

# Advanced Electron Microscopy Imaging and Spectroscopy

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

Clean energy technologies, microelectronics, personalised medicine and new pharmaceuticals are essential for the future of our society and planet. However, widespread implementation and transformational breakthroughs in such fields are only possible if a fundamental understanding of the relationship between the material properties and its physical and chemical structure is achieved at the nano- and atomic scale.

AEMIS user facility at INL wants to be your partner in providing characterisation services. Featuring cutting-edge instrumentation, we can probe physical and chemical structure of matter down to the atomic scale. With wide experience in advanced characterisation techniques, our international team of microscopists and spectroscopists focuses on getting the maximum insight from the structure and composition of your Materials and Life Sciences samples, as well as providing training, technical support, and consultation in electron microscopy and spectroscopy.



Dr. Enrique Carbó-Argibay  
Head of AEMIS



# Glacios™ Cryo-TEM

## Suggested applications

- Single-particle analysis: protein complexes
- Micro-electron diffraction: nanocrystals of chemical compounds, proteins
- Tomography: bacteria, thin cells, sections

## 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.

## Cryo-TEM sample preparation



Leica Plunge Freezer EM GP2



Thermo Scientific Vitrobot Mark IV System

# Double Corrected TEM/STEM Titan Themis

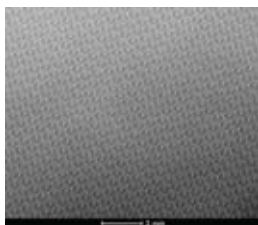
A probe and image-corrected (scanning) transmission electron microscope optimized for elemental analysis and high-resolution TEM/STEM imaging. The microscope is equipped with a monochromator, four energy-dispersive X-ray spectroscopy detectors and a GIF for electron energy loss spectroscopy (Dual EELS).

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

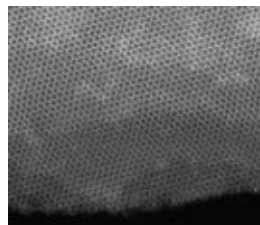
## Capabilities

- 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

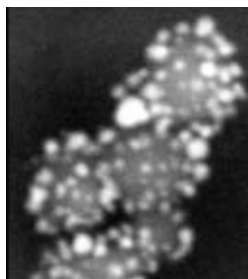


ZnFeO  
(Courtesy of E. Carbo-Argibay, INL)

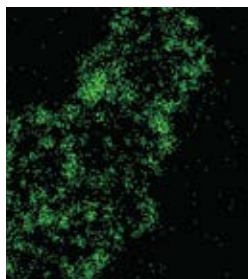


MoS<sub>2</sub>  
(Courtesy of E. Carbo-Argibay, INL)

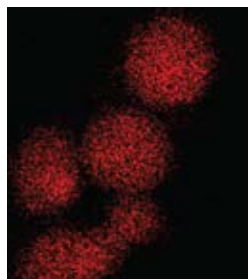
## STEM-EDX



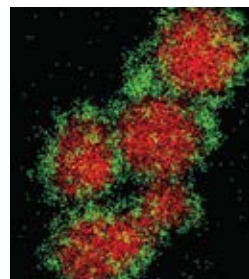
STEM-HAADF



Au



Fe



Au, Fe

# Probe-corrected STEM Titan ChemiSTEM

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

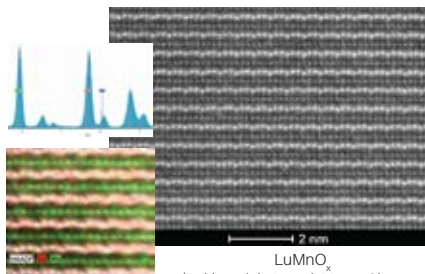
The microscope is equipped with four energy-dispersive X-ray spectroscopy detectors and a GIF for electron energy loss spectroscopy and energy-filtered TEM. The microscope also has a wide pole piece gap and can tilt to  $\pm 70$  degrees, making it suitable for tomography.

## Capabilities

- Voltage: 80 – 200 kv (X-FEG)
- TEM Imaging
- Corrected STEM Imaging (Resolution 80 pm)
- Diffraction (Crystallographic analysis)
- Diffraction (Crystallographic analysis)
- EDX – Super X (Chemical analysis)
- EELS (Energy resolution 1 eV)
- TEM/STEM Tomography (3D Reconstruction)

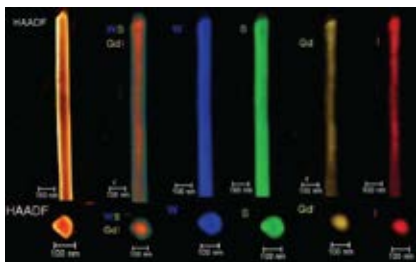


## STEM image coupled with EDX mapping



$\text{LuMnO}_3$   
(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

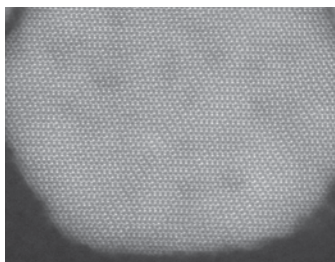


Image of a  $\text{Cu(In,Ga)Se}_2$  nanoplate (Courtesy A. LaGrow and Y. Kolen'ko, INL)

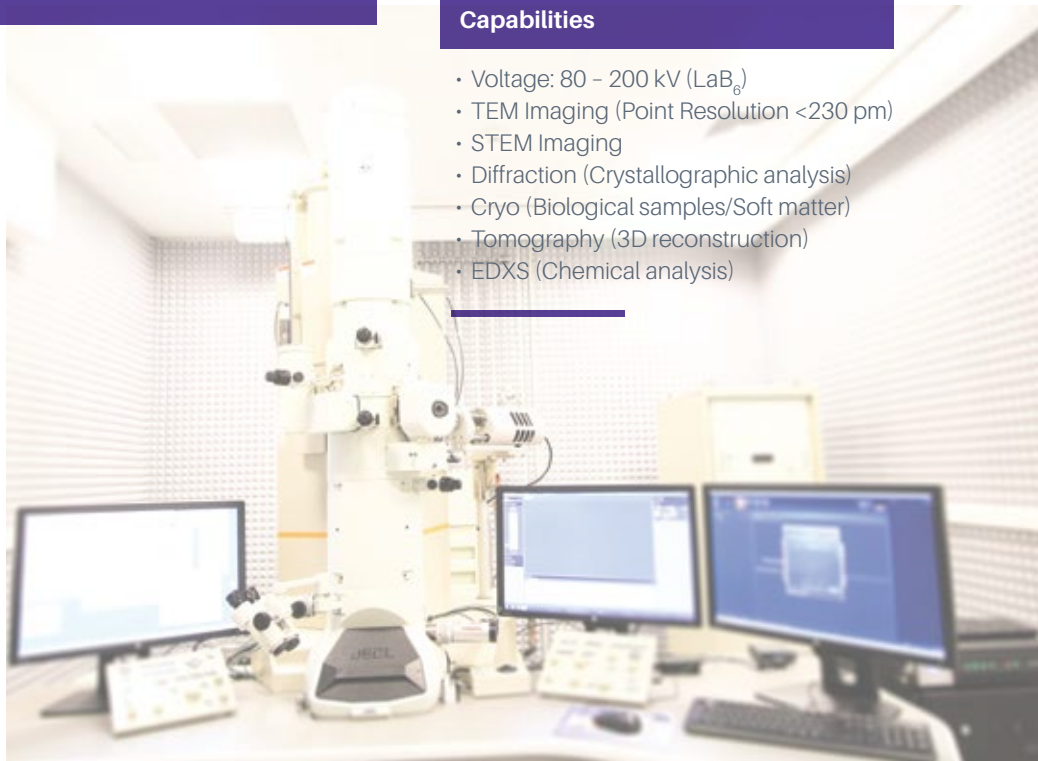
# TEM JEM 2100

A multipurpose electron microscope that is well suited to characterize 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).

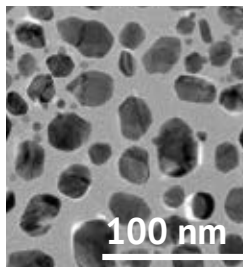
SerialEM is installed on the machine and is used for low-dose imaging, tomogram acquisition and semi-automation.

## Capabilities

- Voltage: 80 - 200 kV (LaB<sub>6</sub>)
- TEM Imaging (Point Resolution <230 pm)
- STEM Imaging
- Diffraction (Crystallographic analysis)
- Cryo (Biological samples/Soft matter)
- Tomography (3D reconstruction)
- EDXS (Chemical analysis)

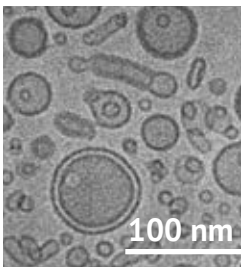


### Bright field Imaging for Morphological Analysis



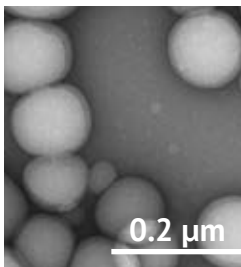
Silver nanoparticles (S. Sampaio et al, 2018 Adv. Nat. Sci: Nanosci. Nanotechnol)

### Cryo TEM



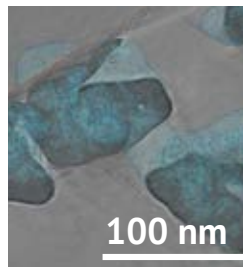
Vitrified Cationic Liposome - DNA lipoplexes (courtesy of B. Silva & D. Stroppa, INL)

### 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

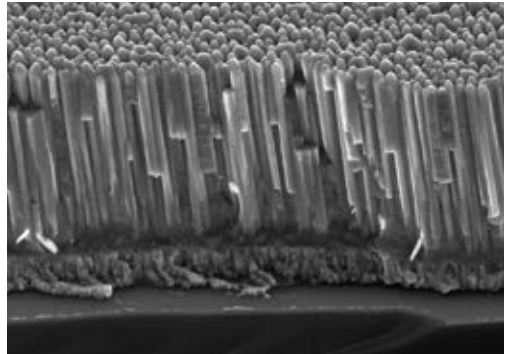
A highly versatile scanning electron microscope that can be used under high, low, and extended vacuum (ESEM) conditions allows the observation of uncoated samples in their original state.

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

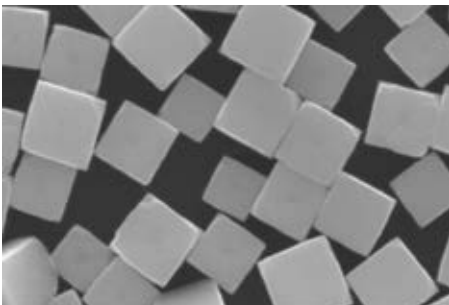
## Capabilities

- 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

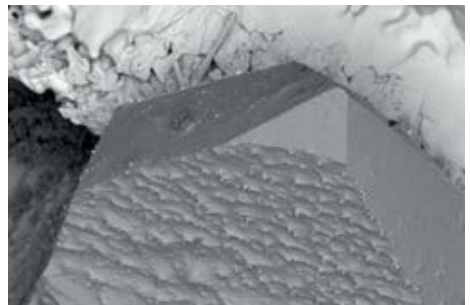


## Secondary electron imaging



Cu<sub>2</sub>O nanoparticles  
(courtesy of Y. Kolen'ko, INL)

## Backscattered electron imaging



Used LaB<sub>6</sub> 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.

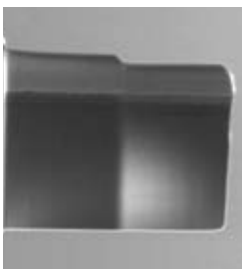
A STEM detector delivers a resolution of 0.8 nm at 30 kV. For element analysis and mapping, an EDX detector is available. An ultra-high resolution (UHR) stage and flip stages are available inside the microscope.

## Capabilities

- Voltage (e-/i+ ): 0.5 – 30 kV
- HRSEM Imaging (Resolution 0.9 nm)
- FIB - Ga<sup>+</sup> 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 (MgSO<sub>4</sub> · 7H<sub>2</sub>O)

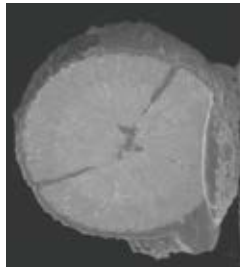


### Lamella preparation for TEM



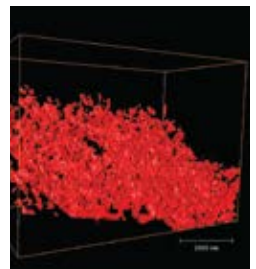
CIGS-Copper Indium Gallium Selenide solar cell  
(courtesy of E. Carbo-Argibay, INL)

### Cross-section imaging



LiNi<sub>1-x-y</sub>Mn<sub>x</sub>Co<sub>y</sub>O<sub>2</sub> electrode material  
(courtesy of C. Alves, INL)

### Slice and view - 3D reconstruction



Pr<sub>2</sub>O<sub>3</sub>/SO<sub>2</sub>-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 analyze 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).

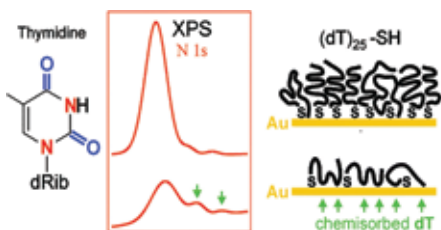


## Capabilities

- Electron Analyser (0 -  $\pm 5000$  eV)
- Detection System (6 channels, 2D position detector)
- X-ray Sources (Monochromatic Al K $\alpha$  and twin anode Mg K $\alpha$ / Al K $\alpha$ )
- Sample Navigation and Manipulation (Automated Sample Manipulator and Azimuthal stage Rotation)
- Heating and Cooling of Specimen
- UV Source
- Flood Sources (Charge compensation and REELS)
- Monoatomic and Gas Cluster Ion Source for depth profiling "soft" (cluster mode) and solid (monoatomic mode) materials

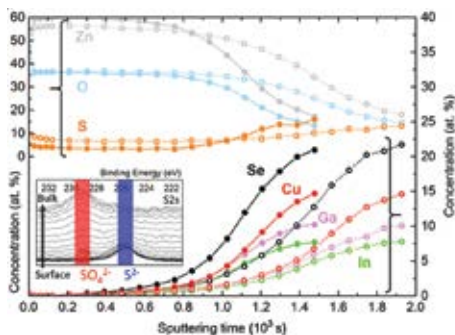


## Elemental analysis



Immobilized DNA (Petrovykh et al, JACS 2003)

## Depth profiling



Zn(S,O)/CIGS (Zutter et al, Phys.Stat.Sol, 2019)



## XPS mapping

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



# OUR TEAM



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+400  
people



+35  
nationalities



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47.000m<sup>2</sup>  
total area



7,500 m<sup>2</sup>  
laboratory space

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