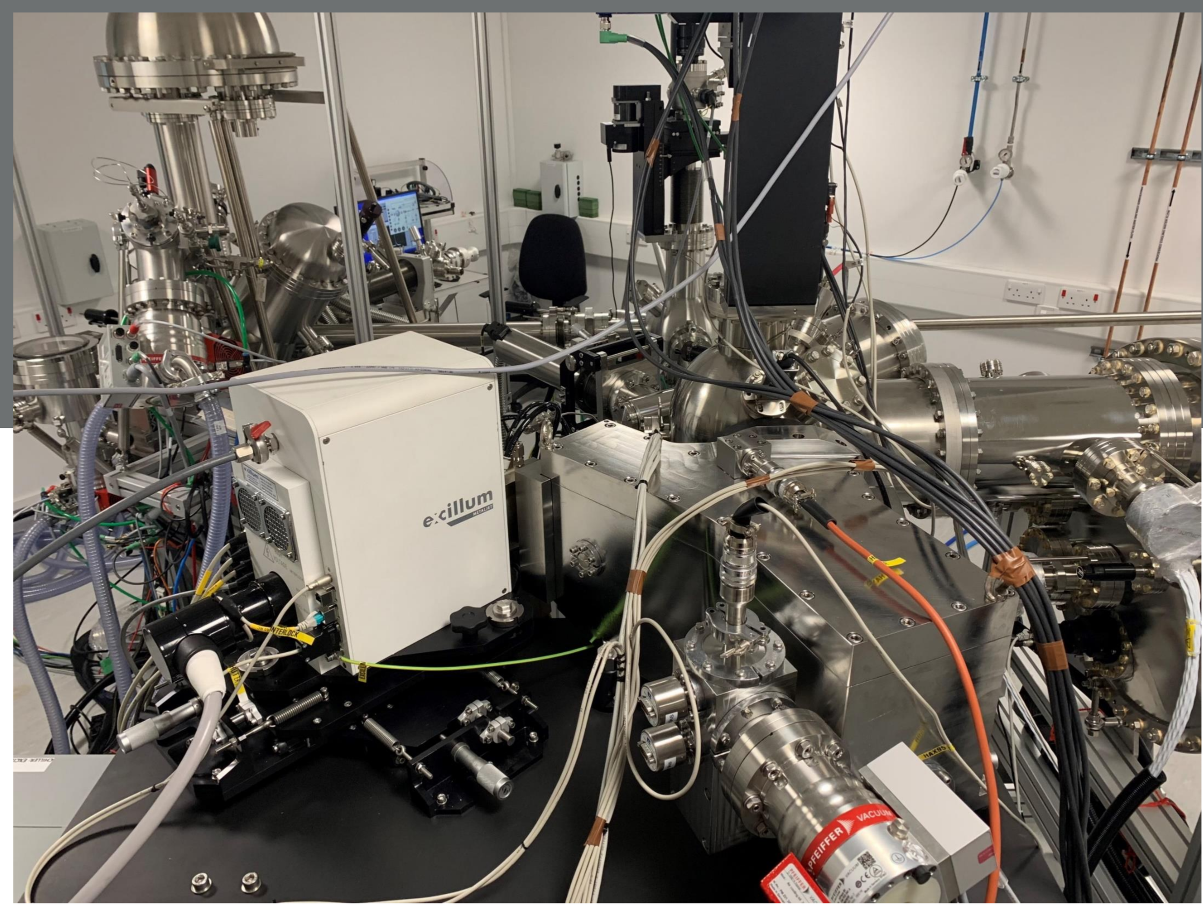
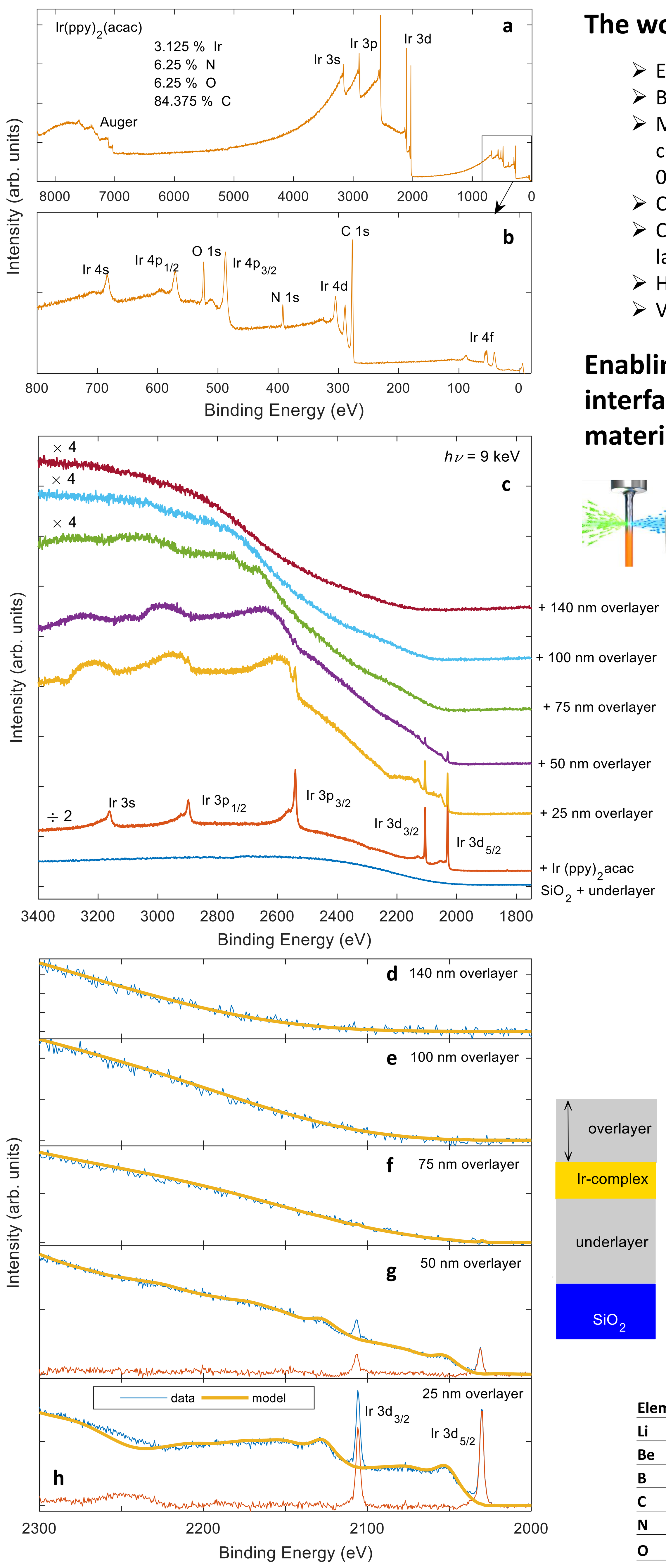
HENRY ROYCE INSTITUTE

ADVANCED MATERIALS CHARACTERISATION



HARD X-RAY PHOTOELECTRON SPECTROSCOPY



Standardisation of HAXPES: sensitivity factors, sampling depths, buried layers

a Survey spectrum ($h\nu$ = 9 keV) for Ir(ppy)$_2$(acac) containing 3% iridium; deeper core levels (3 spd) have very large sensitivity factors. Auger transition peaks are at much lower kinetic energies w.r.t. the photoelectron peaks. *Theoretical RSFs obtain the correct atomic percentages.*

b Ir 4spdf with O, N, C 1s photoelectron peaks.

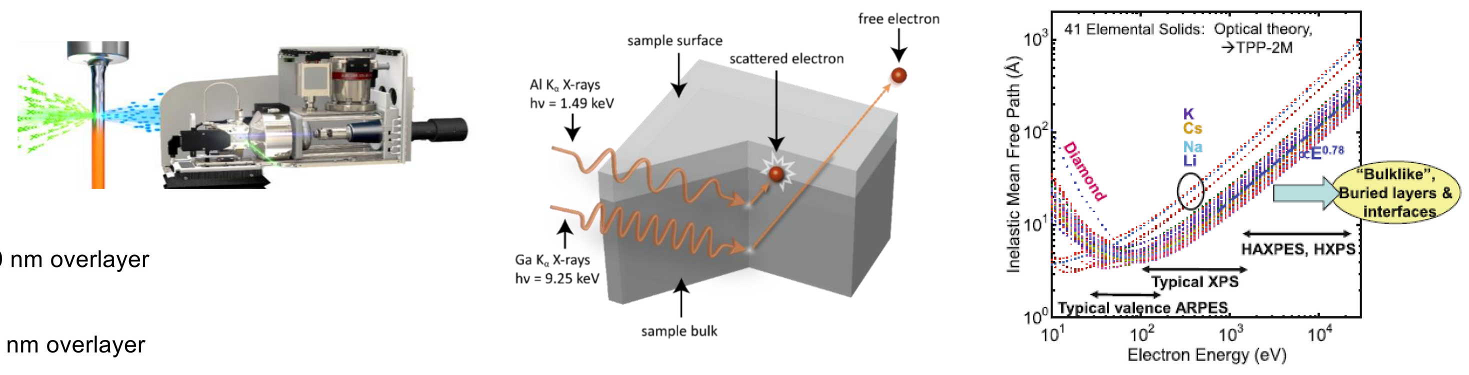
c Ir 3spd region for a silicon wafer with 200 nm organic underlayer (Irganox 1010), then with an 18nm layer of Ir complex, then with various organic overlayers up to 140 nm (as shown in the drawing). Core levels are lost for overlayers > 50 nm thick, however, *the inelastic background associated with Ir 3spd electrons still contains information which can be modelled.*

d-h Ir 3d region for various organic overlayers (140 nm, 100 nm, 75 nm, 50 nm, 25 nm as labelled). The overlayer thickness is obtained by modelling (yellow lines) the inelastic background associated with Ir 3d, using Sven Tougaard analysis (Surf. and Inter. Anal. **11** 453 (1988); Journal of Surface Analysis **24** 107 (2017)) – QUASES software (www.quases.com).

The world's first high-throughput laboratory HAXPES at 9.25 keV

- Excillum gallium metal jet (Ga Kα) X-ray source
- Bespoke monochromator
- Micro-focussed to 50 µm to gain *3 orders of magnitude greater flux* compared to laboratory Al Kα sources (see Regoutz *et al*., Rev. Sci. Instr. **89** 073105 (2018))
- Combined with EW4000 high kinetic energy electron analyser
- Connected under ultra-high-vacuum to a high-throughput XPS with UV lamp, Al/Mg dual anode, monoatomic and cluster etching
- Heating and surface preparation chamber
- Vacuum suitcase for sample transport without exposure to atmosphere

Enabling chemical state information for thin films, buried interfaces and active layers, multi-layered structures, and bulk material information



- HAXPES applies the powerful tool of XPS up to depths of at least 50 nm below the surface
- Analysis of the inelastic background can enable depth-profiling through up to an estimated 20 x IMFP for depths over *hundreds of nm*
- Much less sensitive to surface roughness and contamination

A new tool for advanced materials researchers and industry

- Bringing the technique of HAXPES outside of national-level synchrotron radiation facilities opens up great possibilities for application to a wide range of research across multiple disciplines
- Expect a revolution in uptake as previously occurred with lab-based near-ambient pressure (NAP) XPS
- Establishment of the technique includes a 3-year collaboration between Scienta Omicron, The University of Manchester and the National Physical Laboratory (see data in the left-hand figure)
- Standardisation of HAXPES – establishing sensitivity factors, sampling depths, and analysis protocols for a wide range of materials systems

Access also available through the XPS National Research Facility, www.harwellxps.uk

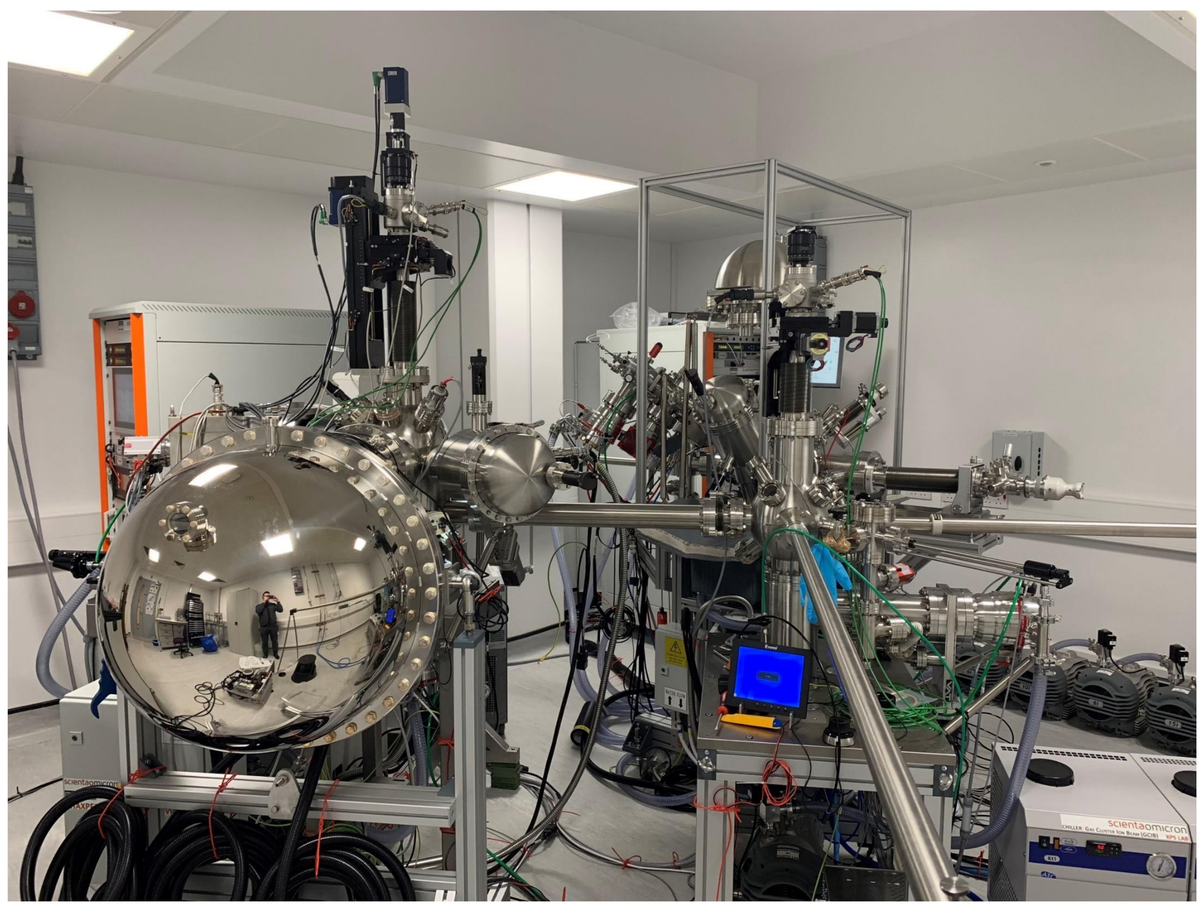
harwellxps

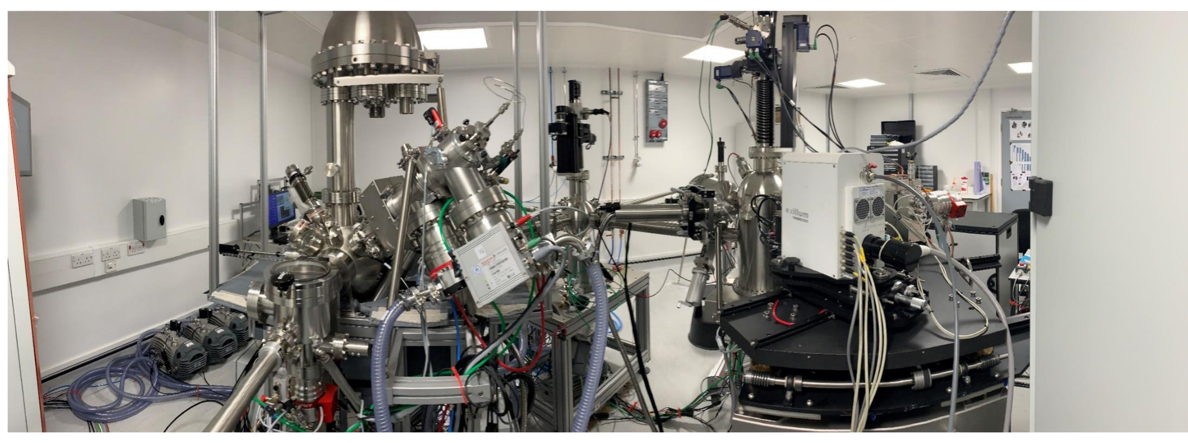
	1s core level			*2p* core level		
Element	BE (eV)	HAXPES	XPS	BE (eV)	HAXPES	XPS
Li	55	66.9	13.5			
Be	112	45.9	9.3			
B	188	45.6	9			
C	285	51.9	9.9			
N	410	51.6	8.7			
O	540	50.1	7.8			
F	695	48.9	6.9			
Ne	870	49.2	5.7			
Na	1070	74.1	6.3			
Mg	1303	47.7	2.4	49	54.3	11.4
Al	1559	37.8		72	44.1	9.3
Si	1839	37.2		99	44.4	9.3
P	2145	37.5		136	46.5	9.3
S	2472	34.8		164	44.4	9
Cl	2822	35.1		200	47.1	9.3
Ar	3205	34.2		248	48	9
K	3608	69.6		294	103.8	18.9
Ca	4038	40.5		346	64.2	11.4
Sc	4492	27.3		398	46.2	8.1
Ti	4966	20.7		453	37.8	6.6
V	5465	16.2		512	32.4	5.4
Cr	5989	13.2		574	29.4	4.8
Mn	6539	11.1		638	28.5	4.5
Fe	7112	8.7		706	27.3	4.2
Co	7709	6.3		778	25.5	3.6
Ni	8333	4.5		852	25.5	3.3
Cu	8979	1.8		932	25.2	3

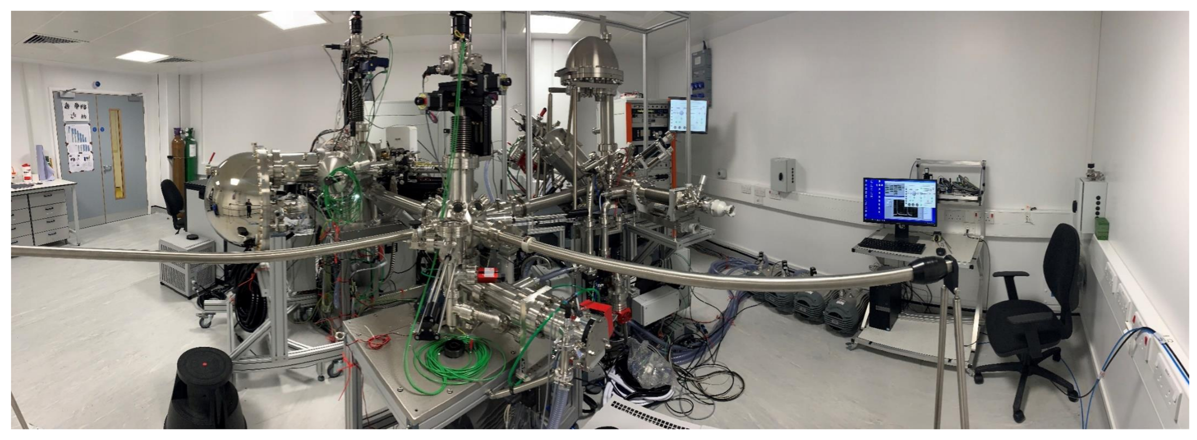
Sampling depth (nm) est. (3 x IMFP)

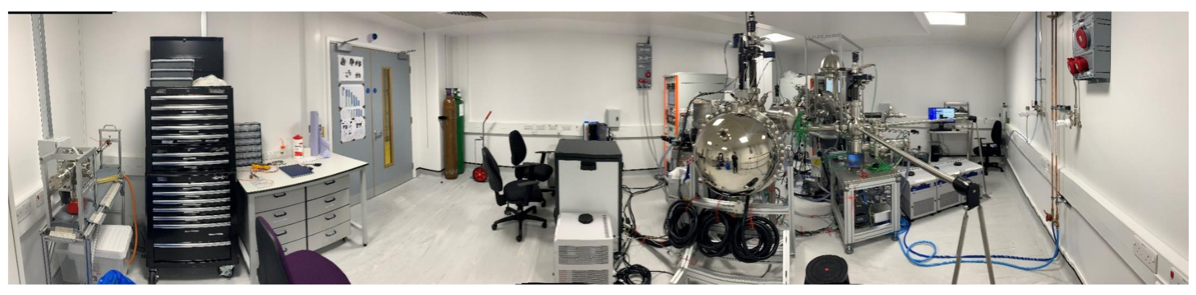
	hv
HAXPES	9252 eV
XPS	1486 eV

HAXPES sampling depths
Using photoelectron peaks (3 x IMFP) compared to Al Kα XPS. HAXPES enables measurement of deeper core levels, and for larger atoms, using different core levels gains information from different depths.







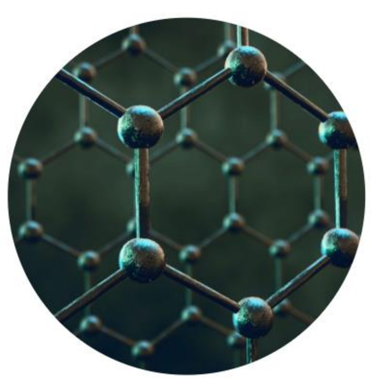


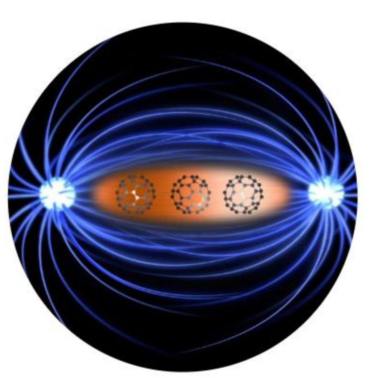
Manufacturer: Scienta Omicron GmbH

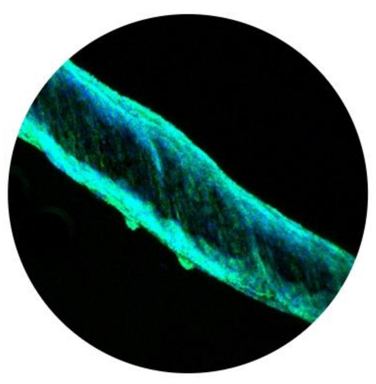
Academic Lead: Prof Wendy Flavell
Technical Lead: Dr Ben Spencer
Research & Facilities Manager: Dr Helen Ryder

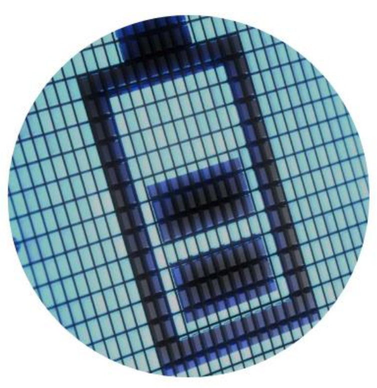
Location: G318 Photon Science Institute, Alan Turing Building, The University of Manchester

"Due to its relatively unlimited electron escape depths, HAXPES has emerged as a powerful tool that has general application to the study of the true bulk and buried interface properties of complex materials systems. Its areas of application are thus growing exponentially compared to more traditional measurements at lower photon energies." **Joseph C. Woicik, NIST**









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The University of Manchester