Surface Analysis,XPS

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