Application Note: 31094

K-Alpha: A New Concept in XPS

Key Words

- Surface Analysis
- Automation
- Integrated Design
- Monochromated, Small Spot XPS
- Optimized Geometry

Thermo Scientific K-Alpha has been designed to maximize the throughput and efficiency of the Surface Analysis laboratory. Many of the repetitive, routine activities have been automated or simplified without compromising the performance or versatility demanded by the XPS expert.

Description

K-Alpha is a compact and fully integrated XPS system. Figure 1 shows the complete instrument, there is no additional electronics rack to add to the configuration, all of the electronics are built into the unit along with the spectrometer. A PC running Windows® XP, connected to K-Alpha via a single USB cable, controls all aspects of the instrument. Figure 2 shows the way in which some of the internal components are arranged within the instrument enclosure.

Figure 1: The new Thermo Scientific K-Alpha

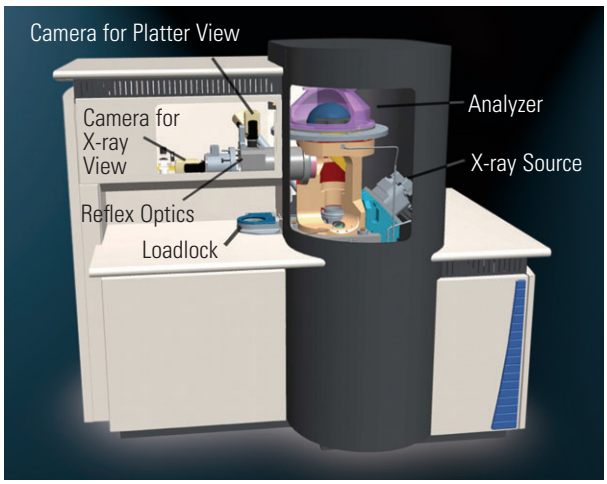

Figure 2: Some of the component parts of K-Alpha

The geometry of all of the analytical components has been given careful consideration in order to optimize performance; their arrangement can be seen in Figure 3. Some of the important features of this arrangement are:

- The axis of the transfer lens is parallel to the sample surface normal, ensuring maximum collection efficiency.
- There is always a live view of the analysis position via the Reflex Optics (patent pending) and this view is also parallel with the sample normal, providing the best possible conditions for identifying the analysis position with great accuracy.
- The height setting camera ensures that the sample is in exactly the right position for small area analysis and at the right height for optimum sensitivity.
- X-rays are delivered from a microfocusing monochromator. This allows the user to define the analysis area and ensures that maximum sensitivity is achieved during small area analysis.
- The new ion gun will provide depth profiles with excellent depth resolution. It has been designed to deliver a beam of low energy ions into a small spot so that resolution is combined with speed of analysis.
- The new flood gun is designed in such a way that the user only needs to switch it on when required for the analysis of insulators, no adjustment or optimization is required.
- Sample illumination is as important as high quality live sample viewing. K-Alpha is equipped with two types of lighting. One is arranged so that the lighting is co-axial with the reflex optics, ideal for samples having a smooth surface. The other light source is off-axis which is ideal for rough samples.

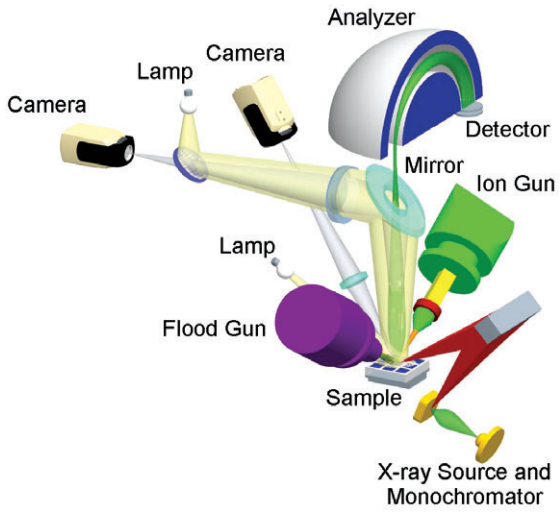

Figure 3: The internal geometry of the analytical components in K-Alpha

The analysis chamber is precision machined from a single billet of nickel-iron alloy. This provides excellent magnetic shielding and the precision of manufacture minimizes the requirement for alignment of the components. It is pumped using a turbo molecular pump, backed by a 'dry' scroll pump, and a sublimation pump.

Sample Introduction

The sample holder, Figure 4, can accommodate samples up to 60 mm x 60 mm and up to 20 mm thick. Samples are easily secured by means of a simple clip, Figure 5.

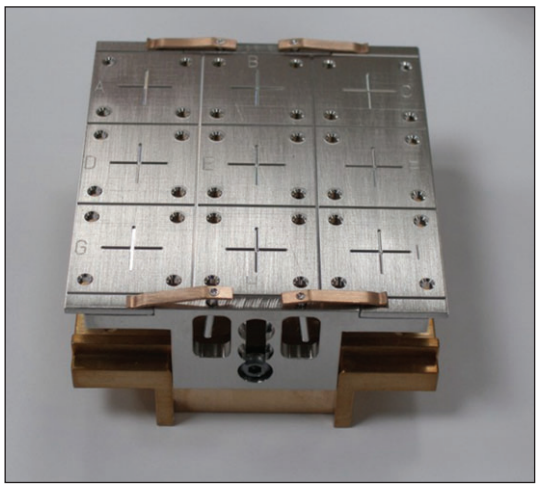

Figure 4: The sample holder for K-Alpha.

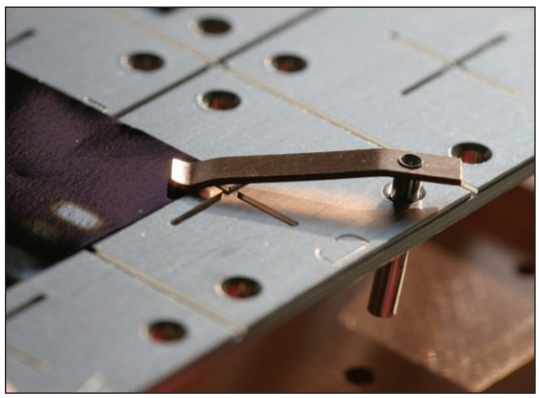

Figure 5: Sample clipped in place on K-Alpha sample holder

Sample transfer is fully automated. Once the sample holder, loaded with samples, is placed in the loadlock and 'Sample Load' is selected from the data system, the loadlock door is automatically closed and pumping begins. Once the loadlock has reached a suitable pressure the sample block is automatically transferred to the analysis chamber.

Selection of the Analysis Position and Analysis Area

An image of the whole sample holder (the 'Platter View') is recorded as soon as the loadlock door is closed. This image is used for the coarse navigation between samples. The analysis positions on each of the samples along with the spectra, linescans, maps or profiles required can be defined using this image. This can be done while the loadlock is pumping so that analysis can begin as soon as the samples are transferred to the analysis chamber.

In addition to the Platter View, a live, high-magnification Reflex View of the sample in the analysis position is always available for the accurate alignment of small features, Figure 6.

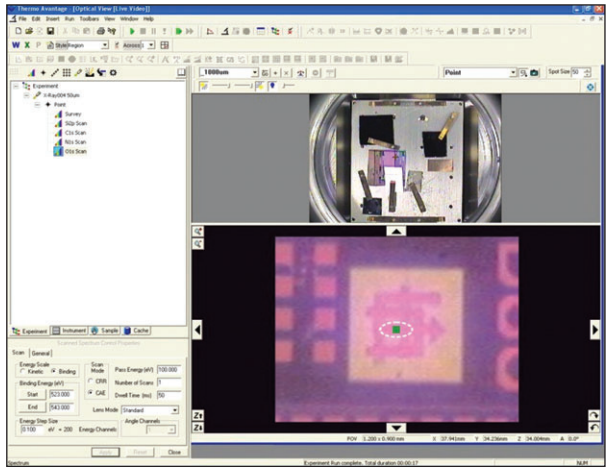

Figure 6: Screen shot from the Avantage data system showing two optical views. The upper image is the Platter View, recorded at sample entry, showing all of the samples loaded onto the sample holder. The lower image is the live Reflex View of the analysis position. In each case the analysis position is clearly marked. Both views are always available to the analyst.

In order to select a position for analysis, the user simply clicks on the appropriate position on either of the two images. Also visible in Figure 6 is a small ellipse surrounding the analysis position. This represents the size of the X-ray spot and hence the analysis area. The spot size is continuously variable and is controlled by the analyst simply by selecting the ellipse with the mouse and dragging it to the appropriate size. This means that the spot size can be accurately matched with the size of the feature to be analyzed, minimizing the analysis time. The maximum spot size is 400 µm and the minimum is 30 µm.

Sample Height Setting

It is important that the sample height is set correctly for three reasons:

1. To ensure that the sample is accurately aligned with the analysis position
2. To optimize the lateral resolution
3. To maximize sensitivity

In order to ensure that the sample height is correct a third optical view is always available to the analyst, this comes from the 'Height Setting' camera which is accurately aligned with the analysis position but is positioned at an angle to the sample normal. This means that the sample is at the correct height when the same feature is centered in both the Reflex Optics view and the view from the Height Setting Camera.

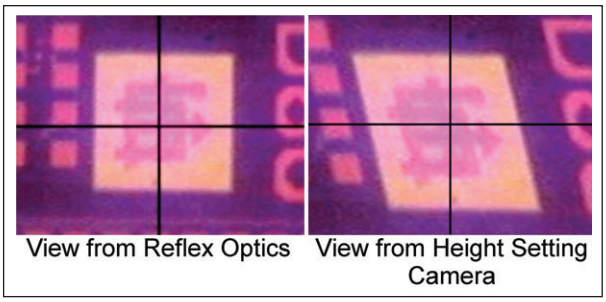

Figure 7: Views of the sample in the analysis position. The same feature is centered in each view and so the sample is at the correct height.

Sample Illumination

When selecting the analysis position, the provision of high-quality illumination is as important as the provision of high quality sample viewing. For that reason K-Alpha has two light sources. One of them is co-axial with the live Reflex Viewing microscope and this is ideal for illuminating smooth, reflective samples. The other is off-axis and is suited to rough samples. Figure 8 illustrates the effectiveness of the two types of lighting for three different samples. This aspect of K-Alpha is discussed in more detail in Application Note AN31091.

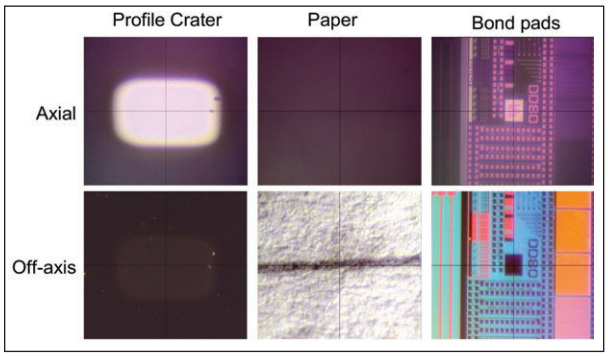

Figure 8: Three sample types each illuminated by axial and co-axial lighting

Insulator Analysis

K-Alpha is fitted with a new flood source for charge compensation. Figure 9 shows high quality C 1s spectra obtained from a series of polymers. All of these polymers were loaded into K-Alpha simultaneously and each analyzed in turn without the need to make any adjustment to the charge compensation conditions.

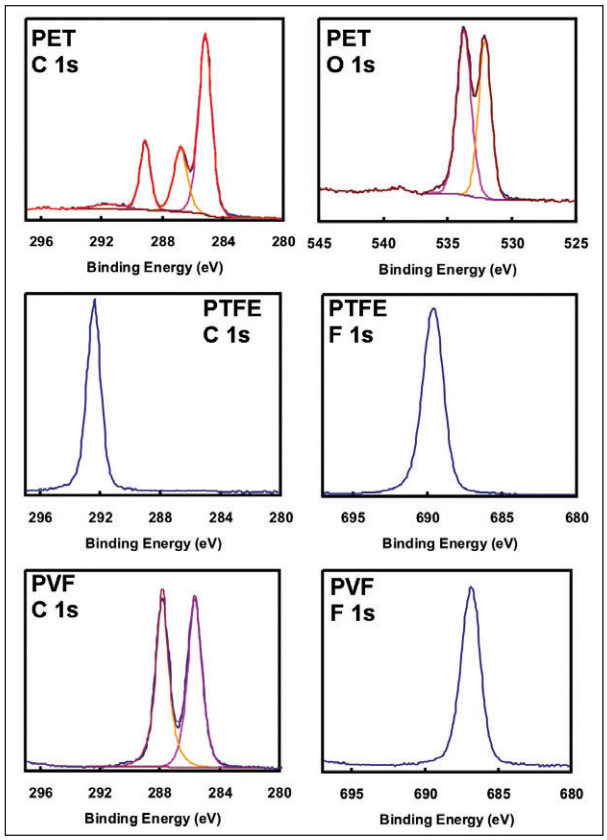

Figure 9: C 1s spectra from a series of polymer surfaces

Chemical State Mapping

The multi-channel detector in K-Alpha means that it can collect high-resolution snapshot spectra. This means that a spectrum can be collected at each pixel of an image. All of the data processing options in the Avantage data system can then be applied at each pixel including peak fitting and non-linear least squares fitting. This allows the analyst to produce quantitative chemical state maps, such as the ones shown in Figure 10. For a more detailed description of the imaging capabilities of K-Alpha, see Application Note AN31093.

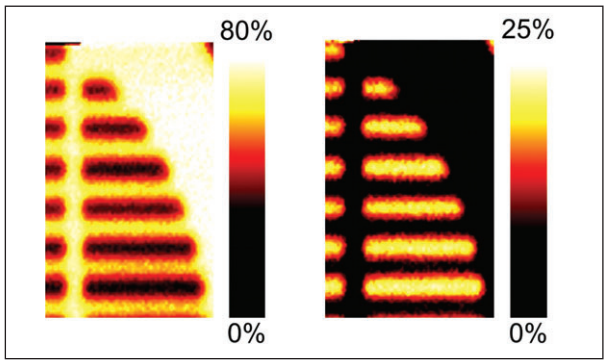

Figure 10: (a) Atomic concentration map from the hydrocarbon peak following peak fitting at every pixel. (b) Atomic concentration map from the fluorocarbon peak.

Depth Profiles

K-Alpha is equipped with a new ion gun with excellent performance, especially at low energy. This means that K-Alpha routinely produces depth profiles having superb depth resolution, see Figure 11. For optimum depth resolution from all sample types, azimuthal rotation can be used during sputtering, if required.

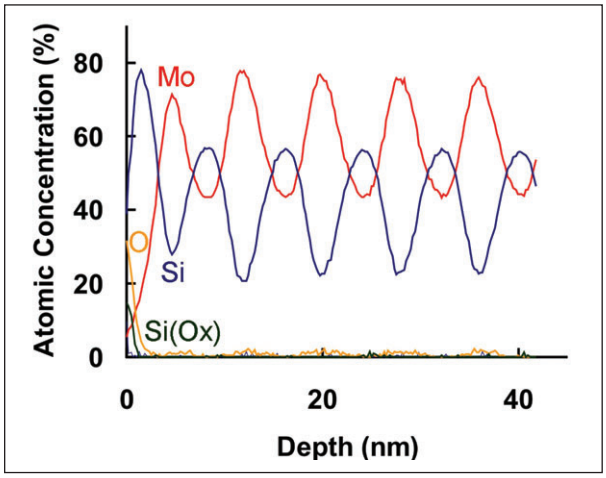

Figure 11: Depth profile of an X-ray mirror having 7 nm repeats. The argon ion energy used for this profile was 200 eV.

Auto-reporting

The Avantage software can remove the need for many of the repetitive tasks associated with the analysis. One example of this is its capability to produce reports automatically. The report contains the images of the samples and of the area analyzed, all of the spectra collected and the quantification table for each of the samples analyzed.

Auto-analysis

K-Alpha also has an 'Auto-analyze' routine. Using this feature, the analyst simply loads the samples and indicates on the Platter View image the positions at which analysis is required. The instrument will then perform the following steps automatically:

4. Pump the loadlock and transfer the sample
5. Collect a survey spectrum
6. Identify the elements present in the survey spectrum
7. Quantify the spectrum
8. Collect high-resolution spectra from the elements identified
9. Perform a chemical state appraisal of the elements identified
10. Move to the next sample and repeat from 2, above
11. When all samples have been analyzed a report is generated
12. Samples returned to loadlock so that K-Alpha can be loaded with a new set of samples

Conclusion

K-Alpha is a new departure for XPS. Every aspect of the analysis process has been simplified or automated with a view to increasing both the throughput of the laboratory and the reliability of the data produced. It is a high-performance instrument that provides the tool you need to get the job done.

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