FIB-SEM

The Storage of Electrochemical Energy (SEE) department conducts fundamental and applied research into battery materials and technologies, with the goal of developing safe, high-performance energy storage. The research focuses on various battery chemistries, including Li-ion, Li-metal, Li-air, solid-state (inorganic and polymer), Mg-ion, Na-ion, and aqueous systems. The focus is on understanding structural evolution and ion mobility, developing new materials and interfaces, and linking experimental results to multiscale modeling.

Detailed analysis of microstructures and interfaces is essential for these research objectives. A FIB-SEM (Focused Ion Beam Scanning Electron Microscope) is required to:

• Create 3D reconstructions of electrode materials and analyze porosity and degradation mechanisms.

• Perform local characterization of interfaces, such as the Solid Electrolyte Interphase, which is crucial for longevity and safety. • Investigate structural changes during battery cycling, supporting the development of new battery chemistries.

• Prepare high-precision samples for Transmission Electron Microscopy (TEM) and operando studies.

The acquisition of this instrument is essential for understanding and improving the link between microstructure, ion transport, and battery performance, and directly contributes to achieving SEE's research objectives.

KO-Criteria

Category	***Demands***
Electron column	*Field Emission SEM with Beam booster & Magnetic/Electrostatic lens combination, 0.02-30kV without stage bias. Magnification range: 12 – 2.000.000x based on polaroid format, 0.2–30 kV, resolution* ≤ 1 nm at 15 kV and ≤ 1.9 nm at 1 kV*
FIB column	*Gallium LMIS-source, 0.5–30 kV, resolution* ≤ 3 nm at 30 kV, current 1 pA–100 nA. Ga-source lifetime >= 1500 hr under normal operating conditions.*
Stage	*True mechanical 6-axis motorized stage, X/Y >=100 mm, Z >= 50 mm, pure mechanical eucentric tilt for highest flexibility, speed and accuracy in change of WD or tilt without loosing or redefining ROI.*
	Stage compatible with in-situ battery cycler for FIB-SEM cross-sectioning while electrochemical cycling. Parameter space: temperature -100/+100°C, pressure 0-125 MPa in steps of +/- 0.1 kPa @ sample size Ø10x2 mm
FOV	*30 mm² on coincident point*

Installed Detectors	*In-lens Secondary Electron and Energy selective Backscatter detectors (ES and EsB)*
	Annular BSD detector
	Secondary Electron Secondary Ion detector (SESI)
	Annular STEM detector (bright-field, dark-field: oriented, annular, and high angular annular)
	EDS detector Oxford UltimMax 65 or equivalent (sensor area and performance)
Gas Injection System	*Minimum of 2 gasses for deposition and etching: Pt, C*
Installed Accessories	*Plasma cleaner*
	Electron Floodgun
	Sample holder for TEM grids
	80 mm Airlock (height 40 mm) with airtight sample transfer shuttle and glovebox interface for air-sensitive samples.
Software & Automation	*Workflow oriented, high-throughput acquisition platform. Fusion of images from multiple instruments, detectors and sessions in one correlative workspace. Import of images (BMP, JPG, TIF, CZI, TXM), flexible layered arrangement of images. Protocol based automated 2D acquisition. Large frame-store up to 50k x 40k, 100ns, 8bit/16bit, 2 channels. xROI imaging (exact regions of interest). Manual stitching of 2D mosaics. Post processing tools like automated stitching, batch stitching, image corrections and advanced import and export functions. Image review and re-shoot. Dual-channel blending. Export to movie.*
	3D Tomography and FIB tomography. Automated acquisition of Gigapixel 3D images with simultaneous high resolution milling and imaging. Accurate and constant slice thickness throughout the entire acquisition process. Isotropic voxel size < 10 nm.
	High-resolution imaging while simultaneously milling, live SEM-based thickness measurements during slicing (every slice), large FOV possible of up to 50k x 40k pixels, <3 nm 3D resolution
	Driftcorrection, endpoint-detection, open API
Compatible options or upgrades	*Femto-second laser for fast and large area ablation or precise patterning, coupled to the 80 mm SEM airlock, allowing direct transfer into the SEM chamber, the laser, or between laser and SEM chamber.*
	Cryo-stage and workflow, cryo-transfer, Cryo LSM, freeze-fracture
	ToF-SIMS
	Lift-out and thinning for TEM-grids, automated workflow
Operational stability	*FIB processing success rate of long runs (multiple days-weeks) >95% by drift correction and automated source management (automated reheating, automated extractor- and condenser adjustment) or better control, fast reshaping (<30 min)*
Available within budget	*<€610.000 EX VAT*
Warranty	*Full*
Model	*Recent*
*	*Resolution based on statistical method, not edge method.*

Conclusion of market research;

• Carl Zeiss is the only supplier with a mechanical (and motorized) 6-axis eucentric tilt stage with very high positioning accuracy (not 5-axis compucentric).

This provides the research team with:

1) Greater flexibility: you can change the tilt or working distance at multiple positions on the sample without losing sight of the ROI.
2) Speed: you don't have to set a compucentric point for each ROI.
3) Accuracy/precision: because nothing needs to be set manually (although it's a purely mechanical solution), the accuracy is much greater and less user-dependent).

• Comes with a stage that is already compatible with a readily available and field-tested in-situ battery cycler. It is suitable for solid-state batteries with a diameter of 10 mm and a thickness of 2 mm, and can reach pressures of up to 125 MPa at temperatures from -100 to +100°C. Electrochemistry and SEM data are integrated into the software.

• High statistical resolution for both electron and ion beams.

• Price within budget