Helios 5 PFIB UXe DualBeam for Battery Research

Enabling microstructural and analytical characterization of battery materials

Understanding battery materials at different length scales in 2D and 3D is critical for the determination of structure-performance correlations in battery development.

The Thermo Scientific™ Helios™ 5 PFIB UXe DualBeam delivers high-quality preparation of large, site-specific cross-sections, large-volume 3D characterization, and Ga+ free sample preparation. Ideally suited for the analysis of advanced battery technology, the Helios 5 PFIB UXe DualBeam provides a high level of insight into the interfaces and 3D structures of battery materials at multiple length scales.

The Helios 5 PFIB UXe DualBeam enables researchers and engineers to prepare ultra-large Ga+ free, site-specific cross-sections on various battery sample types such as NCM cathodes and Li-metal anodes. It can generate representative volumes (> 100 µm field of view) with nanometer-resolution while maintaining high-throughput material removal. It combines a new plasma focused ion beam (PFIB) column with the Thermo Scientific Elstar™ Electron Column, the only monochromated scanning electron microscope (SEM) available in a DualBeam, to deliver advanced focused-ion and electron-beam performance. Intuitive software and an unprecedented level of automation and ease-of-use provide observation and analysis of relevant subsurface volumes.

The PFIB delivers exceptional performance at all operating conditions, enabling you to perform a wide range of challenging and demanding tasks at the micro-scale. The Helios 5 PFIB UXe DualBeam is an all-in-one instrument; there is no need to choose between ion columns (each of which might only be suited for limited applications) and there is no requirement for complex accessories that improve cut-face quality (e.g. milling curtain mitigation). The innovative Elstar Electron Column with high-current UC+ technology provides extreme high-resolution imaging and high materials contrast. The latest chemistries developed by Thermo Fisher Scientific are available via the optional Thermo Scientific MultiChem™ or GIS Gas Delivery Systems, further enhance milling throughput, precision, and control.

In addition to advanced electron and ion optics, the Helios 5 PFIB UXe DualBeam incorporates a suite of state-of-the-art software to facilitates simple and consistent sample preparation

Key Benefits

- Quickly access large and representative Ga+ free cross-sections through the battery electrode and other battery samples using the next-generation 2.5 µA xenon plasma-FIB column
- Access high-quality, multi-modal subsurface and 3D information with precise targeting. Use optional Auto Slice & View 4 and Avizo Software to characterize the 3D structure of battery electrodes, including components distribution, particle cracks, surface area, particle size distribution, connectivity, and tortuosity
- High-quality Ga+ free (S)TEM sample preparation thanks to a novel PFIB column, which offers ultra-low-energy polishing while delivering excellent performance at all operating conditions
- Fast and easy automated, multisite cross-sectioning and (S)TEM sample preparation using optional AutoTEM 5 Software
- Fast access to nanoscale information with SmartAlign and FLASH technologies for users of any experience level
- Reveal nanoscale details using the Elstar Electron Column with high-current UC+ monochromator technology, enabling sub-nanometer performance at low energies
- Complete sample information with sharp, refined, and charge-free contrast obtained from up to six integrated in-column and below-the-lens detectors
- Advanced capabilities for electron- and ion-beam induced deposition and etching with optional MultiChem or GIS Gas Delivery Systems
- Precise sample navigation tailored to individual application needs thanks to the high stability and accuracy of a 150 mm Piezo stage and optional in-chamber navigational camera
- Artifact-free imaging based on integrated sample cleanliness management and dedicated imaging modes such as SmartScan and DCFI

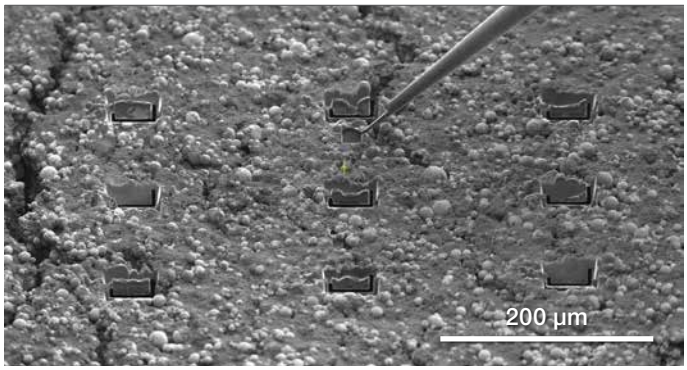

Figure 1. Using AutoTEM 5 Software enables automated *in situ* TEM sample preparation of 9 TEM lamella on an NCM cathode.

for high-resolution TEM/STEM, as well as high-throughput and quality for large-volume subsurface and 3D characterization, even on challenging samples.

High-quality large-volume subsurface and 3D analysis

Subsurface or 3D characterization is often required to better understand the material properties of a sample. In many cases large volumes, inaccessible by conventional Ga+ FIB instruments, are necessary to obtain representative and relevant results. The excellent high-current performance of the Helios 5 PFIB UXe DualBeam, combined with optional Thermo Scientific Auto Slice & View™ 4 Software, enables high-quality, fully automated acquisition of large-volume 3D datasets in a multitude of modalities, including: BSE imaging for maximum materials contrast, energy dispersive spectroscopy (EDS) for compositional information, and electron backscatter diffraction (EBSD) for microstructural and crystallographic information. Combined with Thermo Scientific Avizo™ Software for visualization, it delivers a unique workflow solution for high-resolution, advanced 3D characterization and analysis at the nanometer scale.

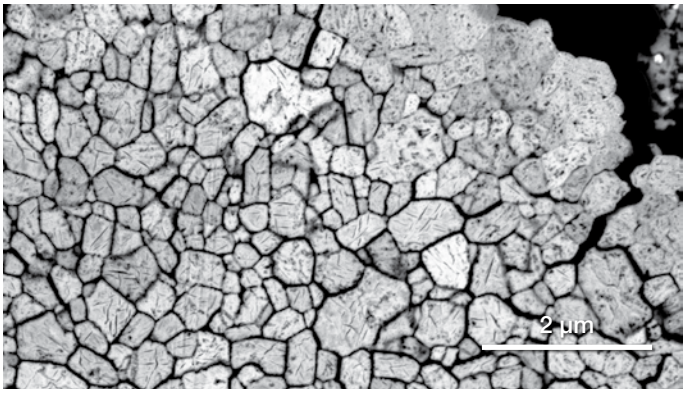

Figure 3. NCM primary particles with defects.

High-quality Ga+ free TEM sample preparation

To understand new materials or to find the root cause of failures, scientists and engineers constantly face new challenges that require highly localized characterization of increasingly complex samples with ever smaller features. The latest technological innovations of the Helios 5 PFIB UXe DualBeam, in combination with easy-to-use, comprehensive software and our application expertise, enable fast and easy preparation of site-specific, high-quality samples for high-resolution (S)TEM from a wide range of materials. In order to achieve high-quality results, final polishing with low-energy ions is required in order to minimize surface damage on the sample. Our new PFIB column not only delivers high-resolution imaging and milling at high voltages, it now extends PFIB performance to accelerating voltages as low as 500 V, enabling the creation of exceptionally high-quality, ultra-low-damage TEM lamella.

The combination of the Helios 5 PFIB UXe DualBeam with Thermo Scientific AutoTEM™ 5 Software enables automated

Figure 2. Helios 5 PFIB UXe DualBeam.

in situ TEM sample preparation. This allows users of any experience level to achieve high-quality results and significantly increases productivity through unattended sample preparation during the day or overnight.

High-resolution with precise materials contrast

The Helios 5 PFIB UXe DualBeam features an ultra-high-brightness electron source with next-generation UC+ monochromator technology, reducing the beam energy spread below 0.2 eV for beam currents up to 100 pA. This enables sub-nanometer resolution and excellent surface sensitivity at low landing energies. Fast, accurate, and reproducible results are obtained thanks to the Elstar Column's unique column design, which includes advanced auto-alignments, constant power lenses for higher thermal stability, and electrostatic scanning for higher deflection linearity and speed. The Elstar Electron Column serves as the foundation of the system's unprecedented high-resolution imaging capability. It can reveal nanoscale details across a wide range of working conditions, whether operating at 30 keV (to access structural information) or at lower energies (to obtain charge-free, detailed information from the surface).

With its unique detection system located inside the column, along with an immersion mode, the system is designed for simultaneous detector acquisition for angular and energy-selective secondary-electron (SE) and backscattered-electron (BSE) imaging. Fast access to detailed nanoscale information is available, not only top-down, but also on tilted specimens or cross-sections. Additional below-the-lens detectors and the electron beam deceleration mode ensure fast and easy simultaneous collection of all signals to reveal even small features in material surfaces or cross-sections.

The Helios 5 PFIB UXe DualBeam introduces novel SmartAlign technology, which eliminates the need for any user alignments

of the electron column. This not only minimizes maintenance but also increases your productivity. Generally, to achieve the best results on different materials, fine tuning of the beam would be required. This typically consists of an alignment sequence (focusing, lens centering, stigmation) that can be challenging and time consuming. To address this, the Helios 5 PFIB UXe DualBeam introduces FLASH technology, a new fine image tuning capability. With FLASH technology, you only need to perform a simple mouse-gesture in the graphical user interface, a procedure similar to focusing the image, and the instrument will introduce any necessary corrections "on-the-fly" to the stigmators and lens centering, while also bringing the image into focus. On average, FLASH technology can result in an up to 10x improvement in the time required to obtain an optimized image.

Specifications

Electron optics

- Extreme high-resolution field emission Elstar Electron Column with:
  - Magnetic immersion objective lens
  - High-stability Schottky field emission gun that provides stable, high-resolution analytical currents
  - UC+ monochromator technology
- User-alignment-free SmartAlign technology
- 60-degree dual objective lens with pole-piece protection allows for tilting of larger samples
- Automated heated apertures ensure cleanliness and touch-free aperture exchange
- Electrostatic scanning for higher deflection linearity and speed
- Thermo Scientific ConstantPower™ Lens for high thermal stability
- Integrated fast beam blanker*
- Beam deceleration with stage bias from 0 V to -4 kV*
- Minimum source lifetime: 12 months

Electron beam resolution

- At optimum working distance (WD):
  - 0.7 nm at 1 kV
  - 1.0 nm at 500 V (in-column detector)
- At coincident point:
  - 0.6 nm at 15 kV
  - 1.2 nm at 1 kV

Electron beam parameter space

- Electron beam current range: 0.8 pA to 100 nA
- Accelerating voltage range: 350 V to 30 kV
- Landing energy range: 20 eV* to 30 keV
- Maximum horizontal field width: 2.3 mm at 4 mm WD

Ion optics

High-performance PFIB column with inductively-coupled Xe+ plasma (ICP)

- Ion beam current range: 1.5 pA to 2.5 µA
- Accelerating voltage range: 500 V to 30 kV
- Maximum horizontal field width: 0.9 mm at beam coincidence point

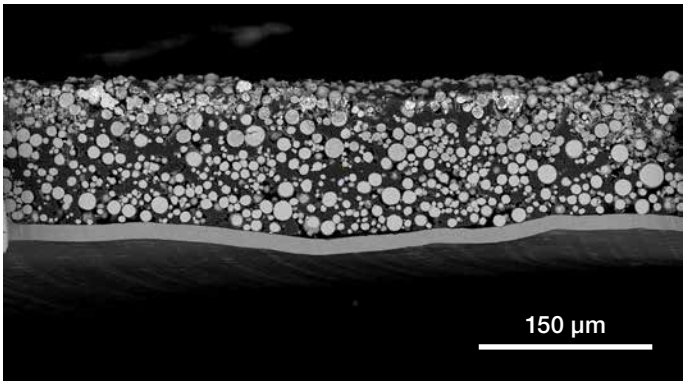

Figure 4. 600 µm wide cross-section of a Li-battery NCM cathode sample prepared by PFIB.

Ion beam resolution at coincident point

- <20 nm at 30 kV using preferred statistical method
- <10 nm at 30 kV using selective edge method

Detectors

- Elstar in-lens SE/BSE detector (TLD- SE, TLD-BSE)
- Elstar in-column SE/BSE detector (ICD)*
- Everhart-Thornley SE detector (ETD)
- IR camera for viewing sample/column
- High-performance in-chamber electron and ion detector (ICE) for secondary ions and electrons
- In-chamber Thermo Scientific Nav-Cam Sample Navigation Camera*
- Retractable low-voltage, high-contrast directional solid-state backscatter electron detector (DBS)*
- Integrated beam current measurement

Stage and sample

High precision 5-axis motorized stage, with piezo-driven XYR axis

- XY range: 150 mm
- Z range: 10 mm
- Rotation: 360° (endless)
- Tilt range: -38° to +60°
- XY repeatability: 1 µm
- Max. sample height: 55 mm clearance to eucentric point
- Max. sample weight at 0° tilt: 500 g (including sample holder)
- Max. sample size: 150 mm with full rotation (larger samples possible with limited rotation)
- Compucentric rotation and tilt

Vacuum system

- Complete oil-free vacuum system
- Chamber vacuum: $<2.6 \times 10^{-6}$ mbar (after 24 h pumping)
- Evacuation time: <5 minutes

Chamber

- Electron- and ion-beam coincidence point at analytical WD (4 mm SEM)
- Ports: 21
- Inside width: 379 mm
- Integrated plasma cleaner

Sample holders

- Multi-purpose specimen holder with adjustable height
- Vise specimen holder to clamp irregular, large, or heavy specimens to the specimen stage*

- Universal mounting base (UMB) for stable, flexible mounting of many sample and holder combinations such as flat and pretilt stubs, or row holders for TEM grids*
- Various wafer and custom holder(s) available by request*

Image processor

- Dwell time range from 25 ns/pixel to 25 ms/pixel
- Up to 6144 × 4096 pixels
- File type: TIFF (8, 16, 24-bit), BMP, or JPEG standard
- SmartScan (256 frame average or integration, line integration and averaging, interlaced scanning)
- DCFI (drift compensated frame integration)

System control

- 64-bit GUI with Windows® 10, keyboard, optical mouse
- Up to four live images showing independent beams and/or signals. Live color signal mixing
- Local language support: check with your local Thermo Fisher Sales representatives for available language packs
- Two 24-inch widescreen monitors (1920 × 1200 pixels) for system GUI and full-screen image
- Microscope-controlling and support computers seamlessly share one keyboard, mouse, and monitors
- Joystick*
- Multifunctional control panel*
- Remote control and imaging*

Supporting software

- "Beam per view"' graphical user interface, with up to 4 simultaneously active quads
- Thermo Scientific SPI (simultaneous FIB patterning and SEM imaging), iSPI (intermittent SEM imaging and FIB patterning), iRTM (integrated real time monitor), and FIB immersion mode for advanced, real-time SEM and FIB process monitoring and endpointing
- Patterns supported: lines, rectangles, polygons, circles, donuts, cross-sections, and cleaning cross-sections
- Directly import BMP files or stream files for 3D milling and deposition
- Material file support for "minimum loop time," beam tuning and independent overlaps
- Image registration enabling sample navigation in an imported image
- Sample navigation possible on an optical image

Accessories*

- GIS (gas injection system) solutions
  - Single GIS: up to four independent units for enhanced etching or deposition
  - MultiChem Gas Delivery System: up to six chemistries on the same unit for advanced etching and deposition controls
- GIS beam chemistry options**
  - Platinum deposition
  - Tungsten deposition
  - Carbon deposition
  - Insulator deposition II
  - Gold deposition
  - Enhanced Etch (iodine, patented)
  - Insulator enhanced etch (XeF2)
  - Delineation Etch (patented)
  - Dx Delayering
  - Empty crucibles for user-supplied materials approved by Thermo Fisher Scientific
  - More beam chemistries available upon request
- Thermo Scientific EasyLift™ Nanomanipulator - fully integrated for precise *in situ* sample manipulation
- FIB charge neutralizer
- Analysis: EDS, EBSD, WDS
- Thermo Scientific QuickLoader™ Load Lock for fast sample exchange without breaking system vacuum
- Cryo-solutions for DualBeam
  - Exclusive Thermo Scientific CryoMAT Loader for cryogenic material science applications
  - Solutions from external vendors
- Thermo Scientific acoustic enclosure
- Thermo Scientific CryoCleaner

Software options*

- AutoTEM 5 Software for automated (S)TEM sample preparation
- Thermo Scientific AutoScript™ 4 Software: advanced automation suite for DualBeams
- Thermo Scientific Maps Software for automatic acquisition of large images and optional correlative work
- Thermo Scientific NanoBuilder™ Software – advanced proprietary CAD-based (GDSII) solutions for FIB and beam deposition, optimized for nanoprototyping of complex structures
- Auto Slice & View 4 Software – automated sequential milling and viewing lets you collect a series of slice images as well as EDS or EBSD maps for 3D reconstruction
- Avizo Software – 3D reconstruction and analysis software
- CAD navigation

Warranty and training

- 1 year warranty
- Choice of service maintenance contracts
- Choice of operation/application training contracts

Documentation and support

- Online user guidance
- User operation manual
- Prepared for RAPID remote diagnostic support

**Optional*

***Some beam chemistries may only be available on MultiChem or single GIS*

Learn more at **thermofisher.com/heliospfib**