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Opportunities and Challenges of Ultra Short Pulsed Lasers with Dual Focused Ion Beams for Characterization of Full-Scale Electronic Devices

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Microscopy and Microanalysis Conference

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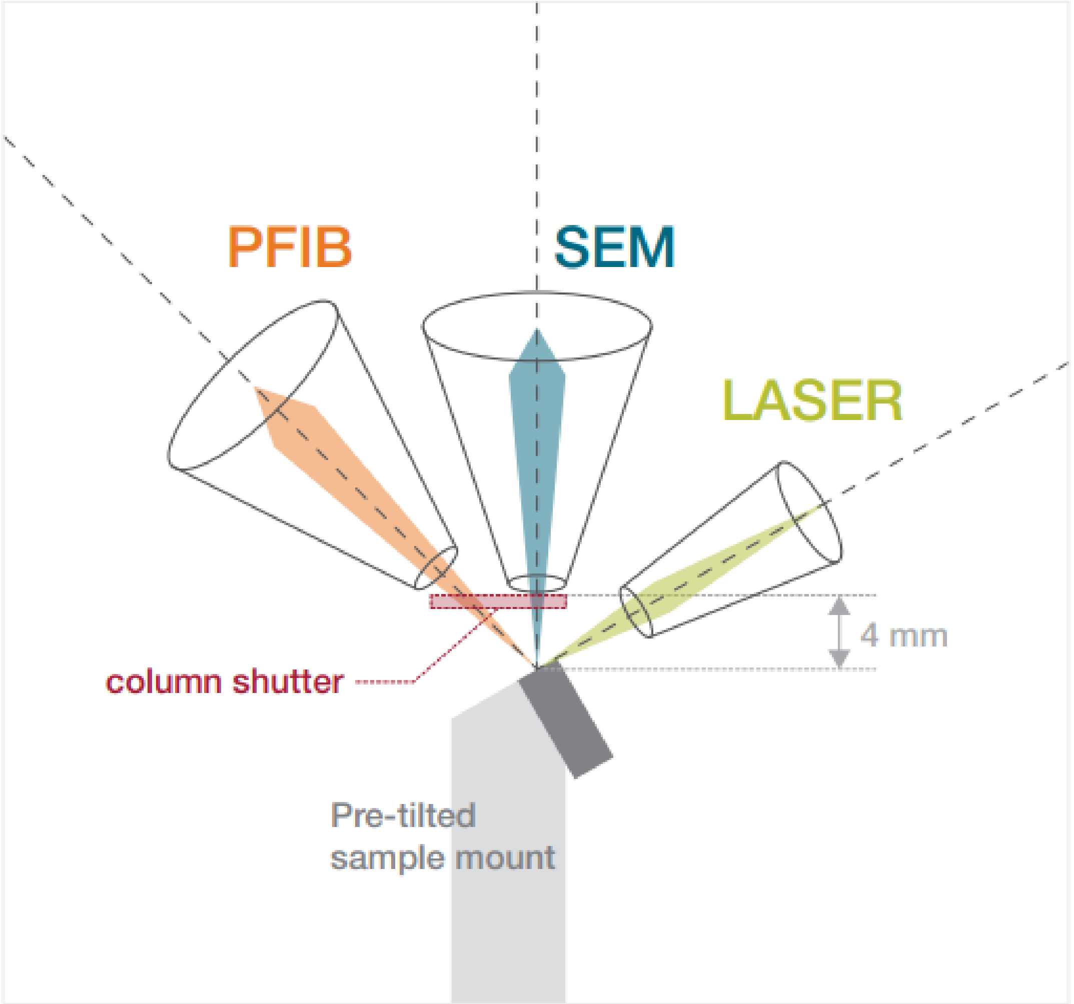
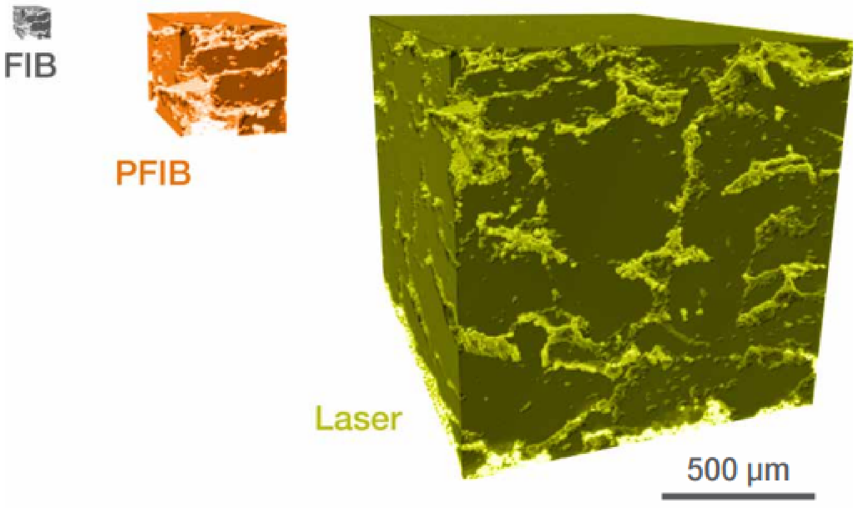
Laser Plasma Focused Ion Beam (PFIB)



Helios G5 Laser Plasma FIB



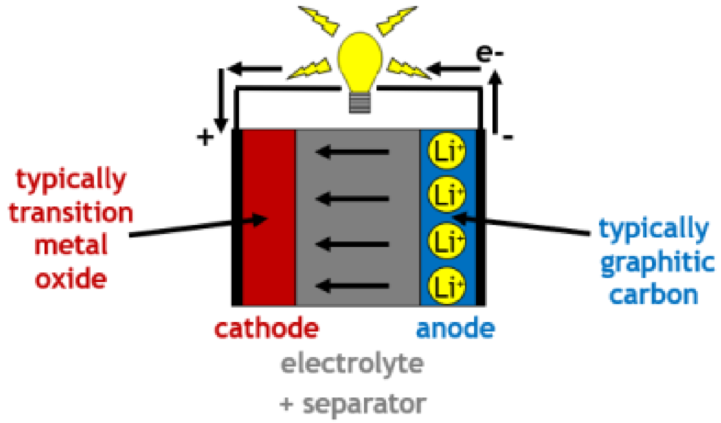
- New laser capability on plasma focused ion beam (PFIB) makes characterization of volumes up to 4x2x1 mm^3 possible
- Unique combination of nanoscale characterization over large length scales – usually is a tradeoff between spatial resolution and area/volume available to characterize

ThermoFisher SCIENTIFIC

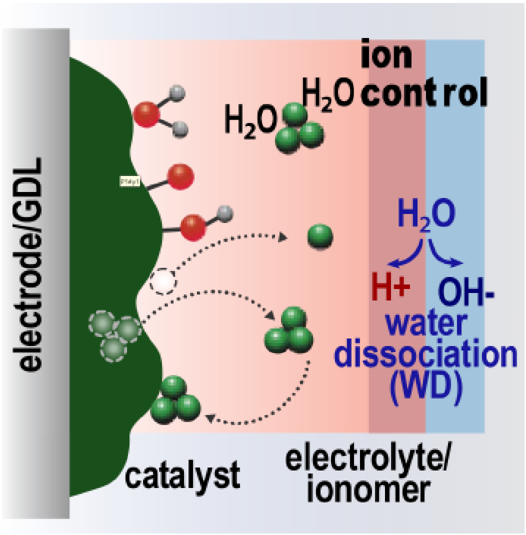
Laser PFIB Utility for Electronics

Particularly advantageous for electronics where site specific information over large volumes gives valuable failure mechanism details

Batteries



Electrolysers



Capacitors

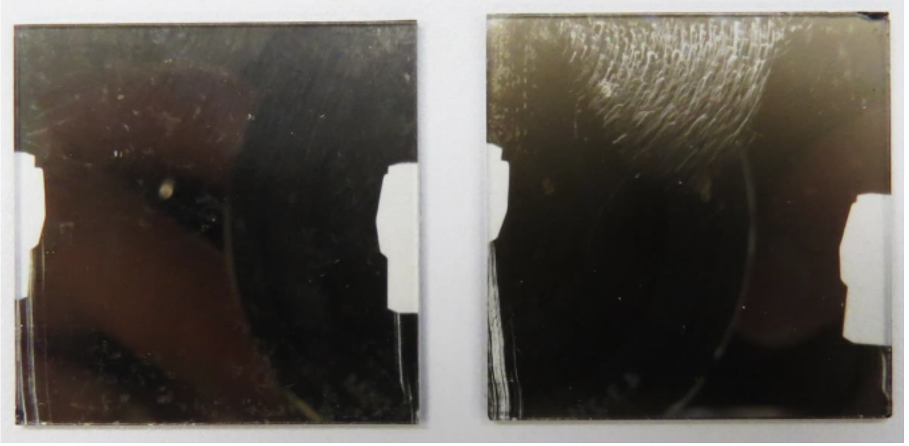
Requires cryogenic capabilities

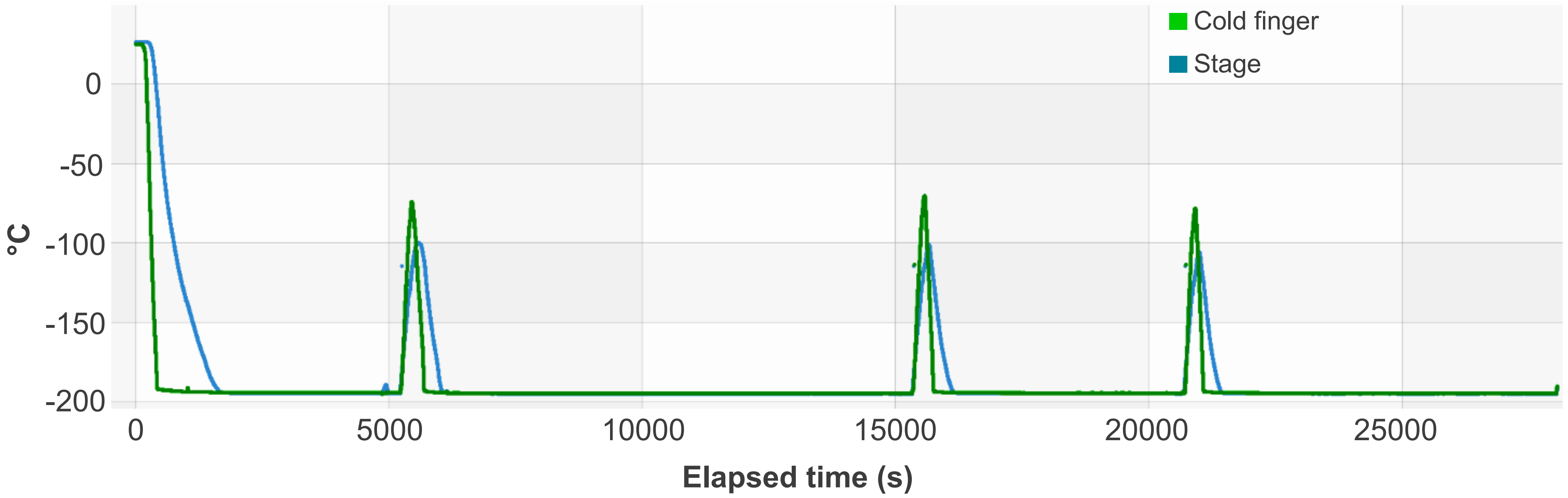
Cryogenic Considerations

Glass slip between laser and chamber prevents ablated material going back into the laser column

Must be changed periodically through cross-sectioning milling

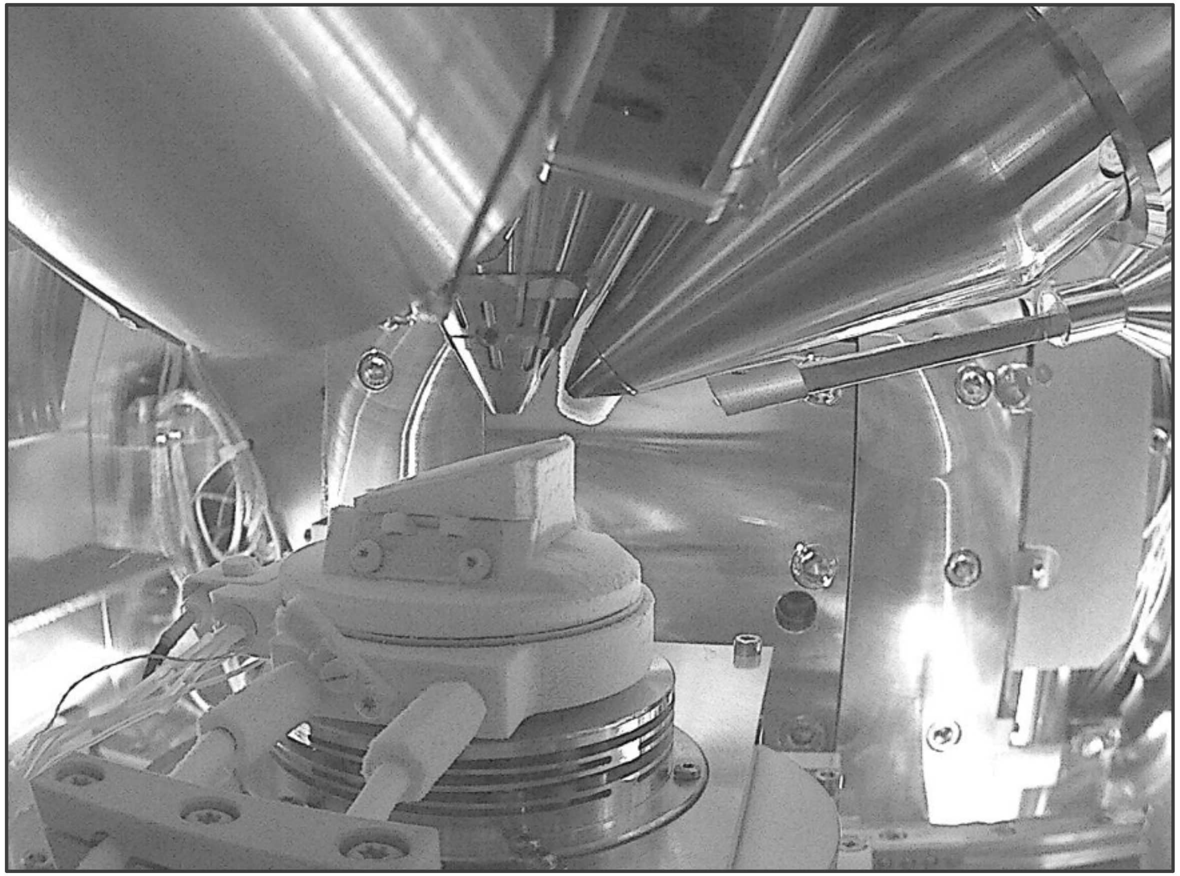
Glass slip after laser milling





Cryogenic Considerations

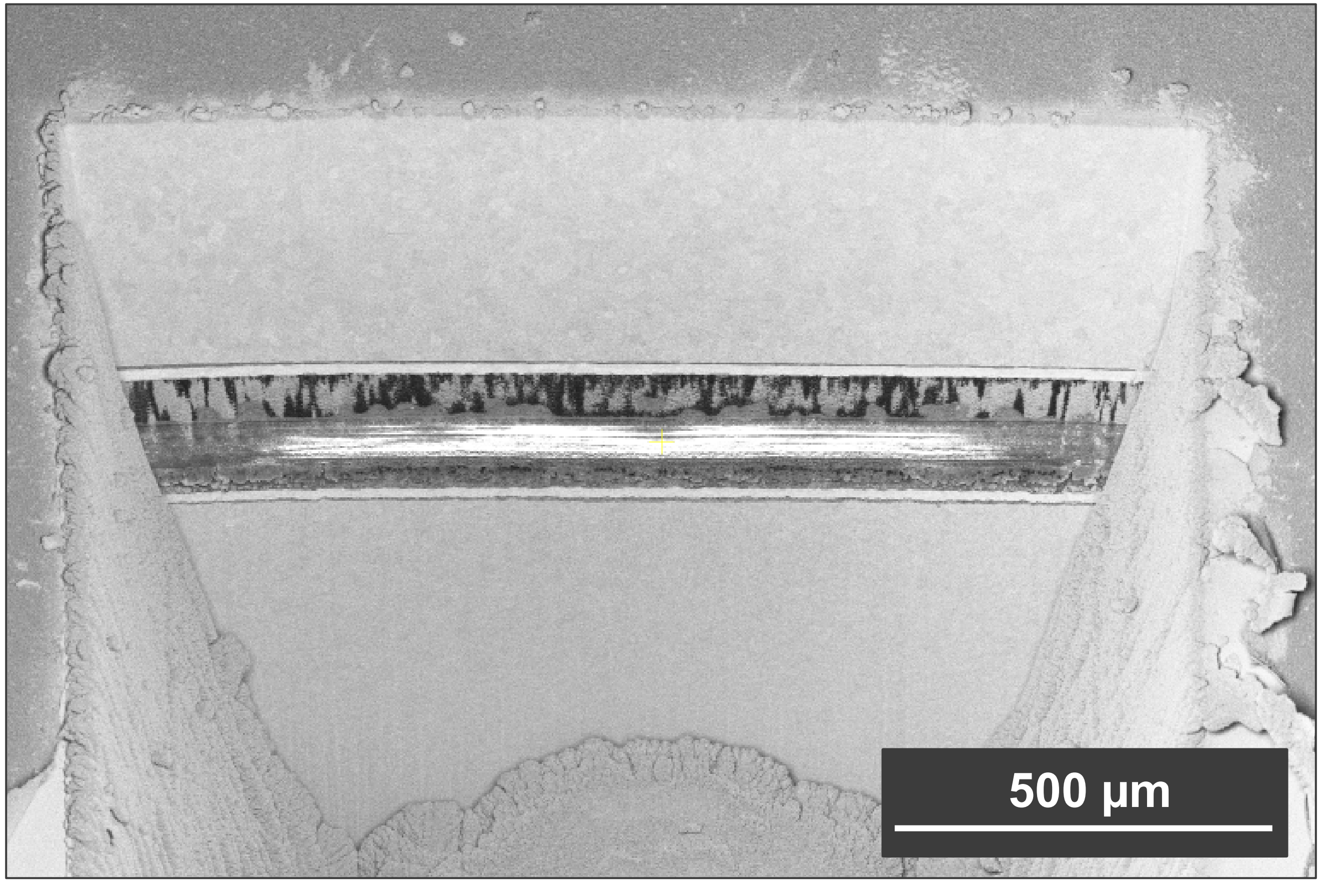
The result…



- When opening the chamber, frost builds on sample from outside moisture
- Using a glove bag with nitrogen partially mitigates the issue
  - House nitrogen still has enough moisture for some frost buildup
  - UHP nitrogen does not lead to frost buildup but we run out rather quickly…
  - Next step to try purifier for house nitrogen

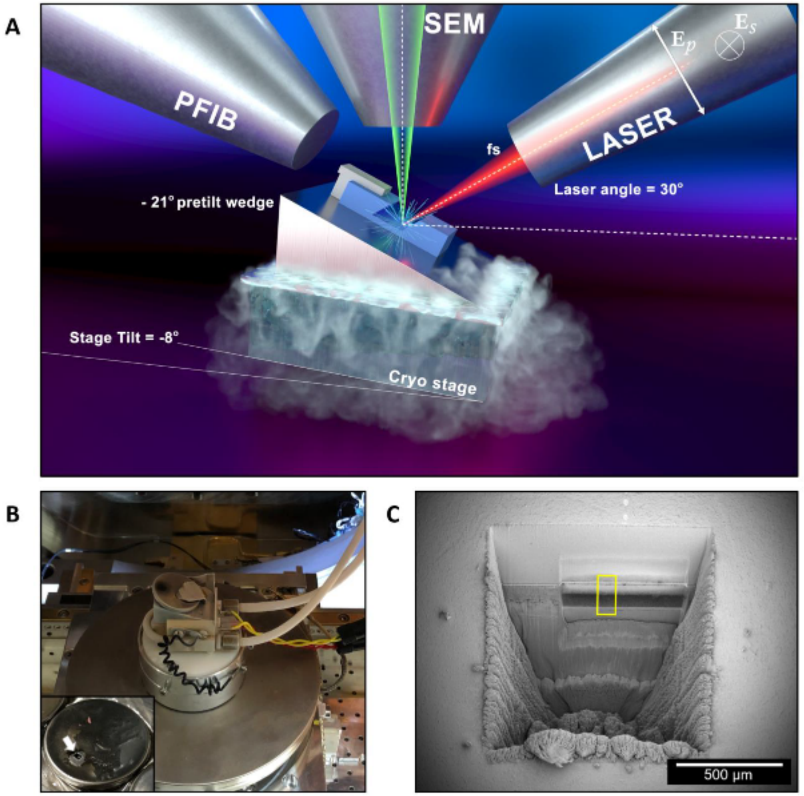
Cryogenic Considerations

- Charging
  - Depositing from multichem challenging at cryogenic temperatures
    - Putting multichem in halfway helps somewhat
  - Easylift Needle doesn't help with porous material
  - Imaging with lower AV and current helps, but not ideal for collecting EDXS data

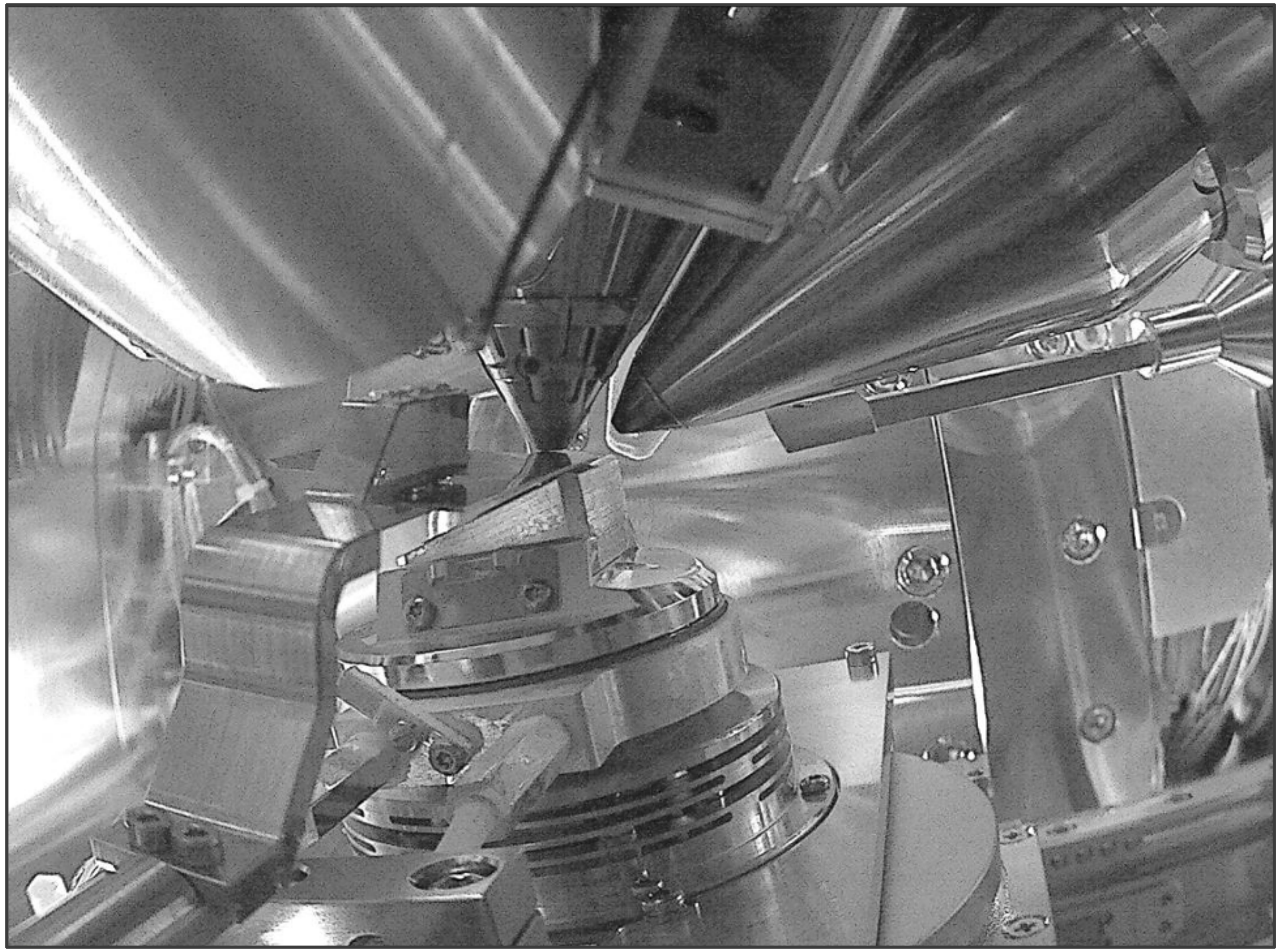


Cross-sectioning without Battery Disassembly

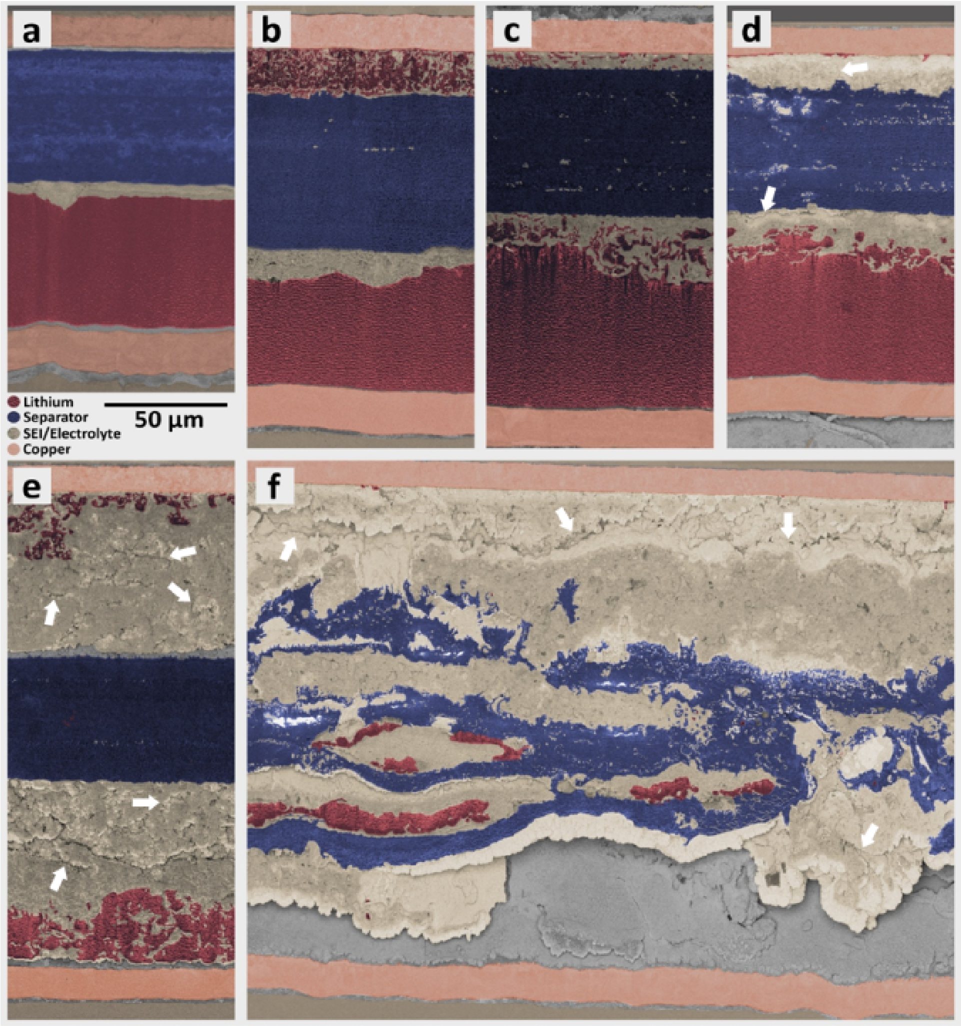
Coin cell



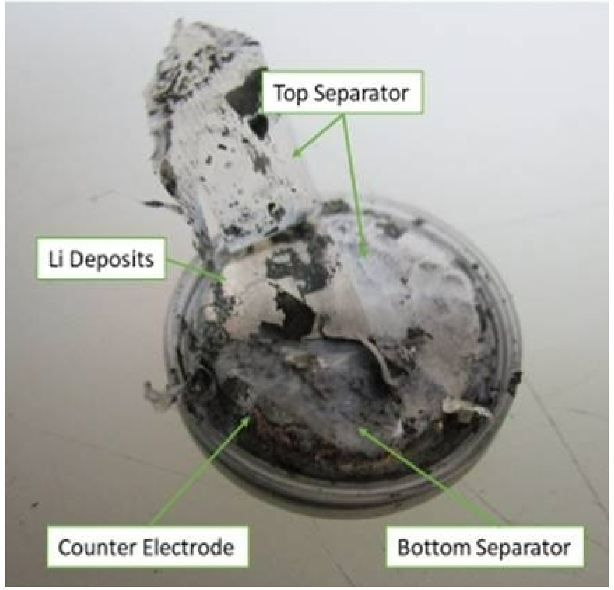
Jungjohann et al., ACS Energy Lett. 6, 2138 (2021)
DOI: 10.1021/acsenergylett.1c00509



Failure after Cycling



- Cryo laser PFIB can image intact coin cells without disassembly, characterization provides:
  - Structure of the separator-Li interface
  - Quantify Li inventory, Li morphology, cracking in SEI, and SEI thickness
  - Under high-rate cycling: Separators are damaged or destroyed
    - Li and SEI grow between separators and tri-layers of separators



2.8 M LiFSI in DME
Two Celgard 2325 separators
Cycled at 1.88 mA/cm^2
Capacity: 1.88 mAh/cm^2

Jungjohann et al., ACS Energy Lett. 6, 2138 (2021)
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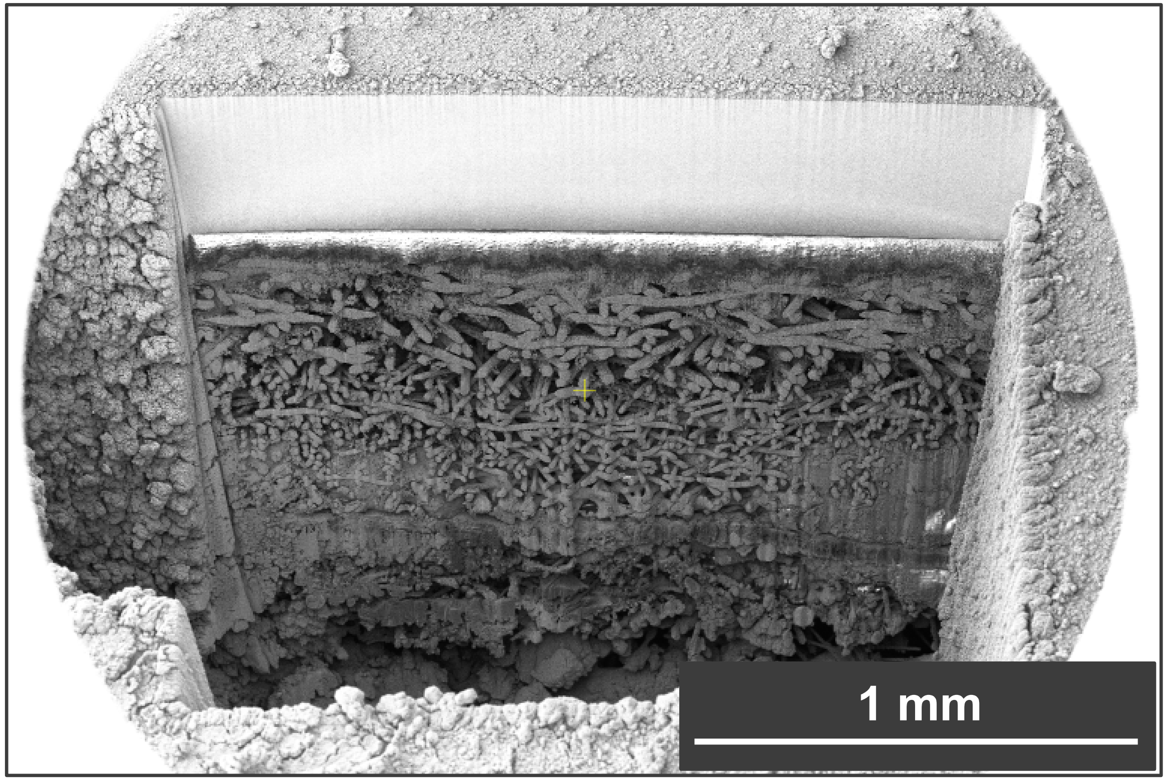
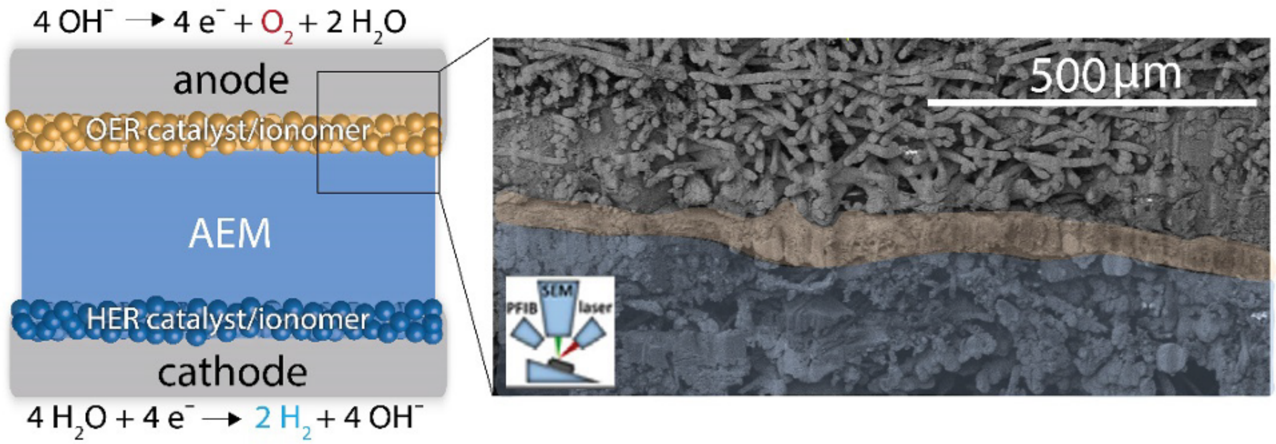
Electrolyser Samples

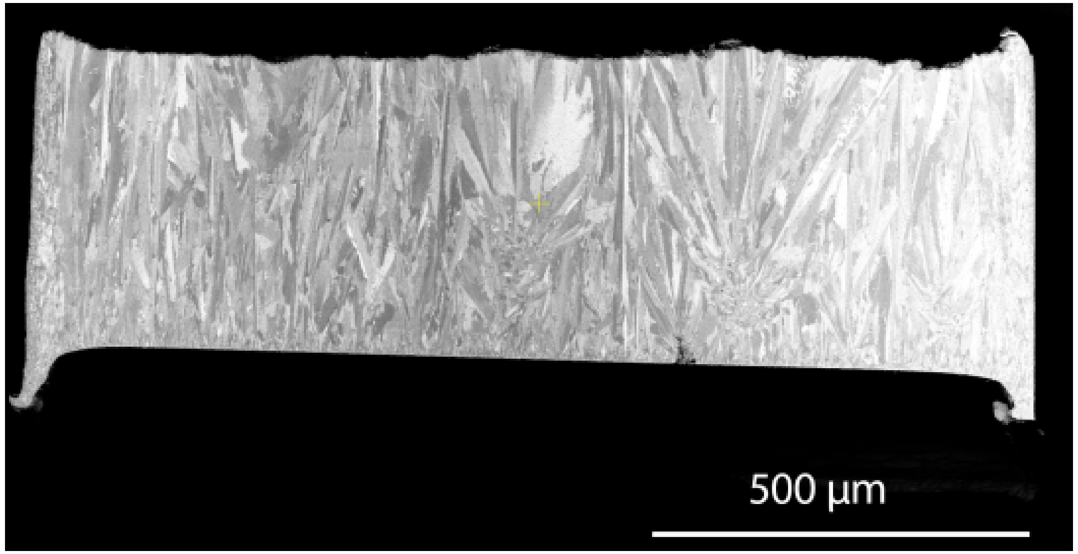




- Can distinctly image various layers in AEM (anion exchange membrane) electrolyser devices
- Challenging to reach layers more than 1mm below surface of the sample
  - Dynamic focus not effective across such large z values
  - Material in front blocks view
  - EDXS shadowing
- 515 nm clean up with laser after 1030 nm bulk trenching

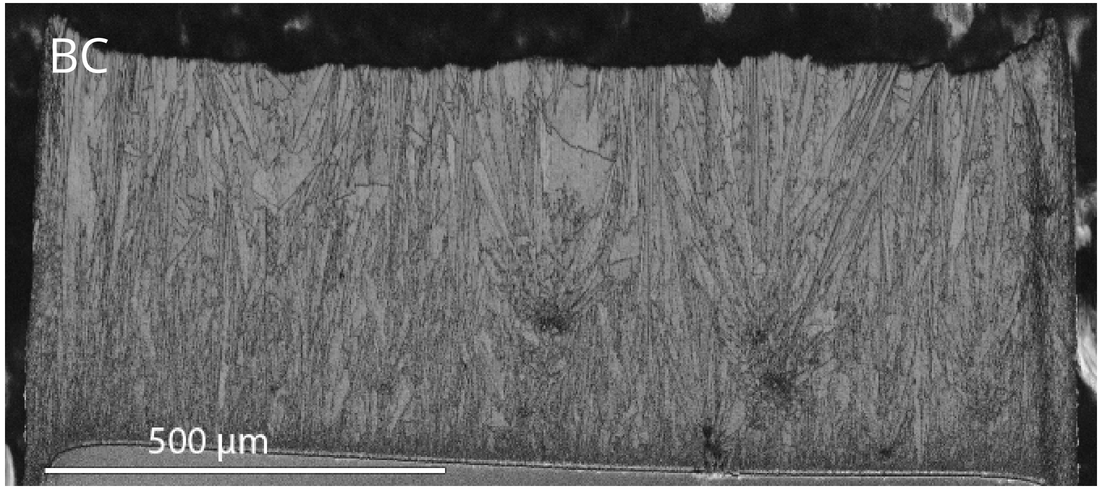
Clean-up steps sample dependent

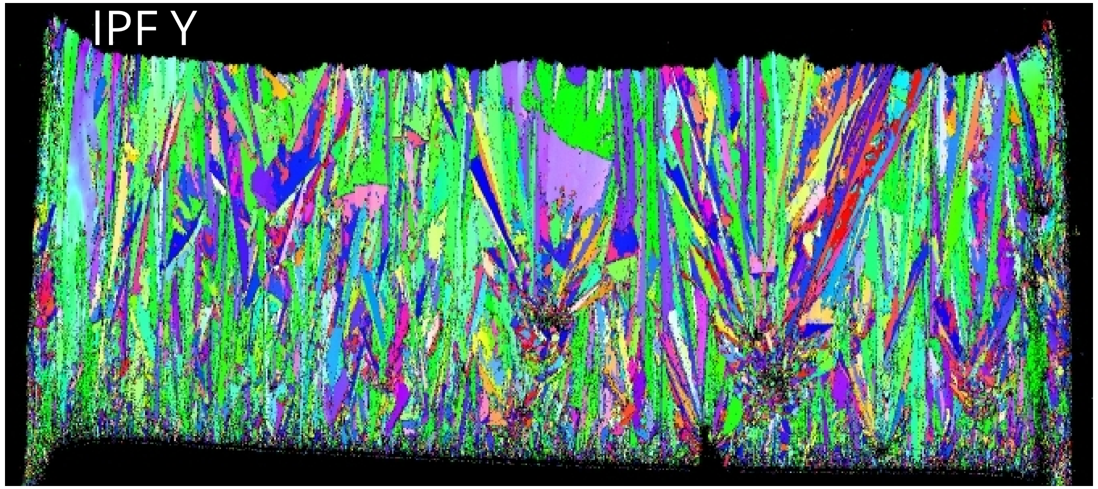
Laser prepared cross section of electroplated Pb



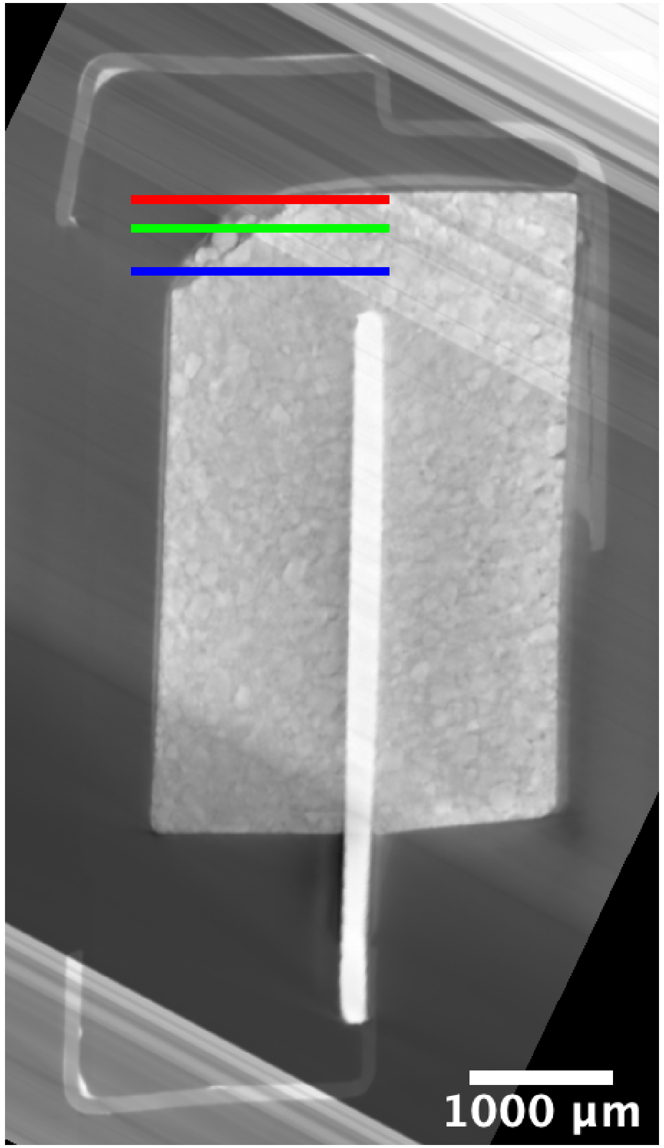
Total milling time was about 5 minutes at room temperature using 1030 nm laser wavelength.

Laser prepared surface great for imaging or EBSD directly with no further preparation!

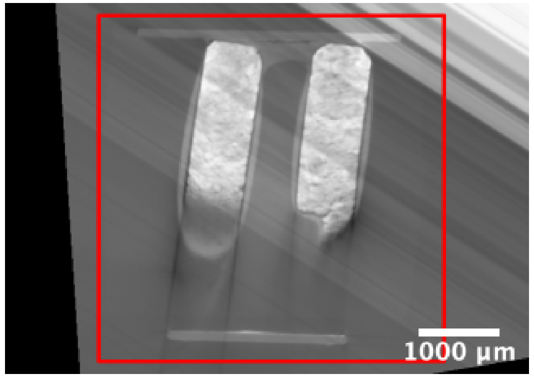


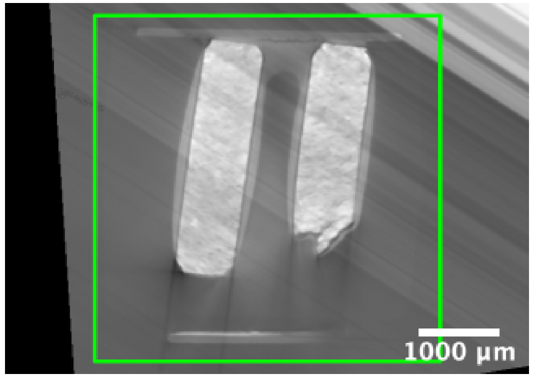


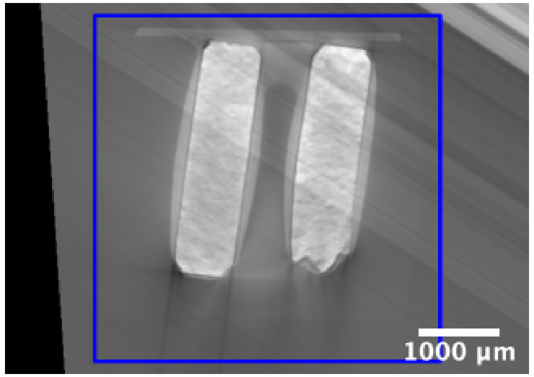
Ta Capacitor Failure



- Failed Ta capacitor was investigated with microCT which identified multiple regions of further interest
- To address this gap, automated 3D serial sectioning with the laser necessary



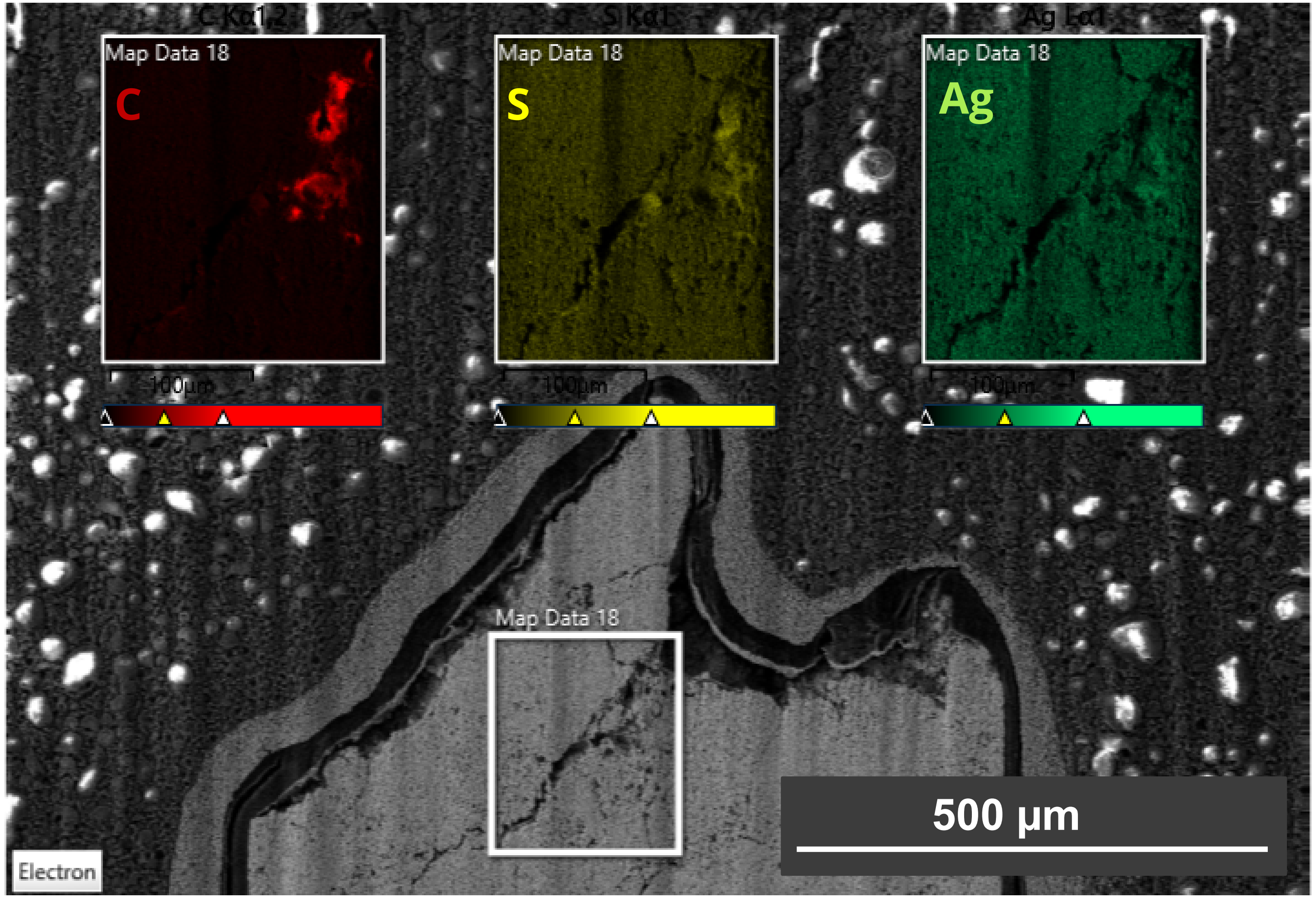




Ta Capacitor Failure

- Automated routine developed using iFAST scripting on laser PFIB
- Max width approx. 1.5 mm, so multiple slices were taken and resulting images stitched together to obtain large field of view
- 2 micron slice thickness, ~400 nm resolution in plane, 720 µm total sectioning depth

EDXS at Various Slices

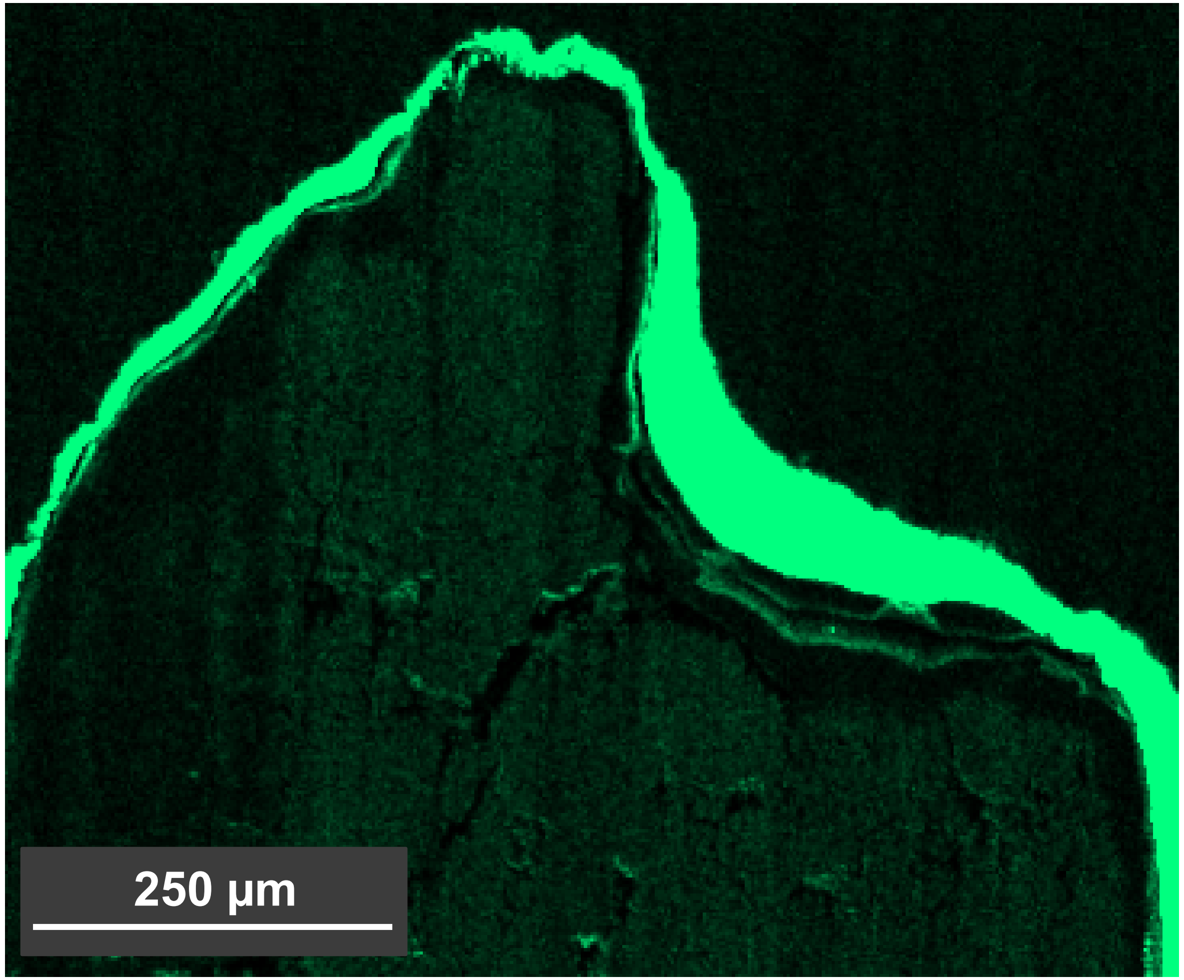


- With iFast scripting, not able to get automated EDXS data collection
- Arrested automation at various slices within regions of interest identified via microCT
- Currently working on Python code to automate this step
- In slice shown here, silver and sulfur found in one of the prominent cracks – potentially a melted region

Silver Incursions

- Noted silver incursions just after cutting through the silver and PEDOT layers
- May be related to failure mechanism
- Attempting again on separate failed capacitor with different CT results and Python automated scripting

Ag, slice 210



Summary and Acknowledgements

- **Still challenges to overcome with 3D automation - particularly EDXS and EBSD collection - and cryogenic operation**
- **Initial efforts on batteries and Ta capacitors show key nano/microscale compositional details related to failure**
- **Laser PFIB, particularly coupled with cryogenic capabilities, offers cutting-edge insight into electronic failure mechanisms**

Acknowledgements

Dennis Nelson, John Witham, Jon Bock, and Clif Aldridge for analysis of capacitor failure

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Thank you!

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