Application Note: 31056

XR5: Microfocus X-ray Monochromator

Key Words

- Surface Analysis
- ARXPS
- Source Defined SAXPS

Introduction

The Thermo Scientific microfocused X-ray monochromator, Figure 1, produces an X-ray beam with a very narrow energy spread.

Figure 1: XR5 microfocus monochromator

Source Defined Analysis Area

The microfocusing capability of the XR5 provides maximum sensitivity combined with excellent energy resolution from selected areas, over a wide range of spot sizes. Its major features include:

- 500 mm Rowland circle
- Twin toroidal crystals
- Crystals aligned independently
- Spot size range 120 µm to 650 µm
- Moveable anode
- Focusing electron gun replaces filament as cathode
- Digital control using *Avantage* data system

This monochromator is based on the design used for both the ESCALAB 250 and MultiLab 2000 instruments, see for example Figure 2.

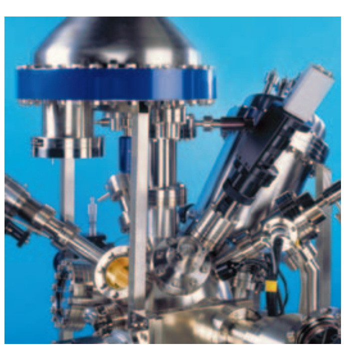

Figure 2: MultiLab 2000 equipped with the XR5 X-ray monochromator

The XR5 monochromator focuses X-rays to provide small area XPS capability with very high sensitivity and resolution.

Small area XPS measurements can be made without reducing the sensitivity of the spectrometer. Consequently, the sensitivity is improved and the analysis time reduced.

Only that part of the sample which is being analyzed is exposed to X-rays. Sensitive samples do not therefore become damaged in areas remote from the analysis position.

In angle-resolved measurements, the whole of the X-ray spot is within the analysis area over the range of sample angles.

Operation of the X-ray Monochromator

Figure 3 illustrates the way in which the XR5 operates. The electron gun focuses high-energy electrons onto a water-cooled aluminum anode causing X-rays to be emitted. Some of these X-rays are intercepted by the twin quartz crystals, which allow only a narrow range of X-ray wavelengths to be diffracted and reach the sample. Additionally, the crystals are toroidally curved so they focus the X-ray beam to produce a small spot at the sample. The size of the X-ray spot on the sample is then determined by the size of the electron spot on the anode. It is possible, therefore, to control the analysis area by selecting the appropriate X-ray spot size.

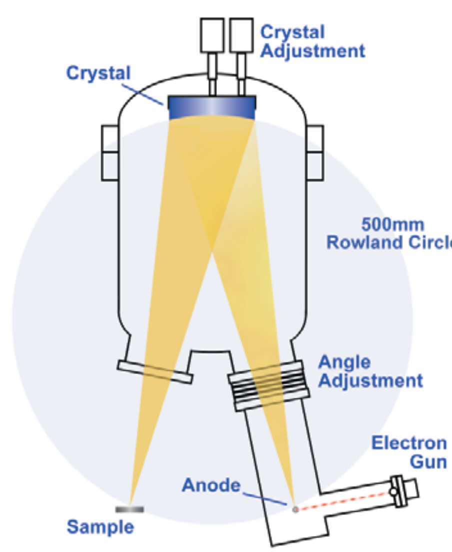

Figure 3: Schematic of the XR5 X-ray monochromator

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Electron Gun

The integral electron gun focuses the electron beam onto the X-ray anode. This provides control of the X-ray spot size. Maximum flux density can be obtained at the sample resulting in short acquisition times. The electron gun defines the area of X-ray emission from the anode, and, in consequence, the X-ray spot size on the sample. The analysis area is defined by the source rather than the analyzer lens, providing very high sensitivity and exceptional energy resolution.

Moveable Anode

To extend the lifetime of the water-cooled, aluminum anode, it is fitted with a linear drive mechanism to enable alteration of the anode position relative to the electron gun beam. This allows the emission point of the anode to be altered at least 20 times, greatly increasing service intervals. The position may be adjusted without breaking the vacuum.

Crystals

The XR5 features a twin, toroidal crystal design, providing high X-ray flux and improved energy resolution. Independent manipulators position the crystals. This ensures precise alignment of the X-ray spots for optimum small area analysis and maximum flux density.

Digital Power Supply

The XR5 is controlled from a digital power supply using the *Avantage* data system. This provides digital control over all aspects of the source, including spot size and power.

The digital power supply for the XR5 also operates the XR3 and XR4 sources. Two sources can be connected to, and controlled by, a single power supply.

Advantages of Using an X-ray Monochromator

There are a number of advantages in using an X-ray monochromator for XPS measurements.

Better Energy Resolution

A monochromator produces an X-ray beam with a narrower energy spread than a non-monochromated source. This can be seen in Figure 4 which compares the X-ray spectra from non-monochromated and monochromated Al Kα radiation. For XPS, this means that the peaks can be much narrower if a monochromator is used.

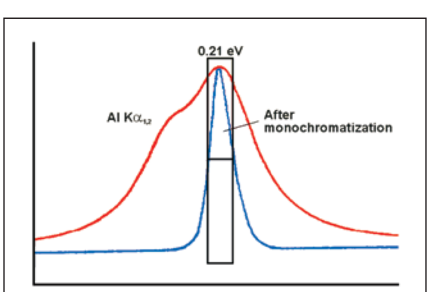

Figure 4: X-ray spectra from non-monochromated and monochromated Al Kα radiation

Removal of Interference from X-ray Satellites

The monochromator removes X-rays other than Al Kα from the beam. These X-ray satellites cause additional XPS peaks to appear in the spectrum.

Reduced Background Signal

By removing the bremsstrahlung radiation from the X-ray beam, the XPS spectra have a lower background leading to improved detectability.

The above benefits are all illustrated in Figure 5, which shows the Ag 3d XPS spectrum from metallic silver. The analyzer settings were the same in each case.

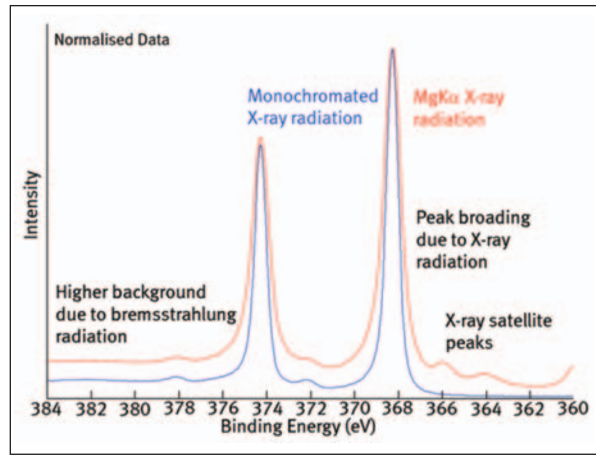

Figure 5: XPS spectra from silver, acquired using both monochromated and non-monochromated X-rays

Improved Chemical State Information

Narrower peaks and a lower background mean that subtle changes in the chemistry of a surface can be more easily identified.

Access to Valence Bands Using XPS

The improved energy resolution combined with a high X-ray flux means that the additional information available from the valence band becomes available to the analyst. Figure 6 shows the valence band and Fermi edge from metallic silver, illustrating both the sensitivity and resolution available using the XR5 monochromator.

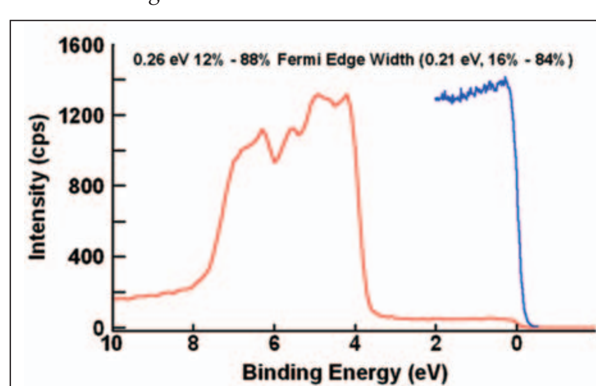

Figure 6: Ag 4d and Fermi edge spectra from metallic silver

In this case, the Fermi edge resolution is 0.21 eV, using the 16% to 84% definition.

Reduced Sample Heating

When using a monochromator, the distance between the X-ray source and the sample is much larger than when using a non-monochromatic source. This means that there is much less radiant heat reaching the sample and consequently less risk of sample damage.

Increased Space for Additional Multi-technique Components

When using a non-monochromatic X-ray source, it is usually necessary to place the source very close to the sample in order to optimize sensitivity. The need to do this restricts the solid angle available for other components. The XR5 places much less of a restriction on the solid angle.

Advantages of Using a Microfocus Monochromator

In general, small spot X-ray monochromators also provide better energy resolution than large spot monochromators. A microfocusing monochromator has additional advantages.

Maximum Sensitivity for Small Area Analysis

When making small area XPS measurements, it is not necessary to reduce the transmission of the electron spectrometer in order to define the analysis area properly.

The benefit of source-defined small area XPS, compared with lens-defined small area XPS, increases with decreasing analysis area.

Shorter Analysis Times

The greater sensitivity also leads to decreased analysis times.

Improved Angle-resolved XPS

The whole of the X-ray spot remains in the analysis area over a wide range of sample tilt angles. This leads to improved angle resolved XPS measurements.

X-ray Spot Sizes

Typically, the monochromator is supplied having the spot size/power combinations shown in Table 1.

X-RAY POWER	NOMINAL SPOT SIZE*
200W	650 μm
150W	500 μm
100W	400 μm
75W	320 μm
55W	200 μm
45W	150 μm
30W	120 μm

Table 1: The spot size/power combinations available from the XR5 monochromator

*Spot size is measured by translating a silver knife edge through the spot and recording the distance the knife edge must move for the signal to change from 20% to 80% of its maximum value.

Other spot sizes within the range 120 to 650 μm can be provided if required.

High Energy Resolution XPS

The source enables fast analysis due to a high photon flux combined with very high energy resolution, as illustrated in Figure 7.

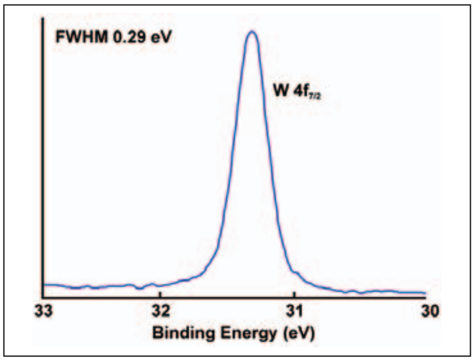

Figure 7: XPS spectrum of WSe_2, acquired using the XR5 monochromator on an ESCALAB 250 instrument

Angle Resolved XPS

The microfocus monochromator is the ideal source for angle resolved measurements. The whole of the X-ray spot remains within the analysis area over a wide range of sample angles, Figure 8.

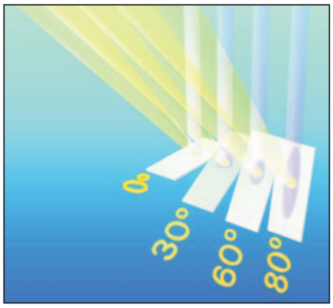

Figure 8: ARXPS using the XR5 monochromator, showing that the X-ray spot remains within the analyzed area over a wide range of angles

Figure 9 shows Si 2p spectra from a thin layer of SiO_2 on Si. The spectra were acquired at two angles (normal electron emission and grazing emission) and the spectra were normalized to elemental silicon. A clear enhancement of the oxide peak is seen in the spectrum taken at grazing emission. The energy resolution available using the XR5 monochromator is illustrated here by the splitting of the two peaks from elemental silicon.

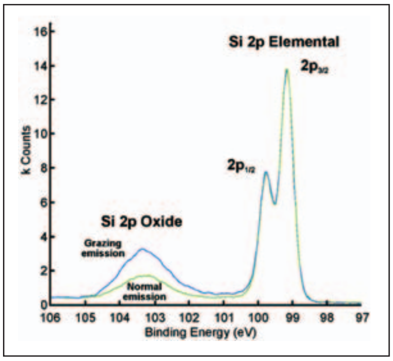

Figure 9: Si 2p spectra acquired from a thin layer of SiO_2 on SI at each of two tilt angles

Small Area XPS

Figure 10 shows a series of aluminum bond pads on silicon. Using the 250 µm spot size from the monochromator, the spectrum in Figure 11 was acquired from the center of one of the pads.

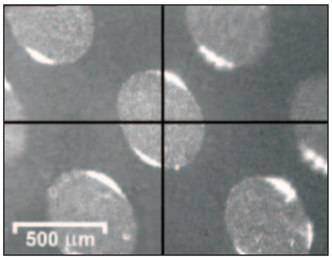

Figure 10: Aluminum bond pads on silicon

Figure 11 also shows the peak fitting capability of the *Avantage* data system. The Al 2p peaks were fitted using asymmetric peaks.

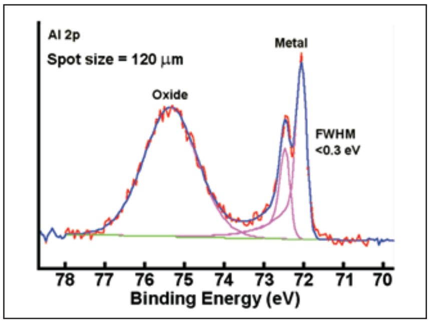

Figure 11: The Al 2p spectrum from one of the bond pads shown in Figure 10

Charge Compensation

When analyzing insulating samples with a monochromatic source, it is usually necessary to use electron flooding to avoid the build up of a positive charge at the sample surface.

For this purpose, the XR5 microfocused monochromator can be supplied with an optional FG01, Figure 12. This is a combined electron and ion source for optimum charge compensation, for details see Application Note AN31071.

Figure 12: FG01

Specifications

The XPS performance that can be achieved using the XR5 monochromator will depend on the analyzer being used and the geometry of the analyzer, monochromator and sample. As a guide, Figure 13 shows the performance available from the monochromator when used with an Alpha110 analyzer on a MultiLab 2000 instrument. To obtain the data in Figure 13, the silver sample was mounted with its surface normal parallel with the analyzer's lens axis. The intensity shown in this figure is that of the Ag $3d_{5/2}$ after subtraction of the background.

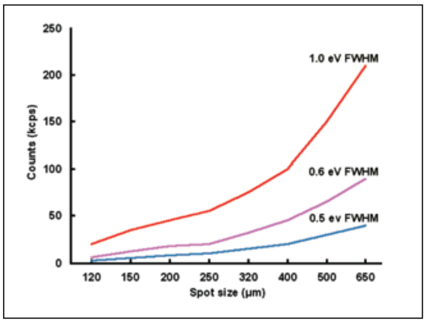

Figure 13: Performance of the XR5 monochromator when used on a MultiLab 2000 instrument with an Alpha110 analyzer

Dimensional information

The dimensions of the XR5 are shown in Figure 14.

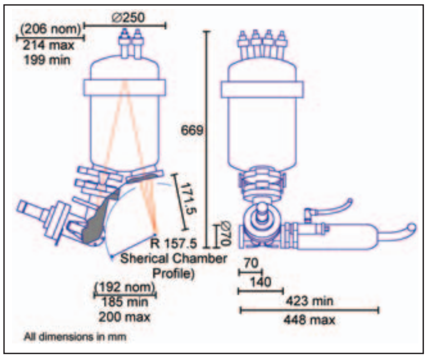

Figure 14: Dimensions of the XR5 microfocus monochromator

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