

Electron Microscopy and X-Rays

A core multi-user facility that features cutting-edge instrumentation, techniques and expertise required for the characterisation of samples in the physical and life sciences.

Clean energy, microelectronics, personalised medicine, and new pharmaceuticals are vital to our future. Achieving breakthroughs in these fields requires a deep understanding of how material properties relate to their physical and chemical structure - especially at the nano- and atomic scale.

The **EMX user facility** at INL is your partner in advanced characterisation services. With cutting-edge instrumentation, we probe the physical and chemical structure of matter down to the atomic scale.

With extensive expertise in advanced characterisation, our international team of microscopists and spectroscopists delivers deep insights into the structure and composition of materials and life sciences samples, while also offering training, technical support, and consultation in electron microscopy and spectroscopy.

Glacios™ Cryo-TEM

Technical highlights

- High-brightness X-FEG electron gun
- 200 kV accelerating voltage
- Cryo-Autoloader for up to 12 autoGrids
- $\pm 70^\circ$ alpha tilt
- Falcon 4i high resolution camera
- CETA-D 16M CMOS camera
- Low dose software (suited for minimised electron dose during cryo-TEM operation in single particle acquisition, tomography and micro-ED experiments)



The Thermo Scientific™ Glacios™ Cryo-Transmission Electron Microscope (Cryo-TEM) is a dedicated 200 kV cryo-microscope, that features a 12-grid Autoloader and a state-of-the-art direct electron detector. The Glacios™ Cryo-TEM is set up for single-particle analysis, cryo-electron tomography (cryo-ET) and micro-electron diffraction (MicroED).

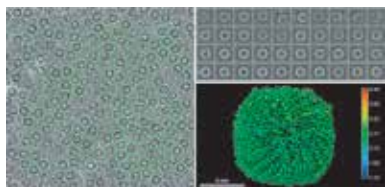
The acquisition of the cryo-TEM was achieved by the National Network of Advanced Electron Microscopy for Health and Life Sciences (CryoEM-PT). It is the first Cryo-Electron Microscope in Portugal. The CryoEM-PT network is organised with a Central Node at INL, where the microscope is installed, and several nodes throughout the country, which are intended to allow for local preparation of samples and data processing.

Tomography



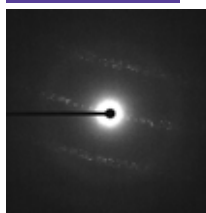
Liposomes
(courtesy of O. Schraidt, INL, and Bluepharma)

Single particle reconstruction



Apoferritin sample and reconstruction $< 1.8\text{\AA}$
(courtesy of O. Schraidt, INL, and D. A. Semchonok, ITQB NOVA)

MicroED



Lysozyme
(courtesy of O. Schraidt, INL, and P. M. Matias, ITQB NOVA)



Double-Corrected TEM/STEM Titan Themis

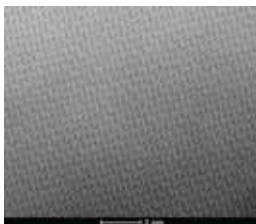
A probe and image-corrected (scanning) transmission electron microscope optimised for elemental analysis and high-resolution TEM/STEM imaging. The microscope is equipped with a monochromator, four energy-dispersive X-ray spectroscopy detectors and a GIF for electron energy loss spectroscopy (Dual EELS).

It also has a bi-prism for electron holography. An *in-situ* sample holder allows heating and biasing experiments.

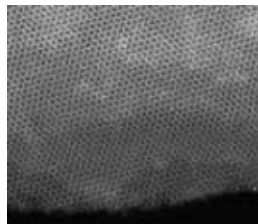
Capabilities

- Voltage: 60 – 300 kV (X-FEG)
- Monochromator
- Corrected TEM imaging (resolution 63 pm)
- Corrected STEM imaging (resolution 63 pm)
- Diffraction (crystallographic analysis)
- EDX – Super X (chemical analysis)
- Dual EELS (energy resolution 190 meV)
- Electron holography
- Lorentz microscopy
- *In-situ* sample holder (heating/biasing)
- Differential phase contrast (DPC) imaging

STEM-HAADF

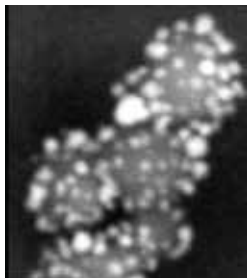


ZnFeO
(courtesy of E. Carbo-Argibay, INL)



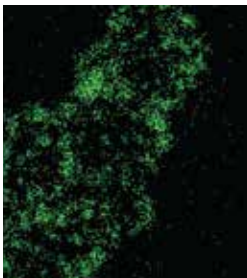
MoS₂
(courtesy of E. Carbo-Argibay, INL)

STEM-HAADF

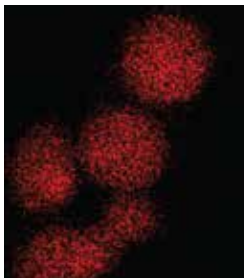


Fe₃O₄@Au nanoparticles
(courtesy of E. Carbo-Argibay, INL)

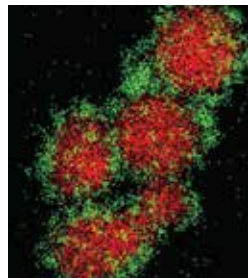
STEM-EDX



Au



Fe



Au, Fe

Probe-corrected STEM Titan ChemiSTEM

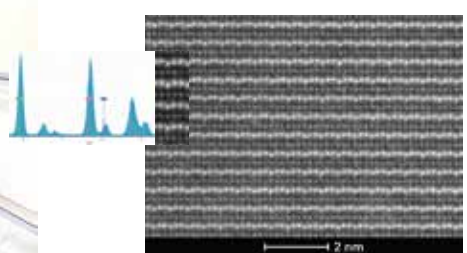
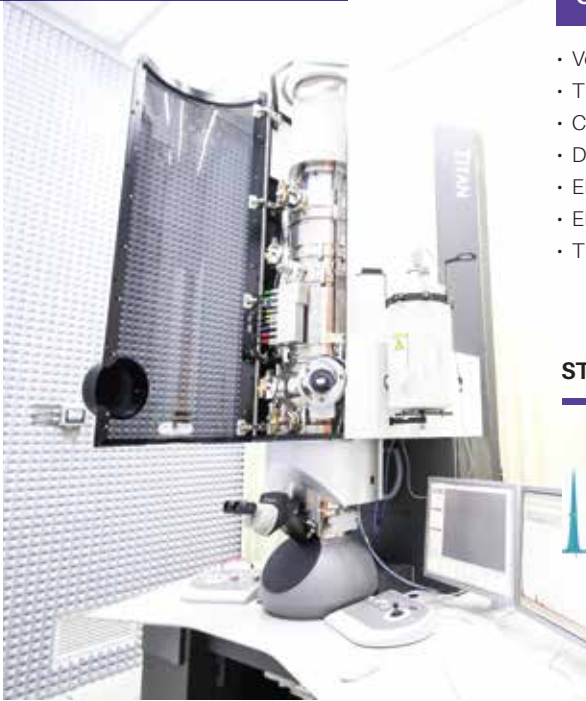
Titan ChemiSTEM is a probe-corrected (scanning) transmission electron microscope optimised for elemental analysis and high-resolution imaging.

It is equipped with four energy-dispersive X-ray spectroscopy detectors and a GIF for electron energy loss spectroscopy and energy-filtered TEM. The microscope also has a wide pole piece gap and can tilt to ± 70 degrees, making it suitable for tomography.

Capabilities

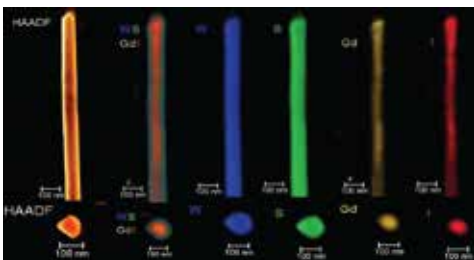
- Voltage: 80 – 200 kV (X-FEG)
- TEM imaging
- Corrected STEM imaging (resolution 80 pm)
- Diffraction (crystallographic analysis)
- EDX – super X (chemical analysis)
- EELS (energy resolution 1 eV)
- TEM/STEM tomography (3D reconstruction)

STEM image coupled with EDX mapping



LuMnO₃
(Bakhizadeh A. et al, Mater. Charact. 2018)

3D Tomography coupled with EDX mapping



Core-shell nanotubes
(Deepak F.L. et al, Nanoscale 2016)

High resolution HAADF-STEM

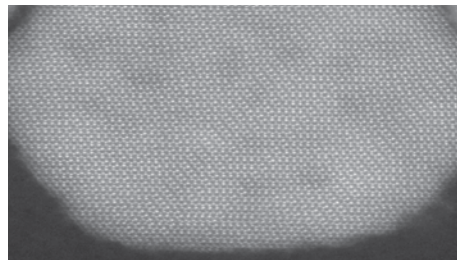


Image of a Cu(In,Ga)Se₂ nanoplate
(courtesy of A. LaGrow and Y. Kolen'ko, INL)

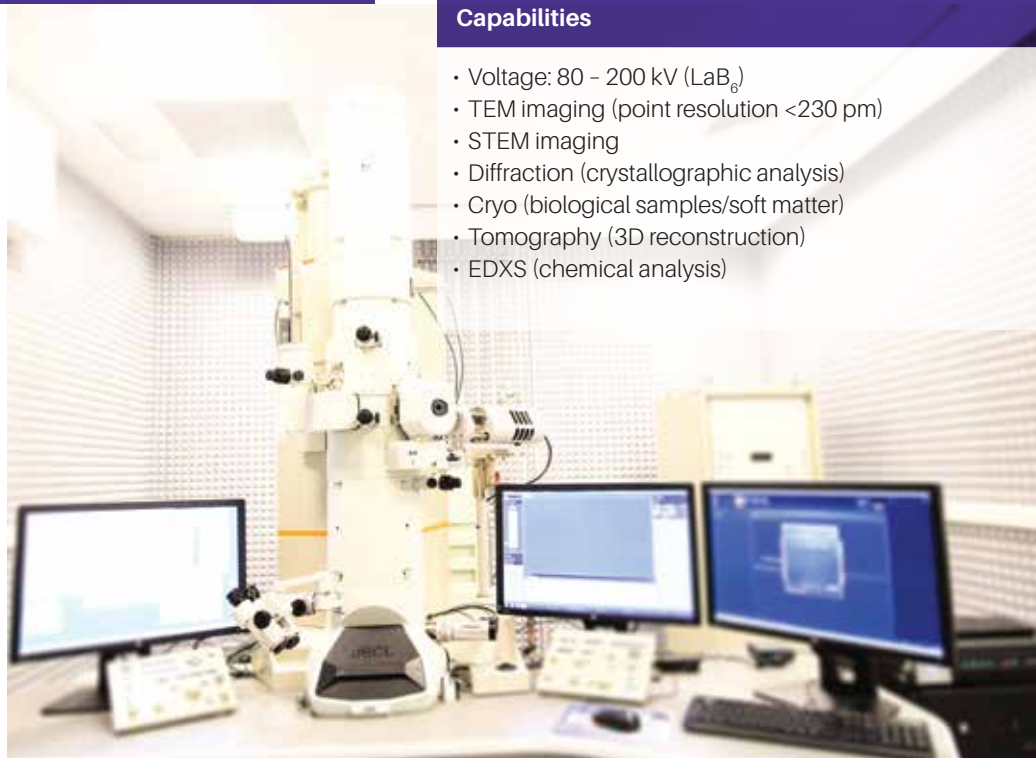
TEM JEM 2100

A multipurpose electron microscope that is well suited to characterise materials and biological samples. A highlight of the JEM 2100 is its fast-readout "OneView" 4k x 4k CCD camera that operates at 25 fps (300 fps with 512 x 512 pixels).

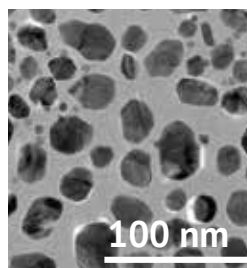
SerialEM is installed on the machine and is used for low-dose imaging, tomogram acquisition and semi-automation.

Capabilities

- Voltage: 80 – 200 kV (LaB₆)
- TEM imaging (point resolution <230 pm)
- STEM imaging
- Diffraction (crystallographic analysis)
- Cryo (biological samples/soft matter)
- Tomography (3D reconstruction)
- EDXS (chemical analysis)

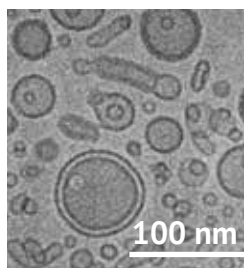


Morphological Analysis



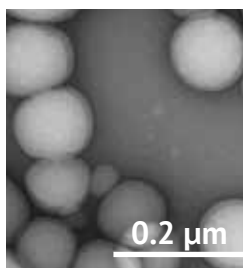
Silver nanoparticles
(S. Sampaio et al, 2018 Adv. Nat. Sci. Nanosci. Nanotechnol.)

Cryo TEM



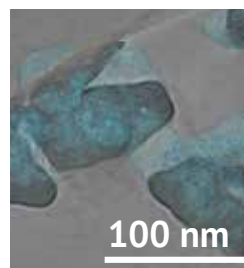
Vitrified Cationic Liposome - DNA lipoplexes
(courtesy of B. Silva & D. Stroppa, INL)

Negative stain Imaging



Vesicles imaged by negative stain TEM
(courtesy of O. Schraidt, INL)

3D Tomography



AuAg NPs, visualization of dual-tilt tomography reconstruction
(courtesy of O. Schraidt, INL)

SEM/ESEM Quanta 650

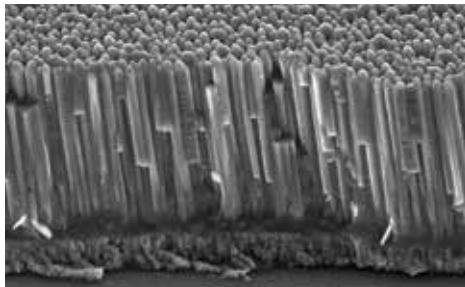
A highly versatile scanning electron microscope that can be used under high, low, and extended vacuum (ESEM) conditions allows the observation of uncoated samples in their original state.

It operates with a Schottky field emission gun that provides high beam intensity and stability. The electron acceleration voltage ranges from 1 - 30 kV.

Capabilities

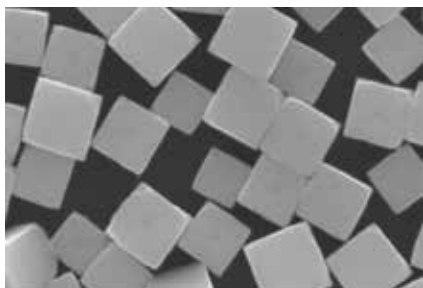
- Voltage: 1 - 30 kV
- SEM Imaging (resolution 1 nm)
- Low vacuum/"environmental" SEM imaging
- (Biological samples/soft matter)
- EDXS (chemical analysis)
- Cooling/heating stage (in-situ) » -20 °C - 1500 °C
- SE/BSE detectors (topographical/structural analysis)

Cross-sectional secondary electron imaging



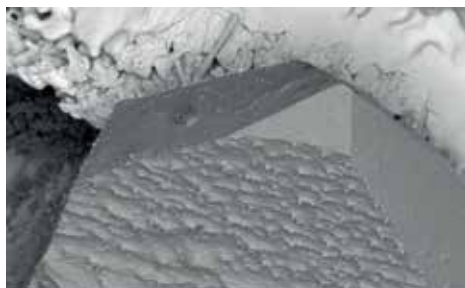
Magnetron sputtered TiAlSiN thin film
(courtesy of S. Calderón, INL)

Secondary electron imaging



Cu₂O nanoparticles
(courtesy of Y. Kolen'ko, INL)

Backscattered electron imaging



Used LaB₆ filament
(courtesy of O. Schraidt, INL)

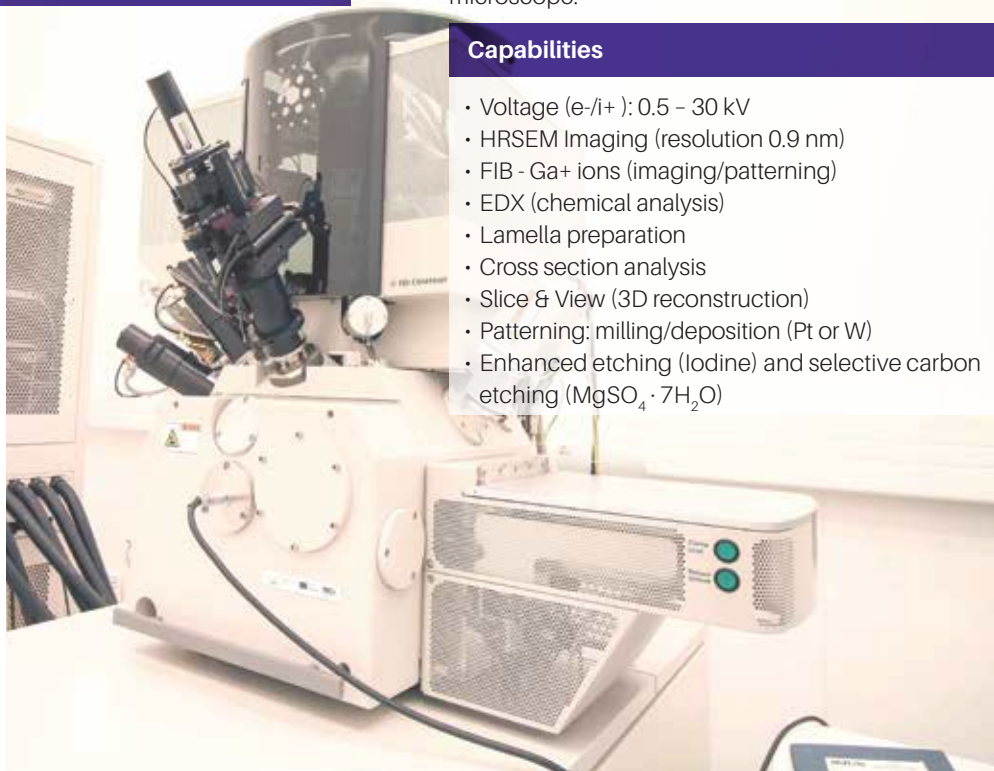
Dual Beam FIB-SEM Helios 450S

A highly versatile tool that combines SEM with a focused ion beam (FIB) of gallium ions. The SEM operates with a field emission gun (FEG) that provides high beam intensity and stability. Imaging can be done with secondary (for SEM and FIB) and backscattered (for SEM) electrons.

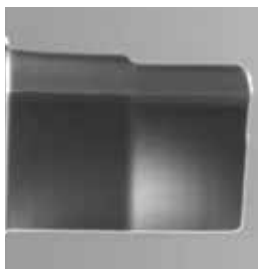
A STEM detector delivers a resolution of 0.8 nm at 30 kV. For element analysis and mapping, an EDX detector is available. An ultra-high resolution (UHR) stage and flip stages are available inside the microscope.

Capabilities

- Voltage (e-/i+): 0.5 – 30 kV
- HRSEM Imaging (resolution 0.9 nm)
- FIB - Ga⁺ ions (imaging/patterning)
- EDX (chemical analysis)
- Lamella preparation
- Cross section analysis
- Slice & View (3D reconstruction)
- Patterning: milling/deposition (Pt or W)
- Enhanced etching (Iodine) and selective carbon etching ($\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$)

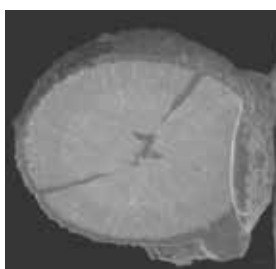


Lamella preparation for TEM



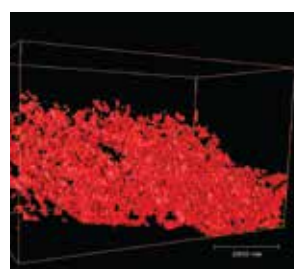
CIGS-Copper Indium Gallium Selenide solar cell
(courtesy of E. Carbo-Argibay, INL)

Cross-section imaging



$\text{LiNi}_x\text{Mn}_y\text{Co}_z\text{O}_2$ electrode material
(courtesy of C. Alves, INL)

Slice and view - 3D reconstruction



$\text{Pr}_2\text{O}_3\text{SO}_4$ -LSCF cathode
(F.J. A. Loureiro et al, J. Mater. Chem. A 2015)

XPS/UPS/AES/ISS /REELS ESCALAB 250Xi

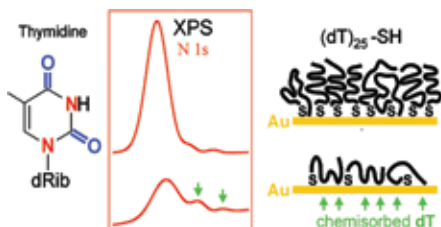
An ultra-high vacuum (UHV) system to analyse the chemical composition of samples by means of X-ray Photoelectron Spectroscopy (XPS). It has a depth resolution of 1-10 nm via depth profiling and lateral resolution down to $\sim 1 \mu\text{m}$.

The complementary techniques that are also available are reflection electron energy loss spectroscopy (REELS), ion scattering spectroscopy (ISS) and ultraviolet photoelectron spectroscopy (UPS).

Capabilities

- Hemispherical energy analyser ($0 \pm 5000 \text{ eV}$)
- Detection system (6 channel electron multipliers and 2D microchannel plates)
- X-ray sources (monochromatic Al $K\alpha$ and twin anode Mg $K\alpha$ /Al $K\alpha$)
- Sample navigation and manipulation (automated samples manipulator and azimuthal stage rotation)
- Sample temperature control in the range 170 -1000 K
- Glovebox integrated to the vacuum system to handle air sensitive samples
- UV Source
- Flood guns (electron and ion) for sample charge compensation, electron gun for REELS
- Monoatomic and gas cluster ion source for depth profiling "soft" (cluster mode) and solid (monoatomic mode) materials
- RGA with mass range 1-100 amu
- Techniques: XPS, UPS, REELS, ISS

Elemental analysis



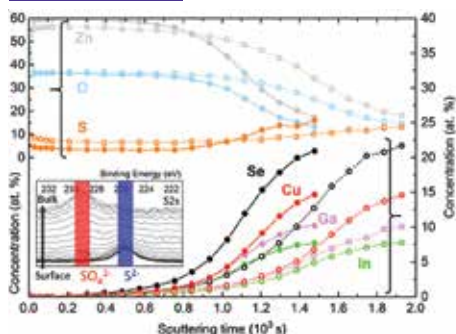
Immobilised DNA
(Petrovykh et al, JACS 2003)



XPS mapping

Bi on Ni foam
(courtesy of O. Bondarchuk, INL)

Depth profiling



X-Ray Diffraction System - X'Pert Pro MRD

The XRD X'Pert PRO Panalytical, a diffractometer used for characterisation to provide sample structural information using various methods such as RSP (Reciprocal Space Mapping), XRR (X Ray Reflectivity), and GIXRD (Grating Incident X Ray Diffraction).

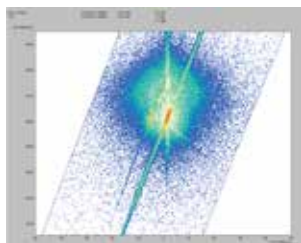
The system includes a six axes goniometer, a Cu X-ray tube, and all the accessories for different configurations of the incident and diffracted beams, such as monochromator, mirror, parallel plat collimator, triple-crystal/rocking curve prefix and crossed slits.



Capabilities

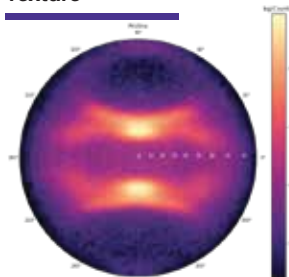
- Crystallographic Orientation
- Texture
- Stress
- Film Thickness
- Composition
- Mismatch
- Structure Parameters
- Density

Reciprocal space maps



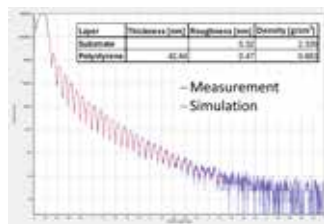
FeO (110) on top of MgO (100)
(courtesy of M. Bañobre, INL)

Texture



Pole figure around (111) peak of a
pristine 25 μm thin copper foil.
(courtesy of T. Pereira, INL)

X-Ray reflectometry (XRR)



Polystyrene film 40nm thick on glass
(courtesy of E. Paz, INL)

Small Angle X-Ray Scattering - SAXSess mc²

Small-angle X-ray scattering is a well-established method for nanostructure analysis and contributes to a deeper understanding of the relationship between a materials structure and physical, chemical and biological properties. It is used to investigate structures from 1 nm to 80 nm present in many different kinds of liquid (e.g. colloids, proteins, surfactants) nanoparticles and solid (e.g. polymers, fibres, nanocomposites) samples.

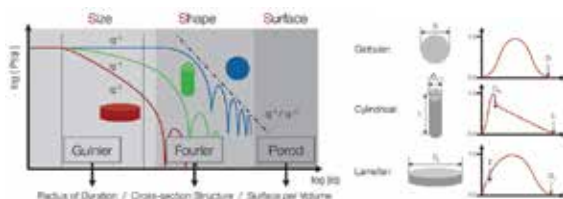
The SAXSess mc² system allows time- and temperature-dependent experiments to be performed fully automatically. Different types of samples liquids, particles and solids as well as grazing-incidence scattering (GI-SAXS) studies of nanostructured surfaces easily possible. The powerful SAXSquant software controls all system devices and effortlessly processes large scattering data sets using customisable templates.



Capabilities

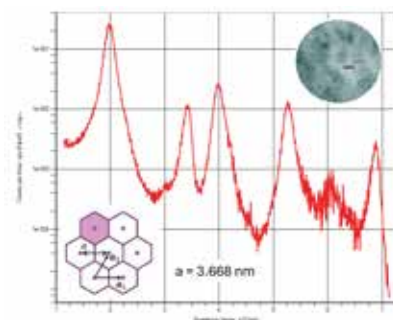
- Nanostructure Analysis (shape, size)
- Specific Surface (porosity)
- Crystallinity and Orientation Effects
- Aggregation Studies
- Dispersion Stability Testing
- Investigation of Particle Growth Processes
- GI-SAXS Studies of Nanostructured Surfaces

Shape and Size



Theoretical spectra and PDDF of particles with different shape.
(The SAXS Guide – Getting acquainted with the principles, by Heimo Schnablegger and Yashveer Singh, Anton Paar)

Crystallinity



COF – Covalent Organic Frameworks. Hexagonal lattice with a lattice parameter $a = 3.668$ nm (courtesy of J. Araújo, INL)

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& innovation

Your Partner in Characterisation





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