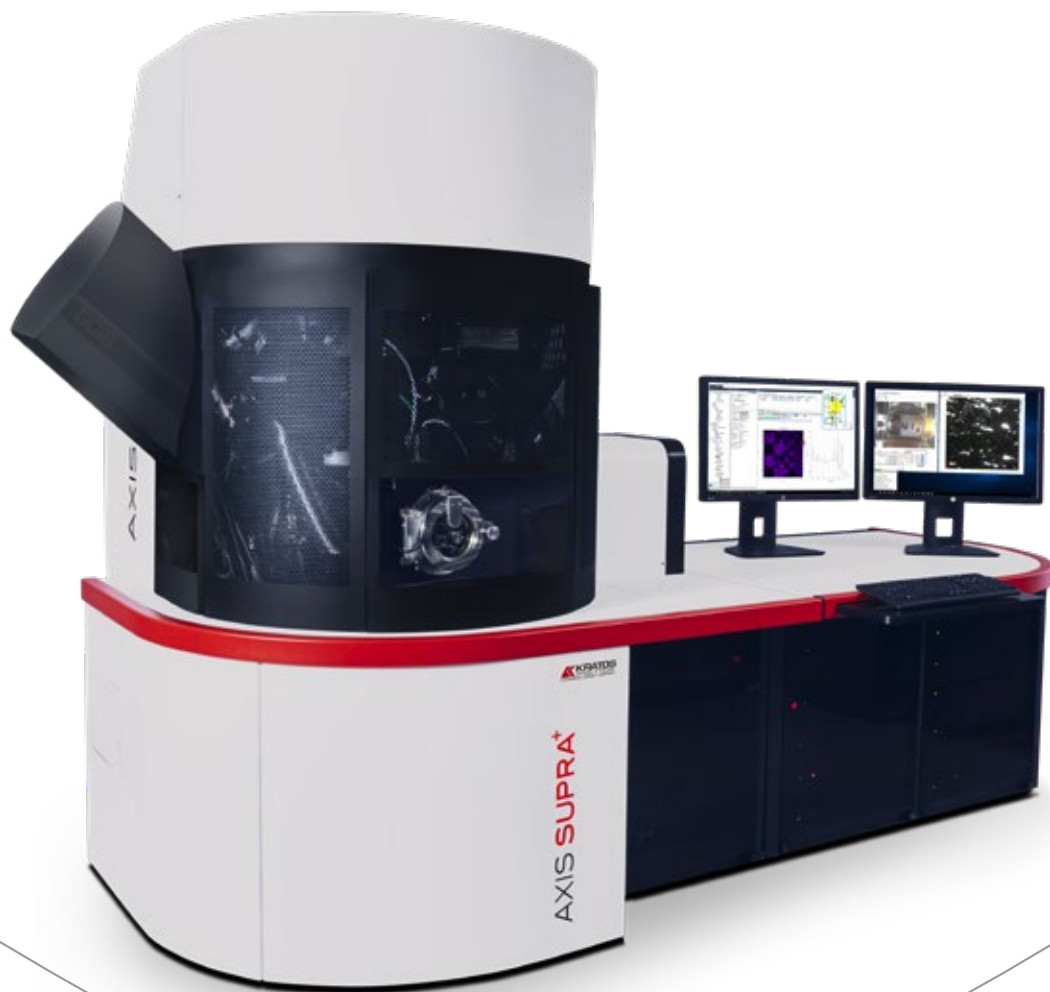


Automated, multi-technique imaging X-ray photoelectron spectrometer

AXIS SUPRA⁺



X-RAY PHOTOELECTRON SPECTROSCOPY

X-ray Photoelectron Spectroscopy (XPS), also known as Electron Spectroscopy for Chemical Analysis (ESCA) is a widely used analytical technique for the surface characterisation of materials. Typically using monochromated Al K α excitation, XPS provides quantitative chemical information from the uppermost 10 nm of a material. The technique is used in both industrial and academic research, characterising surfaces with technological applications as diverse as organic light emitting diodes or superhard, inorganic coatings.

THE AXIS SUPRA+ – IMPROVED PERFORMANCE

The next generation AXIS Supra+, with enhanced performance over its predecessor, combines market leading spectroscopic and imaging capabilities with automation to ensure high sample throughput and ease of use. Unrivalled large area spectroscopic performance allows photoelectron spectra to be acquired. Fast, high spatial resolution XPS imaging reveals the lateral distribution of surface chemistry and aids further characterisation with selected area analysis.

ESCApe integrated acquisition and processing software allows the AXIS Supra+ to perform to its maximum capability and provides an easy interface with the spectrometer.

The AXIS Supra+ is set apart from any other spectrometer by complete automation through computer control of sample handling and instrument parameters. Unattended sample holder transfer and exchange during analysis is achieved by coordination of the Flexi-lock sample magazine and sample analysis chamber autostage.

CAPABILITIES OF THE AXIS SUPRA+

LARGE AREA, HIGH SENSITIVITY XPS

The AXIS Supra+ is optimised for chemical state X-ray photoelectron spectroscopy. Efficient collection of photoelectrons combined with high transmission electron optics ensures unrivalled sensitivity and resolution at large analysis areas. As well as conventional scanned acquisition, spectra may be acquired in fast, unscanned snap-shot mode in less than a second making use of the 128 channel Delay-Line Detector (DLD).

Key attributes include:

- Easy detection of light elements.
- Excellent signal-to-noise, even at low concentrations.
- Fast data acquisition.
- Scanned or snap-shot spectral acquisition modes.



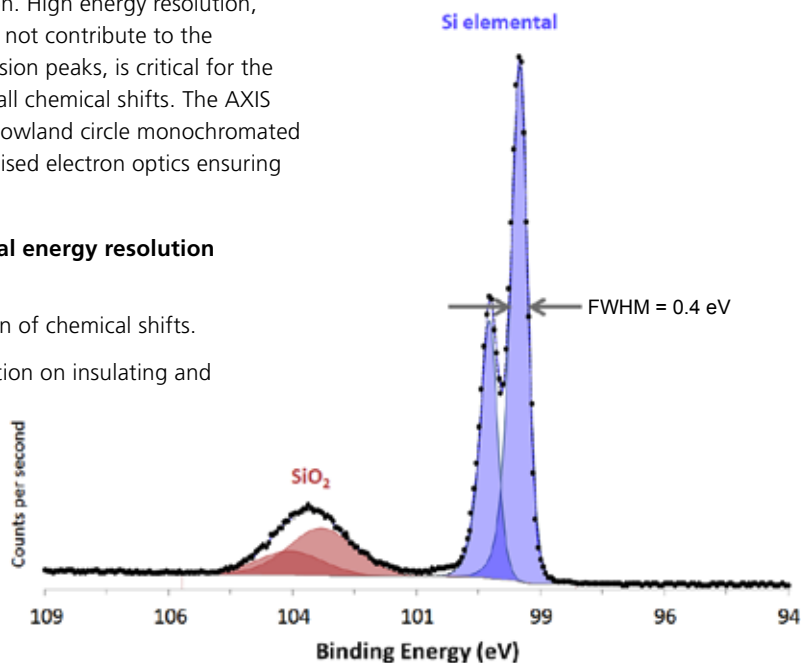


HIGH ENERGY RESOLUTION

The fundamental requirement of any spectrometer is the best possible energy resolution. High energy resolution, where the spectrometer does not contribute to the broadening of the photoemission peaks, is critical for the accurate measurement of small chemical shifts. The AXIS Supra+ has a large 500 mm Rowland circle monochromated Al K α X-ray source and optimised electron optics ensuring excellent chemical resolution.

Advantages of high spectral energy resolution include:

- Unambiguous identification of chemical shifts.
- Guaranteed energy resolution on insulating and conducting samples.



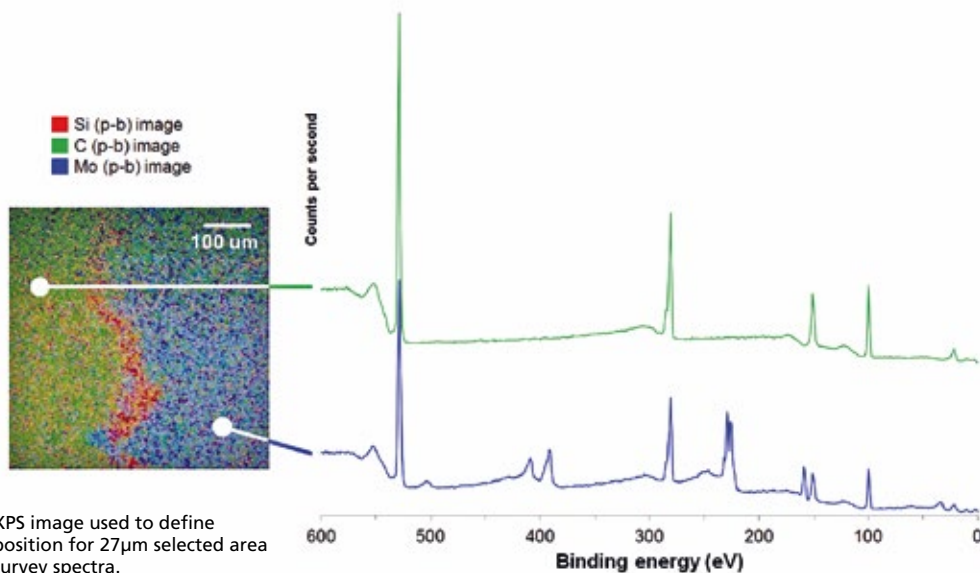
Si 2p region from native oxide on Si substrate acquired from large area with high energy resolution.

SMALL SPOT, SELECTED AREA SPECTROSCOPY

Improvement of the AXIS Supra+ small spot performance guarantees high sensitivity in small spot mode by optimising X-ray illumination of the sample with the analysis area selected. An optional brite-X monochromatic source can be specified for applications that require extremely high sensitivity performance from small selected areas.

Key selected area spectroscopy attributes include:

- Pre-defined small spot analysis areas.
- Optimised X-ray illumination for improved selected area performance.
- Automated aperture and iris in the electrostatic lens column to form a virtual probe at the sample surface.
- Selected area spectra can be acquired using either the monochromatic or achromatic X-ray sources.
- μ -boost acquisition mode for extremely high sensitivity at small area mode (option).



XPS image used to define position for 27 μ m selected area survey spectra.





ES300 photoelectron spectrometer and associated control units circa 1975.

FIFTY YEARS OF EXCELLENCE AND INNOVATION



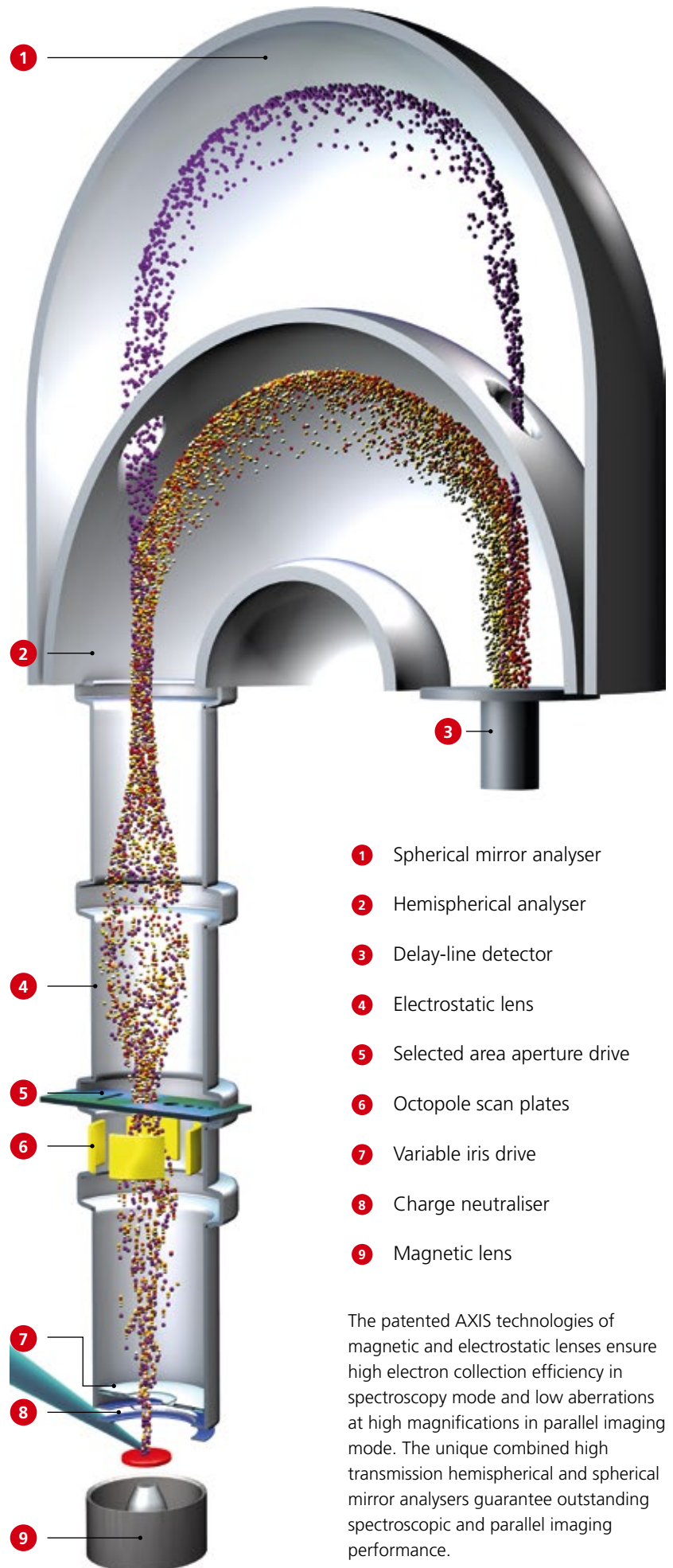
In 1969 Kratos, then AEI, introduced the first commercially-available electron spectrometer, the ES100. The wide range of possible applications included catalysis study, corrosion research, structural organic chemistry and ceramics technology. The ES-series of spectrometers proved popular and established Kratos' position as a leader in surface analysis instrumentation. Developments led to the XSAM instruments of the 1980's and then in the 1990's, the AXIS series.

Fifty years later the commitment to improve and develop our spectrometers still drives the company. The AXIS Supra+ is the culmination of that innovation and is the finest automated, imaging X-ray photoelectron spectrometer currently available today.



Fifty Years of XPS

THE AXIS SUPRA+: IMAGING X-RAY PHOTOELECTRON SPECTROMETER COMBINING EASE OF USE WITH STATE-OF-THE-ART PERFORMANCE.



- 1 Spherical mirror analyser
- 2 Hemispherical analyser
- 3 Delay-line detector
- 4 Electrostatic lens
- 5 Selected area aperture drive
- 6 Octopole scan plates
- 7 Variable iris drive
- 8 Charge neutraliser
- 9 Magnetic lens

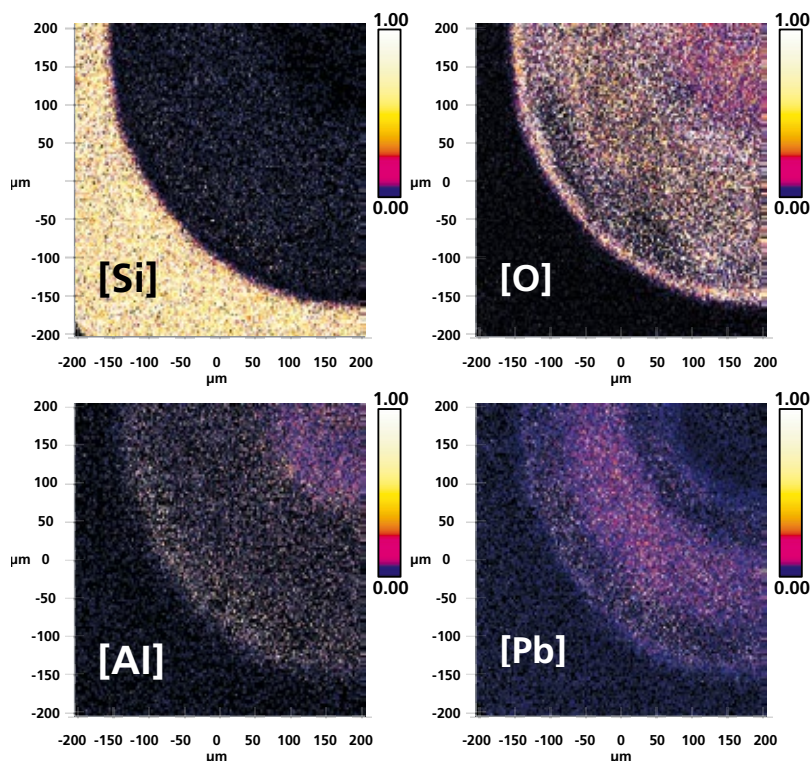
The patented AXIS technologies of magnetic and electrostatic lenses ensure high electron collection efficiency in spectroscopy mode and low aberrations at high magnifications in parallel imaging mode. The unique combined high transmission hemispherical and spherical mirror analysers guarantee outstanding spectroscopic and parallel imaging performance.

FAST PARALLEL IMAGING

The lateral distribution of elements or chemistry at the surface is measured by XPS imaging. The AXIS Supra⁺ acquires fast, high spatial resolution parallel images. Parallel image acquisition has the advantage that it is significantly faster and achieves higher spatial resolution than the more conventional rastered beam approach. Parallel imaging may also be combined with stage movements to acquire a 'stitched' image, capable of generating images over several millimetres with spatial resolution of several microns.

Capabilities provided by parallel imaging include:

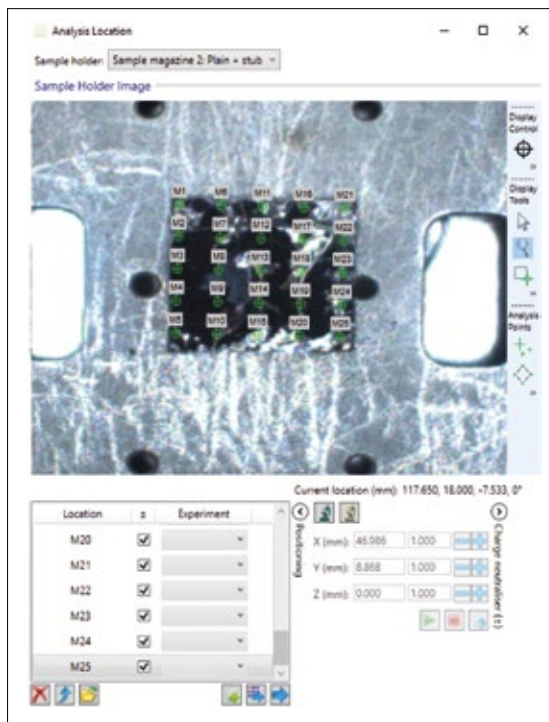
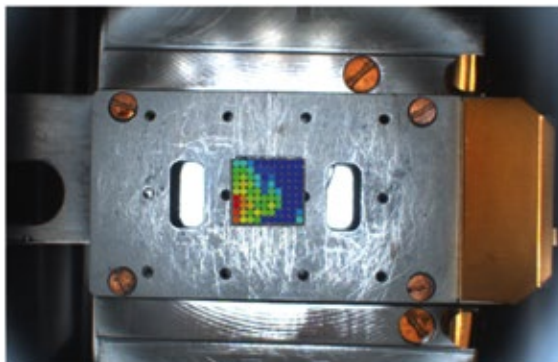
- Ultimate spatial resolution of 1 μm at the highest magnification.
- High energy resolution, chemical state imaging.
- Quantitative imaging – the unique spherical mirror analyser and delay-line detector can provide quantitative chemical state images.
- Spectromicroscopy – easy acquisition of spectra from image datasets providing a spectrum at each pixel.



Quantitative images where colour scale represents relative amount of each element present at the surface.

VERSATILE ESCAPE SOFTWARE FOR ACQUISITION AND PROCESSING

ESCAPE has been developed to make User interaction with the spectrometer as simple as possible, integrating acquisition and processing to fully exploit automation of the hardware. An example of this is the group array analysis capability within ESCAPE. This functionality allows the User to drag an area across a sample and define an array of analysis points, from which spectra are acquired. Automated peak identification and quantification allows easy generation of a colour concentration map of identified elements over the sample surface.



[Cu 2p]



[Na 1s]



[C 1s]



[C 1s]



[O 1s]



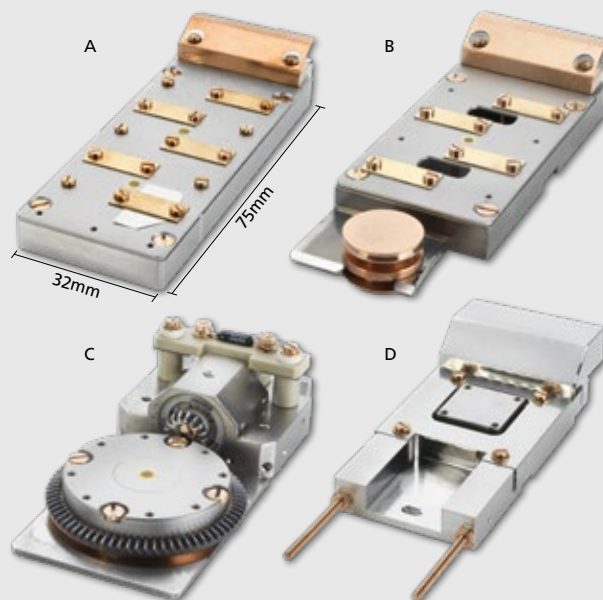
Group array analysis of an inhomogeneous distribution of Cu-nanoparticles on a graphite substrate. Colour scale used to represent relative elemental concentration.

HIGH THROUGHPUT SAMPLE HANDLING – TYPICAL WORK FLOW

To guarantee high sample throughput, up to 3 sample holders may be placed on the Flexi-lock sample magazine. An optical image is acquired of each sample holder from which the sample analysis positions are identified during the automated 'pump' cycle. An acquisition method is chosen defining all requirements for the data acquisition. A method may define simple spectroscopy or more complex experiments such as sputter depth profiling or angle-resolved XPS. Subsequent analysis from samples on different sample holders can be added to the analysis queue with the automated sample handling system exchanging the sample holder to progress through the analysis queue.

Key attributes of AXIS Supra+ automated sample handling:

- Automated, unattended sample holder exchange.
- High throughput, rapid sample analysis.
- Ideal for a multi-User environment.
- Sample mounting area up to 7200 mm² (2400 mm² x3 sample holders) with maximum sample thickness 19 mm (when using dual height sample holder).



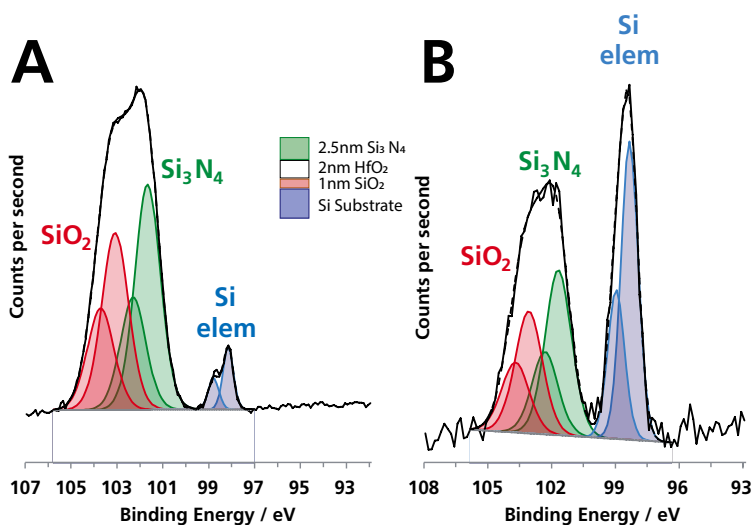
AXIS Supra+ sample holders: (A) Dual height sample holder. (B) Combination sample holder, including 15mm diameter stub for use with accessories. (C) Azimuthal rotation sample holder. (D) Heat and cool sample holder.

MULTI-TECHNIQUE CAPABILITY

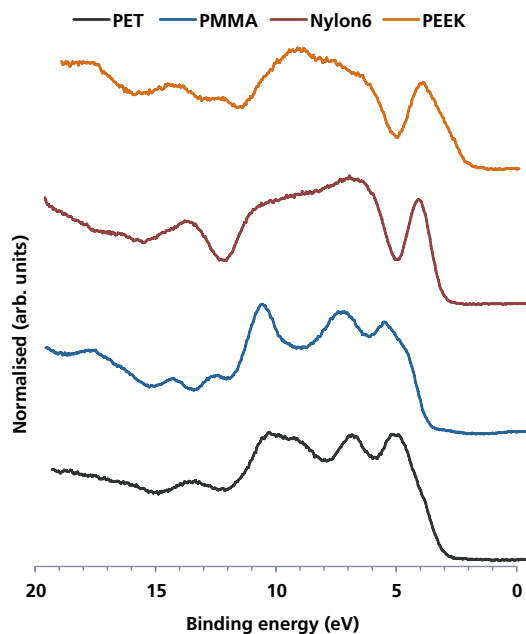
Optional excitation sources include an achromatic Al/Mg X-ray source, improved Helium discharge lamp for ultraviolet photoemission spectroscopy (UPS) and a high energy Ag L α monochromated X-ray source. Addition of a field-emission electron source adds Auger electron spectroscopy (AES), scanning Auger mapping (SAM) and secondary electron microscopy (SEM) capability to the instrument. These additional techniques are coincident with the XPS analysis position giving complementary insight to the sample. Importantly addition of these techniques does not compromise the market leading XPS performance.



Sample preparation and surface modification options can be accommodated on the introduction chamber, also known as Flexi-lock. These options include sample heat and cool, air sensitive sample transporter, broad spot ion source, crystal cleaver and glove box. A third chamber can be configured to provide a dedicated UHV environment for surface science studies. Typical configuration equips this chamber with a manual stage and optional characterisation techniques including low energy electron diffraction (LEED), inverse photoemission spectroscopy (IPES), quadrupole secondary ion mass spectrometry (SIMS).



Si 2p spectra acquired from thin film multilayer sample (courtesy of IMEC) shown in the schematic figure using (A) monochromated Al K α radiation and (B) monochromated Ag L α . The greater information depth of the Ag L α excited Si 2p spectrum is demonstrated by the larger Si elemental substrate component.



He II excited UPS valence band spectra of four common polymers.

INTO THE BULK – ION SPUTTER SOURCES

The AXIS Supra+ is configured with the Minibeam 4, high flux Ar⁺ monatomic ion source or the Minibeam 6 Ar_n⁺ gas cluster ion source (GCIS). Both ion sources are fully integrated into the ESCAPE acquisition software for sputter cleaning or depth profile experiments. Gas handling is fully automated for sputter profiling and includes pump/purge sequences to facilitate changing to Helium gas for ion scattering spectroscopy (ISS).

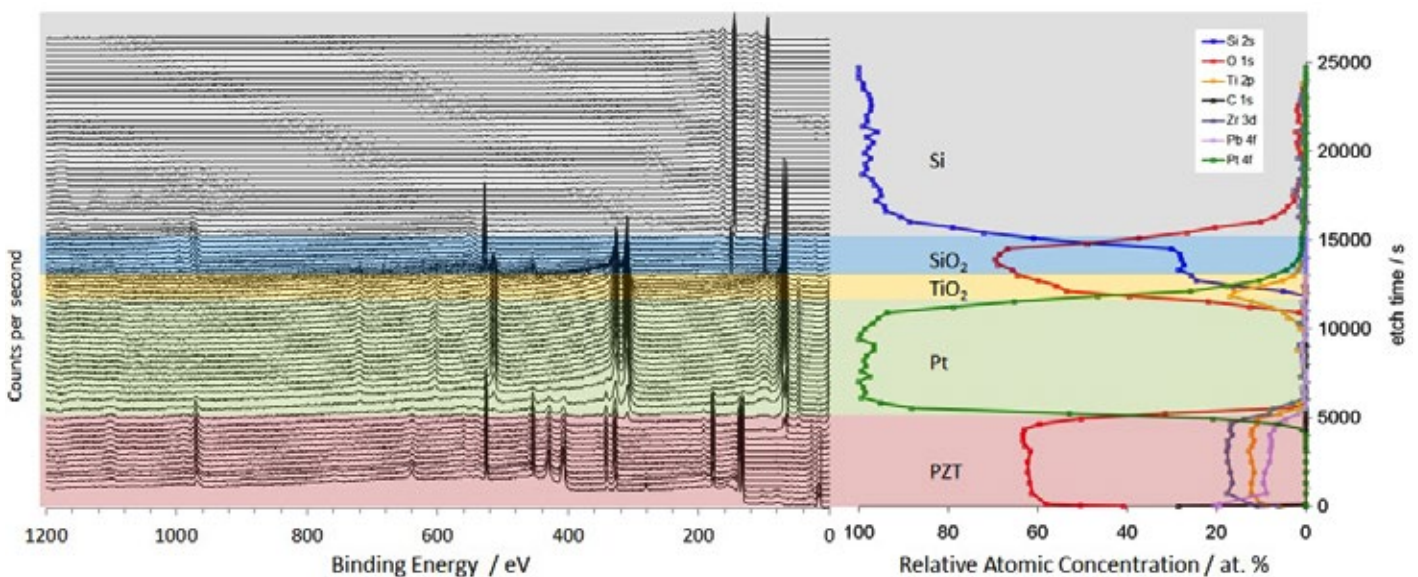
The Minibeam 6 is a multi-mode ion source which can operate in Ar_n⁺ cluster mode and Ar⁺/He⁺ monatomic ion mode as required. Recent development of gas cluster ion sources has opened up the possibility of depth profiling organic materials with retention of chemistry throughout the profile. Depth profiles through complex multi-layer materials such as organic LEDs and flexible electronic devices are now possible with Ar_n⁺

For more information, click here.

cluster ions. A unique capability of the Minibeam 6 to generate 20 keV Ar_n⁺ cluster ions has allowed the successful etching of inorganic materials such as titanium dioxide with significantly less ion induced chemical damage.

Where cluster ion depth profiling is not required the Minibeam 4 ion source is supplied. This ion source operates with continuously variable beam energies between 500 eV and 4 keV. The design produces a high ion flux at low energy for improved interface resolution whilst retaining a high sputter rate.

- Choice of monatomic or multi-mode gas cluster ion sources.
- Automated gas introduction and pressure regulation.
- Both sources capable of operating with He⁺ ions for optional ISS mode.



Sputter depth profile survey spectra (left) and elemental concentration as a function of etch time (right) through a PZT/Pt/TiO₂/SiO₂/Si multilayer sample using 20 keV Ar₅₀₀⁺ ions.

UNRIVALLED INSTRUMENT AUTOMATION

The AXIS Supra+ has complete automation of sample handling which sets it apart from any other spectrometer. Auto sample exchange at the end of an experiment allows continuous operation. Unprecedented levels of automation extend to routine maintenance aspects such as computer controlled bake-out and subsequent degassing of filaments.

For more information, click here.

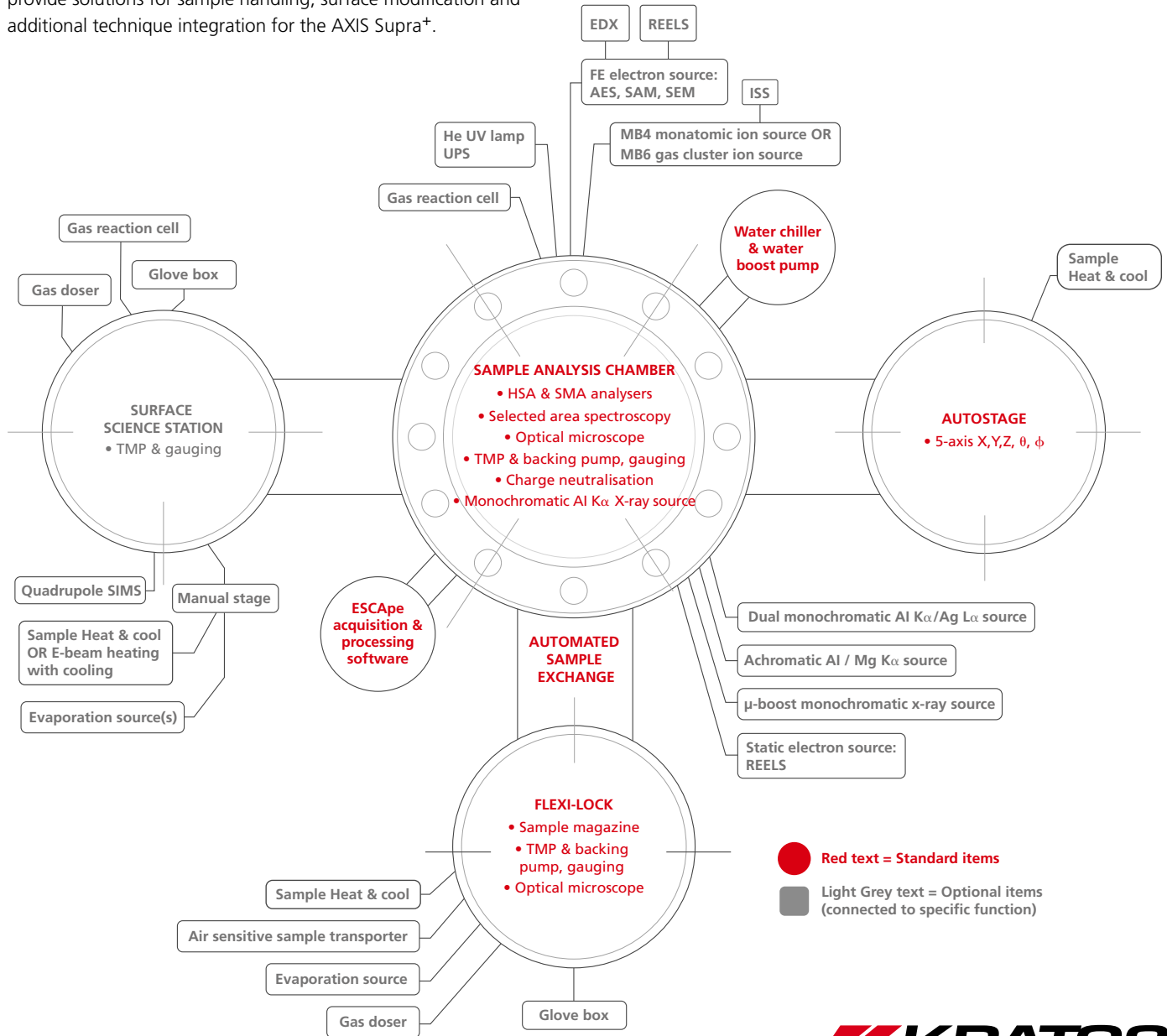
Automation also includes the X-ray source and monochromator mirror so that calibration and switching between Al K α and optional Ag L α excitation sources is completely computer controlled. Furthermore, continued optimised performance is ensured by the motorised multi-position anode.

Analysis						
Acquisition Mode: Queue						
	Mode	Sample ID	Location ID	Submitted	Estimated Time	Status
✗	User sequence	SiNx	SiNx	21/08/2019 08:44:17	00:04:30	Completed
✗	Spectroscopy	SiNx	SiNx	21/08/2019 08:57:02	00:02:00	Completed
✗	Depth profile	WO ₃ -surface modified	WO ₃ -surface modified	21/08/2019 09:46:30	00:37:10	Pending
✗	Image	WO ₃ -surface modified	WO ₃ -surface modified	21/08/2019 09:48:56	00:09:00	Pending
✗	Angle resolved	OLED	lens	21/08/2019 09:52:19	00:07:59	Pending

Start Next Manual Auto

SYSTEM CONFIGURATION OVERVIEW

The AXIS Supra+ offers the highest levels of automation combined with flexibility to add complementary sample preparation and/or surface analytical techniques. Many of the optional items detailed below are available as upgrades post-installation. We have a proven track record in development of surface analysis instrumentation hardware. Kratos Physicists and Mechanical Engineers are able to provide solutions for sample handling, surface modification and additional technique integration for the AXIS Supra+.



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Kratos Analytical, a wholly owned subsidiary of Shimadzu Corporation of Kyoto, Japan, has been manufacturing surface analysis instruments since 1969. Throughout this period Kratos has continued to lead the development of new technologies related to X-ray photoelectron spectrometers and associated accessories for surface and materials characterisation.

All Kratos products are designed, assembled and supported from our headquarters in Manchester, UK.

