HENRY ROYCE INSTITUTE

ADVANCED MATERIALS RESEARCH & INNOVATION

ROYCE

IMAGING & CHARACTERISATION

FACILITIES

The Imaging and Characterisation Research Area enables high-impact discoveries and innovation, with specialised expertise in analysing the structure, properties, and chemistry of advanced materials.

VISION

The Henry Royce Institute Imaging and Characterisation Research Area offers access to advanced techniques that support all the other Research Areas within Royce.

With specialised expertise in analysing the structure, properties, and chemistry of a wide range of advanced materials, we enable high-impact discoveries and innovation.

The vision of the Imaging and Characterisation Research Area is to:

- Support Royce Partners in providing state-of-the-art equipment and facilities for the imaging and characterisation of a diverse set of advanced materials.

- Leverage and champion the expertise of our technical colleagues to develop and support a comprehensive national network of imaging and characterisation researchers. Strengthen collaboration by providing accessible training in communication, management and technical skills, and promote broader awareness and understanding of advanced techniques and their applications.

- Increase external engagement across academia and industry with Royce imaging and characterisation technologies by organising workshops and other networking events.

CONTENTS

ELECTRON MICROSCOPY

SCANNING ELECTRON MICROSCOPY (SEM)

SEM is a surface sensitive technique that uses a focused beam of electrons to produce high-resolution images of a sample's topography and composition. SEMs often have multiple additional detectors to help characterise a sample.

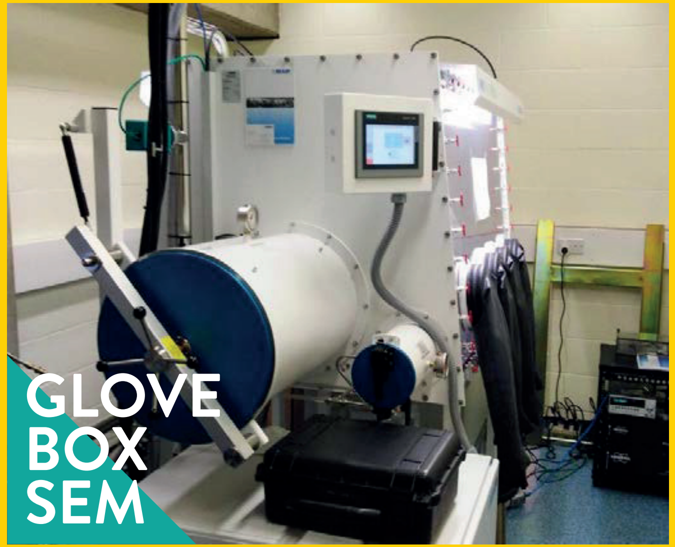
These include:
Energy Dispersive X-ray Spectroscopy (EDS), a technique to identify and map the elemental composition of a sample by detecting the energy of characteristic X-rays emitted when the sample is bombarded with the electron beam, and **Electron Backscattered Diffraction (EBSD)**, a technique to determine the crystallographic orientation, and phase of materials by analysing the diffraction patterns produced when electrons are scattered by a crystalline sample surface.

SEM CAPABILITIES AT ROYCE INCLUDE:

Field Emission Gun (FEG) SEM for high-resolution SEM imaging. Many instruments are also fitted with EBSD and EDS detectors for crystallographic information and high-resolution imaging of surfaces for bulk materials.

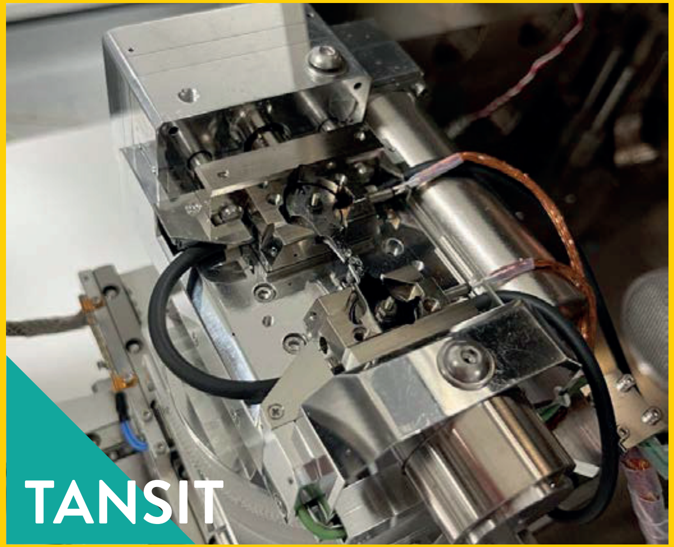
Variable Pressure SEM suitable for imaging non-conductive materials.

Glove Box SEM allows transfer to the SEM from sample preparation in inert and controlled environments. Also has in-situ mechanical testing capabilities.



TANIST automated in-situ testing capabilities with EBSD and EDS detectors with both mechanical testing and/or heating capabilities. High efficiency detectors are used for analysis of dynamic processes with greater temporal resolution.

High Throughput SEM with specialist workflows for automatically mapping multiple samples with EBSD and EDS.



Electron probe micro-analyser (EPMA) is a specialist SEM with multiple **Wavelength Dispersive Spectroscopy (WDS)** detectors. Used to determine the elemental composition of a material with high accuracy and sensitivity by measuring the wavelengths of characteristic X-rays, offering better detection of low concentrations than EDS. It can offer ~100 nm spatial resolution and sensitive quantification of elements (Be to U) when used with calibration standards.

ELECTRON MICROSCOPY

FOCUSED ION BEAM (FIB)

Focussed ion beam instruments use a beam of ions focused on a specific position to precisely ablate materials. FIBs are used to sculpt, mill or modify materials and are usually combined with an SEM column to combine the accurate milling of the FIB with the imaging and analytics of an SEM. Such FIB-SEMs are available at several Royce Partners, with some laboratories having specialist expertise in specific nanofabrication tasks, such as sample preparation for transmission electron microscopy (TEM) or atom probe microscopy, large area or high-speed milling, and 3D-characterisation through serial sectioning tomography.

FIB CAPABILITIES AT ROYCE INCLUDE:

Gallium FIB-SEM offers cross-sectional and 3D characterisation with EDS and EBSD detectors of regions up to ~30 µm in depth. Particularly suited for sample preparation for TEM, Atom probe and so on. Several Royce partners have FIB-SEM instruments which are suitable for milling of active samples. ield Emission Gun (FEG) SEM

Cryo FIB-SEM feature rapid immersion freezers for cryogenic imaging and lower damage TEM sample preparation.


GALLIUM FIB-SEM

Plasma FIB (PFIB) SEM allows faster milling of larger areas using Xe ions compared to conventional Ga ion beam FIBs with typical milling depths up to ~300 µm.

Plasma FIB-SEM with Secondary Ion Mass Spectrometry (SIMS) combines fast milling with detailed compositional analysis through SIMS, and is suited for SIMS analysis of active materials.


PLASMA FIB-SEM

Femtosecond Laser PFIB Tribeam combines a PFIB with laser milling to provide even faster milling with typical milled cross sections over one millimetre in size. Can be combined with EBSD and EDS for tomographic volume microscopy.


PFIB TRIBEAM

ELECTRON MICROSCOPY

TRANSMISSION ELECTRON MICROSCOPY (TEM)

TEM uses a beam of electrons transmitted through an ultrathin specimen to produce high-resolution images and spatially resolved diffraction patterns, which can provide detailed information about the morphology, crystal structure and composition of materials at the atomic scale.

Most instruments also have Scanning Transmission Electron Microscopy (STEM) capabilities, advanced FEG electron sources and can perform complementary energy dispersive X-ray spectroscopy (EDS) for quantitative elemental mapping, with atomic resolution elemental imaging possible for sufficiently stable samples. TEMs with accelerating voltages from 80-300 keV are widely accessible.

Many TEMs have specialist capabilities to allow imaging and analysis of active samples.

TEM CAPABILITIES AT ROYCE INCLUDE:

In situ holders for TEM enable experiments to be performed while cooling or heating, in vacuum, liquids or gases, while biassing the sample, or even while performing electrochemical cycling in liquids. Specialist in situ TEM expertise for different types of materials exists across the Royce partners.

Precession electron diffraction (PED) in the TEM delivers electron diffraction patterns which are more readily interpretable to uncover the atomic structure of complex nanosized crystals, particularly when combined with tomographic data collection.


IN SITU

Spatially resolved electron diffraction studies (4D-STEM) can retrieve complex phase information about a material (ptychography), as well as to probe electric/magnetic field variations, crystal defects, and phase heterogeneity at the nanoscale.

High-Throughput TEMs allow efficient and rapid TEM analysis.


4D-STEM

Electron Energy Loss Spectroscopy (EELS) in the TEM is a technique used to measure the energy lost by electrons as they pass through a sample, providing detailed information about the material's elemental distribution, bonding, oxidation state, optical properties and electronic structure at the nanoscale.


HIGH THROUGHPUT

MASS SPECTROMETRY

Mass spectrometry techniques determine the chemistry of a sample by measuring the mass-to-charge ratio of the atoms or molecules in the sample.

Both Secondary Ion Mass Spectrometry (SIMS) and Atom Probe Tomography are available at Royce. SIMS is an analytical technique that measures the composition of a surface by sputtering it with a primary ion beam and analysing the secondary ions ejected from the sample using mass spectrometry to determine its elemental and isotopic composition.

CAPABILITIES AT ROYCE INCLUDE:

NanoSIMS offers high spatial resolution SIMS analysis with a beam that can be focussed down to 50 nm. It can measure 7 ionic species simultaneously from the same sputtered volume. It is complementary to chemical mapping in the SEM as it is able to map light elements (hydrogen, lithium, boron), trace elements (parts per million level) and can map isotopes allowing tracking of uptake or incorporation of elements overtime. Any sample (including biological samples) can be measured as long as it is high vacuum compatible.

Time of Flight-SIMS (ToF-SIMS) allows surface chemical imaging through parallel mass spectrometry detection of atomic and molecular secondary ions up to m/z ~ 2000 Da. In-depth profiling and 3D analysis to few microns in depth. High-energy gas cluster ion beam for non-destructive molecular profiling and imaging with ~4 um lateral resolution. C60 ion beam for hybrid/inorganic analysis with ~1 um lateral resolution. Any sample (including biological samples) can be measured as long as it is high vacuum compatible.

Plasma FIB SEM with Secondary Ion Mass Spectrometry enables detailed compositional through SIMS, and is suited for SIMS analysis of active materials.

Atom Probe Tomography (APT) is a high-resolution technique that uses a high electric field or laser pulses to evaporate individual atoms from a sample. These atomic species are identified via time-of-flight mass spectrometry to provide 3D atomic-scale imaging and composition analysis of materials. Although, samples are typically ultra-fine needles, Royce has APT instruments suitable for a range of materials including catalytic nanoparticles, metal alloys, and semiconductors.

SCANNING PROBE MICROSCOPY

Scanning Probe Microscopy (SPM) uses a physical probe to scan the surface of a sample, measuring properties like topography, mechanical response, or electrical signals at the nanoscale.

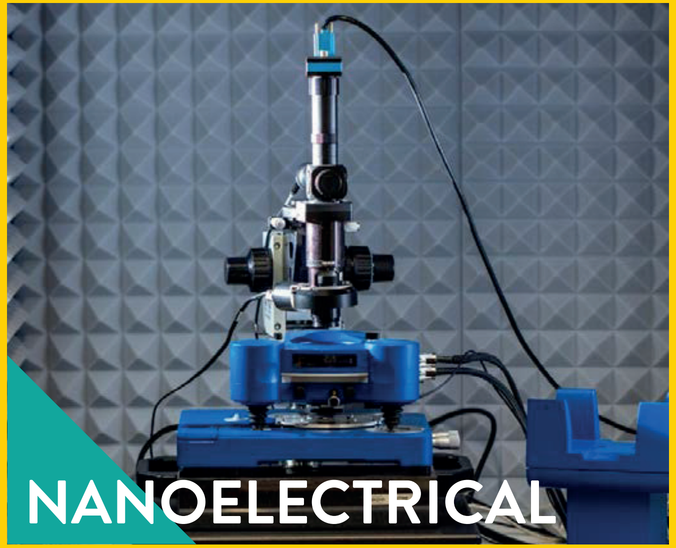
CAPABILITIES AT ROYCE INCLUDE:

Atomic Force Microscopy (AFM) uses a sharp tip on a flexible cantilever to scan a sample's surface, measuring forces between the tip and the surface to create topographic and material property maps at the nanometer scale.

Electrochemical AFM under an inert argon atmosphere.

AFM on an inverted optical microscope and large scanner suitable for mechanical and topographic analysis of biological samples from molecules to cells and tissue.

Nanoelectrical AFM for comprehensive nanoscale electrical, electrochemical, mechanical, magnetic and topographic characterisation of semiconductor, ceramic piezoelectric, ferroelectric and magnetic materials. It has various uses, including investigation into molecular structures and interactions, surface topography, mechanical properties, conductivity and magnetic properties, corrosion, nanodevice fabrication, photovoltaics and energy storage.



Suite of **NanoIR** equipment for the combination of AFM with infrared spectroscopy, including fluid system compatibility and within a controlled environment (gases, inert transfer, humidity and temperature control).

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NANOINDENTATION

Nanoindentation can obtain precise measurements of hardness, modulus, and creep behaviour, along with scratching test capabilities, under various ambient conditions, making it ideal for a wide range of materials testing, from soft Hydrogels and biomaterials to ultra hard coatings.

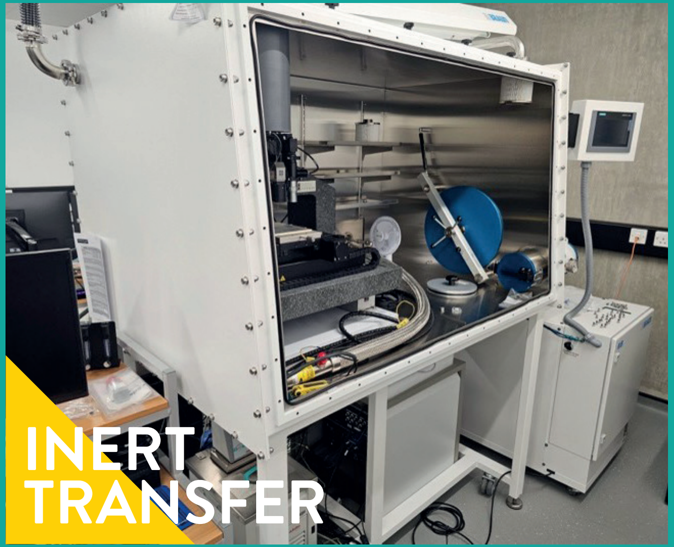
Royce offers a comprehensive suite of instruments capable of almost any nanomechanical testing protocol, offering temperature, humidity and vibration control, and with a ultra-stable environment allowing for long experiments.

CAPABILITIES AT ROYCE INCLUDE:

Humidity controlled nanoindentation

Inert transfer and glove box environment enables nanoindentation to be performed under an inert, oxygen and water free atmosphere with temperature control from -100 to +1000 °C.

Nanoindentation of hydrated and soft/biological specimens is possible with the unique Hysitron Bioindenter. This system is capable of testing very soft (KPa range) and thin samples, such as soft polymer films and biological samples such as tissue histology sections.



High load nanoindenter (up to 1N) offers higher throughput testing for standard samples.

Nanoindentation in liquid and at high temperature with capabilities for scratch testing, automated programmes for nanomechanical mapping, and more.

Performing **in situ nanoindentation in a SEM or TEM** allows indentation experiments or nanoscale tensile testing (typically using MEMS devices) to be performed while imaging. Experimental geometries such as pillar compression, cantilever bending and nano-tensile tests are possible with direct-observation of the nanomechanical testing and simultaneous acquisition of load-displacement data.

OPTICAL SPECTROSCOPY

FOURIER TRANSFORM INFRARED SPECTROSCOPY (FTIR)

FTIR is an analytical technique used to identify and characterise materials by measuring the absorption of infrared light at different wavelengths, providing detailed information about the molecular composition, functional groups, and chemical bonds in a sample.

UV/VIS TRANSMISSION, ABSORPTION & REFLECTANCE SPECTROSCOPY

Enables analysis of liquids, thin films, coatings, high performance glass, solar, and advanced materials across 175 nm to 3300 nm wavelengths. Additional modules enable measurement of diffuse reflectance and angle-dependent specular reflectance.

PHOTOLUMINESCENCE (PL) AND PHOTOLUMINESCENCE EXCITATION (PLE) SPECTROSCOPY

Techniques where the spectrum and intensity of light emitted from a sample is measured following excitation with another, typically spectrally tuneable, light source. This technique provides valuable information on how the material might be applicable in optical devices.

PHOTOLUMINESCENCE QUANTUM YIELD (PLQY) SPECTROSCOPY

PLQY spectroscopy is an analytical technique that quantitively measures the efficiency of the PL process by determining the ratio of light absorbed by a sample to the light emitted as PL.

OPTICAL SPECTROSCOPY

CRYOGENIC ULTRAFAST SCATTERING-TYPE TERAHERTZ-PROBE OPTICAL-PUMP MICROSCOPY (CUSTOM)

The CUSTOM facility consists of a suite of scattering-type scanning near-field optical microscopy (s-SNOM) systems that can operate from the THz to the visible range. The systems offer material characterisation on nanometre length scales with ultrafast temporal resolution, enabling 2D mapping of local dielectric function and time-resolved dynamics.

CAPABILITIES:

Room to low temperature (down to 10K) **operation**

Nanometre (~30 nm) **spatial resolution**

Time-resolved detection (~80 fs temporal resolution)

Operation in MIR (5 – 15 um), **THz** (0.1 – 4THz), **visible** (532 nm, 685 nm) **and NIR** (1064 nm, 1550 nm) **frequency ranges**

Couple custom light sources to SNOM systems

Simultaneous imaging of amplitude, phase and topography

Hyperspectral mapping

Surface-sensitivity

Tapping-mode operation to extract near-field information and perform tomography (3D mapping)

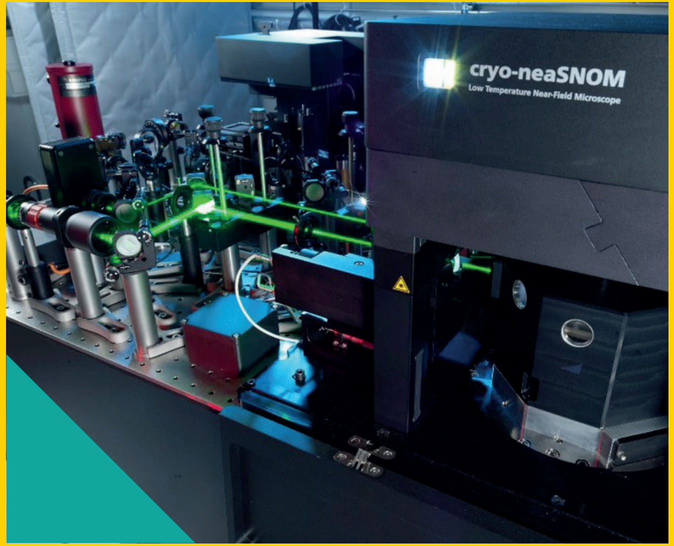
APPLICATIONS INCLUDE:

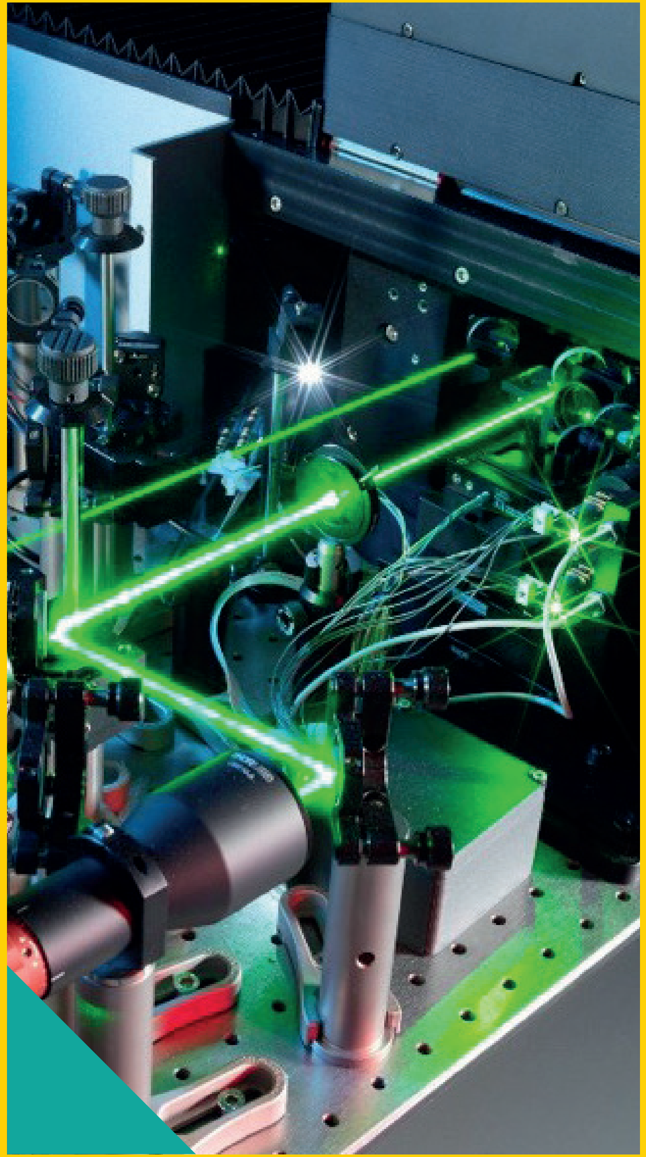
3D conductivity mapping of semiconductor materials and devices

Near-field mapping of metamaterial structures at telecom wavelengths

Chemical composition mapping (nano-FTIR spectroscopy) of surface layers

Nanoscale imaging and spectroscopy of biological cells





OPTICAL SPECTROSCOPY

RAMAN SPECTROSCOPY

Raman Spectroscopy is an analytical technique that measures the scattering of light by a sample to provide information about its molecular vibrations, chemical structure, and bonding, helping to identify materials and analyse their chemical composition and molecular interactions.

CAPABILITIES AT ROYCE INCLUDE:

Inert and controlled atmosphere Raman with focus tracking technology to analyse samples with uneven surfaces.

Confocal Raman Microscope
The coupling of a confocal microscope to a Raman spectrometer enables surfaces to be mapped with high spatial resolution, giving an image of the uniformity (homogeneity) of the material.

Confocal Scanning Laser Microscope combined with Raman, AFM and SNOM capability to build 3D images of a range of samples.

Remotely operated Raman for irradiated materials.

Glove box/fumehood Raman allowing measurements under inert conditions.

X-RAY

X-RAY COMPUTED TOMOGRAPHY (XCT)

X-ray computed tomography (XCT) is a non-destructive imaging technique that uses X-rays to create detailed cross-sectional images of an object's internal structure by combining multiple 2D projections into a 3D model.

This can provide detailed information about porosity, voids, cracks, inclusions and grains within the centre of the material, across multiple length scales.

CAPABILITIES AT ROYCE INCLUDE:

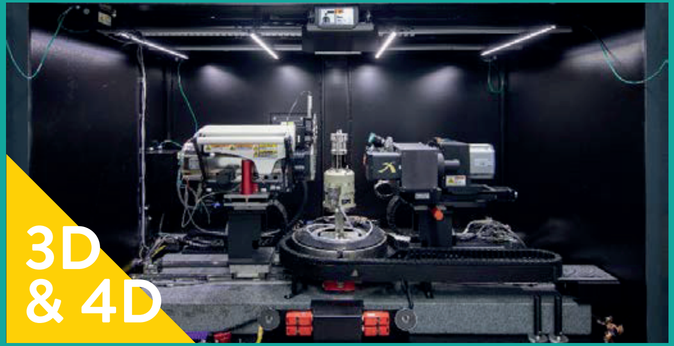
3D and 4D µm-scale XCT for in-situ characterisation of the composition, deformation and damage development of materials.

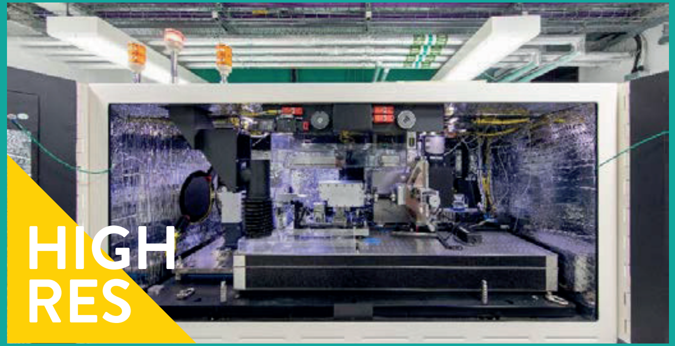
High-flux walk-in XCT for rapid scanning of materials with the capability of in-situ rigs.

High-resolution XCT for imaging down to 50 nm resolution.

XCT with Diffraction Contrast Tomography for the detection of grains within polycrystalline materials by obtaining diffraction spots during the tomography scan.

Colour XCT to determine the chemical composition of samples, utilising state-of-the-art energy sensitive detectors.





X-RAY

X-RAY PHOTOELECTRON SPECTROSCOPY (XPS)

X-ray photoelectron spectroscopy (XPS) is a surface-sensitive analytical technique that measures the elemental composition, chemical state, and electronic state of materials by detecting the kinetic energy of electrons emitted from a sample when irradiated with X-rays.

CAPABILITIES AT ROYCE INCLUDE:

Ultra-High Vacuum XPS (UHV XPS) instruments provide various XPS capabilities including destructive depth profiling (using argon gas cluster etching or monoatomic ion etching) as well as sample heating, cooling, and electrical biasing.

XPS with in-situ / operando electrochemical cycling including lithium deposition.

Near Ambient Pressure (NAP) XPS enables analysis of samples under more realistic atmospheric environments. Gases and gas mixtures up to ~ 10 mbar can be applied along with heating, with some systems even enabling measurements of liquids.

Ultraviolet Photoelectron Spectroscopy (UPS) spectroscopy using a He XUV source (21.2, 40.8 eV) enables valence band and work function measurements.

Hard X-ray Photoelectron spectroscopy (HAXPES) is used for the non-destructive measurement of the bulk chemical & electronic environment and depth-profiling from the surface into the bulk of a material up to depths ~ 100 nm. HAXPES typically uses a Ga metal jet X-ray source (9250 eV) although systems are also available in Royce with a Ag X-ray source (3000 eV).

Inert transfer to XPS instruments from vacuum suitcases and glove boxes is available at various locations. This enables measurement of materials sensitive to atmospheric exposure.

X-RAY

X-RAY DIFFRACTION (XRD)

XRD is a technique used to study the structure of materials by analysing how X-rays scatter off the atoms in a sample, revealing information about its crystallographic structure, phase composition, and other properties.

CAPABILITIES AT ROYCE INCLUDE:

Multipurpose XRD with thin film and polycrystalline material capability.

Air sensitive XRD in a glovebox under nitrogen atmosphere.

High energy XRD for greater penetration of samples.

Transmission XRD for small sample volumes of liquids, thin films and powders that suffer from preferred orientation.

XRD with grazing incidence and high temperature capability, suitable for thin film samples.

Automated XRD with guidance software for irradiated materials.

X-RAY FLUORESCENCE (XRF)

X-ray fluorescence (XRF) is an analytical technique used to determine the elemental composition of materials by measuring the characteristic secondary (fluorescent) X-rays emitted from a sample when it is excited by a primary X-ray source.

Handheld XRF enables rapid and accurate identification of composition.

Benchtop XRF for measuring liquids, powders and solids in a vacuum, helium or air.

OPTICAL IMAGING

Leica SP8 Deep In Vivo Explorer (DIVE) 2-Photon Confocal Fluorescence Microscope – Fluorescence imaging with x63 objective, lateral resolution 139nm, axial resolution 529nm. Tunable laser range: 405nm diode, white light laser 470-670 nm and IR extended wavelength 680-1300 nm. Ideal for a variety of biomedical applications including, 3D imaging of cells on biomaterials; In-vivo imaging, dynamic (time-lapse) studies; imaging of other fluorescent samples; FLIM, FRET.

Stochastic optical reconstruction microscopy (STORM) – fluorescence imaging with a 20 nm resolution in x/y and ~40 nm in z (using a MicAO 3DSR Adaptive Optics device). Samples can be maintained at room temperature in a hydrated environment (Solent incubator), so the apparatus finds many applications in biological physics (e.g. live cells) and soft-condensed matter physics (e.g. peptide aggregates).

Ultra-fast particle tracking microscope - Olympus IX71 microscope Photron Mini UX50 CMOS monochrome camera. 100 frames per second recording.

Optical Coherence Tomography (OTC)
A non-invasive imaging technique that uses a low coherence light source to create cross-sectional images of biological tissues and other scattering media. Analogous to ultrasound imaging, except that it uses light instead of sound. OTC produces high-resolution cross-sectional imaging.

Polarization-Sensitive Spectral Domain OCT Telesto Series Imaging System TEL220PSC2 – 1300nm.

High Resolution Ganymede OCT Imaging System GAN620C1 – 900nm.

EQUIPMENT LOCATIONS

	ROYCE PARTNER									
CAPABILITY	MANCHESTER	SHEFFIELD	LIVERPOOL	LEEDS	CAMBRIDGE	IMPERIAL	OXFORD	STRATHCLYDE	UKAEA	UKNNL
SEM	🟨	🟨	🟨	🟨	🟨	🟨	🟨		🟨	🟨
FIB	🟨	🟨	🟨	🟨	🟨	🟨	🟨	🟨	🟨	🟨
TEM	🟨	🟨		🟨	🟨	🟨	🟨			🟨
SIMS	🟨			🟨						
APT						🟨	🟨			
SPM	🟨	🟨			🟨	🟨	🟨		🟨	
FTIR	🟨		🟨			🟨	🟨			
RAMAN	🟨		🟨		🟨	🟨	🟨		🟨	🟨
XCT	🟨		🟨	🟨	🟨		🟨			
XRD	🟨	🟨	🟨	🟨		🟨	🟨		🟨	
XPS	🟨			🟨	🟨		🟨			
XRF	🟨	🟨	🟨						🟨	
OPTICAL		🟨				🟨	🟨			

ACCESS

Royce is open to businesses of all sizes, to tackle materials challenges and accelerate both innovation and technology translation.

From equipment access for commercially sensitive short-term research to long-term partnerships, Royce can work flexibly and support a range of approaches and needs.

Our team will provide clear options, including costings and available funding, enabling you to understand the model that fits your organisation's needs including wider training requirements.

Through our Equipment Access Schemes, Royce can provide funding to utilise its extensive portfolio of state-of-the-art materials science and engineering equipment.

Royce funded access schemes are open to researchers and students based at UK academic institutions, Research Technology Organisations, and UK-based SMEs.

Industrial Collaboration Programme

Funding for universities, RTOs and companies to explore innovative ideas with a focus on technology translation.

Researcher and Student Access Schemes

Funding for UK-based doctoral and research masters students, and researchers at all stages of their careers.

SME Access Scheme

Open to UK-based SMEs, spin-outs and start-ups, the scheme offers subsidised access to its extensive portfolio of equipment.

Designed to overcome cost barriers, de-risk experimental materials-based R&D, and complete proof-of-concept studies.

To access our services or engage with us, email info@royce.ac.uk and we'll be in touch to see how we might collaborate.

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