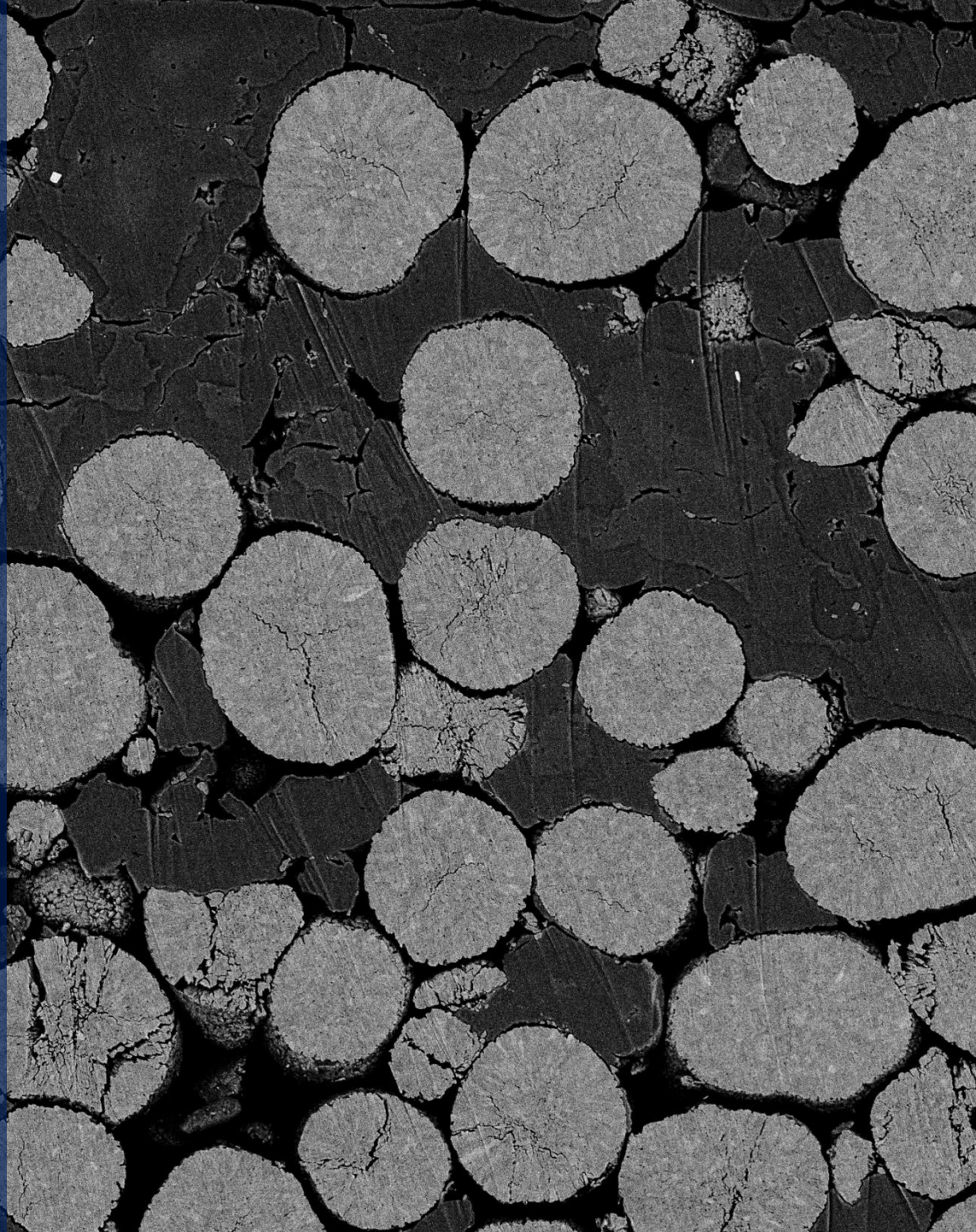
Li-ion battery sample preparation

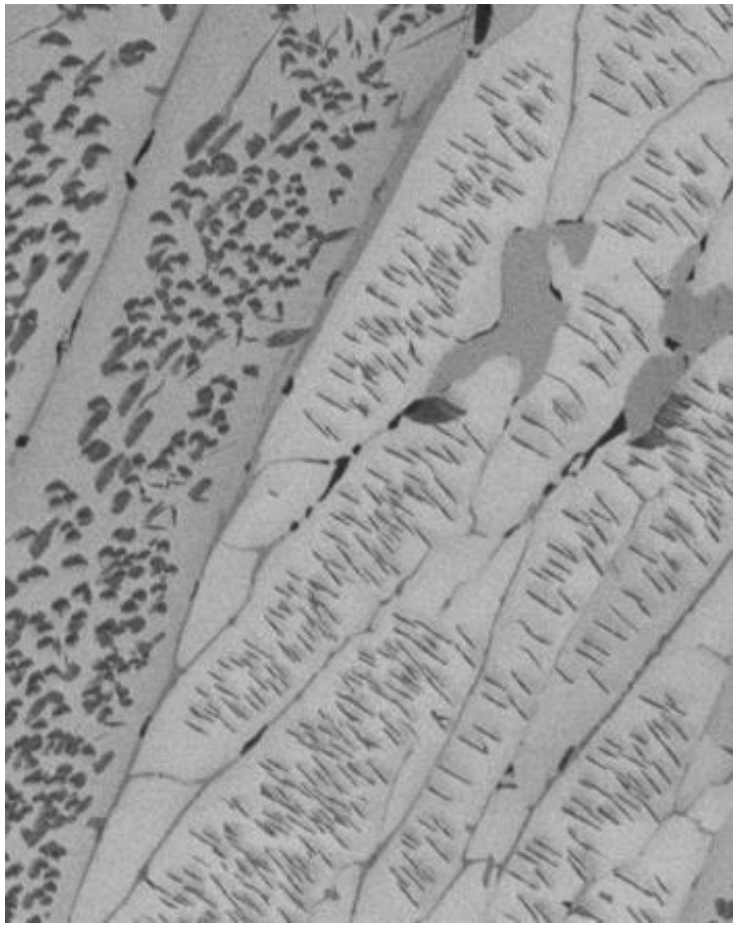
FISCHIONE INSTRUMENTS



Model 1061 and Model 1062 argon ion mills

Versatile broad ion beam mills for SEM applications

- Produces ideal results for both **planar and cross-section** samples
- **Wide-ranging ion energies** allow either rapid milling or gentle polishing on a broad variety of sample materials
- **Artifact-free samples** are readily produced
- **Easy-to-use interface**
- **Fully automated**, including precise sample height detection, for high-throughput applications



Model 1061 SEM Mill

Model 1062 TrionMill

Features

- Produces ideal results for both planar and cross-section samples
- Wide-ranging ion energies from 0.1 to 10 keV
- Adjustable beam size from 300 µm to 5 mm
- Milling angles from 0 to 10°
- Liquid nitrogen stage cooling (optional)

Model 1061 SEM Mill

Model 1062 TrionMill

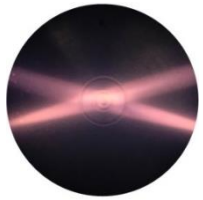
Features

- In situ sample viewing and capture (optional)
- Vacuum or inert gas sample transfer (optional); cryogenic sample transfer (optional, Model 1062 only)
- Fully automated, including precise sample height detection, for high-throughput applications
- None, continuous, or rocking stage motion

Features

Model 1061 SEM Mill

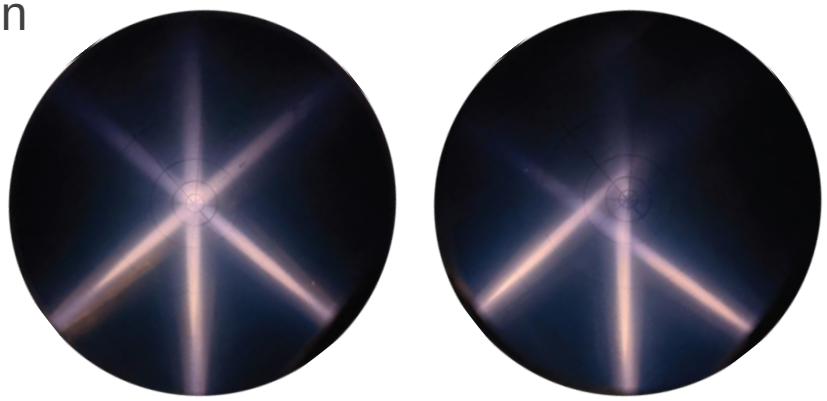
- Two ion sources
- Sample size up to 32 mm diameter



25 mm

Model 1062 TrionMill

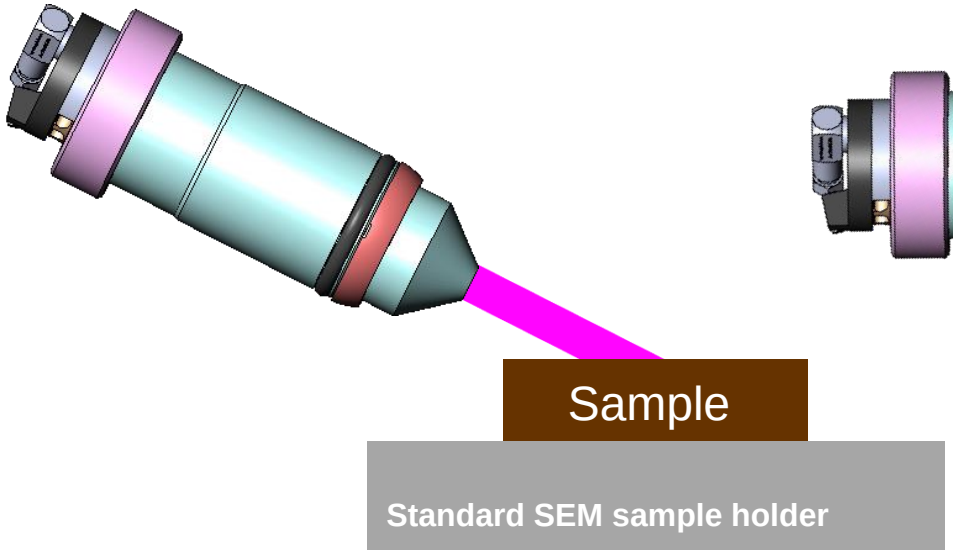
- Three ion sources
- Sample size up to 50 mm diameter
- Flexibility in beam position



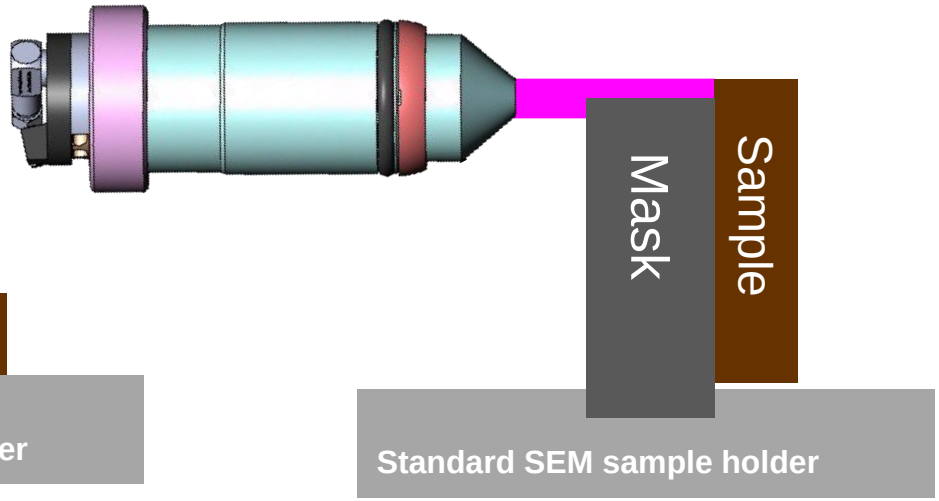
50 mm

Milling modes

Planar milling

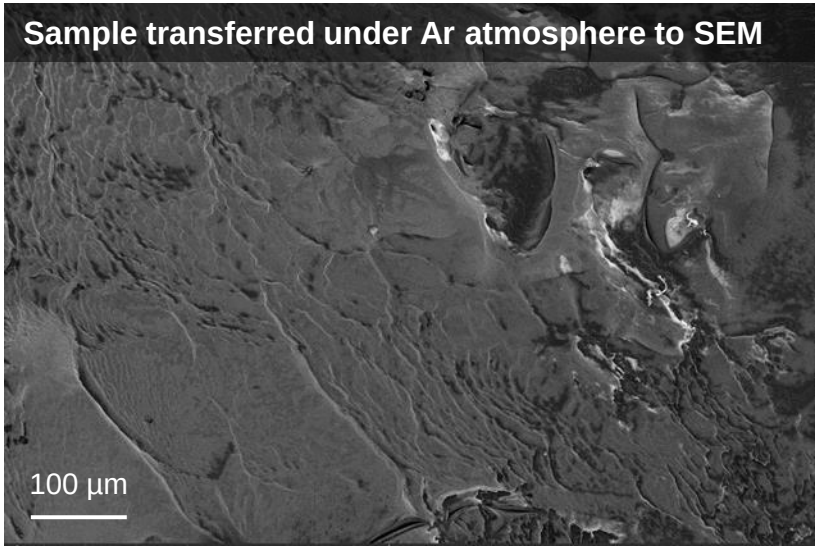


Cross-section milling



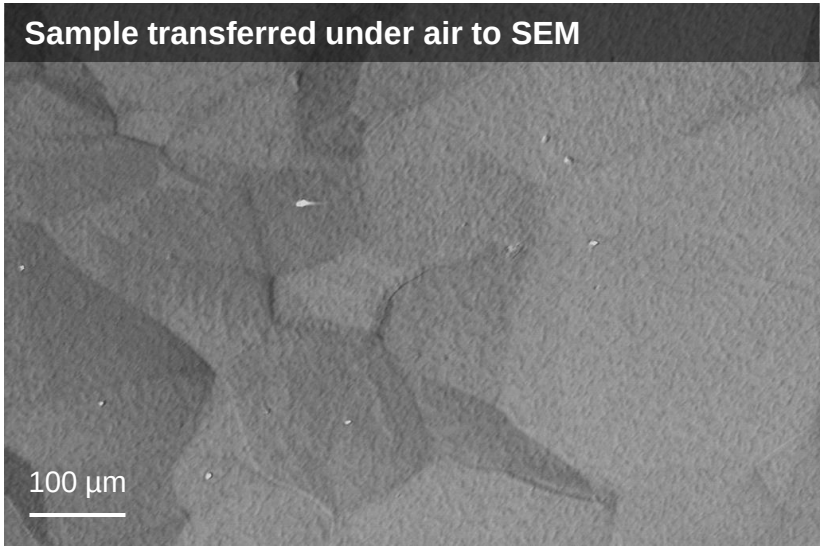
Importance of sample preparation

Ion milling at 6 kV <u>without</u> stage cooling



Thermal damage is observed

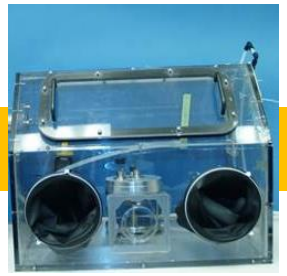
Ion milling at 6 kV <u>with</u> stage cooling



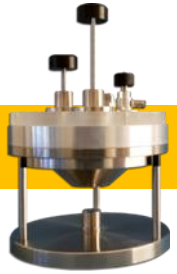
Oxide and hydrocarbon formation on the sample surface

In both cases, no Li microstructure was revealed.

Model 1061 controlled environment workflow



Glove box with Ar positive pressure

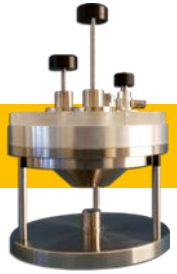


Fischione Instruments
Vacuum/Inert Gas Transfer Capsule

Fischione Instruments
Model 1061 SEM Mill

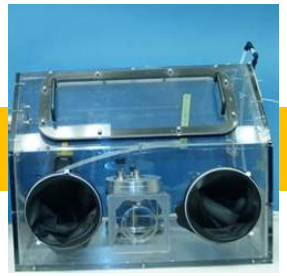
SEM / FIB with **Quorum** PP3004 Airlock

Glove box with **Quorum** transfer chamber



Fischione Instruments
Vacuum/Inert Gas Transfer Capsule

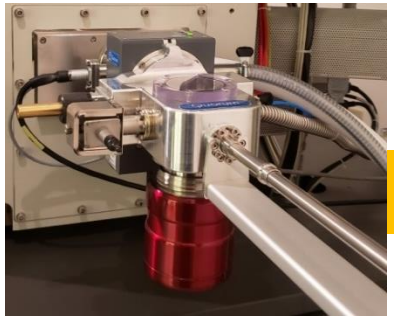
Model 1062 controlled environment workflow



Glove box with Ar positive pressure

Fischione Instruments Model 1062 TrionMill Vacuum/Inert Gas/Cryogenic Transfer

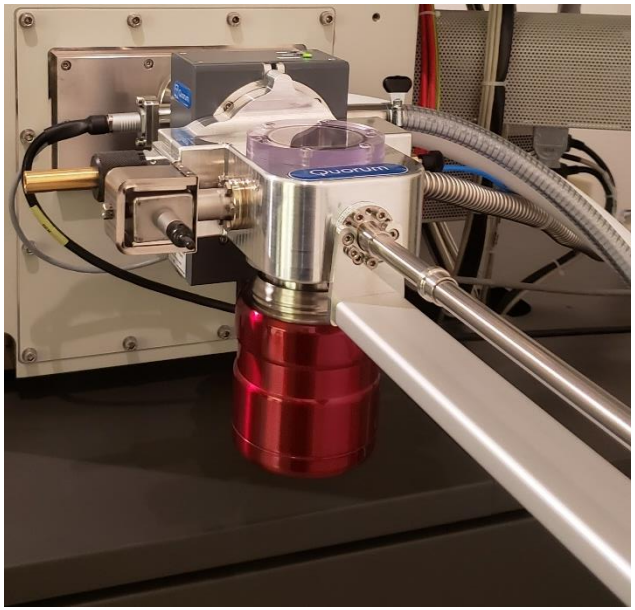
Fischione Instruments Model 1062 TrionMill



SEM / FIB with **Quorum** PP3004 Airlock

Fischione Instruments Model 1062 TrionMill Vacuum/Inert Gas/Cryogenic Transfer

Vacuum/inert gas/cryogenic transfer system



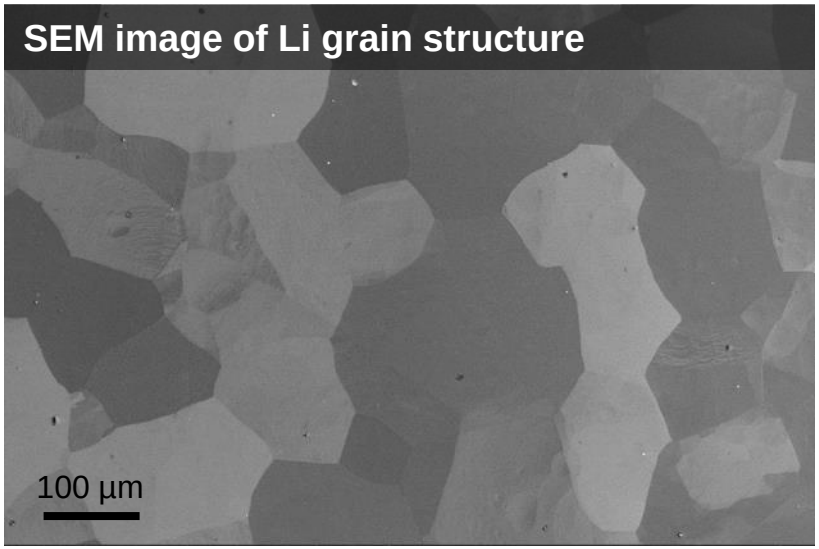
Allows direct transfer of a sample at vacuum, in inert gas, or in a cryogenic environment to the SEM or FIB.

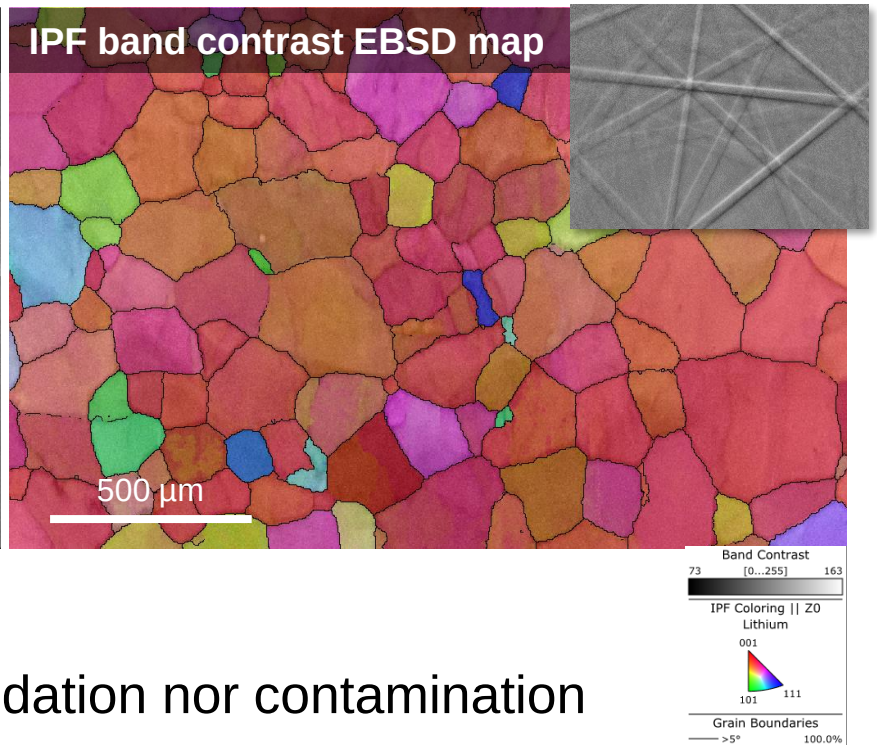
Transfer system design in collaboration with Quorum.

Applications

Li metal characterization

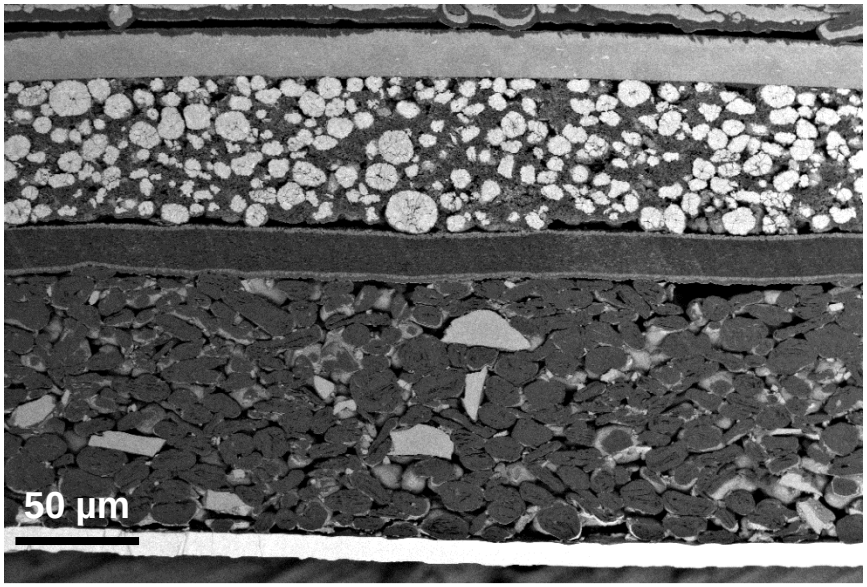
Sample transfer validation

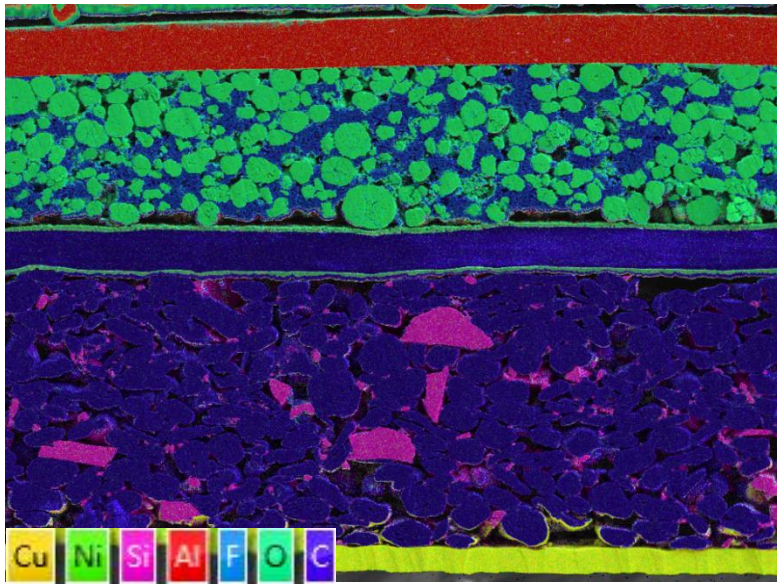




High-quality EBSP confirms no oxidation nor contamination of sample after transfer.

Li ion battery cross-section sample



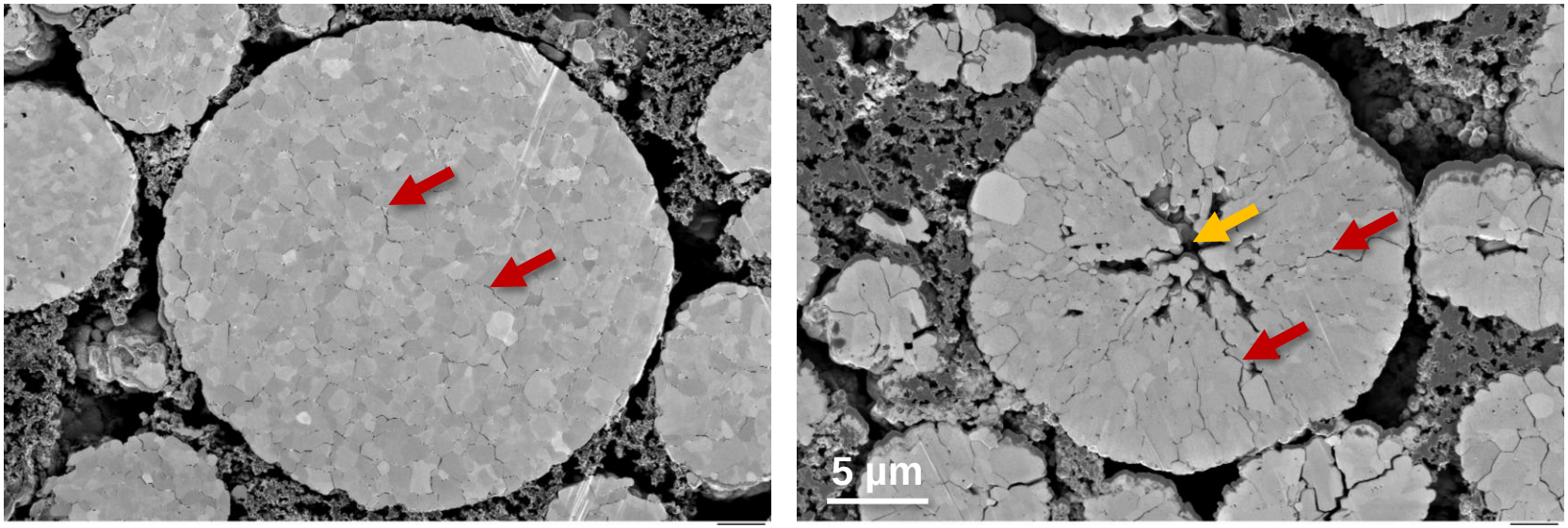


EDS at 3 kV

EDS study shows the chemical composition of different elements of a Li ion battery cell

Li ion battery cross-section sample

NCM cathode particles after 500 cycles

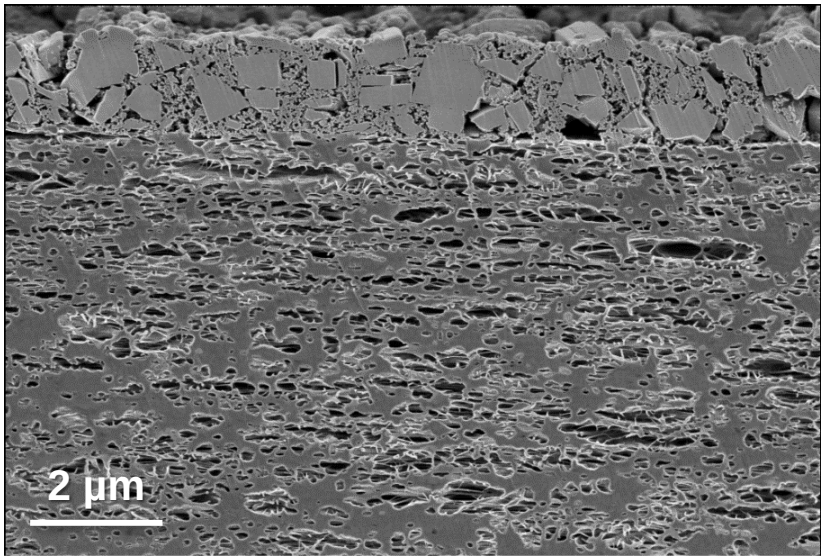


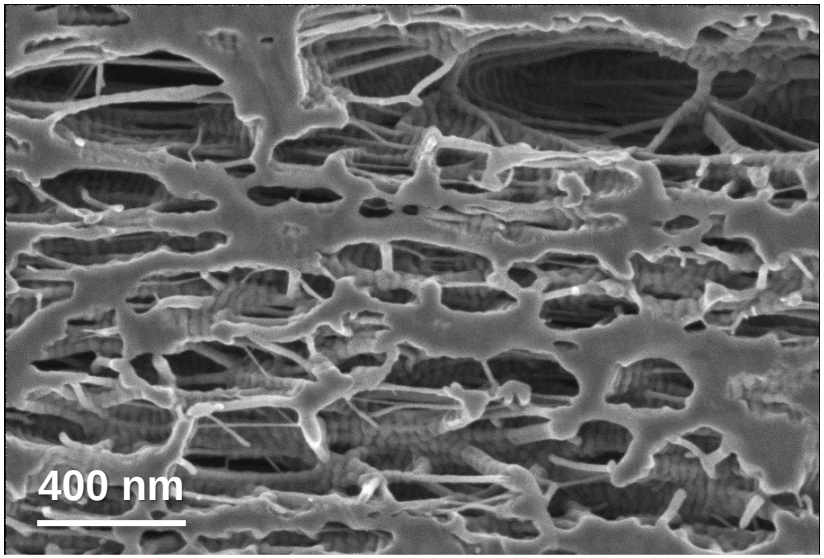
Cracks at the grain boundaries (red arrow) are due to cycled charge and discharge during battery lifetime.

The core voids (gold arrow) come from NCM powder synthesis.

Li ion battery cross-section sample

Ceramic-coated separator

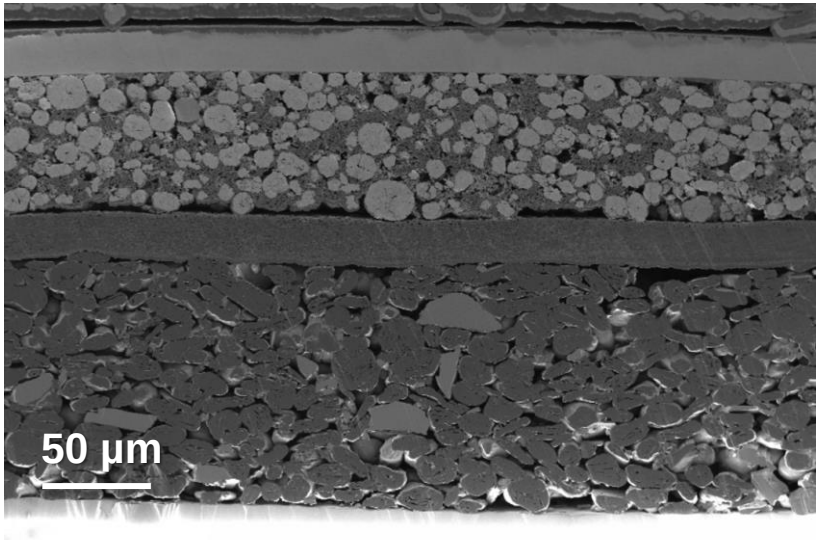


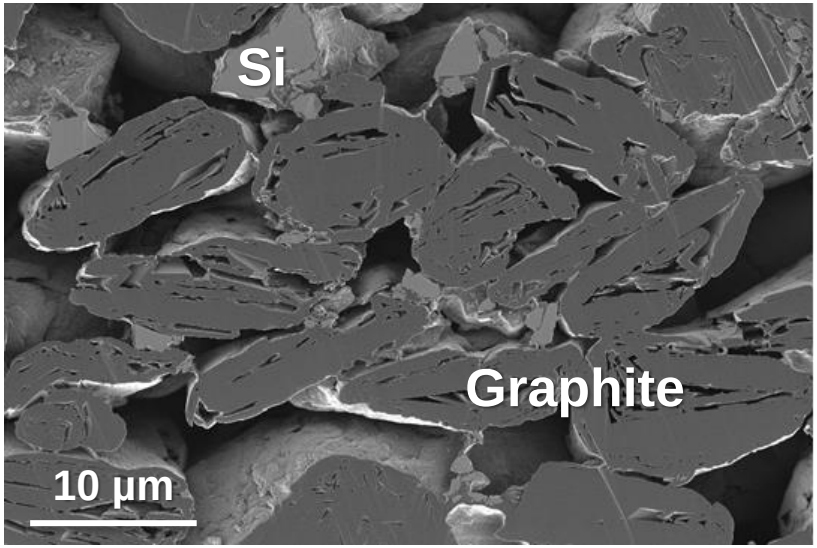


Perfectly preserved porous structure of polymer separator can be observed after ion milling with cryogenic conditions.

Li ion battery cross-section sample

Graphite–silicone anode

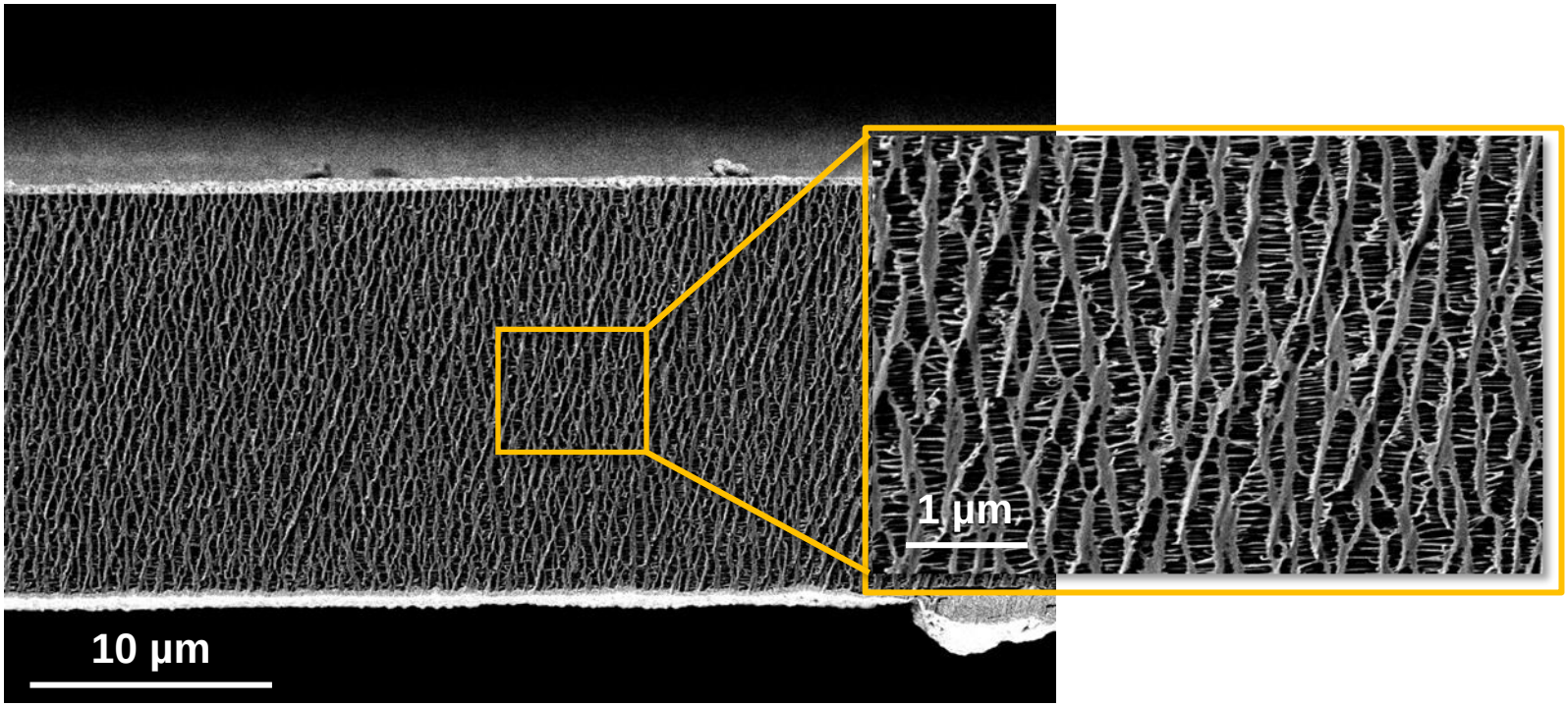




Graphite silicon anode 9:1 ratio

Li ion battery polymer separator

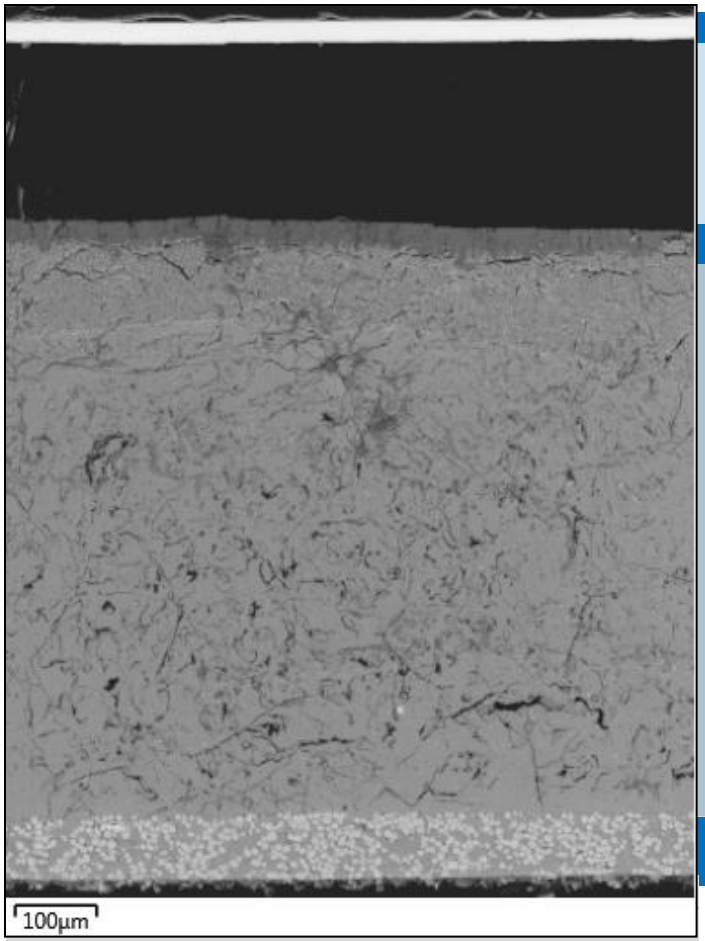
Cross-section sample; 6 keV beam energy and cryogenic milling conditions



The pore structure is completely preserved, which allows accurate porosity measurement.

Solid state Li ion battery

Large cross-section sample



Cu current collector: 25 µm

Li metal anode: 230 µm

Solid electrolyte interface: 30 µm

Solid state electrolite: 600 µm

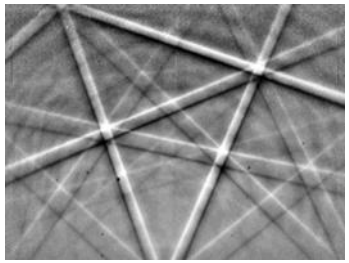
Ni / Co / Mn cathode: 80 µm

LPS
SSE

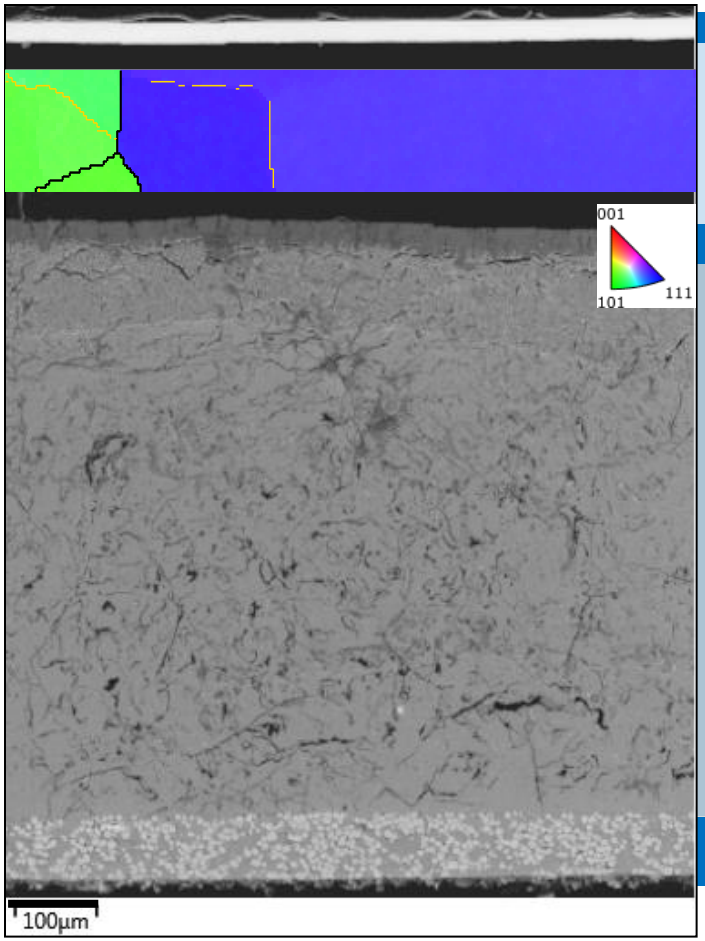
Solid state Li ion battery

Large cross-section sample

EBSD at 20 kV
4 µm step size



High quality EBSP confirms no oxidation nor contamination of sample after transfer



Cu current collector: 25 µm

Li metal anode: 230 µm

SEI: 30 µm

SSE: 600 µm

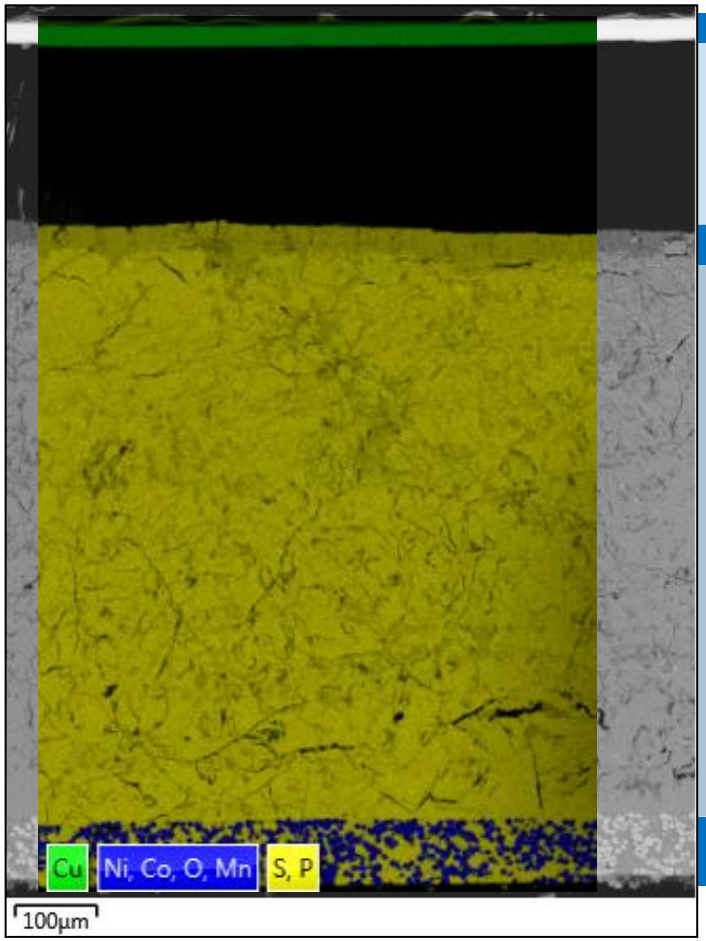
NCM cathode: 80 µm

LPS SSE

Solid state Li ion battery

Large cross-section sample

EDS at 6 kV



Cu current collector: 25 µm

Li metal anode: 230 µm

SEI: 30 µm

SSE: 600 µm

NCM cathode: 80 µm

LPS
SSE

Distribution in the UK & Ireland

Lambda

Characterisation, Measurement & Analysis

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