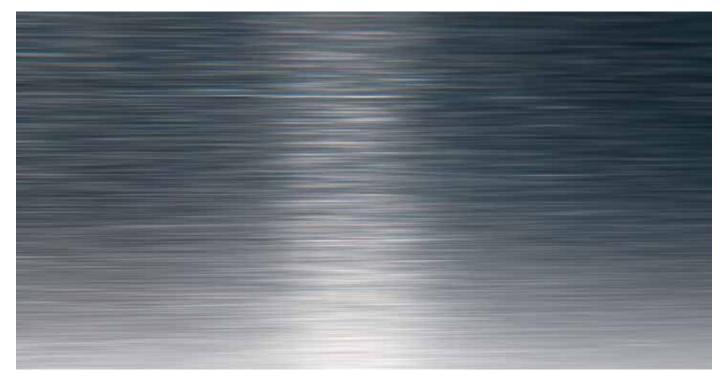thermo**scientific**

Fast, Effective Analysis of Metal Components

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TThe Thermo Scientific™ K-Alpha™ X-ray Photoelectron Spectrometer (XPS) System was used to investigate a tarnished aluminum component. By using X-ray photoelectron spectroscopy (XPS), identification and quantification of chemical differences between optically distinct areas on the sample was possible.

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Introduction

XPS sample analysis can be a very involved process, with operators requiring a high skill level to load samples, collect data and interpret the results. The K-Alpha surface analysis system enables researchers to utilize this powerful analytical technique via a unique combination of hardware design and intuitive software, without compromising information content or performance. The K-Alpha system is controlled by Thermo Scientific Avantage software, which controls all aspects of the sample analysis. Safe sample transfer protocols, and a point-and-shoot user interface via live camera views, are designed to make sample loading, navigation and analysis alignment quick and easy.

When this user-friendly operation is coupled with powerful, integrated analytical tools such as "Multi-spectrum insert", "Survey ID", and full peak deconvolution, performing an XPS point analysis is quite easy to perform.

In this application note we describe how the K-Alpha can be used to analyze a tarnished aluminum part to obtain chemical state information from different regions of the surface and understand the source of discoloration observed on the surface.

Method

The sample was mounted onto the 60 mm × 60 mm sample platter by the use of two supplied spring clips (see Figure 1). Latex gloves were worn to prevent the sample or platter from being contaminated. The sample was then loaded into the fast entry load-lock, and the button pushed in the software to pump down and transfer the sample carrier into the analysis chamber. Sample transfer occurs automatically when the pressure in the load-lock reaches an acceptable level.

Figure 1: Picture of the aluminum sample mounted on the K-Alpha sample block

Once the samples are loaded into the analysis chamber, double-clicking on the platter image moves to a position of interest. A point analysis object can be inserted by first choosing "Point" from the drop-down menu; then, whilst holding down the "control" key on the keyboard, left clicking on the live optical view where you wish the centre of the analysis position to be (see Figure 3).

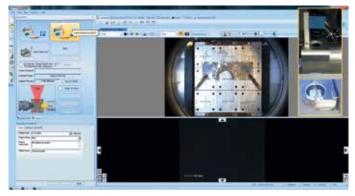Thermo Scientific K-Alpha XPS

Figure 2: Screenshot of the platter image acquisition, with inlay of the platter view camera configuration
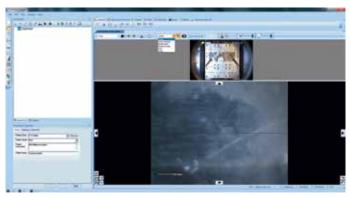

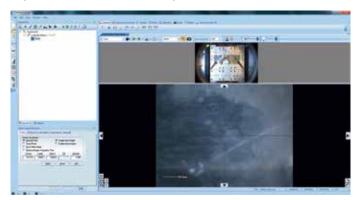Figure 3: Screenshot of inserting a "Point"

Figure 4: Screenshot after inserting a "Point"

This simultaneously inserts an X-ray object and a point object in the experiment tree (on the left of the screen) and on the optical view draws an ellipse, representing the size of the X-ray spot, at the analysis position (see Figure 4). The size of the X-ray spot defines the analysis area because the XPS signal is only obtained from the location on the sample that the X-ray is irradiated. The X-ray spot size is easily adjusted using the mouse-wheel to allow it to be tailored to the size of the feature being analyzed.

Next, the "multispectrum" tool can be used to insert a survey spectrum (see Figure 5) and the experiment can be run. A survey spectrum is a full-range scan that can be used to identify the elements present at the surface, and to produce an elemental quantification. These spectra are usually lower resolution but have high count-rates to facilitate detec-tion of weaker signals. It also enables detection of unexpected elements, and so are used to find all the elements that are present before collecting high-resolution, region spectra.

Once collected, the survey spectrum is dragged into a processing view and the automated "Survey ID" tool is used to analyze the data using an elemental fingerprinting algorithm. The results of this, including the elemental quantification table, are shown in Figure 6.

Based on the results of the survey spectrum analysis, region scans can be inserted using the

"Multi-spectrum insert" tool by clicking on the required elements in the periodic table (see Figure 7 and Figure 8). This allows the user to setup the region scans without having to know the individual peak positions or any other acquisition parameters.

After the experiment has completed, the region scans can be dragged into a processing view for quantification, either by adding or fitting peaks. Adding peaks is done by placing the range cursor at either end of the peak, clicking the "add peak" tool and it will integrate all the peak area between the data and the chosen background type (see Figure 9).
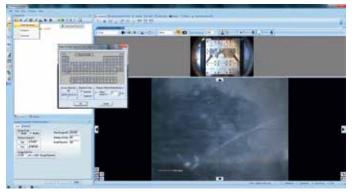

Figure 5: Screenshot of inserting a survey scan using the "Multi-spectrum insert" tool
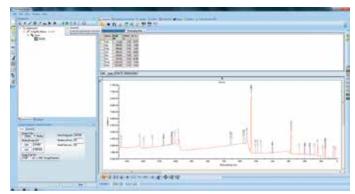

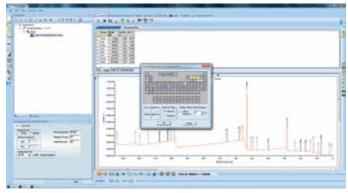Figure 6: Screenshot of survey scan after "Survey ID"

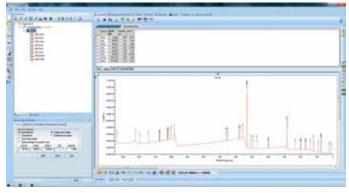Figure 7: Screenshot of using the "multispectrum" tool to insert region scans

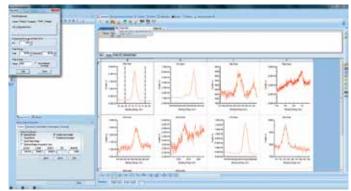Figure 8: Screenshot after inserting region scans

Figure 9: Screenshot of adding peaks to a spectrum

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If a peak fit is required to deconvolve overlapping peaks from different chemical states then, once the range is selected, the "peak fit" tool can be used to add and fit peaks to the spectrum (see Figure 10).

Once all the regions scans have been processed, the full chemical state quantification is shown in the peak table above the spectra (see Figure 11).

This whole process can be repeated to give multiple point analyses in one experiment. Figure 12 shows the experiment setup to run three consecutive point analyses in the brown, grey and shiny parts of the sample. The software can also be configured to run this process automatically (Auto-Analysis mode), where the experiment is created

on-the-fly by the instrument based on analysis of the survey spectrum, and data is exported to a Microsoft® Word® report.

Results

Table 1 shows the comparison table of the chemical state quantification for all three analysis points.

Name	Shiny Area At. %	Grey Area At. %	Brown Area At. %
Al2p Al_2O_3	20.11	21.38	8.64
Si2p SiO_2	0.36	0.23	0.60
S2p sulfate	0.60	0.40	0.23
Cl2p organic	0.25	0.32	0.46
C1s C-C/C-H	17.78	9.51	11.88
C1s C-O	11.43	13.36	10.31
C1s C=O	3.01	2.70	2.63
N1s organic	0.75	0.55	0.59
O1s	45.10	51.08	53.57
Fe2p Fe_2O_3	0.27	0.18	10.14
$Zn2p_{3/2}$ ZnO	0.16	0.18	0.82
Na1s	0.18	0.12	0.14

Table 1: Full chemical state quantification for all three analysis points

The quantification shows that the shiny and grey areas have very similar chemistries, with the only clear differences being the levels of carbon and oxygen. The brown area has over 10% of Fe_2O_3 and a significant increase in the amount of ZnO as compared to the two other analysis points. As the iron oxide is on the surface, the signals from the elements beneath (most notably aluminum) are reduced. This attenuation of the substrate signal allows the thickness of the iron oxide layer to be calculated using another tool in the Avantage™ data system, based on a simple two-layer model. Using this tool, the iron oxide thickness is 1.5 nm. The identification of increased levels of zinc oxide and iron oxide enabled the analyst to determine the cause of the discoloration on the sample and hence the failure of the component.
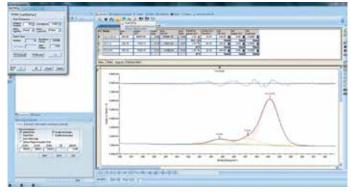

Figure 10: Screenshot of fitting peaks to a spectrum
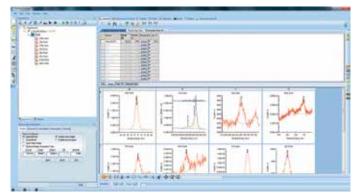

Figure 11: Screenshot of full chemical state quantification after all spectra have been analyzed

Figure 12: Screenshot of the experiment tree setup to run multiple point analyses

Summary

The K-Alpha XPS system can be used to analyze points of interest on the surface of a sample quickly, easily and reliably by making use of the user-friendly experiment setup interface and the integrated data-processing tools.

Find out more at **thermofisher.com/xps**

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