WESTERN SYDNEY UNIVERSITY

AMCF

ADVANCED MATERIALS CHARACTERISATION FACILITY

FACILITY CAPABILITIES

Western Sydney University's Advanced Materials Characterisation Facility (AMCF), is home to a suite of state-of-the-art instruments for analysis and characterisation. The AMCF specialises in assisting researchers, students and industry meet their research needs.

A REPUTATION FOR EXCELLENCE

Western Sydney University is ranked amongst the top two per cent of universities in the world, with a growing international reputation as an impact driven, research-led University

LEADING RESEARCH FACILITIES

Attracting researchers from around the world, our leading research institutes and state-of-the-art facilities provide a research-intensive environment with vibrant, well-resourced hubs for research.

CONTENTS

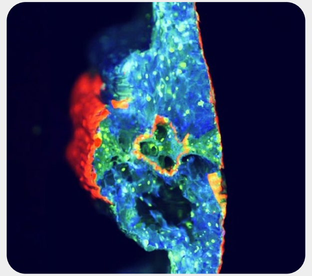
TECHNIQUES AVAILABLE.

ELECTRON MICROSCOPY

Scanning Electron Microscopy (SEM)
  - high vacuum
  - low vacuum

Scanning Transmission Electron Microscopy (S-TEM)

Cathode Luminescence (CL) imaging

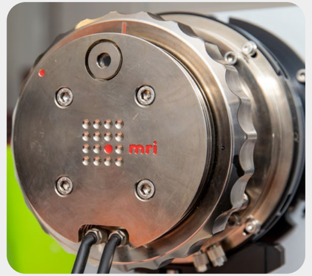


MICROANALYSIS & MAPPING

Energy Dispersive Spectroscopy (EDS)

X-ray Mapping (XRM)

Wavelength Dispersive Spectroscopy (WDS)



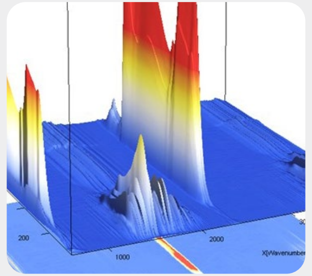
XRAY ANALYSIS

X-ray Diffraction (XRD)
  - parallel beam
  - brag-brentano

High Temperature stage XRD

X-ray Computed Tomography (CT)

X-ray Fluorescence (XRF)



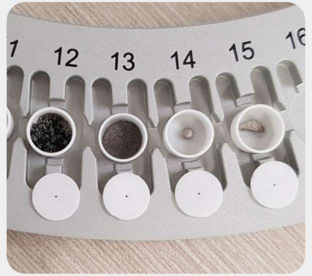
VIBRATIONAL and ELECTRON SPECTROSCOPY

Fourier Transform Infrared Spectroscopy (FTIR)
  - ATR stages
  - KBr/pellet stage
  - variable temperature stage

Raman Spectroscopy

UV-Visable Spectroscopy (UV-Vis)

FTIR Microscopy



THERMAL ANALYSIS

Thermo-Gravimetric Analysis (TGA)

Differential Scanning Calorimetry (DSC)

Thermo-Mechanical Analysis (TMA)

Cold-stage DSC and TMA options

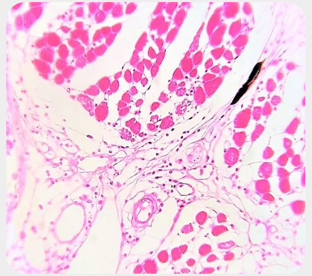
Evolved Gas FTIR Spectroscopy

SURFACE ANALSYIS

Accelerated Surface Area and Porosity (ASAP) Analysis

Surface roughness analysis with Phenom SEM

Low accelerating voltage FEG-SEM

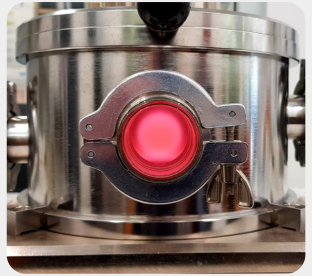


OTHER INSTRUMENTATION

Various optical microscopes with Helicon focus stacking software

Ultramicrotomes and microtomes

Critical Point Drying (CPD)



SUPPORTING EQUIPMENT

Plasma and sputter coaters for SEM sample preparation.

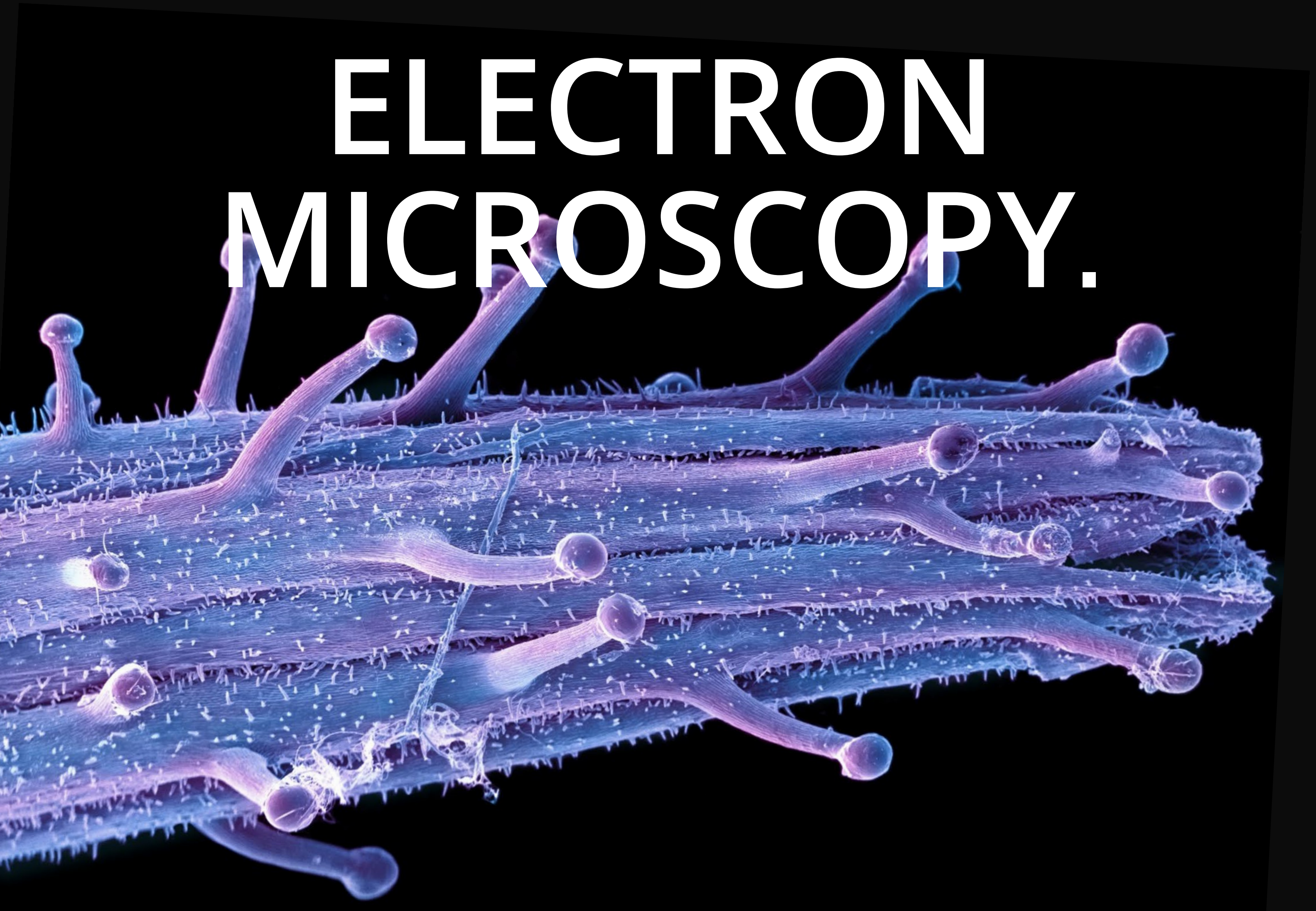
Cutting, grinding and polishing equipment.

Resin and stub mounting

Plasma cleaners, vacuum chambers and ovens

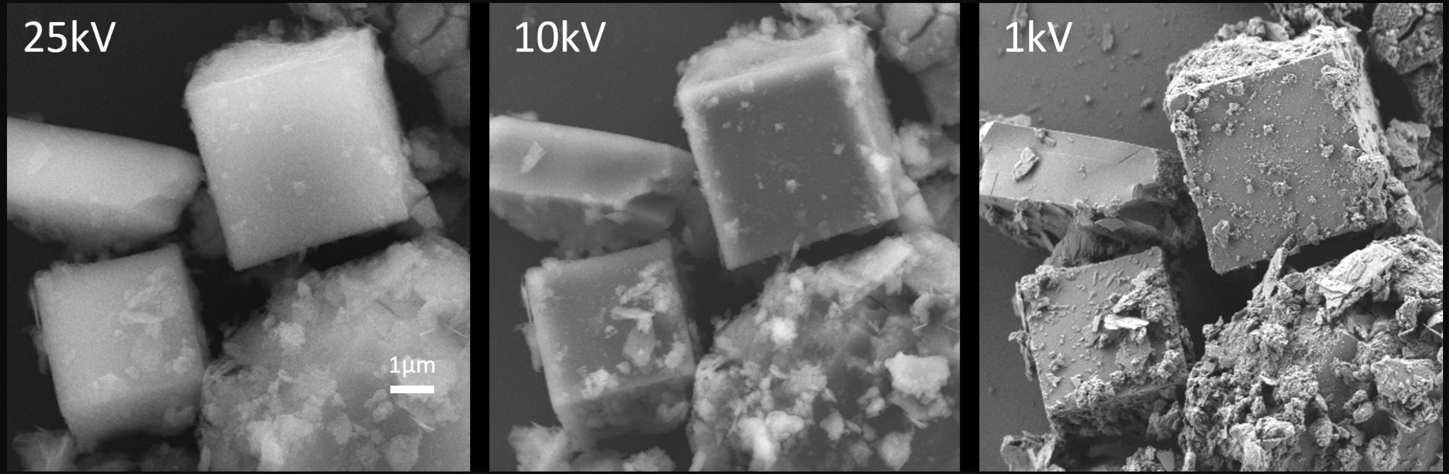
Fumehoods

ELECTRON MICROSCOPY.



The AMCF has a number of **Scanning Electron Microscopes (SEM)** available for use, all of which are equipped with Secondary Electron (SE) and Backscatter Electron (BSE) detectors for imaging, and Energy Dispersive Spectroscopy (EDS) detectors for elemental microanalysis (*see section on Microanalysis & Mapping*). Two of our instruments also have Scanning Transmission Electron Microscopy (S-TEM) capabilities.

Depending on your imaging and analysis needs, we also have a number of specialist holders, detectors and techniques which can be used, and a full range of sample preparation equipment, including coaters, resin mounting, grinding and polishing, critical point drying, microtomes and more (*see Supporting Equipment*).



A sample imaged with a secondary electron detector at different accelerating voltages (kV). Higher accelerating voltages are usually needed for elemental microanalysis, but lower kV's can give more detail of sample surfaces.

Available detectors

<u>Secondary Electron (SE) detector</u> *(all SEMs)*
General imaging under high vacuum.

<u>Backscatter Electron (BSE) detector</u> *(all SEMs)*
Low vacuum imaging of fresh or uncoated samples. Shades of grey also give information on where high and low atomic mass elements or phases are located.

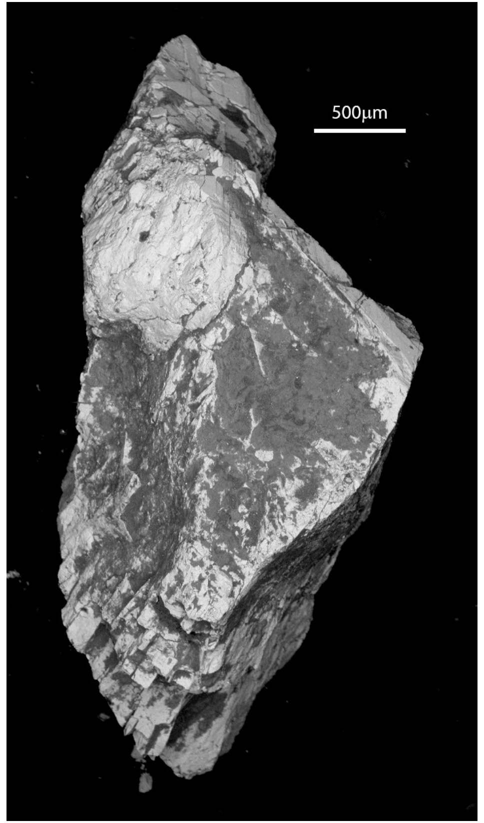
<u>Energy Dispersive Spectroscopy (EDS) detector</u> *(all SEMs)*
Elemental microanalysis of samples for identification, and X-ray mapping (XRM) for spatially locating elements and phases.

<u>InLens detector</u> *(Zeiss Merlin, Jeol 7001 SEMs)*
Great for imaging at low accelerating voltages, where small surface features are of interest.

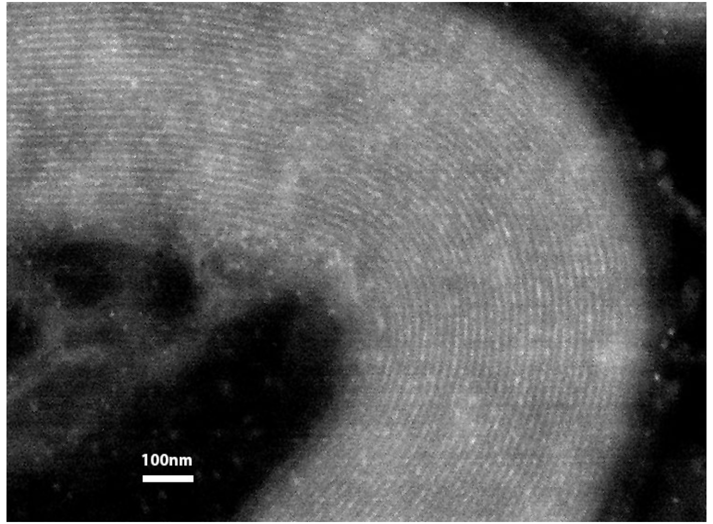
<u>Scanning Transmission Electron Microscopy (S-TEM) detector</u>
(Zeiss Merlin, Jeol 7001 SEMs)
Transmission imaging of sample sections on grids.

<u>Ultra Variable pressure Detector (UVD)</u> *(Hitachi FlexSEM)*
A secondary detector which can be used under low vacuum with fresh or uncoated samples.

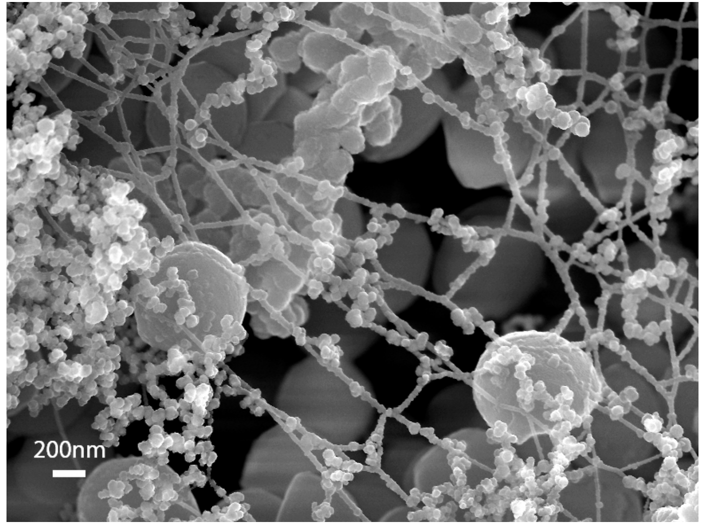
<u>Cathode Luminescence (CL) imaging detector</u>
(Zeiss Merlin, Hitachi FlexSEM)
Imaging of sample areas which emit light.



Backscatter image of a mineral. The bright patches show that there is a heavier element present in these areas, while the darkers areas show the presence of lighter elements.

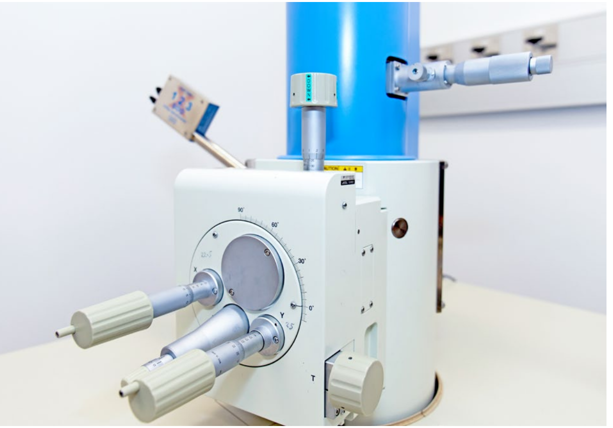


A 10kV Backscatter SEM image of a sensory afferent nerve showing individual myelin rings.



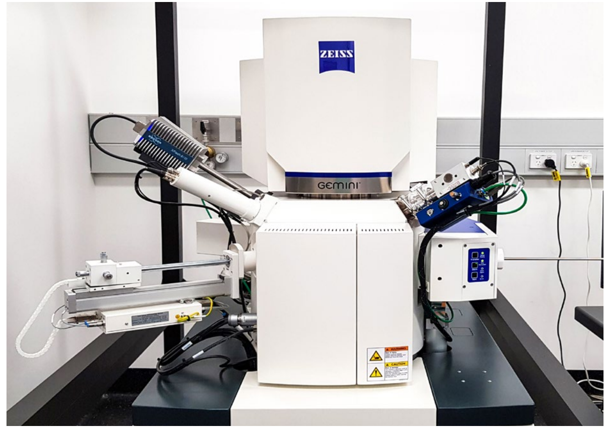
A 10kV InLens SEM image of bacteria on agar.

An example of a tungsten filament (left) used as an electron source in some of our SEMs, a Wehnelt cap which the filament will sit in (middle), and three SEM stubs with uncoated paint chip samples on a sample holder (right) ready for analysis



Jeol JSM 6510LV SEM

Source: Tungsten (W) electron filament.
Vacuum: High and variable low vacuum modes.
Stage Control: Manual 5-axes stage (X, Y, Z, Tilt and Rotate).
Accelerating Voltages: 1 - 30kV
Imaging Detectors: Secondary and Backscatter.
Microanalysis Detector: Amptek SSD C1 EDS detector, supported by Moran Scientific software package for X-ray analysis of elements present (EDS).
Advantages: A great workhorse, good for initial sample examination. This SEM is useful for natural state analysis in low vacuum, requiring minimal or no sample preparation. Its large chamber is great for analysis of bigger samples.



Zeiss Merlin Compact VP SEM

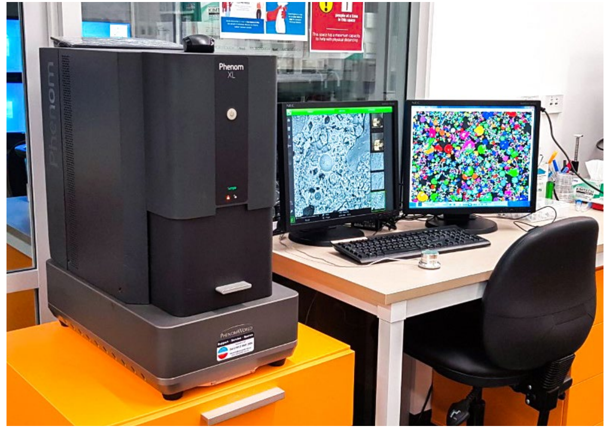
Source: Schottky Field Emission Gun (FEG).
Vacuum: High and variable low vacuum modes.
Stage Control: Motorized 5-axes stage (X, Y, Z, Tilt, Rotate).
Accelerating Voltages: 0.1 - 30kV.
Imaging Detectors: Secondary, Backscatter, InLens, S-TEM (transmission imaging) and CL detectors.
Microanalysis Detector: Bruker Quantax EDS detector, supported by Bruker Espirit software package.
Advantages: With its large range of electron energies and specialty detectors, anything from detailed surface analysis to scanning transmission imaging is possible. Surrounded by a magnetic field compensation system allowing greater field stability, the Zeiss is capable of high resolution and high magnification imaging (up to 2 million times possible), great for work on nano-materials.

Jeol JSM 7001FEG SEM

Source: Shottky Field Emission Gun (FEG)
Vacuum: High vacuum.
Stage Control: Motorized 5-axes stage (X, Y, Z, Tilt, Rotate)
Accelerating Voltages: 1 - 30kV
Imaging Detectors: Secondary, Backscatter, InLens and S-TEM (transmission imaging) detectors.
Microanalysis Detector: Multiple Amptek SSD EDS detectors, supported by Moran Scientific software package for X-ray analysis of elements present (EDS) and X-ray Mapping (XRM)
Advantages: With a FEG electron source, multiple EDS detectors and the latest Moran Scientific software, this instrument specialises in producing high quality X-ray Mapping images of prepared samples.

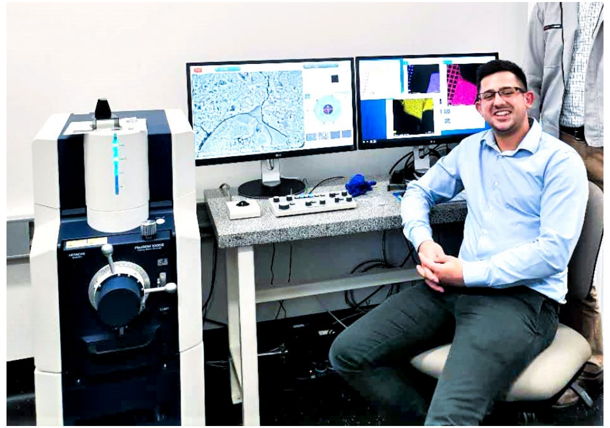
Jeol JXM 8600 Probe

Source: Tungsten electron filament.
Vacuum: High vacuum.
Stage Control: Motorized 5-axes stage (X, Y, Z, Tilt, Rotate)
Accelerating Voltages: 1 - 49kV
Imaging Detectors: Secondary and Backscatter.
Microanalysis Detectors: Amptek SSD C2 EDS detector and Wavelength Dispersive Spectroscopy (WDS) detector. Both are supported by Moran Scientific software package for X-ray analysis of elements present (EDS/WDS) and X-ray Mapping (XRM)
Advantages: Designed for non-destructive X-ray microanalysis and imaging of solid materials. Capable of high spatial resolution and relatively high analytical sensitivity. Has WDS capability for trace elemental analysis.



Phenom XL Desktop SEM

Source: Cerium Hexaboride (CeB_6) electron filament.
Vacuum: High and set low (10 and 60Pa) vacuum.
Stage Control: 3-axes stage (motorised X-Y, and manual Z). Does have scan rotation in software.
Accelerating Voltages: 5 - 15kV
Imaging Detectors: Secondary and Backscatter.
Microanalysis Detectors: EDS detector supported by both the Phenom Pro-suite and the Moran Scientific microanalysis and X-ray mapping software packages.
Advantages: Small, portable and highly capable. The Phenom SEM includes optical microscope imaging for stage navigation. The Phenom Pro-Suite package includes software for elemental analysis, particle/pore sizing, image stitching and surface roughness analysis. Its large stage which can hold over 20 samples is an added bonus.



Hitachi FlexSEM

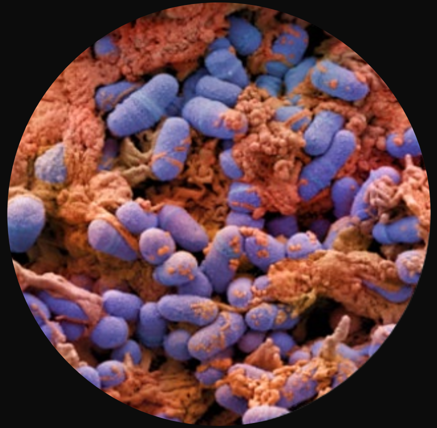
Source: Tungsten electron filament.
Vacuum: High and variable low vacuum modes.
Stage Control: 5-axes stage (motorised X-Y, and manual Z, tilt and rotation).
Accelerating Voltages: 5 - 20kV
Imaging Detectors: Secondary, UVD and Backscatter.
Microanalysis Detectors: Bruker Quantax EDS detector, supported by Bruker Espirit software package.
Advantages: The Ultra Variable-pressure Detector (UVD) allows for high resolution SE imaging of uncoated samples in their natural state in low vacuum mode. With optical microscope imaging for stage navigation this SEM is easy to use.

Colouring Images

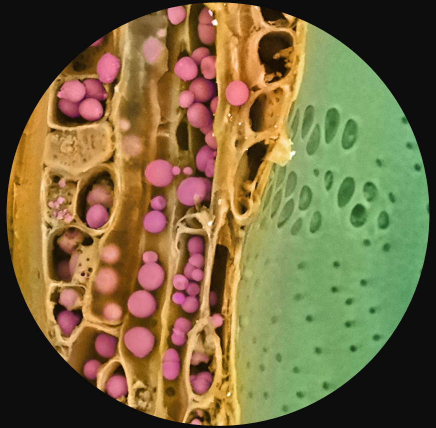
SEM images are inherently black & white. Colouring can be done using software such as Photoshop. Colouring an image can not only look great, but can also be used to help focus attention on specific details of importance in an image.

Plant material
Dr Laurel George

Spider Eggs
Dr Daniel J Fanna

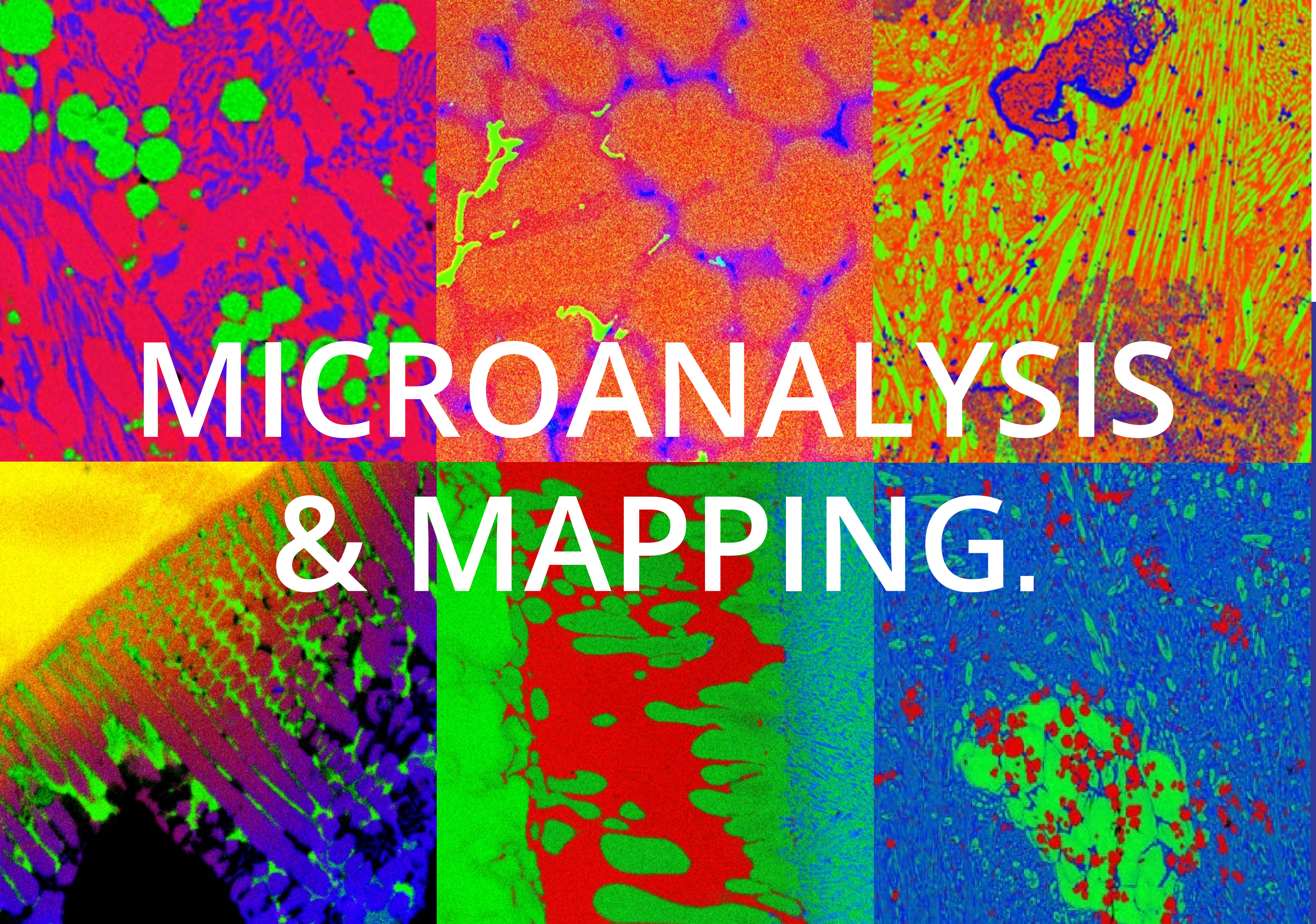


Bacteria (purple) on Flesh
Dr Michael Razieta



Starch (pink) in wood
Dr Sebastian Pfautsch

Pollen (yellow) on moth
Dr Jonathan Finch

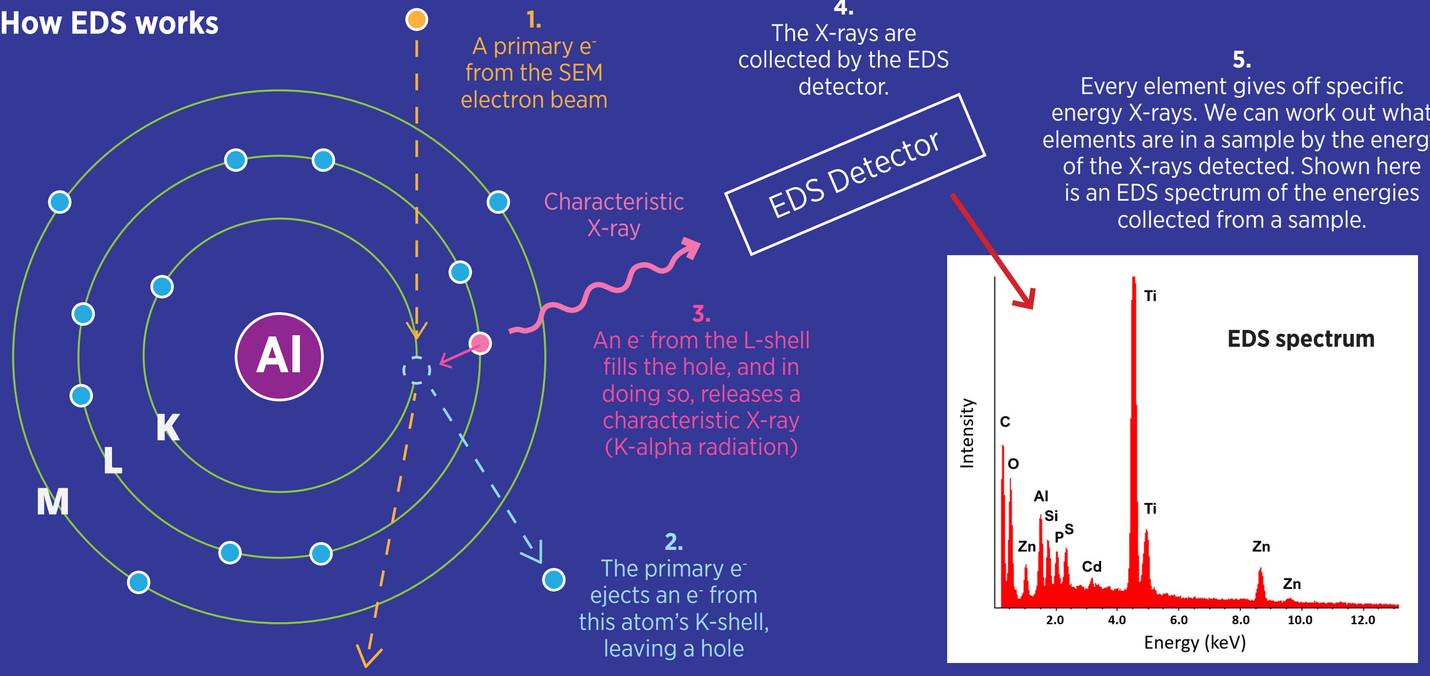


MICROANALYSIS & MAPPING.

All AMCF Scanning Electron Microscopes are equipped with **Energy Dispersive Spectroscopy (EDS)** detectors for elemental microanalysis and X-ray mapping (XRM).

These detectors allow us to generate an EDS spectrum, which is a spectrum of the x-ray energies emitted from a spot or an area of a sample. As every element has specific characteristic X-ray energies, we are able to determine a sample's elemental composition. If the sample is prepared properly, results can be both qualitative and quantitative.

How EDS works



1.
A primary e$^-$ from the SEM electron beam

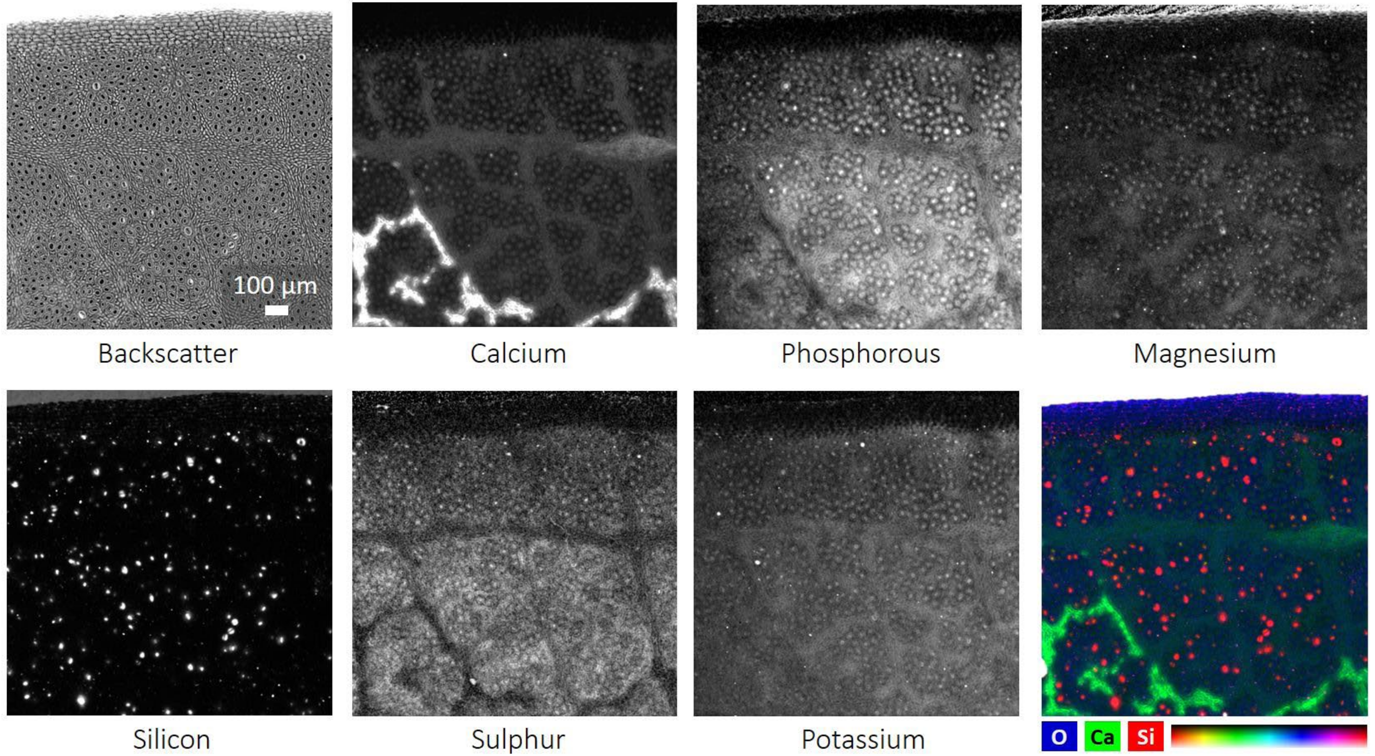
2.
The primary e$^-$ ejects an e$^-$ from this atom's K-shell, leaving a hole

3.
An e$^-$ from the L-shell fills the hole, and in doing so, releases a characteristic X-ray (K-alpha radiation)

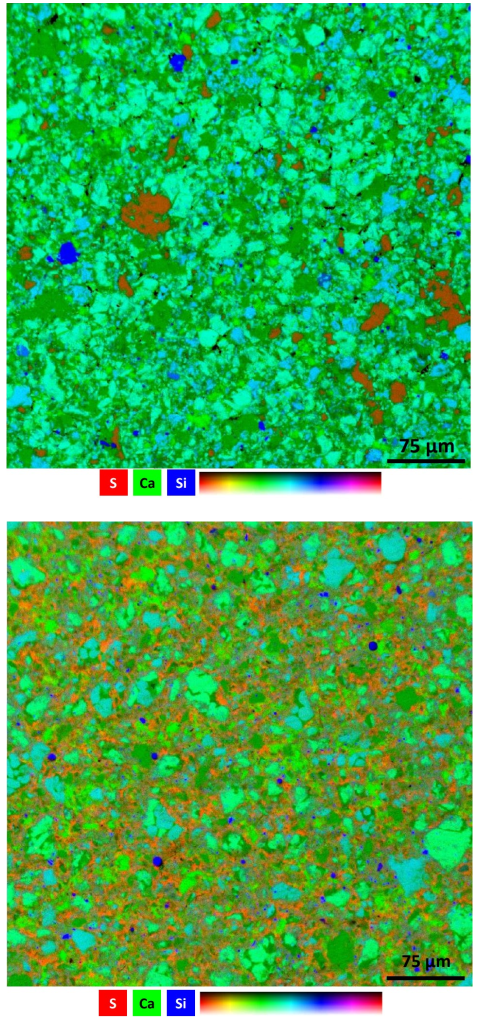
4.
The X-rays are collected by the EDS detector.

5.
Every element gives off specific energy X-rays. We can work out what elements are in a sample by the energy of the X-rays detected. Shown here is an EDS spectrum of the energies collected from a sample.

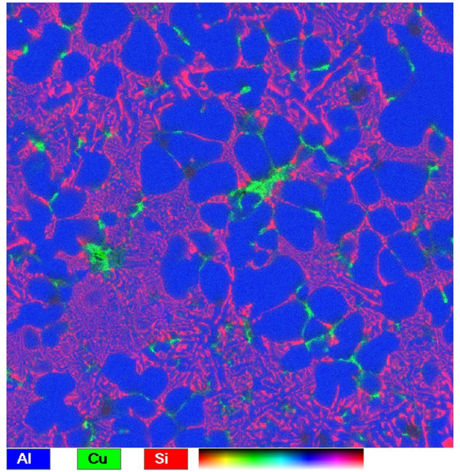
X-ray Mapping (XRM) is the collection of characteristic X-rays as a function of the position of the scanning electron beam on the sample. In other words, we are collecting an EDS spectrum for each pixel in an SEM image. Using an XRM software package, we are then able to extract data from all of the spectra collected, and colour code each pixel in an image to show what elements are present.



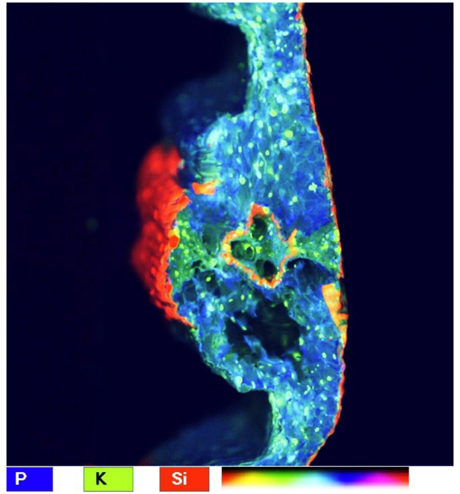
A backscatter SEM image of a Eucalyptus leaf (top left), and some of the corresponding elemental XRM maps collected. Bottom left shows a false coloured image, where elements have been assigned a colour. This allows us to now spatially locate where elements are in a sample.



Pseudo coloured XRMs of an anhydrous Ordinary Portland Cement (OPC) sample (top) and a 7-day hydrated sample (bottom. We can see sulfur (red) has dispersed throughout the cement matrix. Colours inbetween the three assigned show that there are phases present involving at least two of the selected elements (e.g. light blue falls between blue (Si) and green (Ca) on the colour scale, so there may be a calcium silicate phase present)



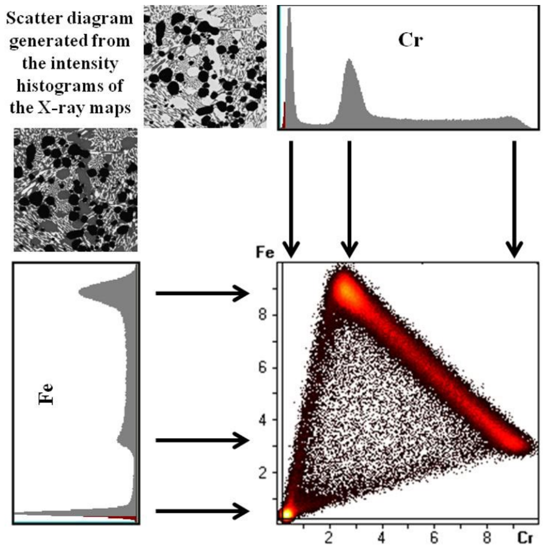
A pseudo coloured XRM of an aluminium alloy used on a motocycle.

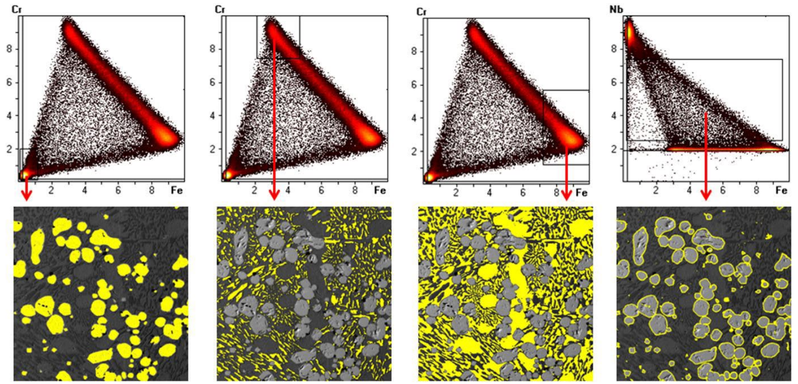


A pseudo coloured XRM of a grass leaf cross section showing the presence of Si (red)

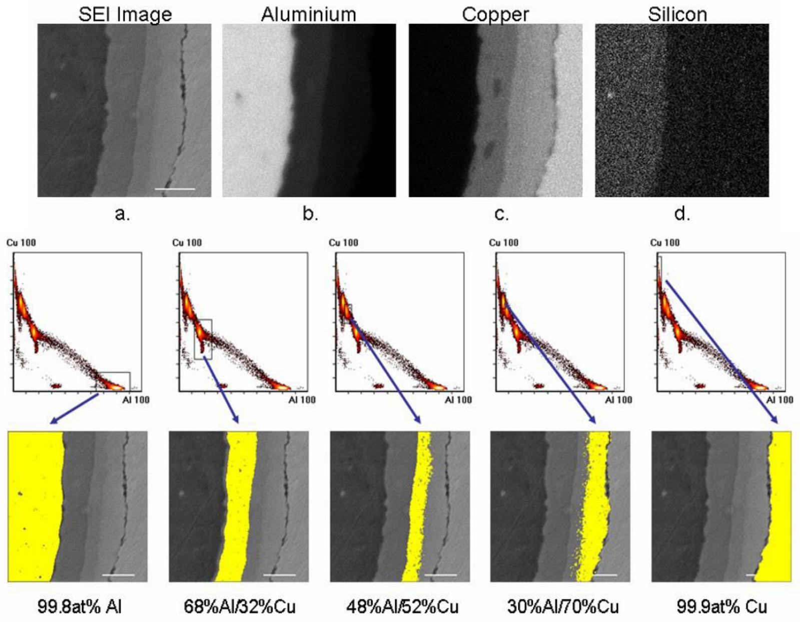
The X-ray intensity distributions of the elements from an X-ray map allow us to generate two dimensional and ternary **scatter diagrams**, converting spatial information into concentration dimensions. This is an important tool for displaying the spatial relationships of elements or correlated elements (phases) in materials. The method to accomplish this is through the use of two and three-dimensional (2D and 3D) scatter (or correlation) diagrams, which are generated from the X-ray maps.

From these scatter diagrams we observe clusters, also referred to as nodes, which correspond to different chemical phases. We can also observe linking (connection) between clusters indicating the boundaries between phases within a material as well as branching from clusters (some links may contain branches).

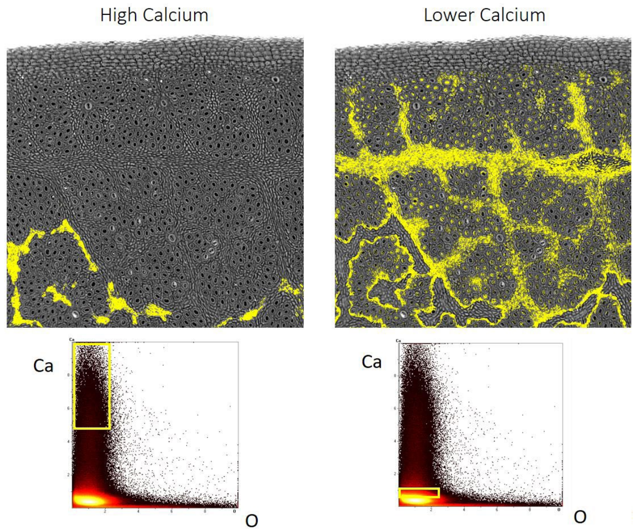




Generation of scatter diagrams for a white cast iron hard-facing material with NbC particles. A set of scatter diagrams for iron versus chromium (a-c) and niobium versus iron (d) showing different cluster selections and the corresponding points then displayed and redrawn on the SEM image.



BSE, XRM and scatter diagrams for copper-aluminium laminate. The images at the bottom are secondary electron images with information from the different clusters of the scatter diagram superimposed over the image

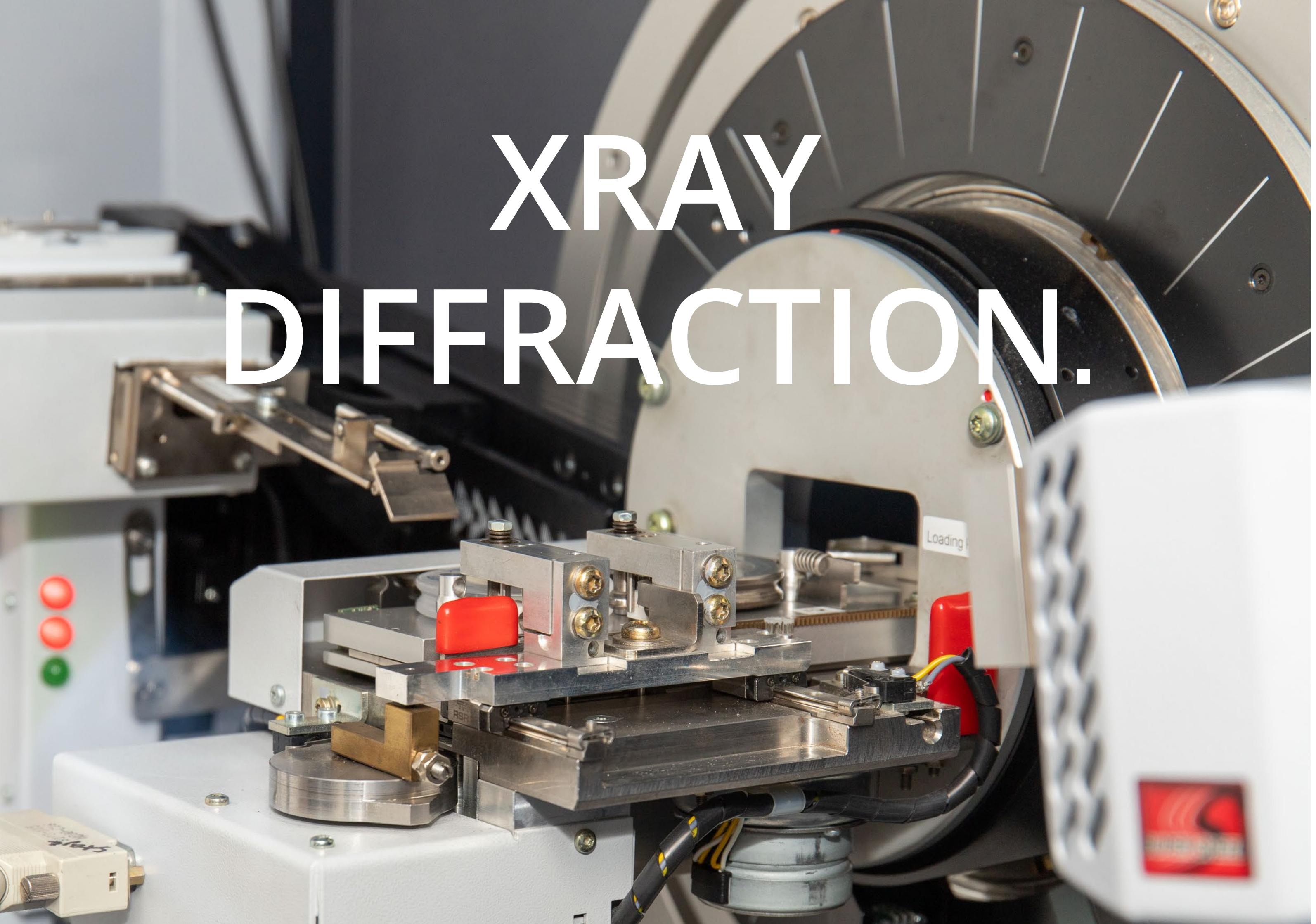


Superimposed BSE images of a Eucalyptus leaf, showing the presence of high and low amounts of calcium in the leaf. This has been done using the information taken from teh scatter diagrams below. The high amounts show where calcium oxalate crystals have formed just below the leaf's surface.

From the clusters observed in the scatter diagram, it is possible to map the phases. This is often referred to as phase mapping, but really it should be called chemical phase mapping (CPM), as phase mapping assumes knowledge of crystal structure and requires diffraction analysis.

Chemical phase mapping (CPM) involves selecting areas on the scatter diagrams and observing where the points or clusters relate on the BSE image and is an important part of the phase identification process. These selected analysis points may then be summed for a more accurate analysis in total or by selecting strategic areas on the image.

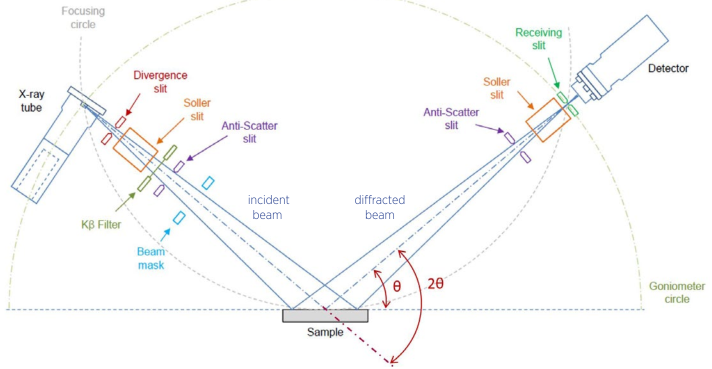
XRAY DIFFRACTION.



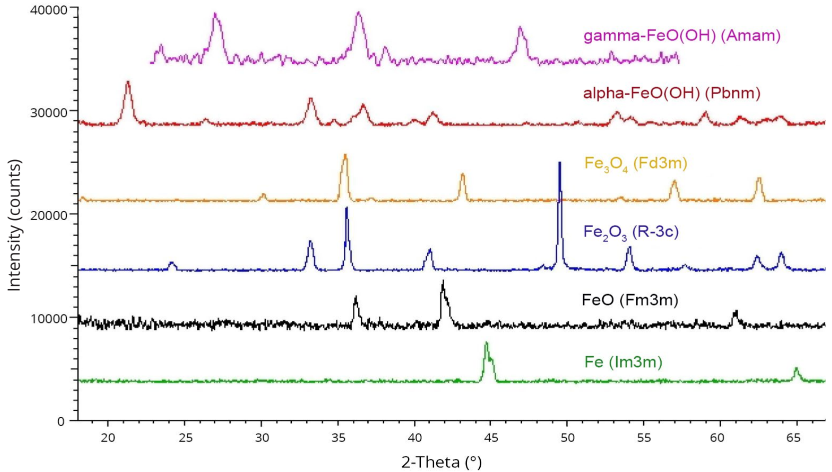
X-Ray Diffraction (XRD) is a non-destructive technique that uses X-rays to investigate and quantify the crystalline nature of materials. It does this by measuring the diffraction of X-rays from the planes of atoms within a material, and is sensitive to both the type and relative position of atoms in the material as well as the length scale over which any crystalline order persists.

It is therefore used to measure the crystalline content of materials, identify the crystalline phases present (including the quantification of mixtures in favourable cases), determine the spacing between lattice planes and the length scales over which they persist, and to study preferential ordering and epitaxial growth of crystallites.

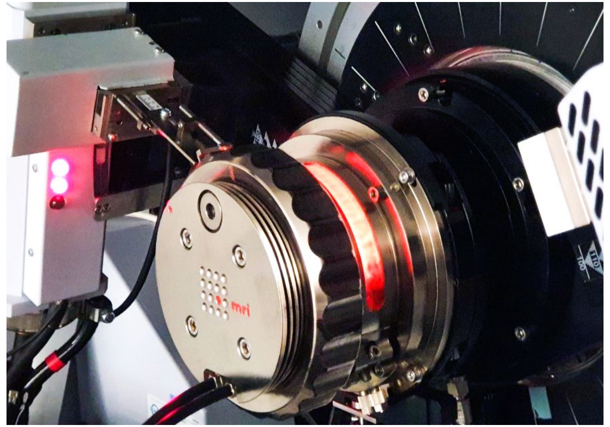
Once spectra are acquired, they can be matched to a library of spectra in order to identify the crystalline phases present.



A typical Bragg-Brentano XRD configuration

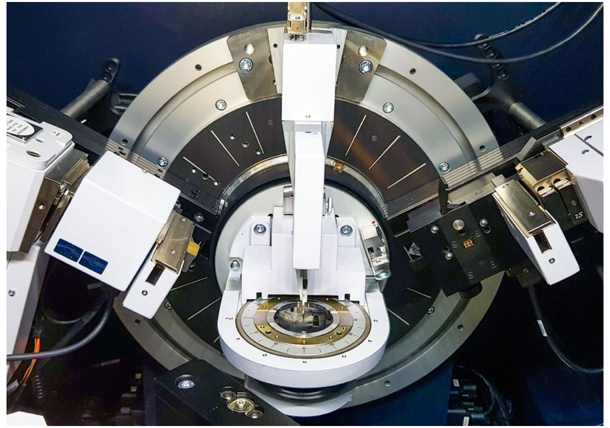


These XRD spectra show how this technique can be used to identify the crystalline phases present in samples with the same elemental composition (Fe and O)



Bruker D8 Advance XRD (Series I)

X-ray Source: Cu K-alpha wavelength
Detector: LYNXEYE position sensitive detector allowing for rapid analysis while reducing artefacts from Cu K-beta radiation.
Configurations: manual switching between Bragg-Brentano and Parallel-beam geometry. Can be configured for use of a high temperature stage to probe the effects heat has on the crystalline properties of your sample.
Advantages: Motorised divergent slit allowing the slit angle to be finely tuned or set to a fixed sample illumination mode. Flip-stick auto changer allows for up to 9 samples to be mounted (only when heat stage is not in use).



Bruker D8 Advance XRD (Series II)

X-ray Source: Cu K-alpha wavelength
Detector: LYNXEYE XE-T position sensitive detector. Energy resolution of < 380eV (FWHM: 160eV at 8 keV) and variable active detector window for rapid analysis while filtering out Cu K-beta radiation and iron fluorescence.
Configurations: TWIN optics for semi-auto switching between Bragg-Brentano and Parallel-beam geometry. permits users to carry out a range of diffraction experiments without complicated alignments.
Advantages: Dynamic Beam Optimisation (DBO) allows for optimisation of measurements at low 2theta angles by tuning the divergent slit opening (Bragg-Brentano), incorporating a motorised anti-scatter screen and a variable field-of-view active detector window. Autosampler with current capacity for up to 45 samples.

XRD accessories, sample holders and other capabilities

The AMCF hosts a range of different sample holders and accessories for our diffractometers, these include:

Sample holders

- Powder sample holders which are the typical sample holders for XRD experiments.
- Bulk/solid sample holders with a larger sample recesses allowing for bulk powder samples or solid samples which cannot be ground (metals, thin films, etc.).
- Low Background Silicon sample holders for very small sample sizes or organics.
- Backloading sample holders to reduce preferred orientation effects on powder samples.
- Orientated Clay mount sample holders for aligning clay crystallites along their basal plane (00l) to assist in identification of specific clay groups.
- Airtight sample holders with transparent domes allowing for environmentally sensitive materials to be analysed in tailored atmospheres.
- We also have the ability to design and 3D print sample holders for specific sample needs.

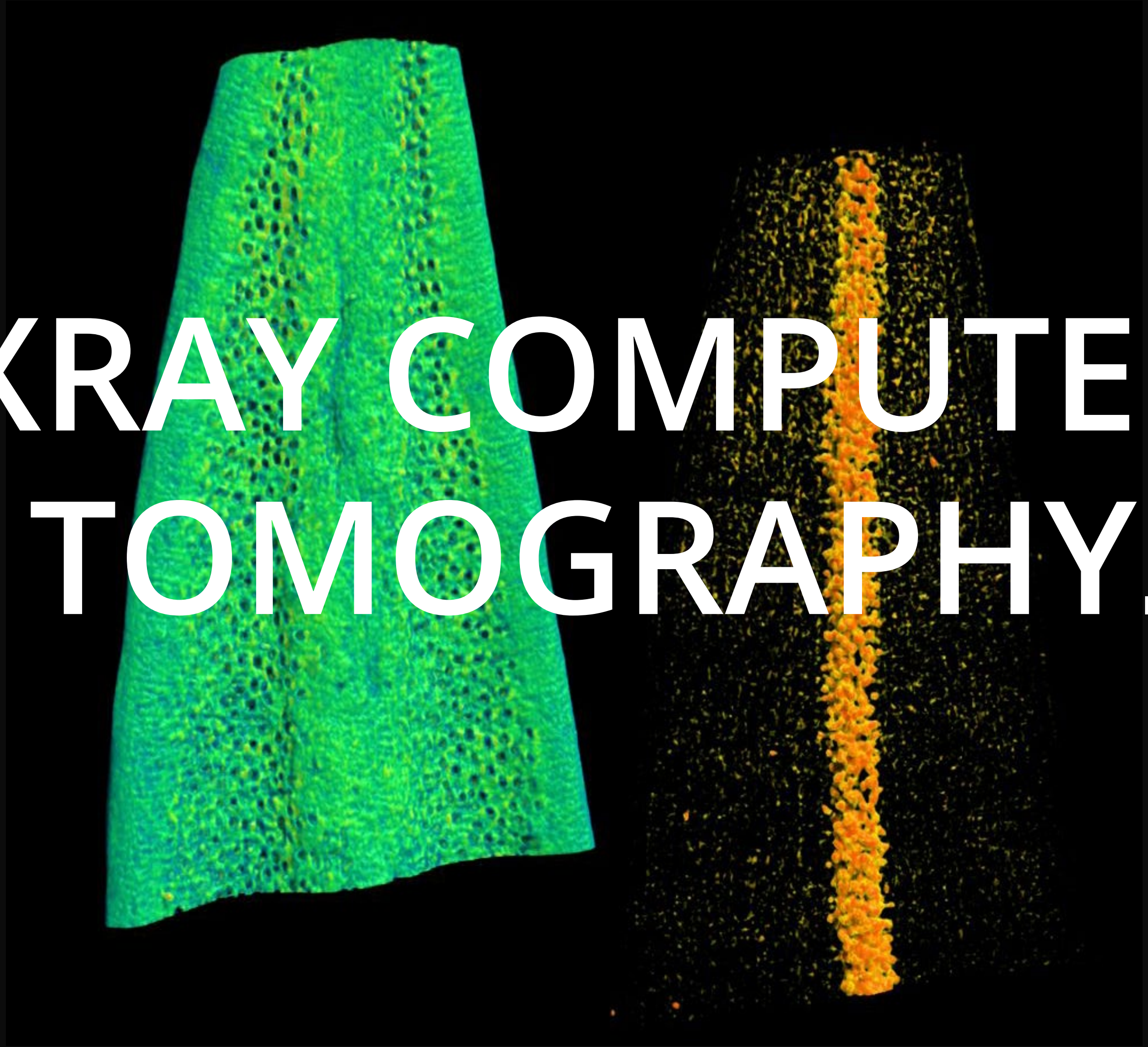
Heating stage accessory

Allows for experiments to be carried on samples from 30 °C and up to 1300 °C (sample depending). Experiments below 300 °C can be carried out in a variety of atmospheres, while higher temperature experiments require the sample to be in held under vacuum. High temperature XRD will allow users to probe the crystalline changes that may occur in their samples with respect to temperature.

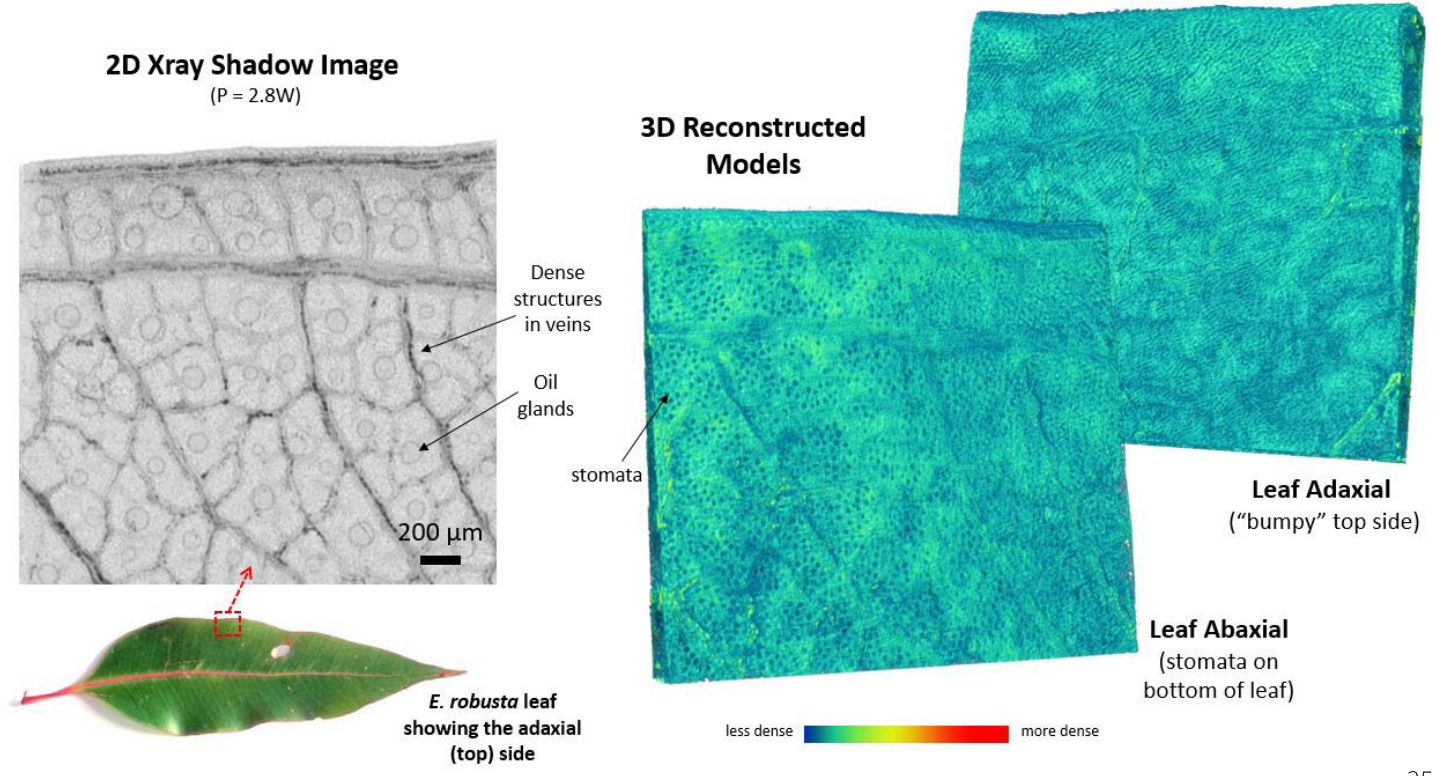
Software and Databases

- **Eva**, Bruker's general XRD data evaluation, processing and phase identification software.
- **TOPAS**, Bruker's advanced XRD processing including phase quantification, Rietveld, Le Bail Pawley refinements and more.
- **ICDD PDF 4+** powder diffraction database the most comprehensive database for inorganic diffraction data.
- **COD** open access database with collection of organic, inorganic and mineral crystal structures.

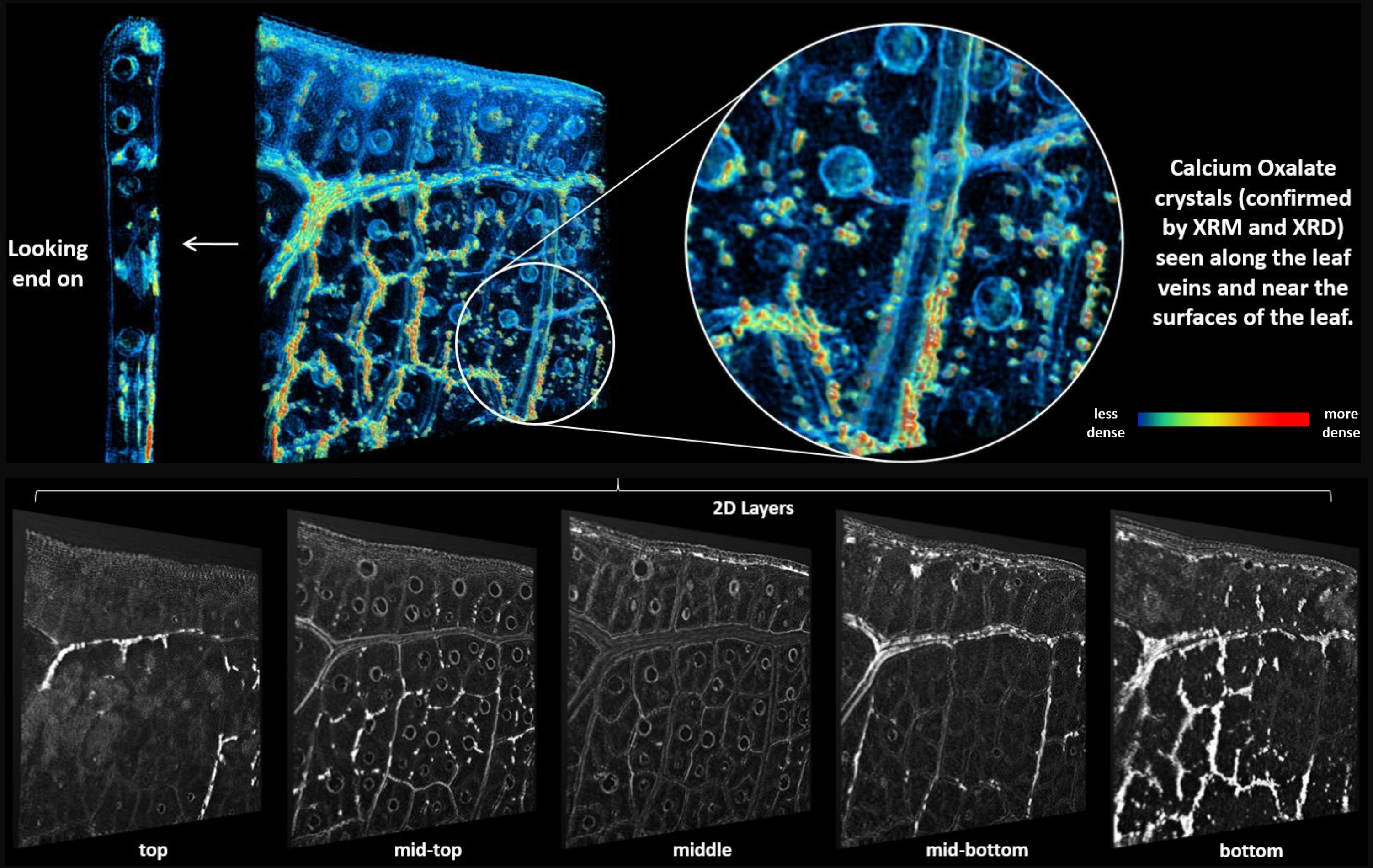
XRAY COMPUTED TOMOGRAPHY.



Using X-rays and a sample rotation system, **micro computed tomography (micro-CT)**, allows us to image cross sections (2D) of an object, and then reconstruct these images into a 3D model using specialised software. This computer model can then be manipulated to show various aspects of interest in the sample, such as cross sectional views, sample transparency to see internal structures and density mapping. This all allows for the non-destructive investigation of the internal structures of samples with high detail.



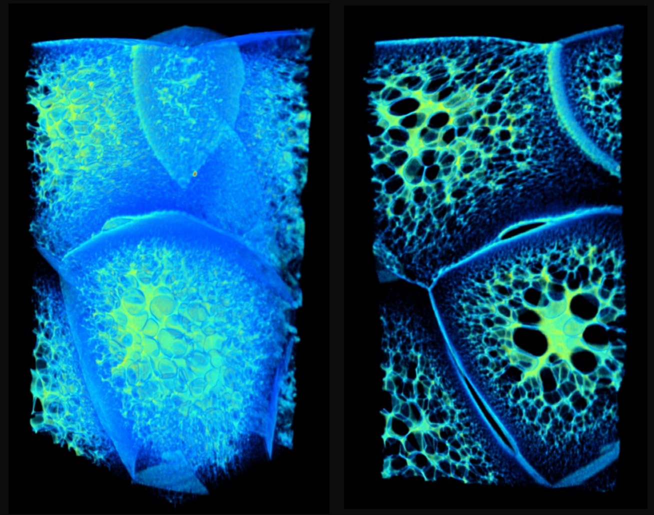
Example of 3D transparent micro-CT models of a Eucalyptus leaf (top), and individual 2D X-rays taken from different layers in the leaf (bottom). These types of views allow us to spatially locate dense structures.



Calcium Oxalate crystals (confirmed by XRM and XRD) seen along the leaf veins and near the surfaces of the leaf.

3D Reconstructed Models

Cross sections of a cactus flower

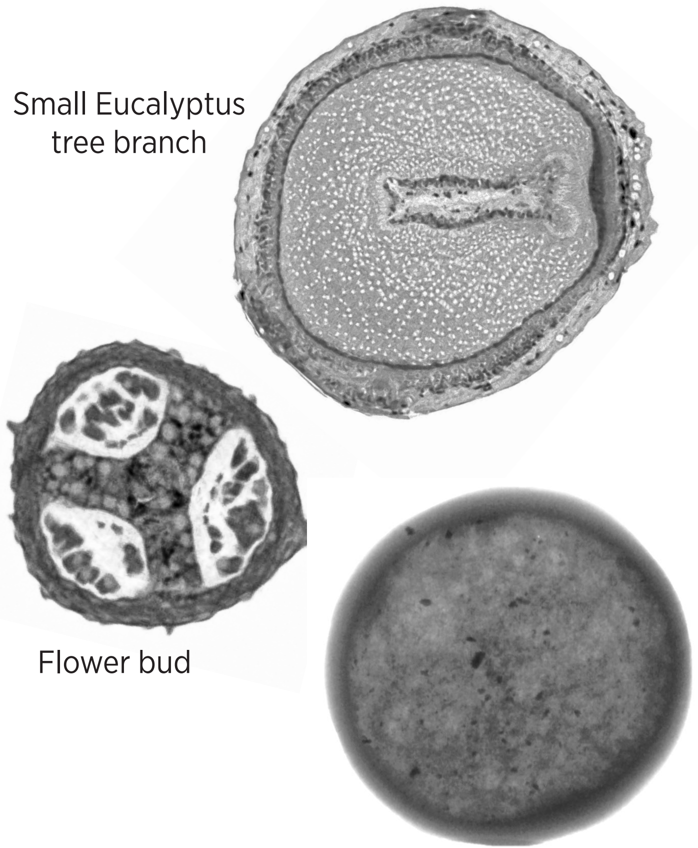


Engineering foam and cross section

Small lizard attacked by ants

Seed pod and cross section

2D X-ray sections



Small Eucalyptus tree branch

Flower bud

Malteser

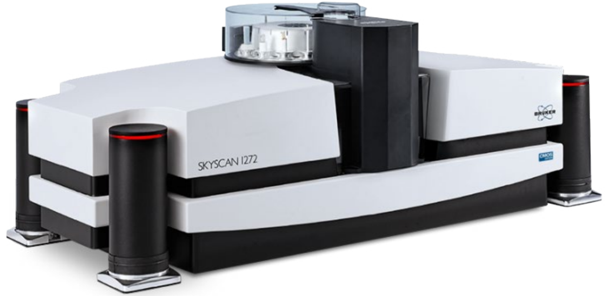
Bruker SkyScan 1072 CT Scanner

X-ray Source: Cu K-alpha wavelength
Operation Voltage: 0.5 - 80kV.
Operation Current: 1pA - 100µA.
Specimen Size: up to 1.5cm diameter and 3cm tall.
Magnification: x15 - 120
Filters: 1mm aluminium
Spacial Resolution: 5-20µm depending on sample material.
Software: Skyscan operating software for data Acquisition, Nrecon for reconstruction and Dragonfly for processing.

COMING SOON

Bruker SkyScan 1273 CT Scanner

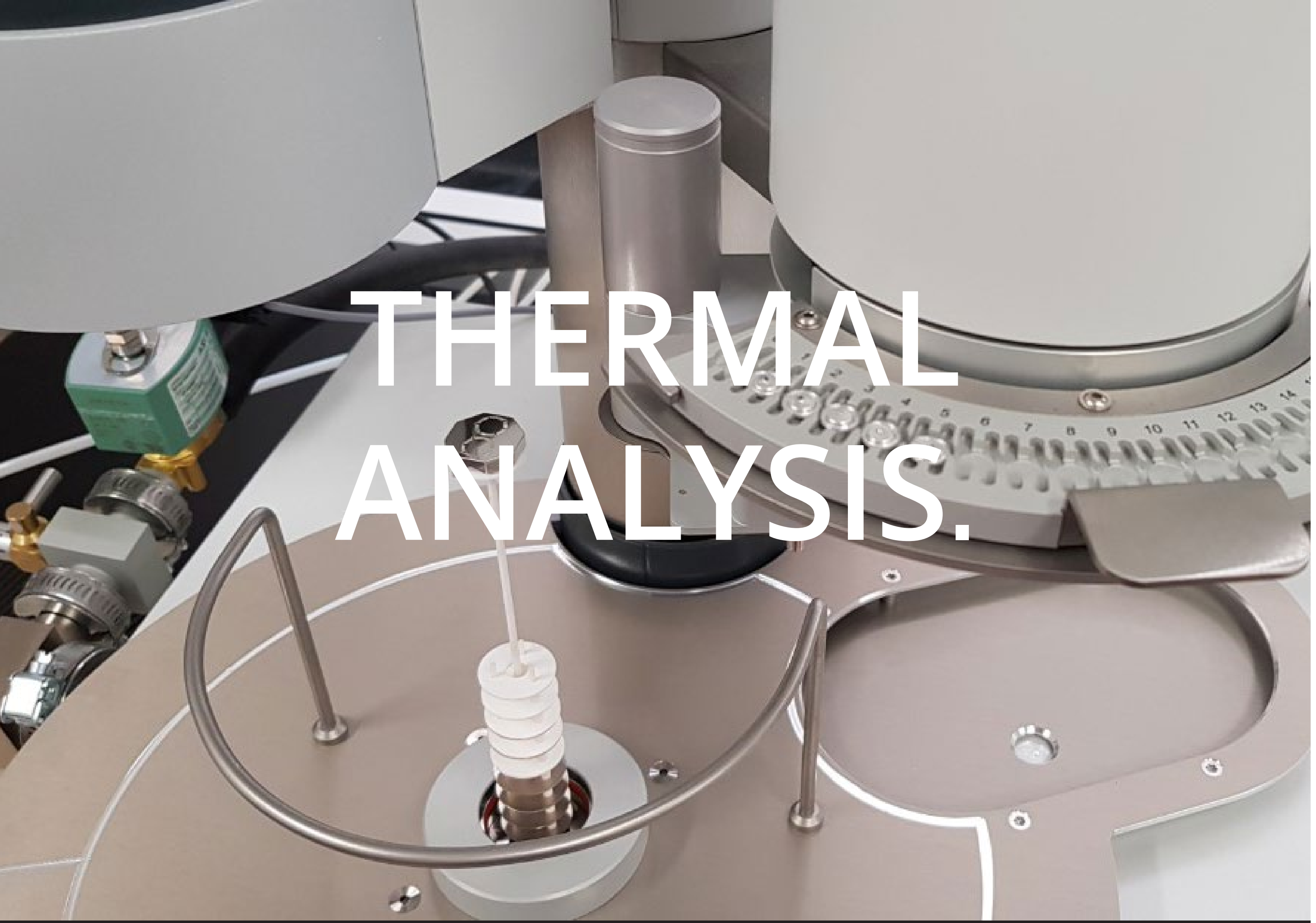
Operation Voltage: 40 - 130kV.
Max. Power: 39W.
Max. Scanned volume: 25x25cm, but chamber can fit up to 30x50cm samples.
Detector size: 5.9Mp
Spot size: < 5µm spot size @ 4W
Advantages: With its ability to go to higher power, this micro-CT scanner will be perfect for denser materials, such as concretes, steels and composite materials to name a few. It also makes use of high aspect ratio scanning, which can make scans 4x faster than regular scanning protocols.
The large chamber means large samples can be scanned, such as a whole small plant in a pot.



Bruker SkyScan 1272 CT Scanner

Operation Voltage: 20 - 100kV.
Max Power: 10W.
Max. Scanned volume: 2.5cm (7.5cm using offset scans) x 8cm high
Detector size: 16Mp
Spot size: <5µm spot size @ 4W
Advantages: This high resolution micro-CT scanner is capable of detecting object details down to 0.45µm. This instrument will also be coming with a 16-position sample changer, complete with auto adjustment of magnification and scanning protocols.
Both of these instruments can also be fitted out with other optional stages (e.g. for heating, colling, compression/tension testing)

THERMAL ANALYSIS.



Thermal analysis involves the characterisation of solid and liquid materials during heating, cooling. or static temperatures. Various thermal events can be monitored and quantified with high accuracy across a broad temperature range. The AMCF houses instruments that can conduct three main thermal techniques:

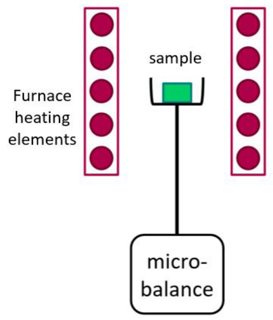
Thermo-Gravimetric Analysis (TGA): Monitors changes in mass as a function of temperature or time (e.g. mass loss due to thermal decomposition or mass gain due to oxidation)

Differential Scanning Calorimetry (DSC): Monitors flow of energy into (endothermic) or out of (exothermic) a sample as it is heated/cooled. Information about thermal events such as glass transitions, melting points, crystalisation temperatures and phase transitions can be found.

Thermal Mechanical Analysis (TMA): Samples are monitored for changes in physical characteristics as a function of temperature. There are different setups which can allow for analysis of characteristics such as expansion, contraction, pentration, softening points, glass and phase transitions, young’s modulus and elasticity to name a few.

TGA

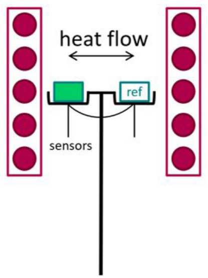
Thermo-Gravimetric Analysis



mass loss

DSC

Differential Scanning Calorimetry

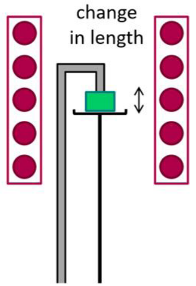


Endothermic: heat absorbed
Exothermic: heat released

heat flow

TMA

Thermo-Mechanical Analysis



Can apply static or dynamic forces

size change

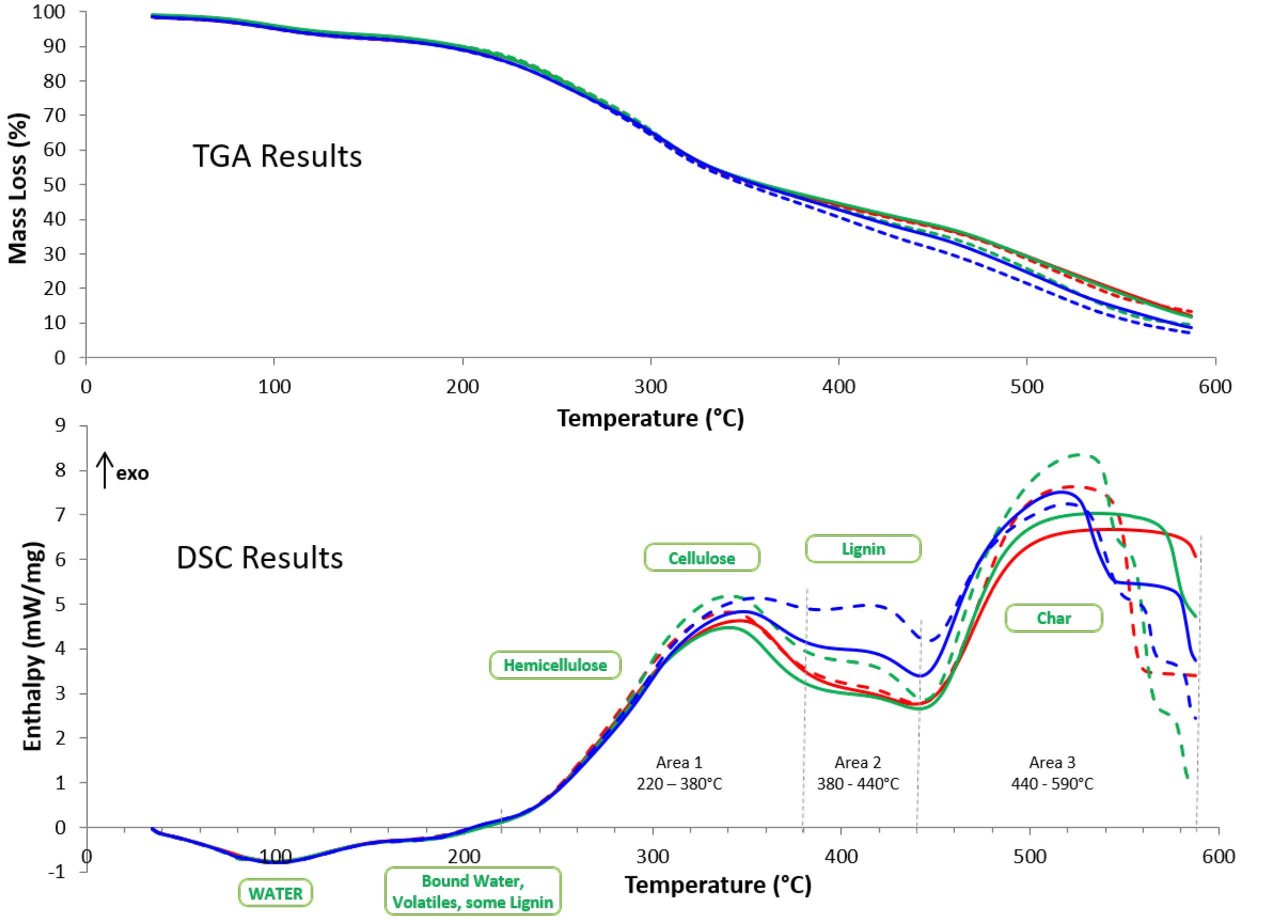
All thermal techniques are complimentary, and data from each can plotted against temperature on the same graph. The AMCF has a suite of thermal instrumentation, including two Simultaneous Thermal Analysers (STA), which can simultaneously perform TGA and DSC at the same time. By analysing these signals together, we can get a better understanding of the thermal events taking place in a sample.

Before beginning an experiment, it is first important choose what has atmosphere you would like to conduct your experiment under, as this will affect how your sample behaves. Which gas you choose will also be influenced by what you are wanting to understand about your sample. For example:

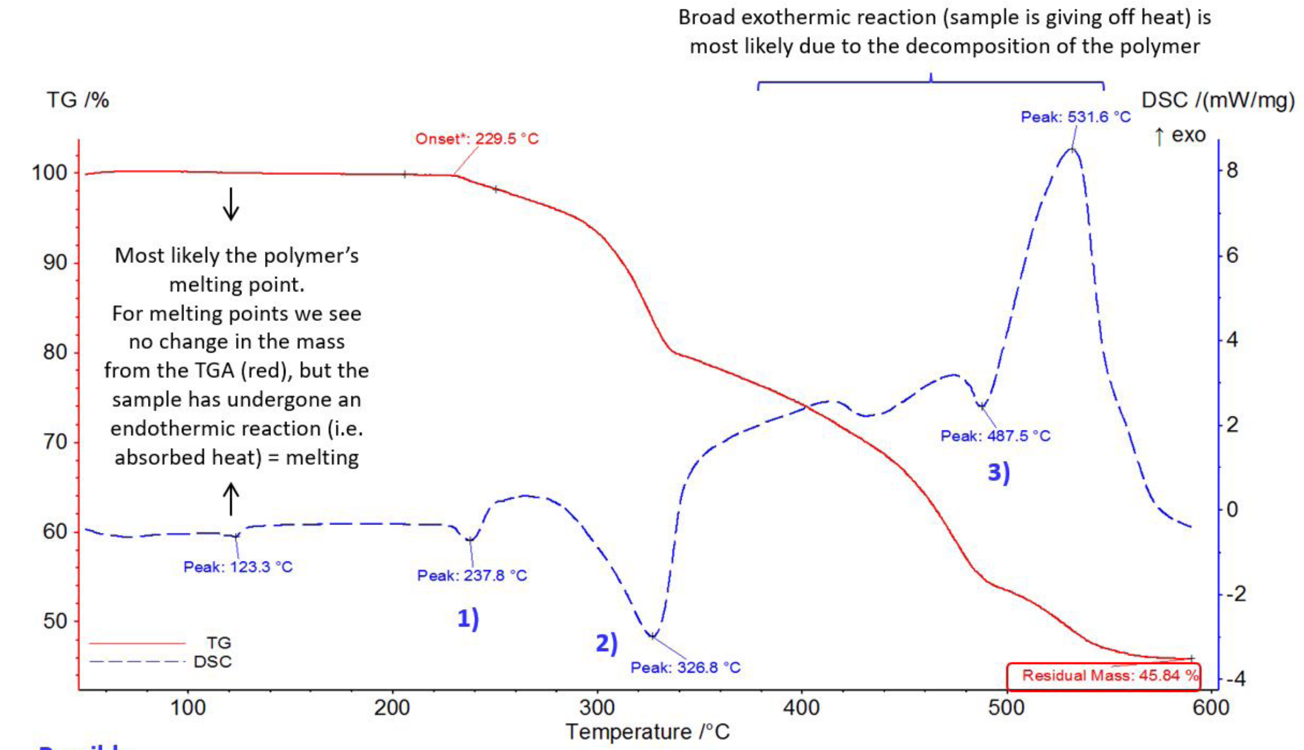
- Is there a real life senario you are wanting to replicate?
- Are you wanting to understand more about the material's structure?

Atmospheres available at the AMCF and how they thermally affect sample behaviour.

Atmosphere	Gas	Thermochemical Reaction	Process	Main Products	DSC Result
Inert	Nitrogen, Argon	Pyrolysis	Breaking molecules down into smaller fragments	Small chain molecules (CH_4), chars, tars, H_2	Endothermic
Oxidising	Air, Oxygen	Combustion	Reaction between material and oxygen to produce heat and light	CO_2, H_2O, CO, NO_2, NO_3	Exothermic



STA results (simultaneous TGA and DSC) of different Eucalyptus leaf samples heated up to 600°C under an Air atmosphere. Here it can be seen that as the sample loses mass, endo- or exo-thermic events can be seen in the DSC results, which help to explain the processes taking place .


Broad exothermic reaction (sample is giving off heat) is most likely due to the decomposition of the polymer

Most likely the polymer's melting point. For melting points we see no change in the mass from the TGA (red), but the sample has undergone an endothermic reaction (i.e. absorbed heat) = melting

Possible *endothermic* peak reactions

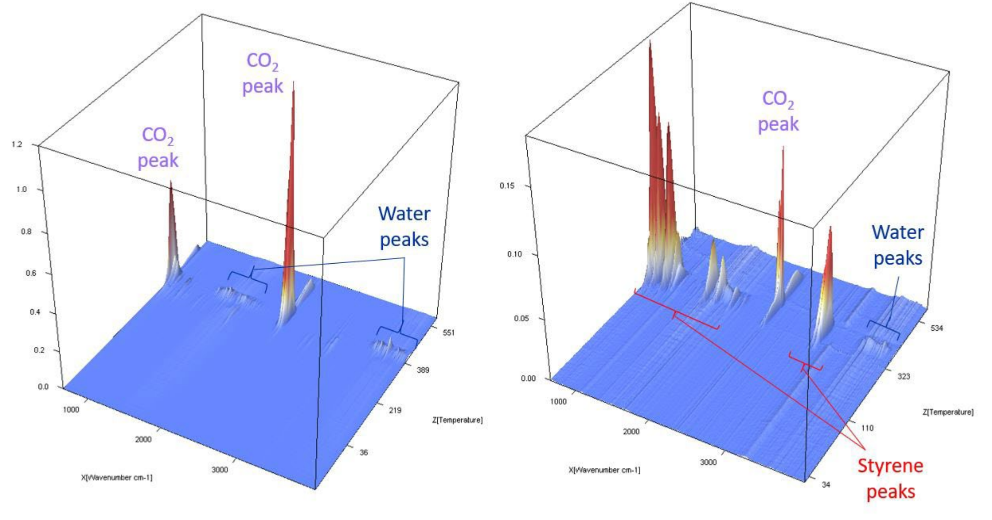
1) $Al(OH)_3 \rightarrow AlOOH + H_20$ — *Gibbsite*, *Boehmite*

2) $2Al(OH)_3 \rightarrow \chi\text{-}Al_2O_3 + 3H_20$ — *Gibbsite*, *amorphous Alumina*

3) $2AlOOH \rightarrow \Upsilon\text{-}Al_2O_3 + H_20$ — *Boehmite*, *Alumina*

STA results (simultaneous TGA and DSC) for a piece of composite building cladding material (polyethylene with aluminium hydroxide filler) heated up to 600°C in an Air atmosphere. Here a melting point becomes apparent in the DSC at 123°C as there is no mass loss in the TGA. The evaporation of water at three points as the alumium hydroxide dechydrates is an endothermic event, and therefore these peaks face down. The thermal degredation of polyethylene is exothermic.

During thermal analysis, one of our STA instruments is able to be connected to a FTIR gas cell for Evolved Gas analysis (EG-FTIR) *(also see vibrational spectroscopy section)*. This allows us to identify not only what gases are coming off, but also profile the temperatures at which they are coming off. The TGA results can then be used to calculate how much has evolved.



Air atmosphere

Nitrogen atmosphere

Shown here are 3D EG-FTIR spectra from polystyrene samples heated up to 600°C in different atmospheres. These show that different thermal decomposition proceses have taken place. Under an Air atmosphere the sample has combusted and released CO_2 and water vapour, whereas under a Nitrogen atmosphere the sample has pyrolysed, and broken up into smalller styrene molecules which have evolved off the sample.

Software Available for Analysis:

Netzsch Proteus
For analysis of STA, TGA, DSC and TMA results

Netzsch Kinetics Neo
For analysis of thermal data for kinetics calculations and modelling

Netzsch Peak Separation Software
For analysis of overlapping thermal events

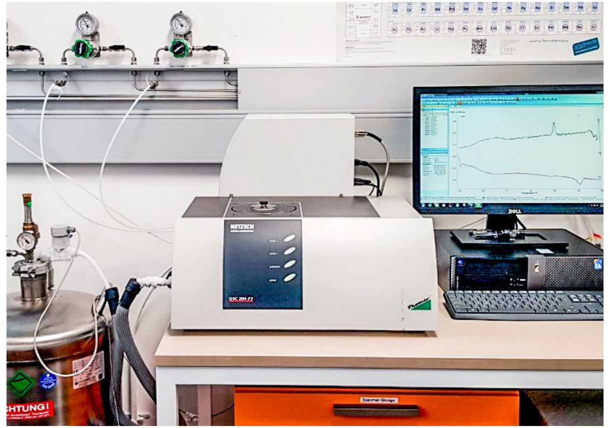
Bruker OPUS 3D Package
Analysis of EG-FTIR data

Netzsch STA 449F3 Multi

Analysis Type: Simultaneous TGA and DSC
Furnace: Silicon Carbide
Temperature Range: Room Temperature up to 1600°C.
Thermocouple: S-type
Gases: Instrument Air, Nitrogen and Argon.
Crucibles: Aluminium, alumina and platinum. Alumina and platinum crucibles may need to be purchased.
Advantages: With a 20 position auto-sampler, this instrument is great for the routine analysis of many samples. This system is supported by Proteus 8 software which includes the Auto-Evaluate feature. Also installed is Netzsch's Kinteics Neo software (for analysis of the thermo-kinetic properties of samples, such as activation energies) and Netzsch's Peak Separation Software, which can be used for thermal, FTIR and mass spec data.

Netzsch STA 449C Jupiter

Analysis Type: Simultaneous TGA and DSC. Can also be setup for TGA-only analysis of large samples, or coupled to gas FTIR for evolved gas analysis.
Furnace: Platinum-Rhodium
Temperature Range: Room Temperature up to 1600°C.
Thermocouple: S-type
Gases: Instrument Air, Nitrogen and Argon.
Crucibles: Aluminium, alumina and platinum. Alumina and platinum crucibles may need to be purchased.
Advantages: This versatile instrument is equipped with a number of different holder types and accessories for more specific experiments. It's metal furnace means its ideal for sensitive specific heat measurements. It can also be coupled to a gas FTIR cell for evolved gas FTIR analysis.

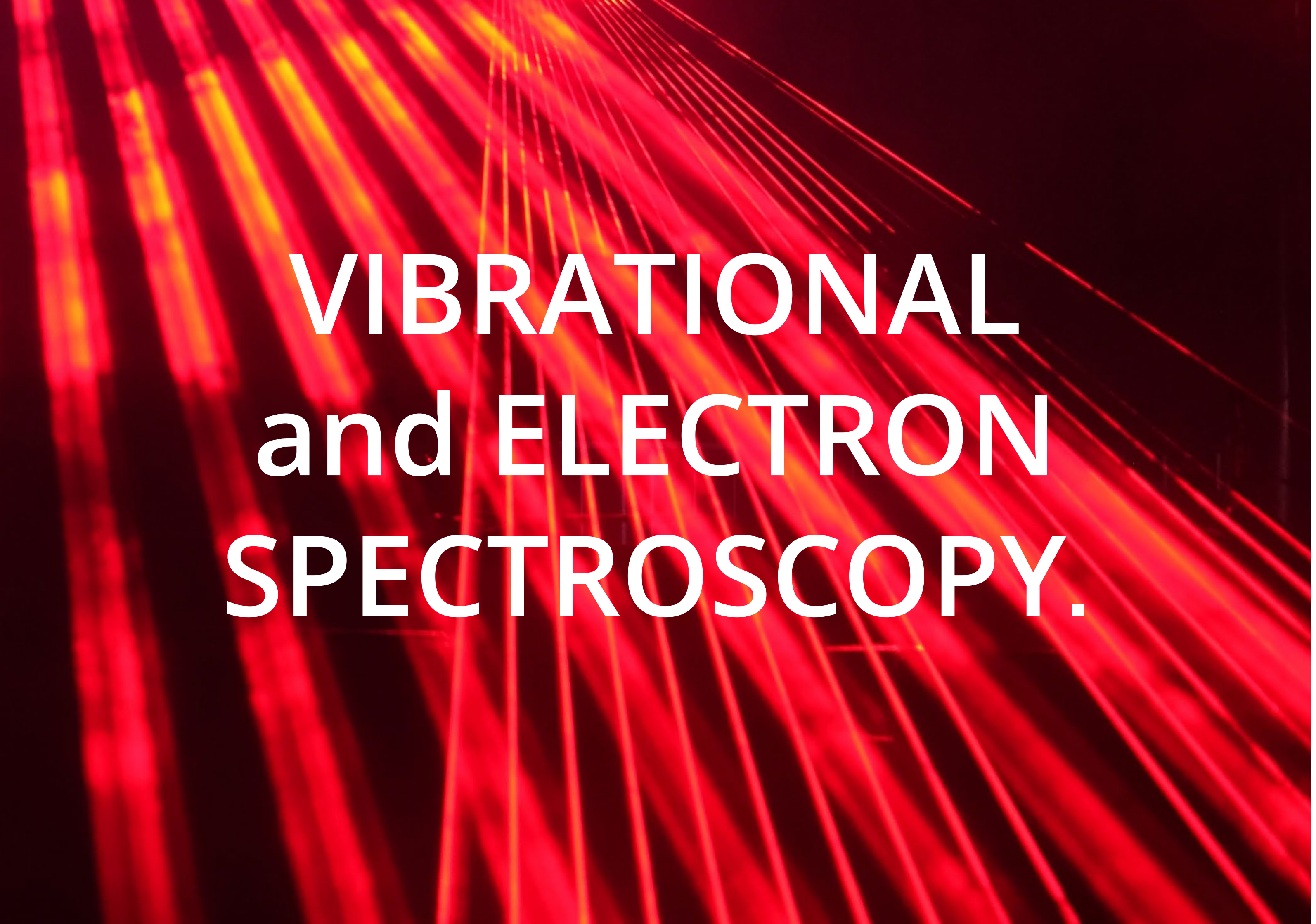


Netzsch DSC (Cold-Stage) 204F1 Pheonix

Analysis Type: DSC
Furnace: Platinum-Rhodium
Temperature Range: -160°C up to 700°C
Thermocouple: K-type
Gases: Instrument Air, Nitrogen and Argon. Helium may also be possible on consultation with AMCF staff.
Crucibles: Aluminium.
Advantages: This stand alone DSC offers the most sensitivity for DSC measurements, handy for analysis of small changes in sample properties which other instruments may not pick up. It is also coupled to a high pressure liquid nitrogen dewer, allowing the user to analyse their samples under negative temperature conditions approaching liquid nitrogen temperatures.

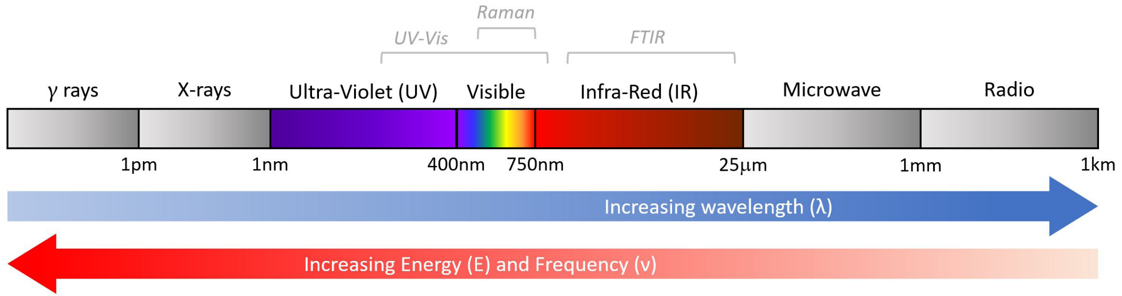
Netzsch TMA 402F1/F2 Hyperion

Analysis Type: TMA with multiple setups including those for *Expansion, Penetration, Tension* and *3-Point Bending*.
Furnace: Choice of Steel or Silicon Carbide.
Temperature Range: -140°C up to 1000°C (Steel furnace) or room temperature up to 1600°C (SiC furnace).
Thermocouple: S-type or K-type (depending on setup).
Sample Holder/rod materials: Quartz for low temperatures and Alumina for high temperatures.
Maximum Force: 3N
Gases: Instrument Air, Nitrogen and Argon.
Advantages: The vertical design of the TMA's furnace with motorized hoist allows for many different sample geometries (rods, squares, plates, films, fibers, powders, liquids). Static and/or dynamic forces can be exerted allowing for the setup of custom experiments as needed.



VIBRATIONAL and ELECTRON SPECTROSCOPY.

Vibrational (FTIR/Raman) and Electron (UV-Vis) Spectroscopy techniques are non-destructive, and are used for determining the chemical bonds, functional groups and components present in a material. It does this by measuring the interaction of a particular electromagnetic radiation source (as shown below) with a sample (e.g. absorption, scattering). This can therefore be used to identify the type of bonds present in the sample.



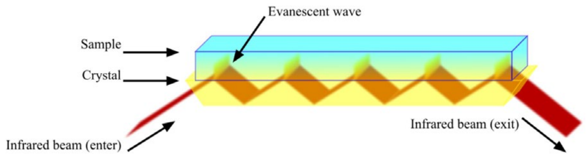
A comparison of vibrationals and electron spectroscopy techniques available at the AMCF.

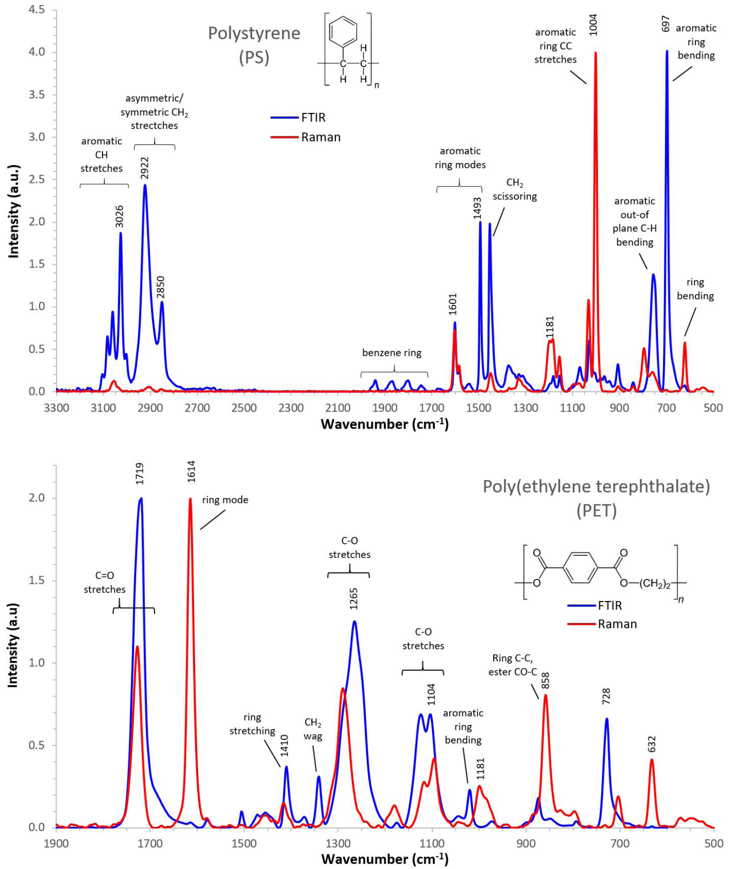
	FTIR	Raman	UV-Visible
Electromagnetic radiation source:	Polychromatic Infrared.	Monochromatic Visible.	Polychromatic Visible/Ultraviolet.
Source wavelengths used:	2500-25000nm	532, 633, 785 and 1064nm	190-900nm
What is Detected:	*Absorption* of radiation at certain wavelengths as molecules vibrate.	Changes in wavelength of *scattered* radiation as molecules vibrate.	*Absorption* of radiation at certain wavelengths as electrons are excited.
What causes a signal:	*Asymmetrical vibration of molecules* (bending, stretching, rocking), which cause changes in a molecule's dipole moment.	*Symmetrical vibration of molecules* (stretching), which cause changes in a molecule's polarizability.	*Electron transitions* as electrons in an atom/molecule are excited to a higher energy level.

FTIR

The most common method for collecting an FTIR spectrum from a sample at the AMCF, is through the use of an Attenuated Total Reflection (ATR) stage. An ATR stage uses a crystal with a high refractive index (e.g. diamond or germanium) on which the sample (solid or liquid) is placed. As an infrared beam passes through the crystal, it is reflected internally many times, in turn creating an evanescent wave that extends beyond the crystal and into the sample placed on top.

The sample may then absorb some energy as molecular bonds vibrate and bend. This energy absorption thereby attenuates (i.e. changes) the infrared beam as it returns to the detector. This signal is then used to create an FTIR spectrum, where we can see at what wavelengths/ wavenumbers energy was absorbed.



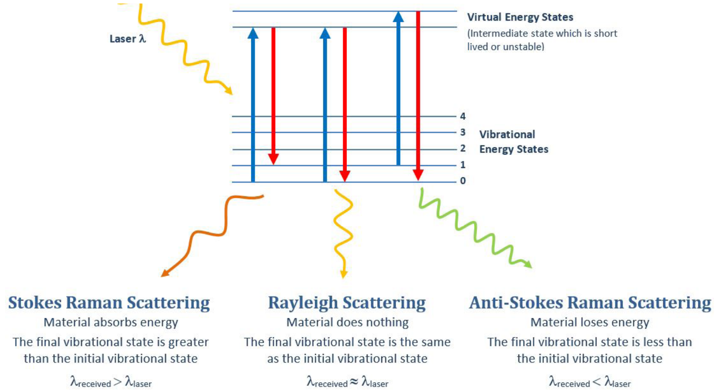


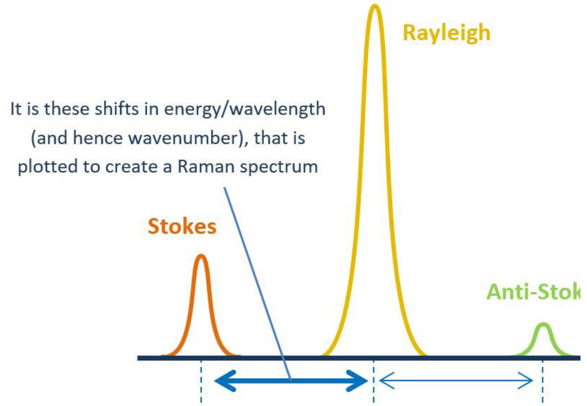
Some comparisons of spectra collected from either FTIR (blue) or Raman (red) spectroscopy for two different polymer samples (top = polystyrene, bottom = poly(ethylene terephthalate)). Assignment of some peaks are shown

Raman Spectroscopy

Raman Spectroscopy relies on the detection of inelastic scattering of monchromatic light. In other words, we are hitting a sample with a specific wavelength of light using a laser, and detecting changes in wavelength of the light that is scattered after hitting the sample. Most light scattered will have the same wavelength as the incident light (rayleigh scattering), but a small amount of light will have a longer wavelength (stokes raman scattering) as it has lost energy when interacting with molecules, causing them to vibrate.

As FTIR and Raman are both analysing molecule vibrations and the energy absorbed, the two techniques are comparable and complimentary. Which technique you should use will depend on the compound you are analysing, and what you'd like to find out. For example, FTIR will give information on bonds with strong dipole moments (asymmetrical vibrations), while Raman will give information on bonds with weak dipole moments (symmetrical vibrations). Some bonds may be seen by both techniques, and some by neither.





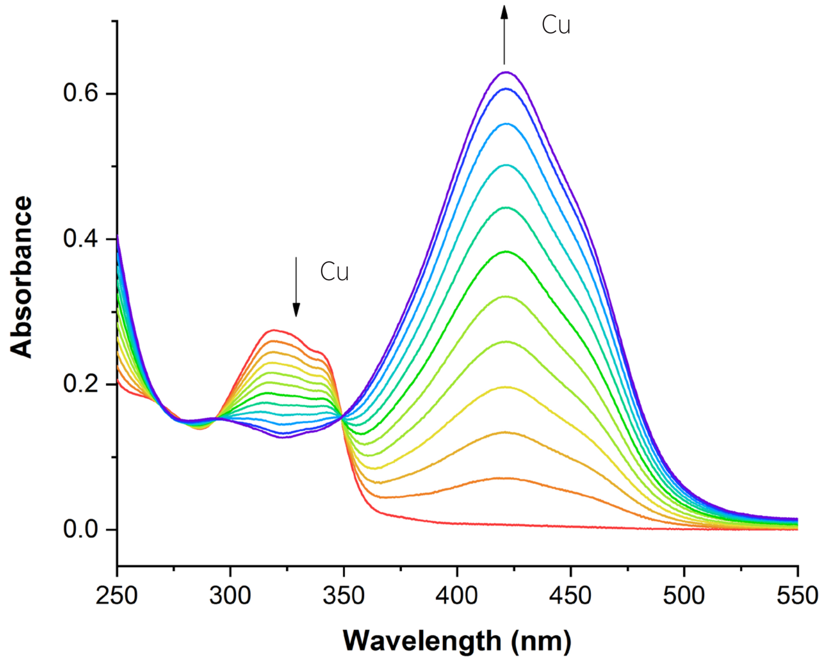
It is these shifts in energy/wavelength (and hence wavenumber), that is plotted to create a Raman spectrum

Signals for each type of scattering are the same distance apart on an energy scale, as quantum amounts of energy are lost or gained.

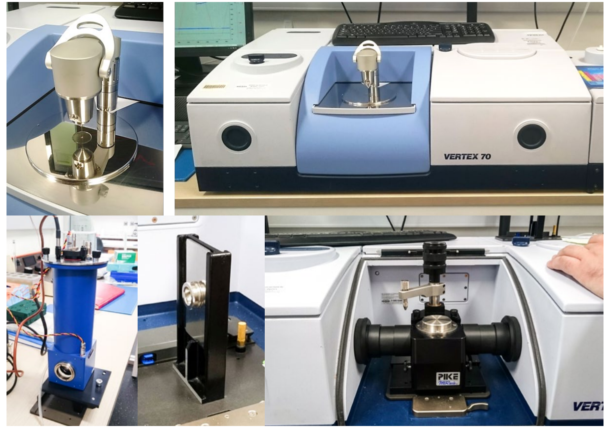
UV-Visible Spectroscopy

When electromagnetic radiation in the form of visible and ultraviolet light (UV-Vis) is absorbed by an atom/molecule, this absorbed energy can result in an electron transitioning from a lower to a higher energy level. This may only happen at specific wavelengths, so a spectrum showing at what wavelength energy is absorbed can be created.

As UV-Vis spectroscopy involves electronic transitions, it is particularly of use for analysing conjugated systems and compounds with transition metal complexes. It is also useful for monitring colour changes in samples, or concentrations in solution samples.

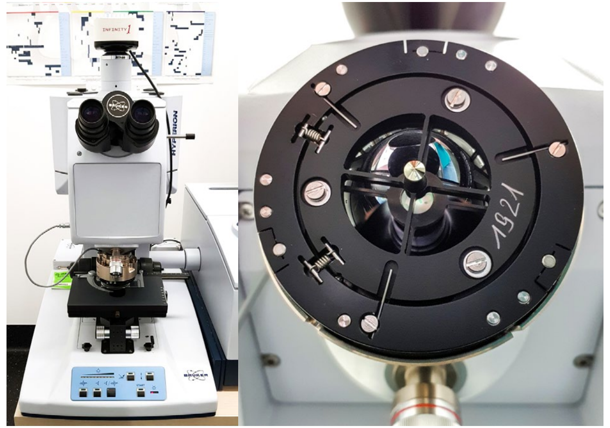


A UV-Vis spectrum showing an organic chemosensor ligand (red line), with increasing amounts of copper titrated. As more copper is added, we see more absorbance at 425nm, and less absorbance at 325nm. The solution is therefore changing colour.



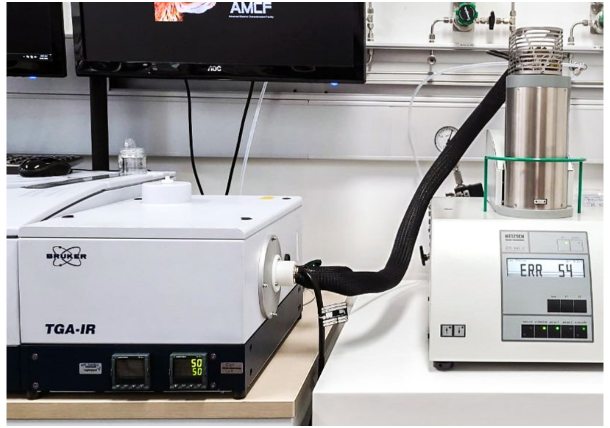
2 x Bruker Vertex 70 FTIR's

Infrared Source: Mid infrared (MIR).
Spectral Range: Best results for 600-4000cm^{-1} range, but 400-6000cm^{-1} is possible.
Detector: Room temperature DigiTect MCT-detector.
Beamsplitters: KBr and CaF (for Raman module) options.
Stages: Diamond ATR, Germanium ATR, Variable temperature stage and a KBr pellet holder for absorption/transmission.
Advantages: The AMCF houses 2 of these FTIRs, as they can be configured for different modes/stages and for the attachment of different modules for more specific experiments. The ATR stages allow for quick and easy analysis of solids and liquids. These instruments are supported by OPUS software package, along with various libraries for spectrum matching.



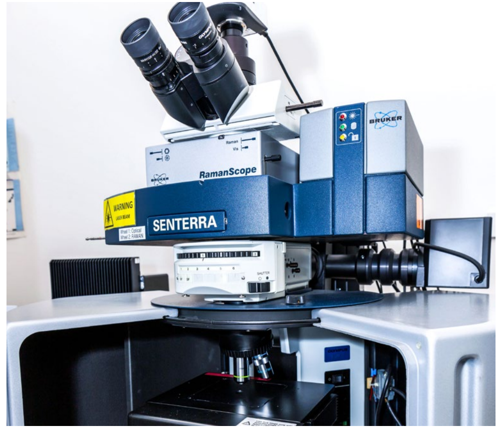
Bruker Hyperion 1000 FTIR Microscope

Infrared Source: Connects and makes use of the Bruker Vertex 70 Mid infrared (MIR) source.
Spectral Range: Best results for 600-4000cm^{-1} range, but 400-6000cm^{-1} is possible.
Detector: LN$_2$ cooled DigiTect MCT-detector.
Beamsplitter: KBr
Attachments: a 4x objective lens, 100µm diameter Ge ATR indenter with internal pressure sensor (can also be used as a 20x objective), 15x transmission/reflectance FTIR objective.
Advantages: The optical microscope can be used for locating small areas of interest to be analysed for FTIR. Bright and dark field illumination with binocular and video viewing. Supported by OPUS software package, along with various libraries for spectrum matching.



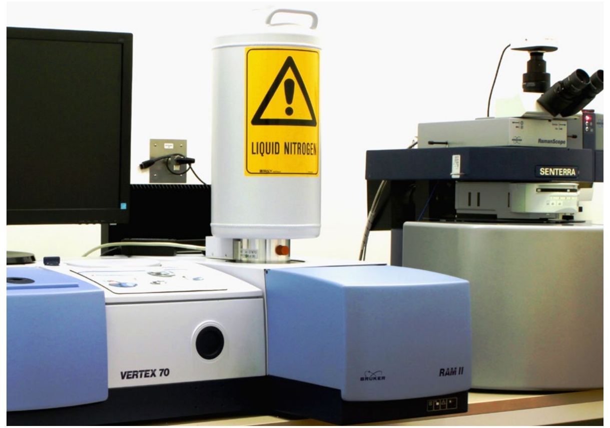
Bruker TGA-IR Evolved Gas FTIR Module

Infrared Source: Connects and makes use of the Bruker Vertex 70 Mid infrared (MIR) source.
Spectral Range: Best results for 600-4000cm^{-1} range, but 400-4000cm^{-1} is possible.
Detector: LN$_2$ cooled DigiTect MCT-detector.
Advantages: This module connects via a transfer tube to a Netzsch TGA/DSC thermal analyser. As samples are heated, any evolved gases are sent to the TGA-IR module's FTIR gas cell for anaysis. This is supported by both Netzsch's Proteus and Bruker's OPUS software working in tandem. By combing these techniques it is possible to find at what temperature particular gases have evovled from the sample as it heats or thermally decomposes. Specific FTIR spectra from the 3D spectrum can be extracted for library matching to identify gases.



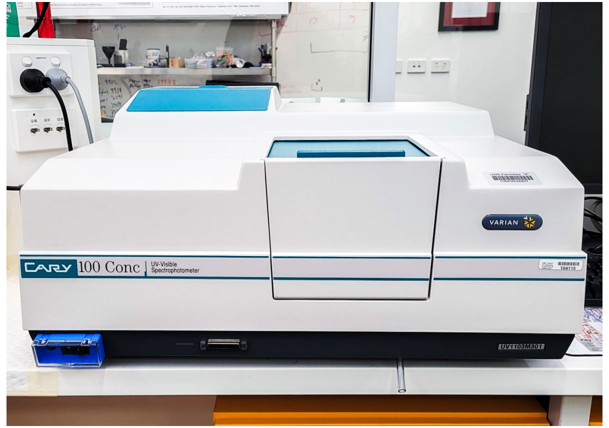
Bruker Senterra Confocal Raman Microscope

Laser Sources: 785nm, 633nm and 532nm. The RAM II 1064nm laser can also be coupled to the spectrometer.
Spectral Range: Best results for 600-4000cm^{-1} range, but 100-6000cm^{-1} is possible.
Optical Objectives: x20, x40 and x50
Advantages: This Fourier Transform (FT) dispersive Raman spectrometer, it is quick and easy to use, particulary when needing to change between lasers to find the best parameters for your sample. With both binocular and video viewing, the optical microscope can be used for locating small areas of interest for analysis. It also features an automatic x-y axis stage, allowing the user to load series of points on a sample for automatic analysis using the supporting OPUS software.



Bruker RAM II Module

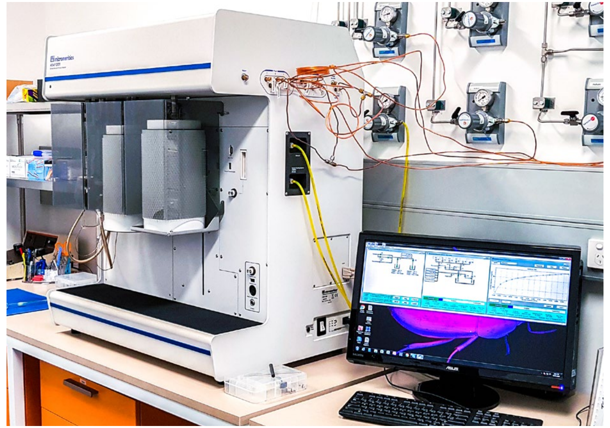
Laser Source: 1064nm laser. Connects to the Bruker Vertex 70 for operation. The laser source can also be fed into the Raman Senterra microscope via a fibre optics cable.
Spectral Range: Best results for 600-4000cm^{-1} range, but 100-6000cm^{-1} is possible.
Detector: LN$_2$ cooled high sensitivity Ge diode detector.
Beamsplitter: CaF
Advantages: For tricky samples, the 1064nm wavelength laser offers better fluorescence suppression. The RAM II has a large sample compartment. This instrument is supported by an OPUS software package, along with various libraries for spectrum matching.



Cary UltraViolet-Visible Spectrometer

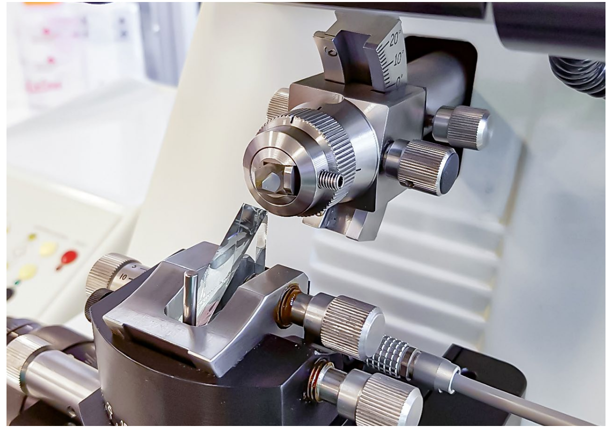
Source: Tungsten-halogen bulb visible source with quartz window, deuterium arc ultraviolet source
Spectral Range: 190 - 900nm
Detector: R928 PMT
Accesories: This instrument has several accessories allowing for measurements on a range of sample types, including; a cuvette holder for solutions, a transparent solid sample holder ideal for glass, films, and other transparent solid samples, a Labsphere DRA-CA-30I diffuse reflectance accessory for non-transparent solid samples, such as powders and other materials.
Advantages: Double beam design which can be operated in single, double, or duel-single beam modes.

OTHER INSTRUMENTATION and SUPPORTING EQUIPMENT.

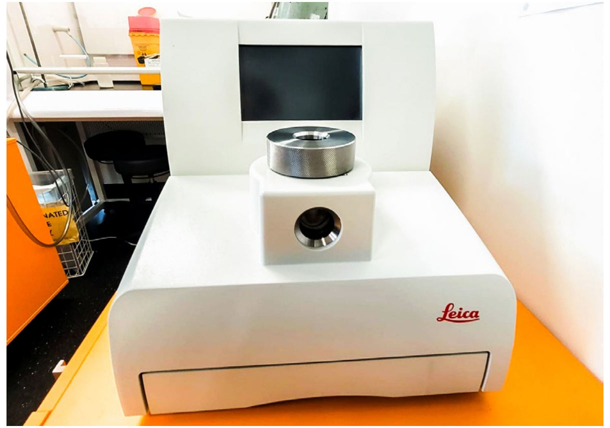


Micrometetics ASAP 2020 Analyser
(Accelerated Surface Area and Pore sizing)

Optical Microscopes and imaging software

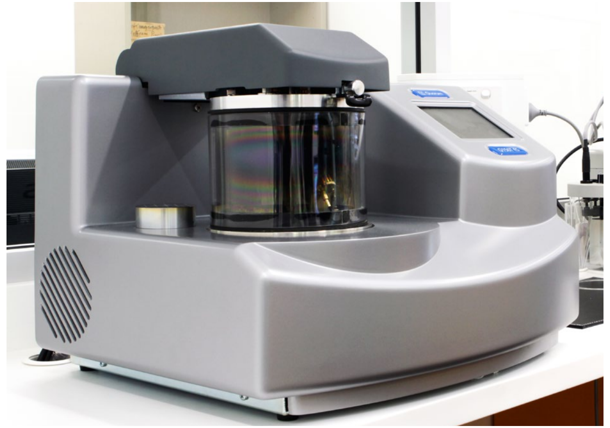


RMC PowerTomeX Ultra-microtome



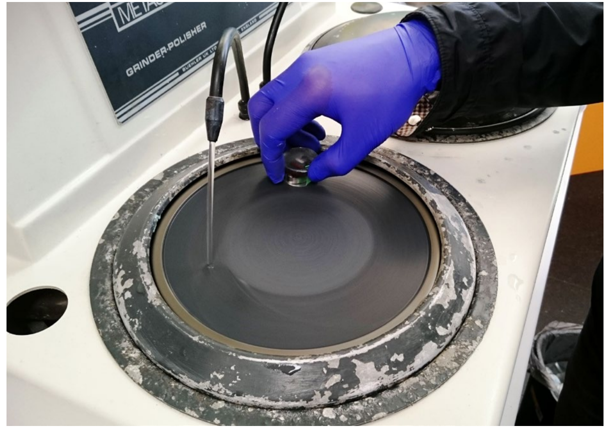
Leica EM300 Critical Point Dryer


Leica EM SCD005 Cool Sputtering Device
(Gold and Carbon coating)


Quorum Technologies Q150T ES Coater
(Chromium coating)


Leica Microtome

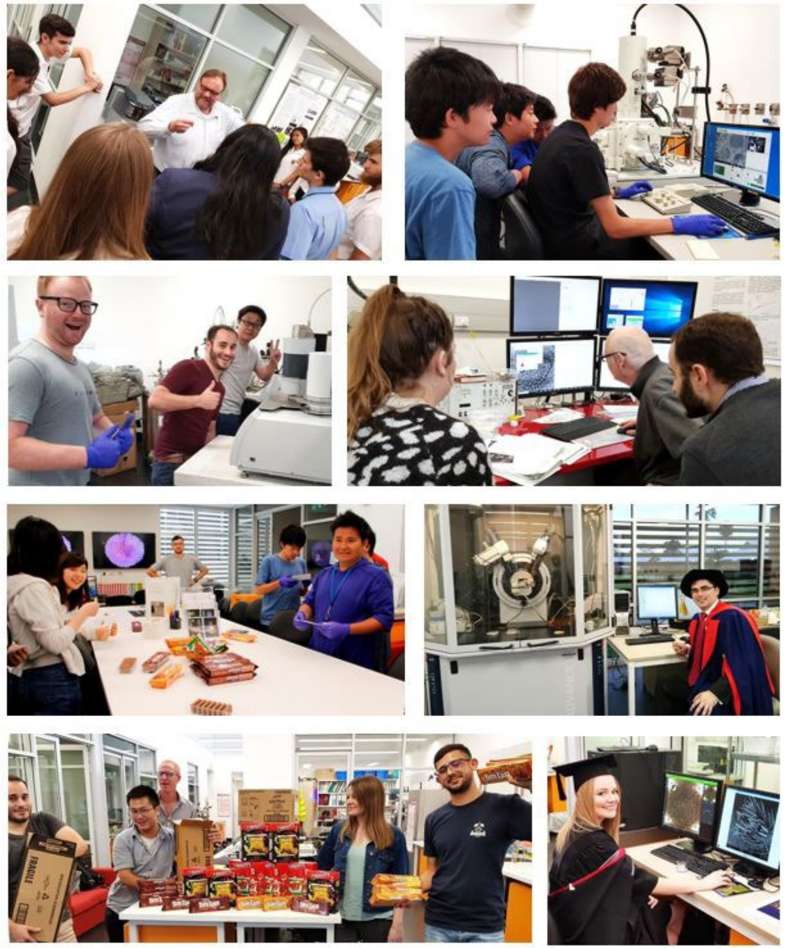

Plasma Cleaner


Cutting Grinding and Polishing


Resin Setting and Sample Mounting


Vacuum Chambers and Ovens


Dynaflow Fumehood



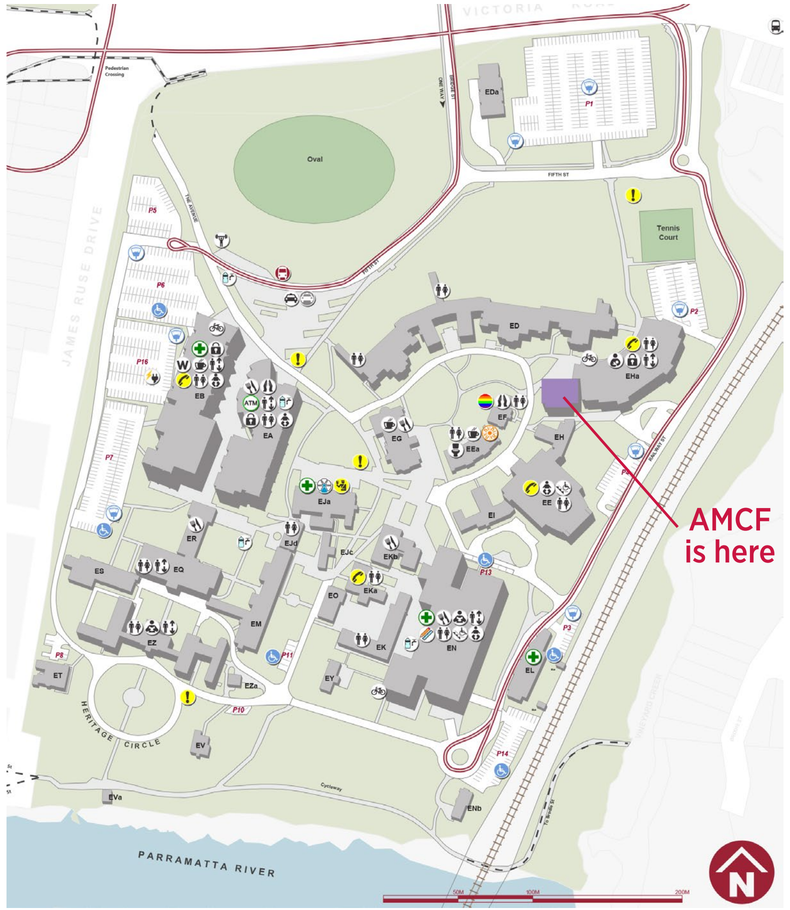
Other Opportunities at the AMCF

The AMCF is a centralised research facility, training and supporting user groups from all schools across the university. We specialise in assisting researchers, students and industry with material analysis and characterisation.

The AMCF:

- Provides training and workshops for users.
- Helps with research, publications and grant applications.
- Gives our students hands on experience, not only with the use of instrumentation, but also on the building, maintenance and servicing of advanced instrumentation.
- Helps develop partnerships and connect our students and researchers with industry.
- Undertakes paid commercial work to analyse unknown materials, help solve production issues and material failure analsyis.
- Works with our instrumentation manufacturers to test new equipment and provide joint workshops.
- Helps to excite school students through open days, youth programs and school visits.

Parramatta South Campus



Our Location

The Advanced Materials Characterisation Facility (AMCF) is located at Western Sydney University's Parramatta South Campus in building EHa.G.49.

Contact us:

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