In-Situ Combination of TOF-SIMS and EDS Analysis During FIB Sectioning

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9\textsuperscript{th} Annual FIB-SEM Workshop, John Hopkins APL, Laurel MD
Outline

- Tescan GAIA @ AIM Lab, UMD Nanocenter
- FIB-STEM holder for liftout grids
- FIB-ORTHO, FIB, and SEM positions
- EDS mapping on thinned sample
- TOF-SIMS mapping prior to final thinning
From Mount Olympus in Greece...

“Gaia was … the primal Greek Mother Goddess; creator and giver of birth to the Earth and all the Universe; the heavenly gods, the Titans, and the Giants were born to her. The gods reigning over their classical pantheon were born from her union with Uranus, while the sea-gods were born from her union with Pontus.”

Wikipedia, 02/15/2016
...by the way of Brno, Czech Republic...
... to UMD Nanocenter AIM Lab
High Resolution Analytical TEM

Model: JEOL 2100 LaB₆ TEM
Electron Gun: LaB₆
Accelerating Voltage: 100 to 200 kV
Resolution: 0.23 nm (P-P)/0.14 nm (L)
EDS (Oxford)
Electron Diffraction: SAD, NBD, CBED
Cameras: CCD and TV Cameras

In-situ Experiments

Ultra High Resolution Analytical SEM

Model: Hitachi SU-70 UHR FEG-SEM
Electron Gun: Schottky Field Emission
Accelerating Voltage: 0.5 to 30 kV
Resolution: 1.0 nm (15 kV), 1.6 nm (1 kV)
Detectors: SEM and BSE;
EDS (Bruker)

Ultra High Resolution TEM/STEM

Model: JEOL 2100 FEG-TEM
Electron Gun: Schottky Field Emission
Accelerating Voltage: 100 to 200 kV
Resolution: 0.194 nm (P-P)/0.14 nm (L)
Electron Diffraction: SAD, NBD, CBED
Cameras: two CCDs
EDS (Oxford)
EELS and Energy Filter TEM (EFTEM, Gatan)

In-situ Experiments

Gallium FIB/SEM (GAIA)

Electron Gun: Schottky Field Emission
Accelerating Voltage: 0.2 ~ 30 kV
Resolution: 1.0 nm (15 kV), 1.8 nm (1 kV)
Ion Gun: Ga Liquid Metal Ion Source
Accelerating Voltage: 0.5 ~ 30 kV
Resolution: < 2.5 nm (30 kV)
STEM, EDS, EBSD, TOF-SIMS, OP-200, 5-GIS, Peltier Stage, Low-Vac

Xenon FIB/SEM (XEIA)

Electron Gun: Schottky Field Emission
Accelerating Voltage: 0.2 ~ 30 kV
Resolution: 1.0 nm (15 kV), 1.6 nm (1 kV)
Ion Gun: Xe Plasma Ion Source
Accelerating Voltage: 3 ~ 30 kV
Resolution: 25 nm (30 kV)
EDS, CL, 5-GIS, Peltier stage, Low-Vac

Office

Vacuum Evaporator

Plasma Cleaner

Ion Mill

Optical Microscope

Carbon Evaporator

Fume Hood

Image Scanner

Laminar Flow Hood
GAIA-3 FIB/SEM

SFE, LMIS, Se-, R-BSe-, IB-Se-, IB-BSe- Si+, STEM, 2xEDS, EBSD, TOF-SIMS, 5-GIS, OP-200, STEM
FIB-STEM Holder

STEM Position

Rapid grid change for:

STEM DF/BF, HR Se-

STEM-EDS with EDS-1

2/19/2016  9th Annual FIB-SEM Workshop, John Hopkins APL, Laurel MD
FIB-STEM Holder Additional Positions

**FIB-ORTHO:**
Cutout, TOF-SIMS

**FIB:**
Thinning, EDS-2

**SEM:**
Lamella Attachment
FIB-STEM Holder

- STEM imaging on round and lift-out grids
- In addition to STEM imaging, the FIB-STEM holder could be positioned for:
  - SEM – attaching lamella to grid
  - FIB – thinning lamella, EDS with enhanced count
  - FIB-ORTHO – TOF-SIMS, lamella cut-out
- Change between any positions in under ~40 Sec
EDS on Porous Ceramic Lamella

Improved spatial resolution on thinned area, prior to final thinning to TEM transparency

EDS: Main Element

EDS: Inclusion
FIB / TOF-SIMS on Thick Lamella

Differences in Sr distribution visible in FIB/SIMS

No differences in Sr distribution detected by EDS

EDS: Sr
Summary

- Developed and demonstrated full-cycle TEM sample preparation process using Tescan FIB-STEM holder
- Utilized increased X-Ray counts in FIB position for high-resolution EDS mapping on thinned lamella
- Utilized higher efficiency of secondary ion collection in FIB-ORTHO position for detecting variation in Strontium distribution near ceramic boundary by in-situ TOF-SIMS
Acknowledgements

Support of University of Maryland NanoCenter and its AIM Lab

Image of STEM-EDS of Solid Electrolyte Fuel Cell provided by Joshua Taillon

Instrumentation slide provided by Director of AIM Lab, Dr. Wen-An Chiou

Challenging sample for FIB/SIMS & EDS provided by Wachsman’s ceramic anode development team:

- Prof. Eric D. Wachsman
- Dr. Mohammed Hussain Abdul Jabbar
- Ke-Ji Pan