Advanced Microscopy in the Study of Tracks in Natural and Synthesized Quarts

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Michigan Ion Beam Laboratory

NSF

MICHIGAN ENGINEERING
MICHIGAN CENTER FOR MATERIALS CHARACTERIZATION

GORDON AND BETTY MOORE FOUNDATION

Outline

- ❑ Why quartz for Dark Matter detection?
- ❑ Strategy for imaging ion tracks using TEM
- ❑ Preliminary data from some natural quartz samples
- ❑ Summary and future-plan
- ❑ New facilities in $(MC)^2$ @ Umich

Mineral Selections in Michigan for DM Detection

❑ Selected natural olivine as the first MD:

- ➢ Advised by Prof. Rod Ewing (Standford) and Jackie Li (Umich)
- ➢ Purchased three pieces of natural olivine (peridot) from two companies from Pakistan during a Michigan mineral show.

❑ Selection of quartz:

- ➢ **Very abundant in the earth**
- ➢ Ebadi et al. (2021) propose using old quartz as large exposure detectors for ***ultra-heavy dark matter*** and note “the age of geological quartz compensates for the low number density of UHDMs, and the distinct geometry of the damage track serves as a high-fidelity background rejection tool.”
- ➢ I have some synthetic single crystal quartz wafers in hands.

Synthetic Single Crystalline Quartz

- ➢ Hydrothermally synthesized High purity (optical grade) single crystal quartz wafers from Vritra Technologies
- ➢ 5inch x 0.5mm double-side polished

Target Mineral DM Detector Candidates Proposed by Prof. Bodnar

Mineral	1	2	3	4	5	6	7	8
Diamond (AM)	✓	✓	✓	✓	✓	✓	✓	✓
Olivine (RFM)	✓	?	✓	✓	✓	✓	✓	✓/x
Zircon (AM)	✓	?	?	✓	✓	✓	✓	x
Muscovite (RFM)	✓	X	✓	✓	?	✓	✓	
Halite (RFM)	✓	✓	✓	✓	✓	✓	?	(H_2O soluble)
Quartz (RFM)	✓	✓	✓	✓	✓	✓	✓	✓
Corundum (AM)	✓	✓	?	✓	✓	?	✓	
Apatite (AM)	✓	✓	?	?	✓	✓	✓	
Monazite (AM)	X	?	?	?	✓	?	✓	
Zirconia (AM, rare)	X	✓	X	✓	?	?	X	

1. Natural samples are relatively abundant and easily obtained
2. Synthetic samples available or can be made
3. Sufficient quantities of samples from ≥1 km beneath the surface can be obtained
4. Low U & Th samples available
5. Radiation damage behavior relatively well known
6. Preliminary studies related to radiation damage have been conducted
7. "Old" samples are available
8. Simple in structure or composition

RFM = Rock-forming mineral (i.e., it is abundant and common); AM = Accessory mineral (i.e., less common)

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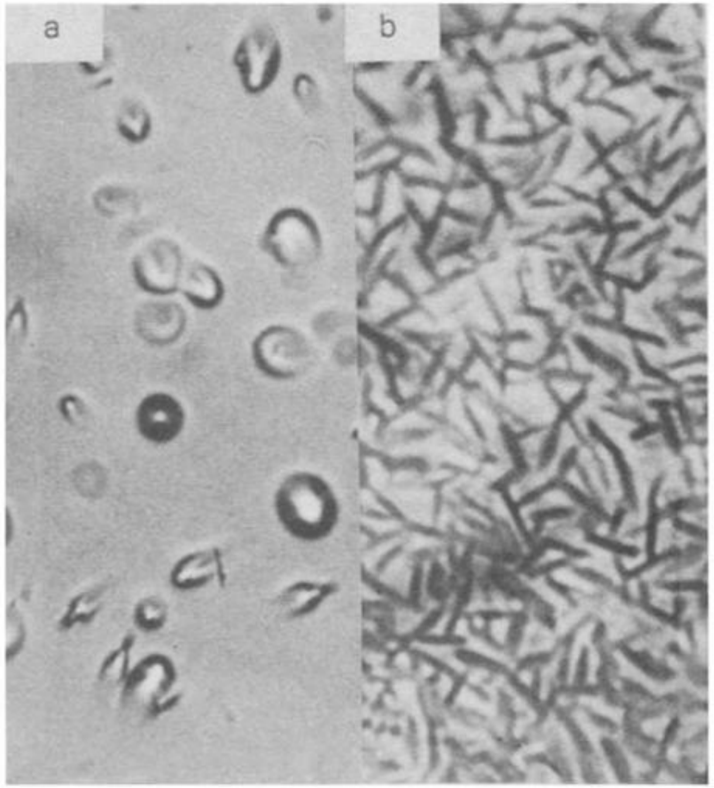
Previous Studies on Fission Tracks in Quartz



S. Koul et al. in 1991: Chemical etching

Fig. 3 Fission tracks in (a) amorphous, (b) crystalline SiO_2 ; both etched.

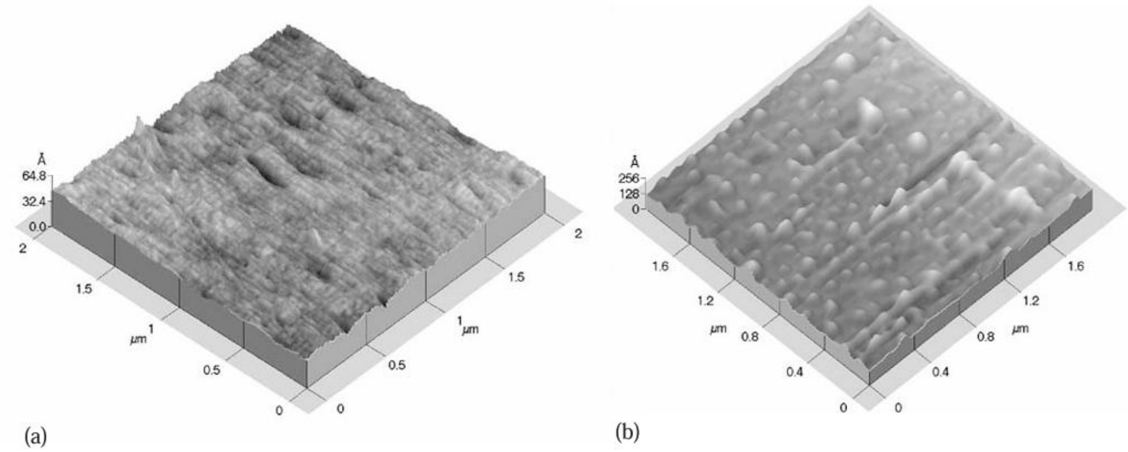
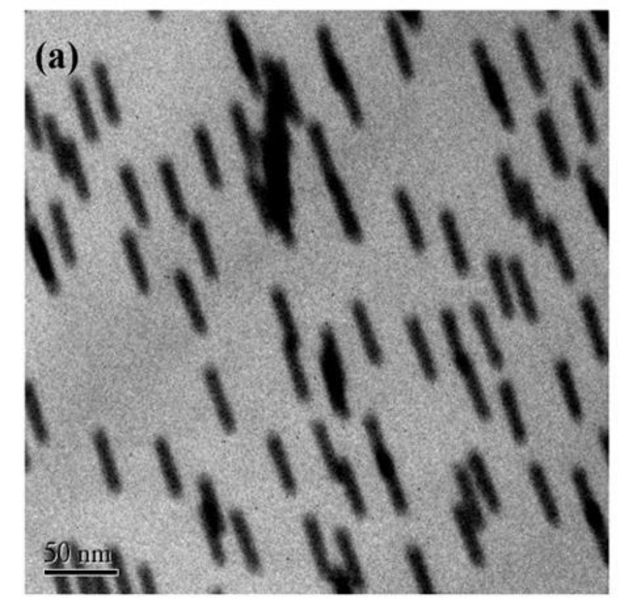


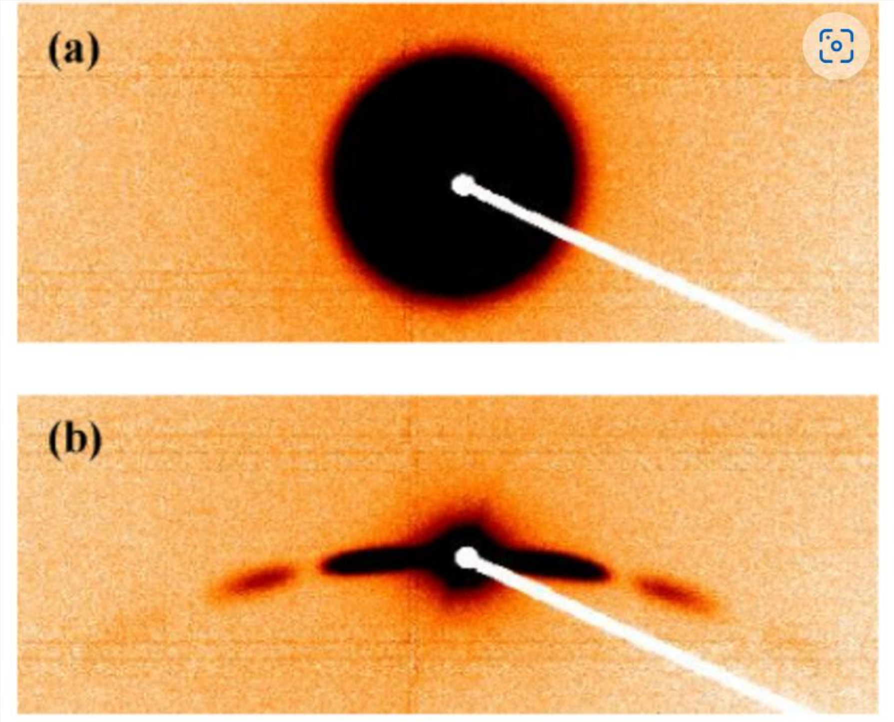
Fig. 2. Microphotography obtained by the AFM, showing the surface topography of the crystalline quartz: (a) before exposure to radiation, and (b) after being exposed to fission fragments source.

- Chemical etching was used +Optical or AFM imaging
- Advantage: large area sampling and imaging the same tracks at different depths with well controlled etching and positioning..

Previous Studies on Ion Tracks in Quartz



- Plan-view TEM image of ion tracks in quartz generated with 1 GeV Pb ions of fluence 5×10^{10} ions cm^{-2}. Ion tracks were tilted with respect to the electron beam.
- TEM specimen was prepared by traditional mechanical grinding, polishing and broad beam ion milling.

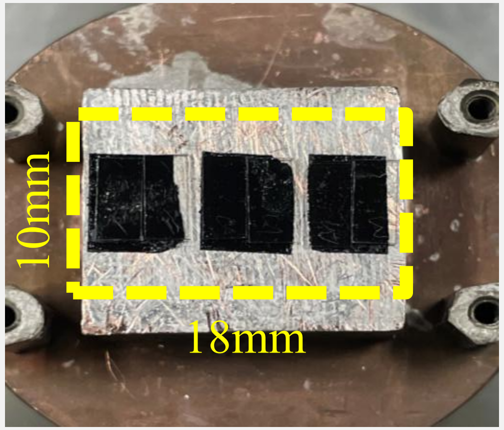


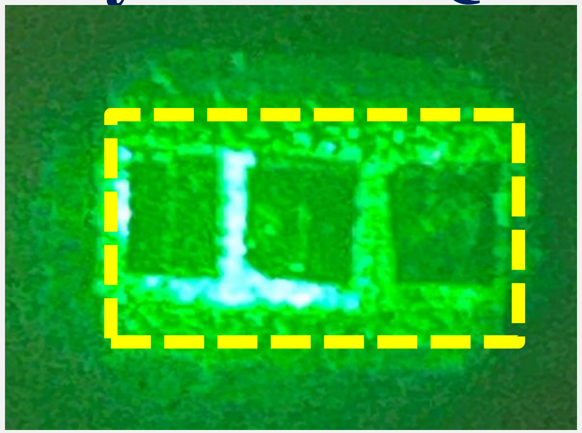
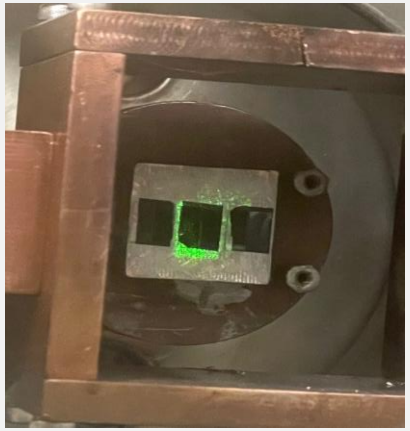
SAXS images of a quartz sample irradiated by 1.6 GeV Au ions to a fluence of 5×10^{10} ions cm^{-2}, (a) with the x-ray beam parallel to the ion tracks, (b) with the ion tracks tilted by 5° with respect to the x-ray beam.

B Afra *et al*. SAXS investigations of the morphology of swift heavy ion tracks in α-quartz, 2013 *J. Phys.: Condens. Matter* **25** 045006

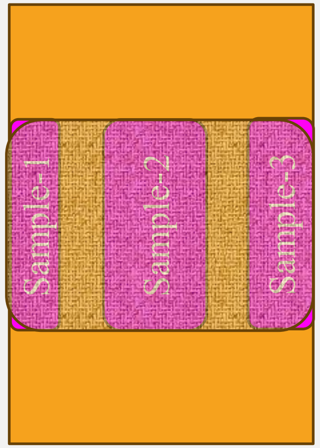
15 MeV Au ion Irradiation to Synthetic Quartz

- **Ion beam parameters**: 15 MeV Au^{5+} ion beam with current of 5nA (~ 6.25 x 10^9 Au^{5+}/s and a 180mm^2 raster area (~ a fluence of 3.47 x $10^9 Au^{5+}/cm^2 s$) at room temperature.
- Three groups of samples (~2mm x 5mm) were irradiated for one experiment with the samples loaded on the ion beam stage illustrated to the bottom right.

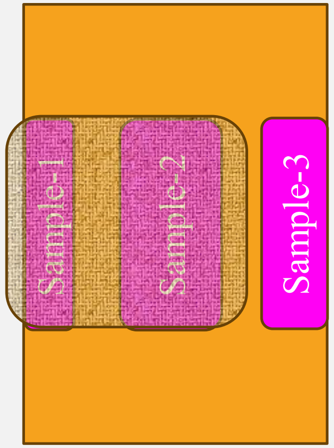


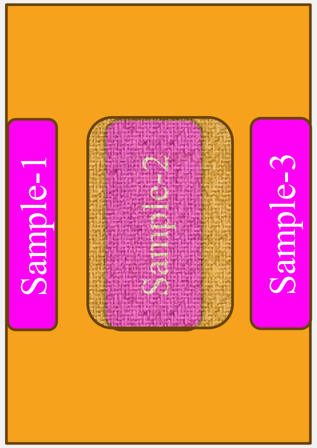
Samples on the ion beam stage



Sample-3: 5.21 x $10^{10} Au^{5+}/cm^2$



Sample-1: 1.04 x $10^{11} Au^{5+}/cm^2$

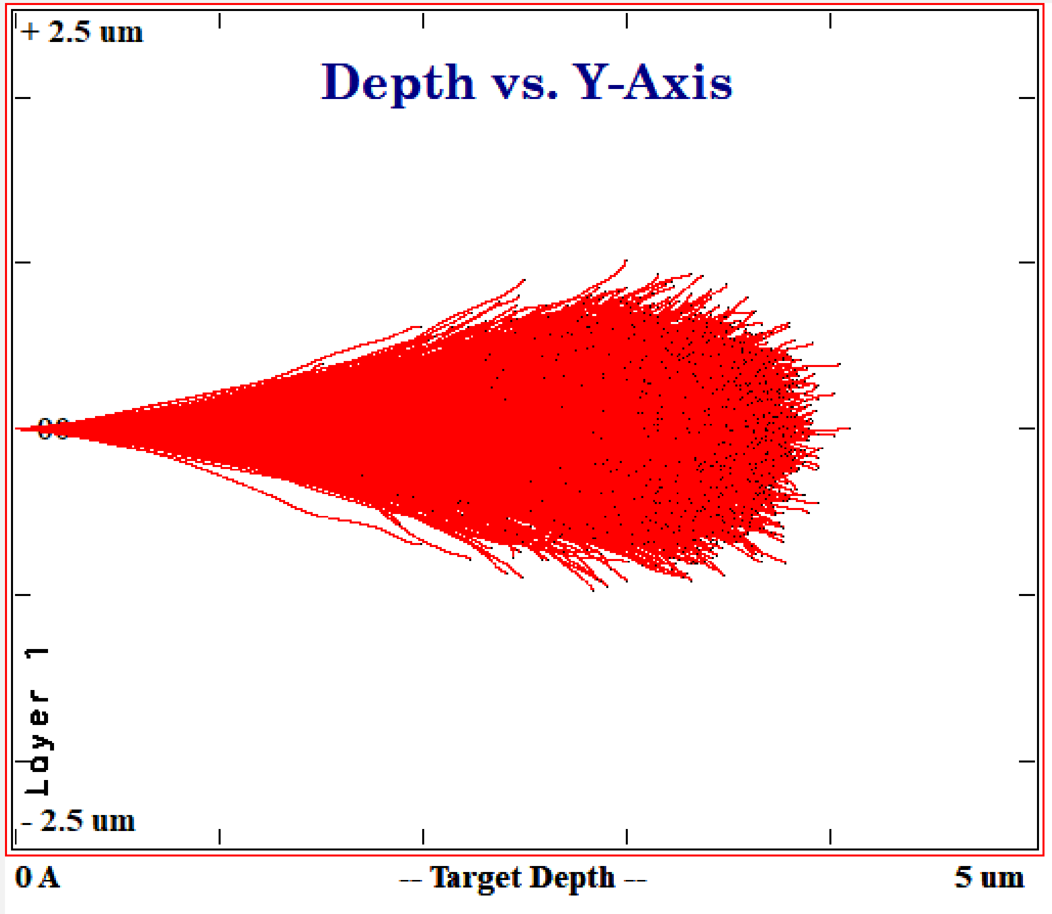


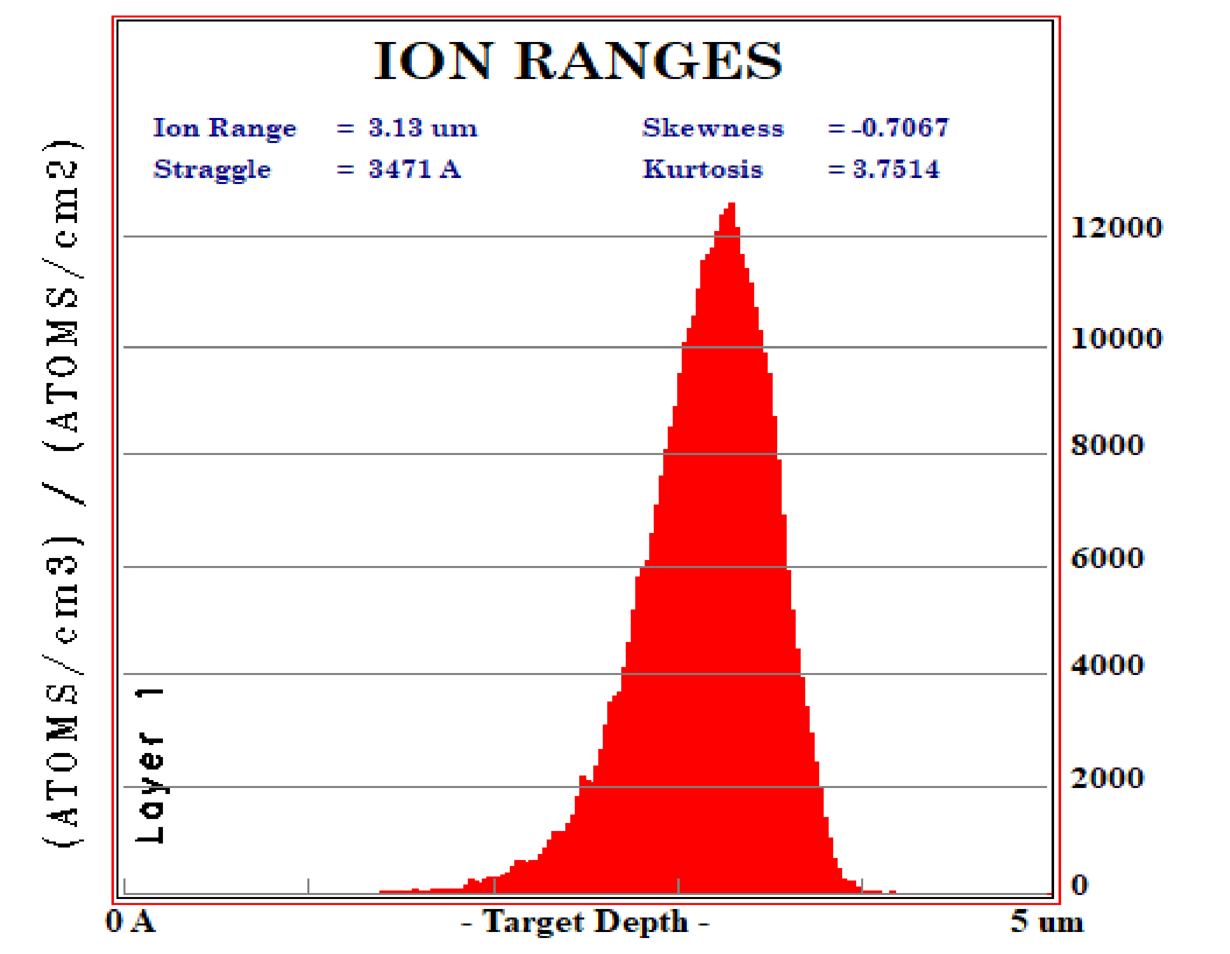
Sample-2: 1.56 x $10^{11} Au^{5+}/cm^2$

- **Time was controlled using iPhone clock with roughly ~1s error!**
- **To reach the ~10^6 ions/cm^2, only 1 μs is needed for such a beam current.**

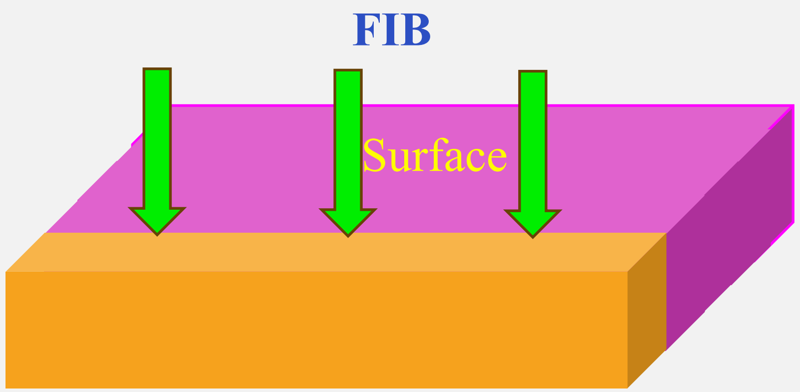
Au Ion Penetration Depth by SRIM Simulations

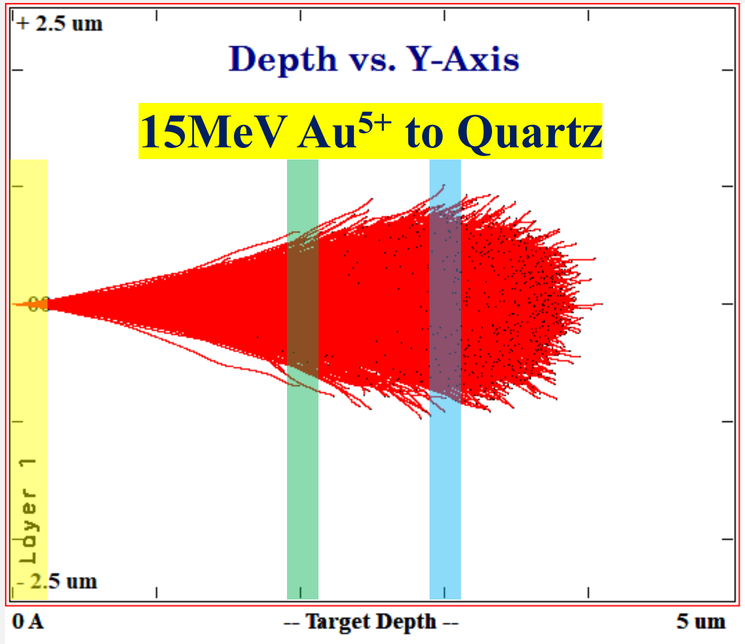
15MeV Au^{5+} to Quartz

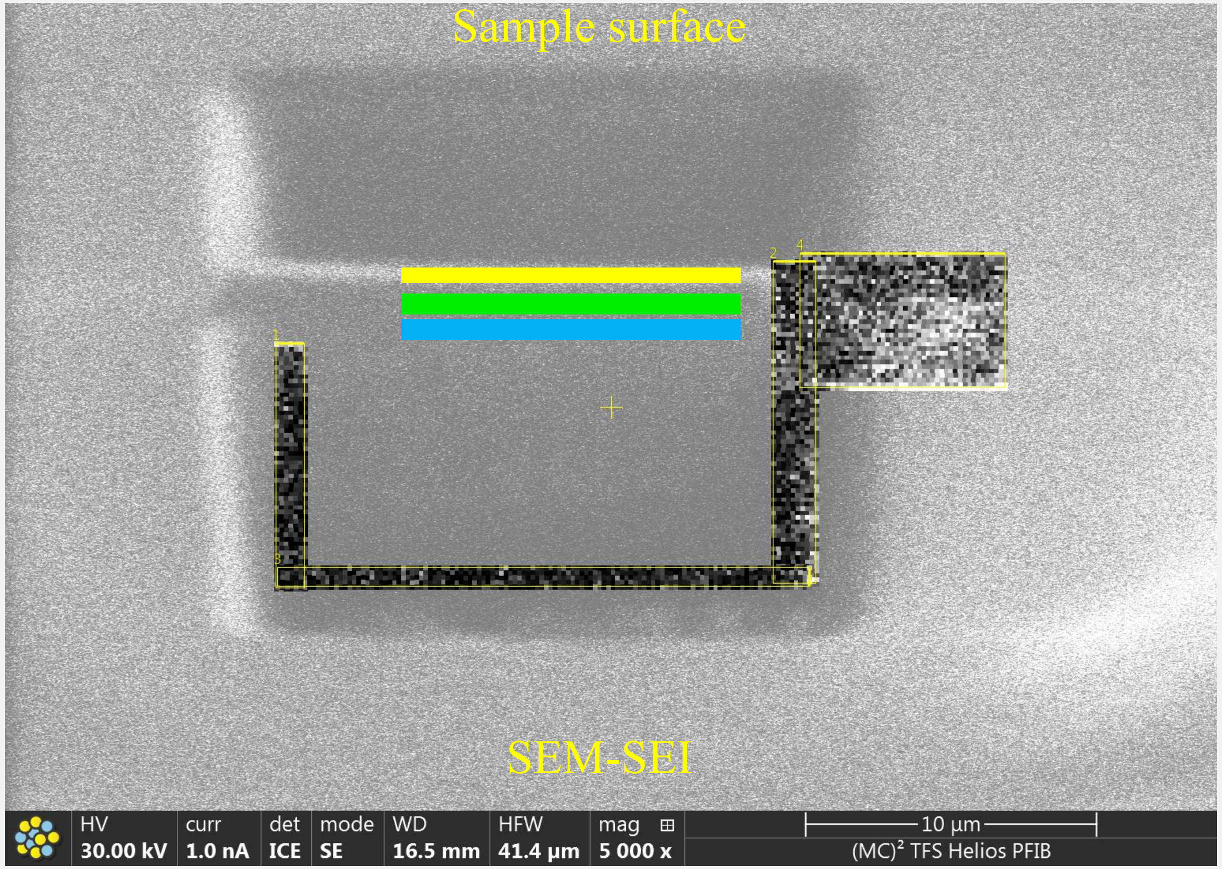




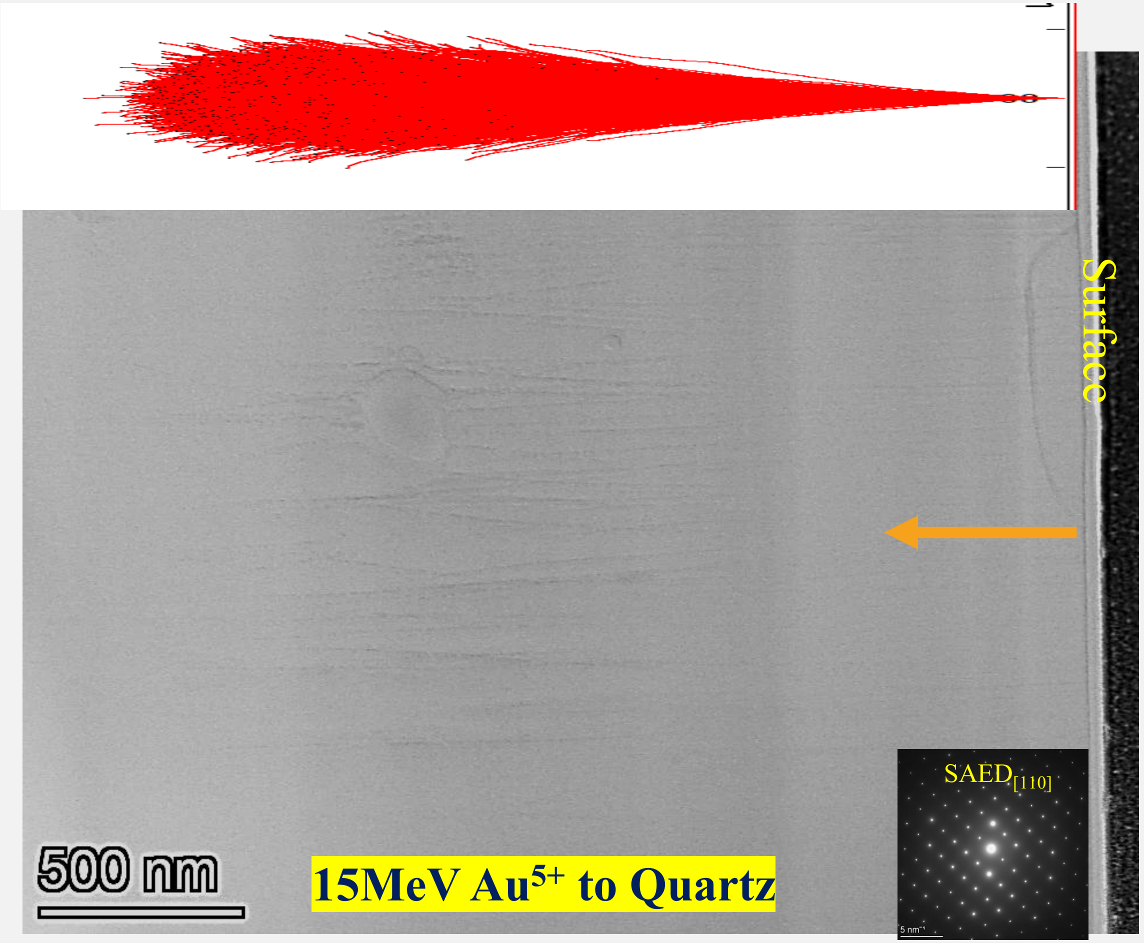
FIB Sampling: For Cross-sectional View Imaging



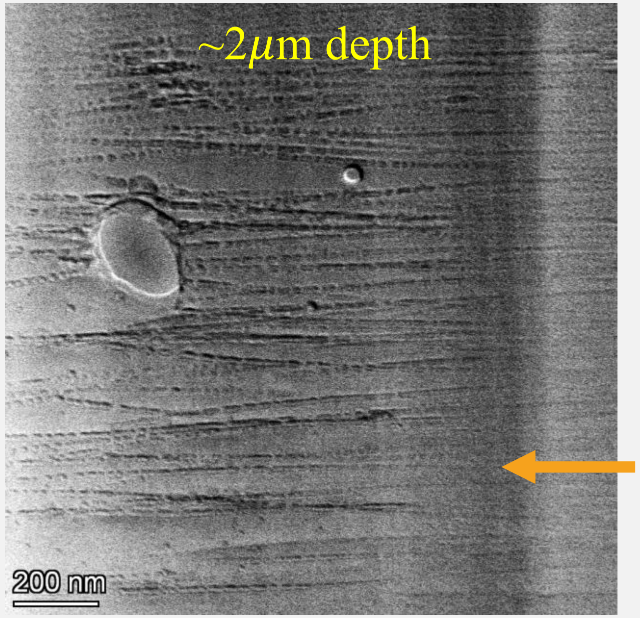


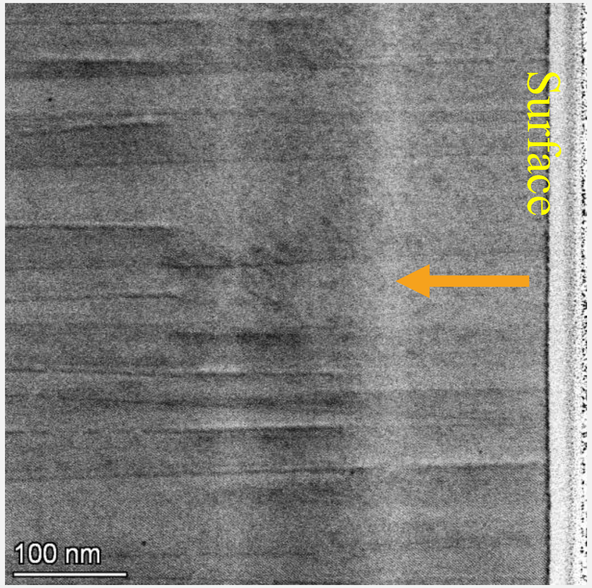


X-STEM Imaging of Au Ion Tracks in Synthetic Quartz



Au^{5+} ion beam direction



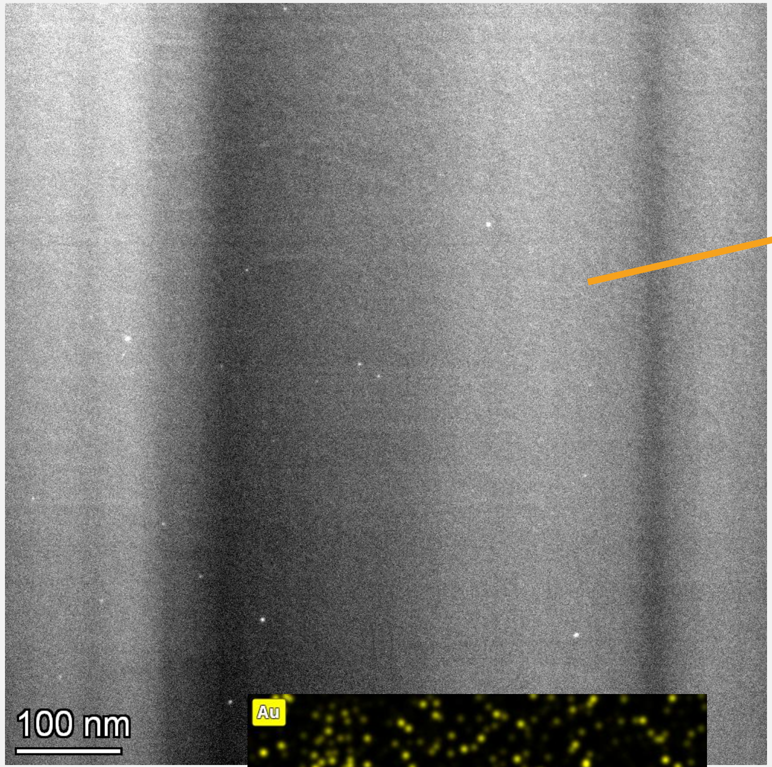


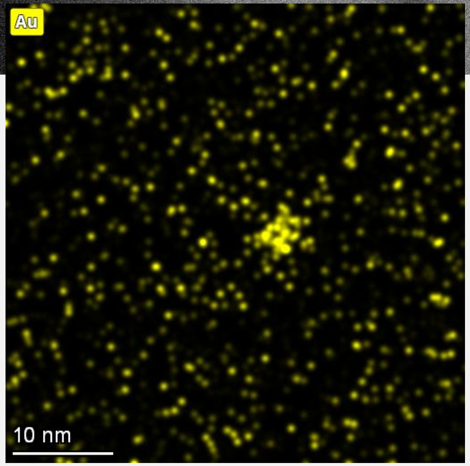
STEM-BF Images

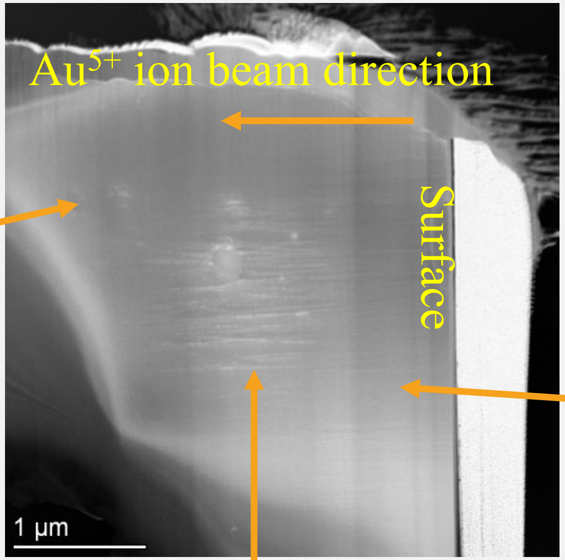
Sample-1: 1.04 x 10^{11}Au^{5+}/cm^2

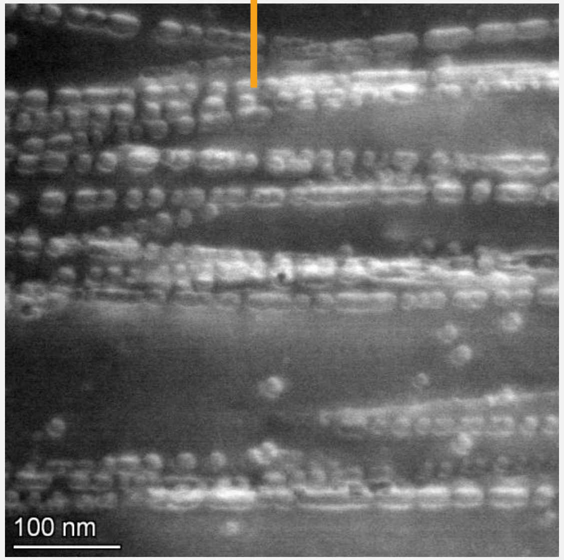
- Advantage: See the whole lengths of the tracks from the surface down to the ends
- Disadvantage: Too many tracks causing image overlaps

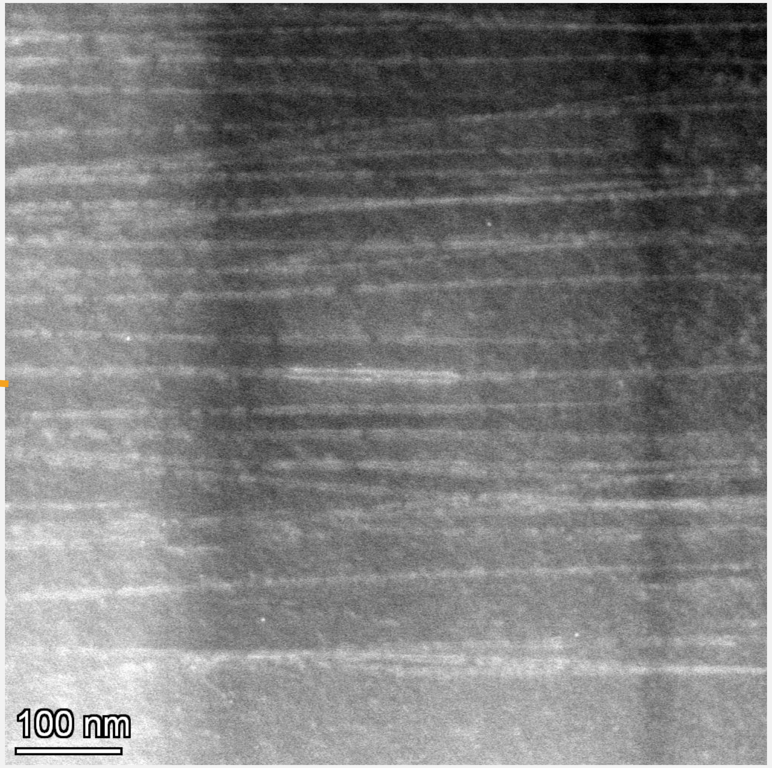
X-STEM Imaging of Au Ion Tracks in Synthetic Quartz







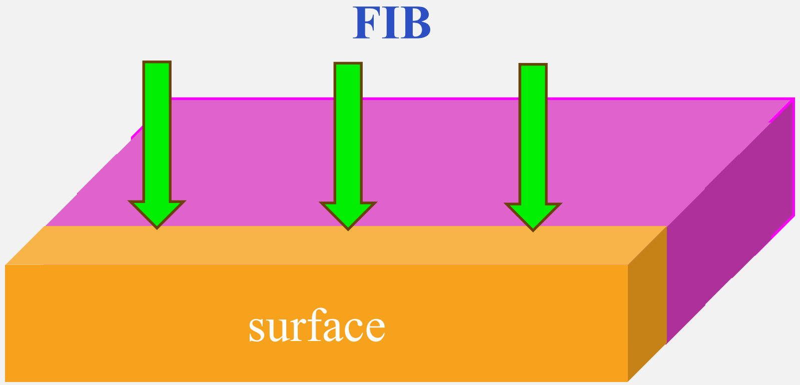


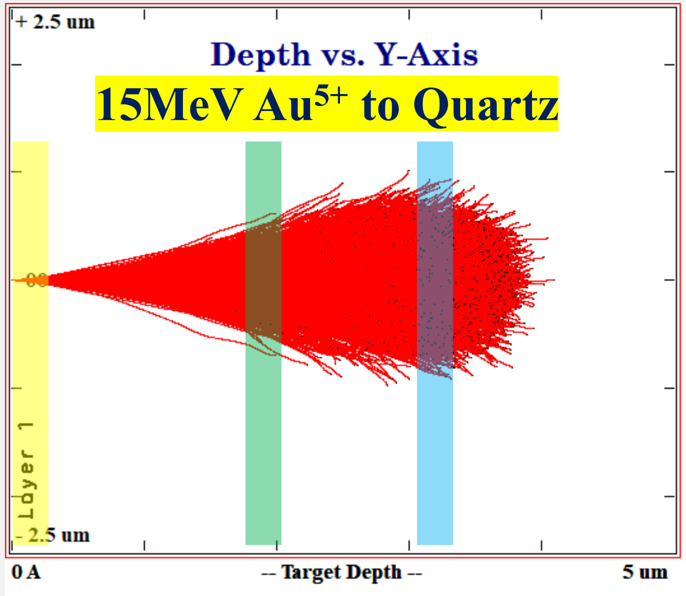


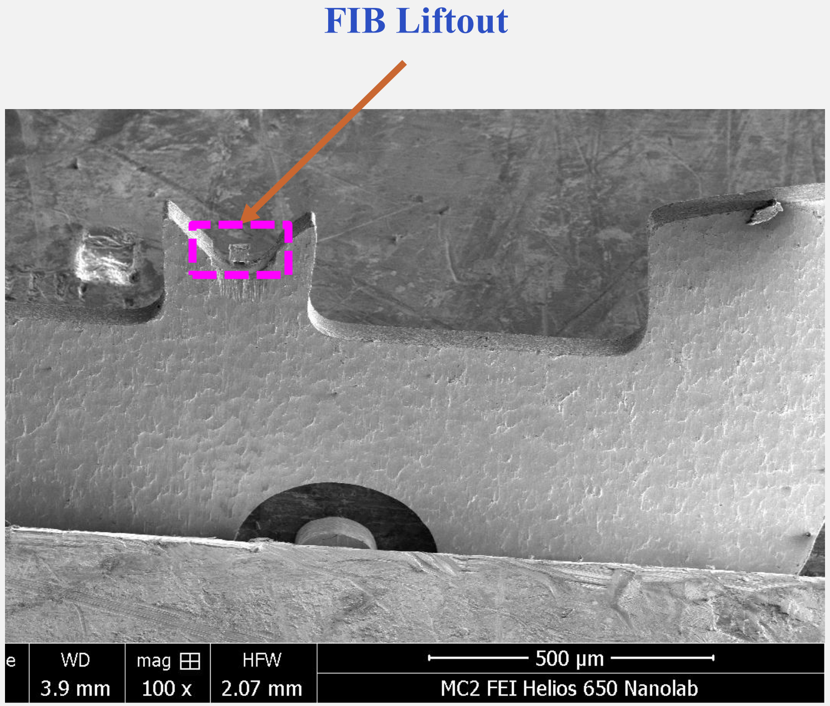
STEM-HAADF Images

- Au clusters can be seen
- Probably we could see U/Th in fission tracks

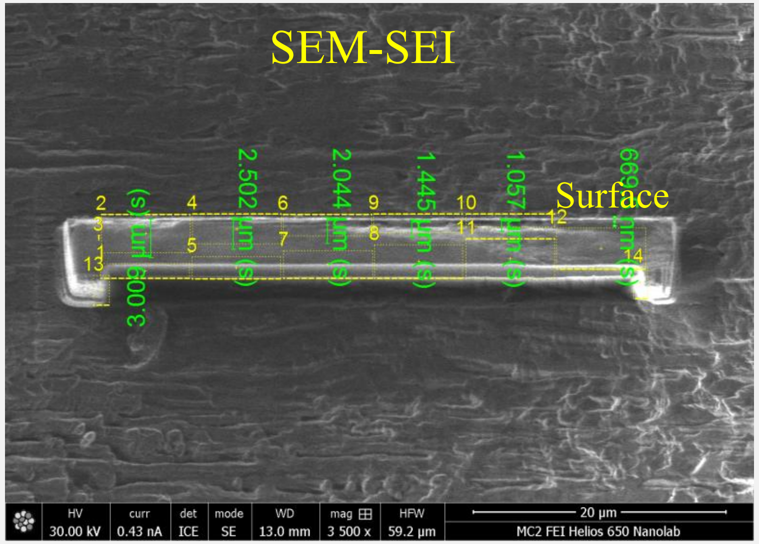
For Plan-view Imaging-Pseudo Volume Electron Microscopy

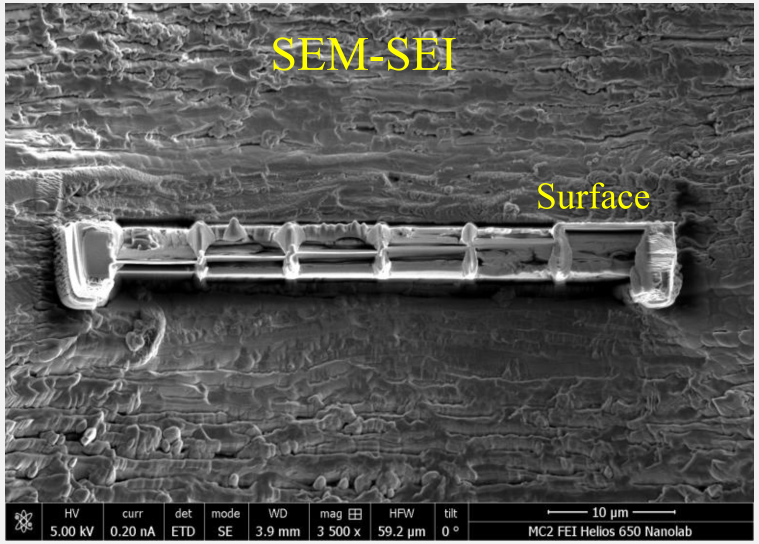






FIB grid





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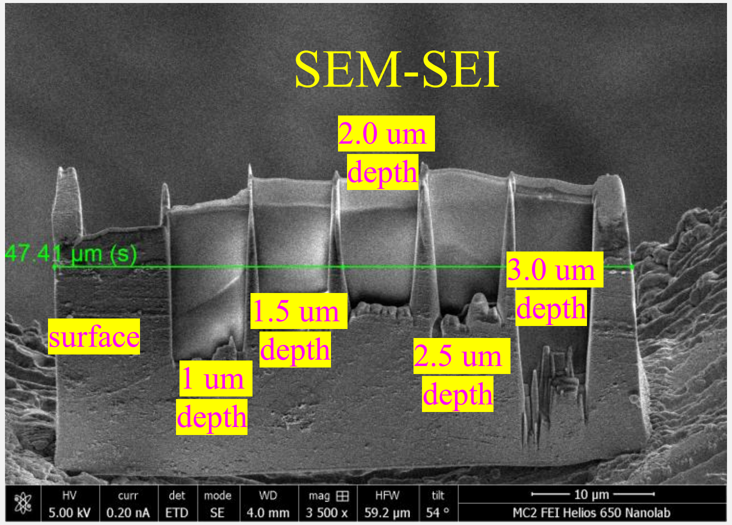
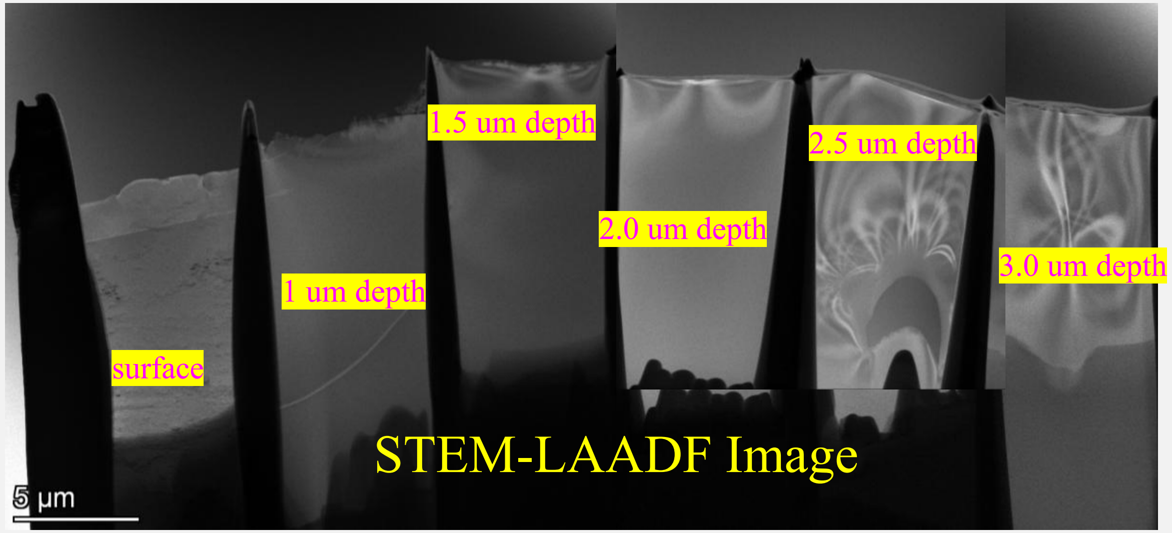
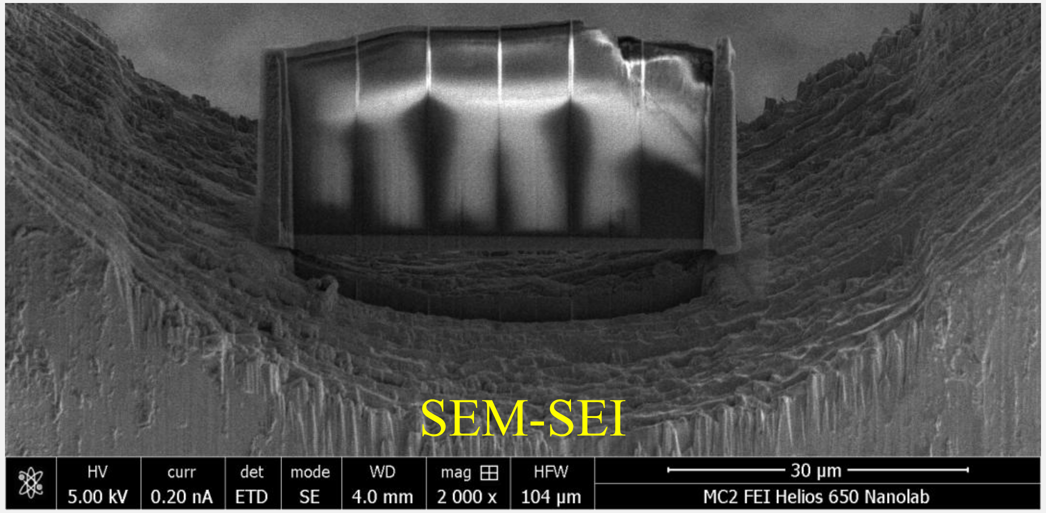
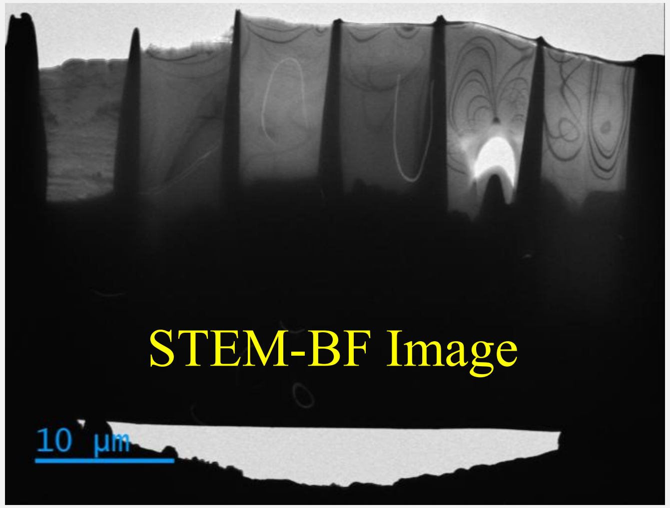
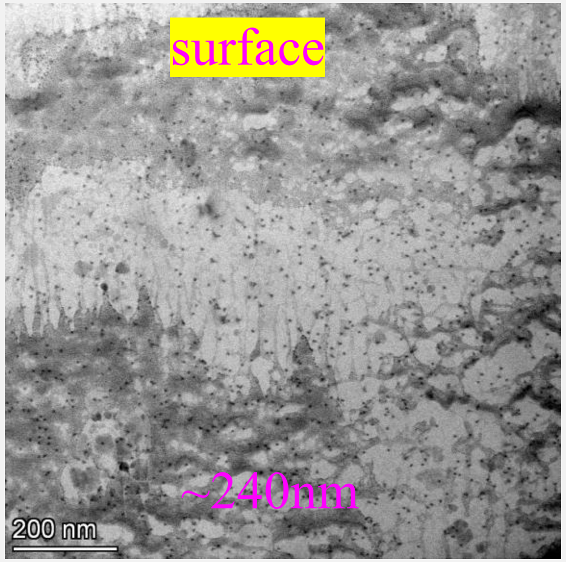


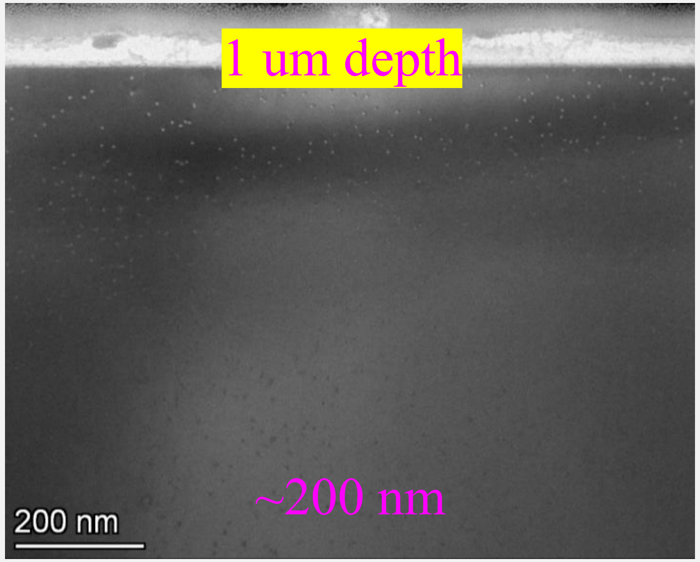


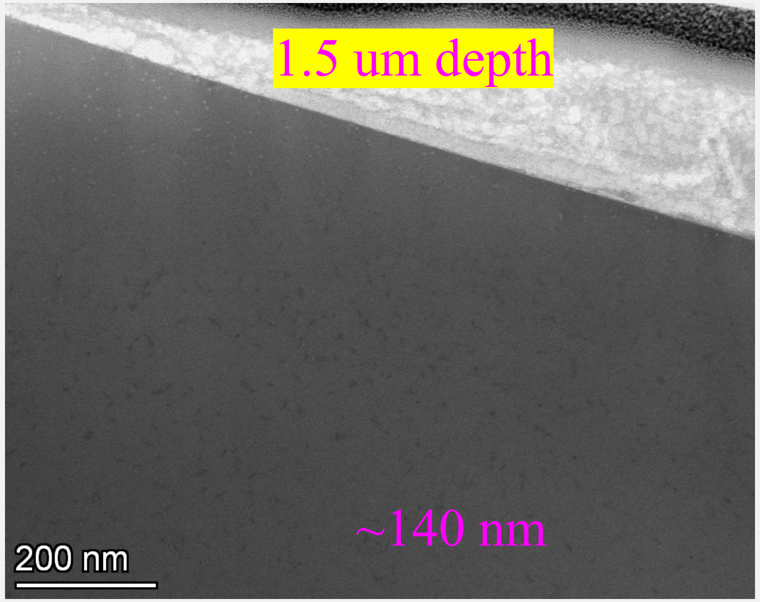


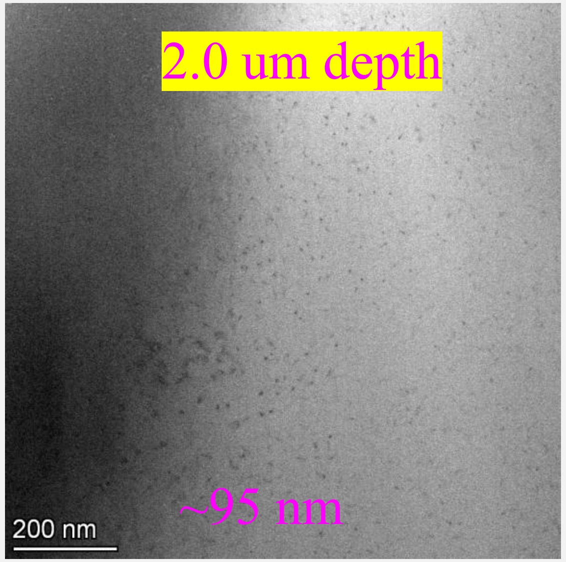


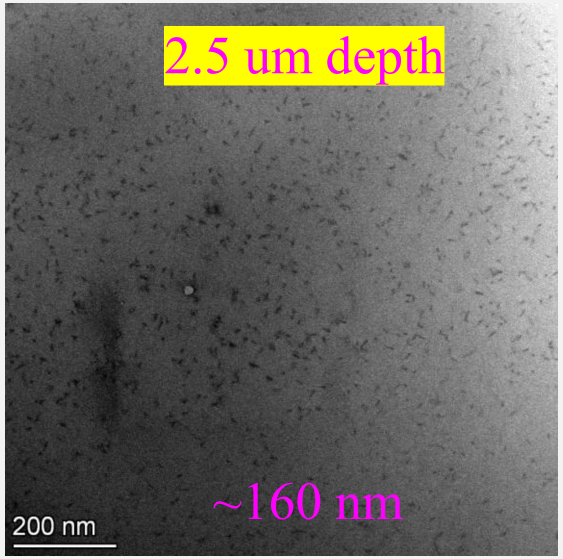
X-STEM Imaging of Au Ion Tracks in Quartz at Different Depths



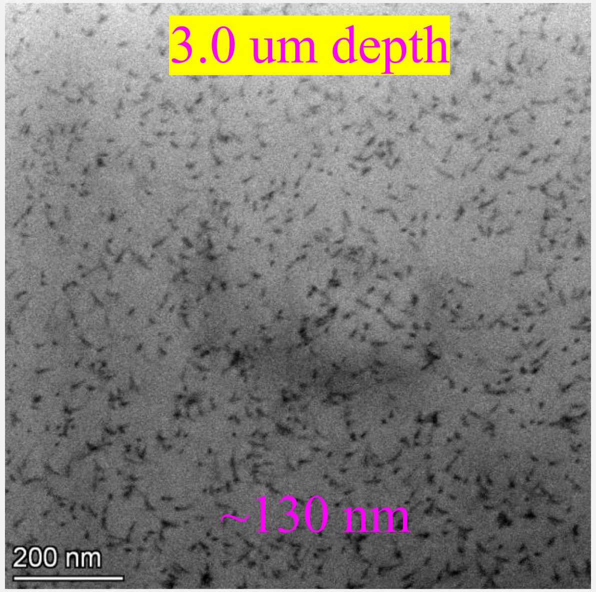




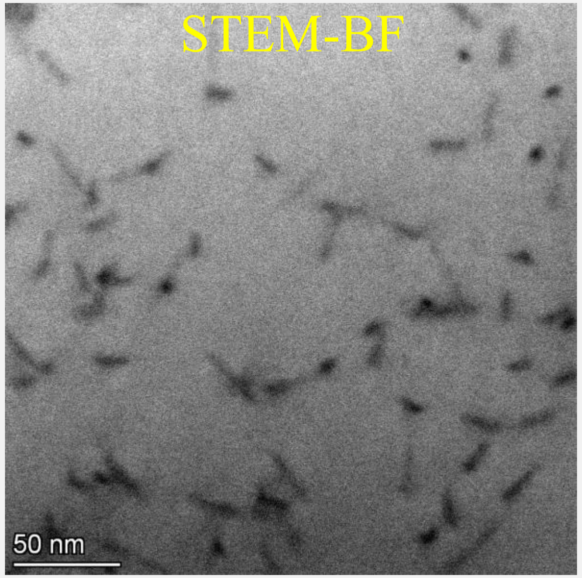


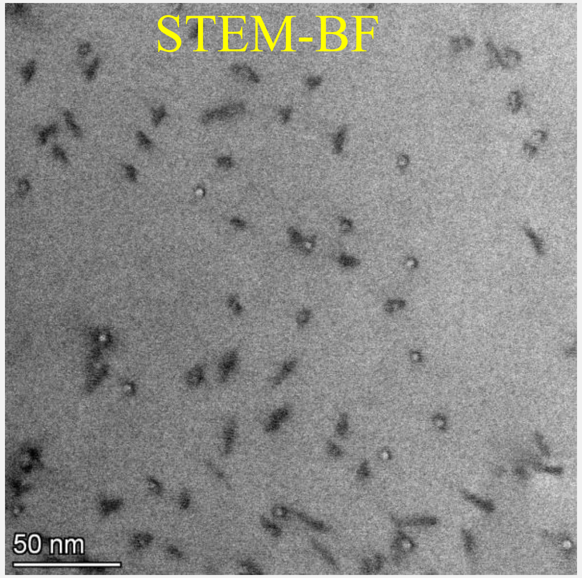


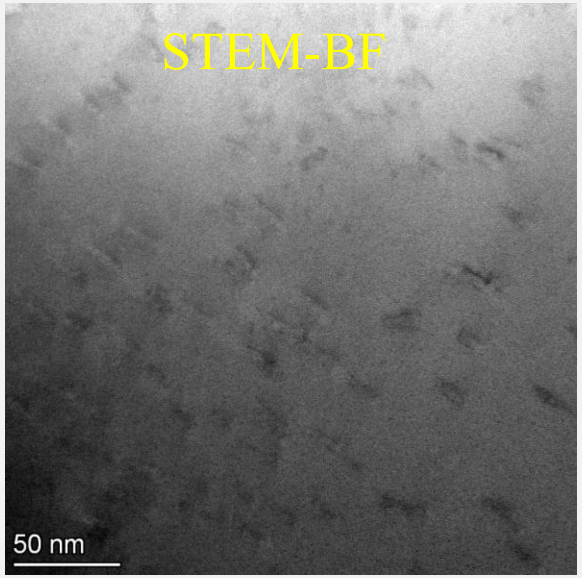
STEM-BF Image

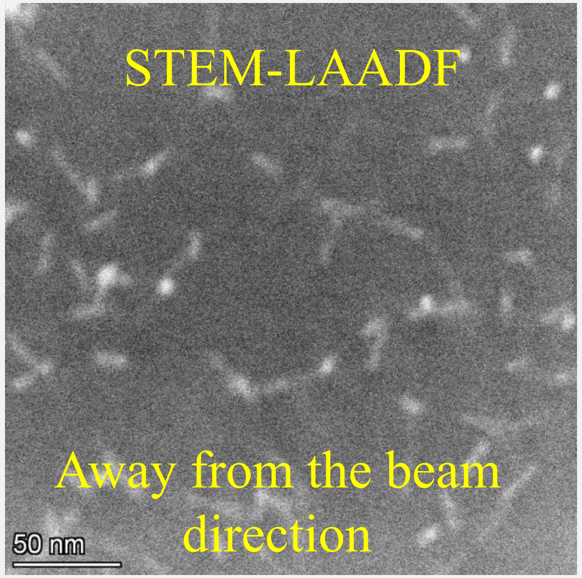


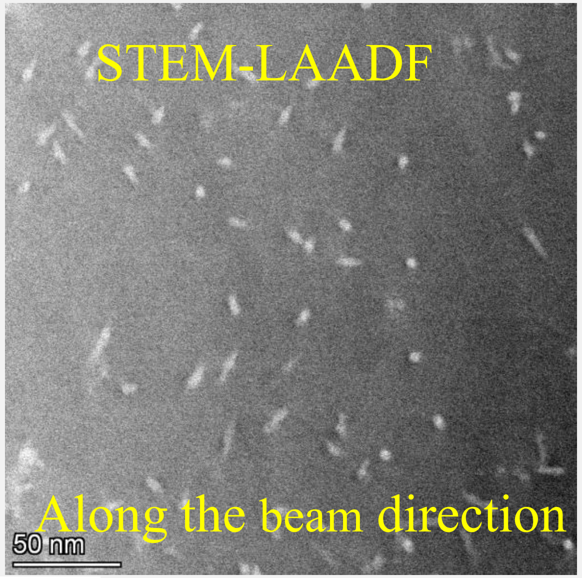
Au Ion Tracks in Synthetic Quartz Imaged at Different Orientations

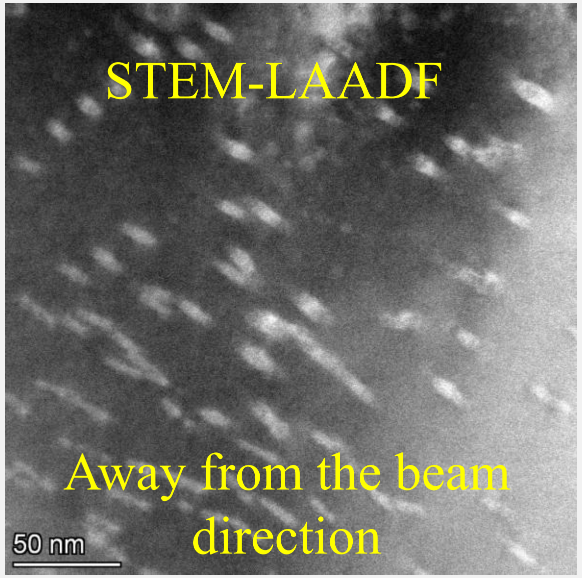






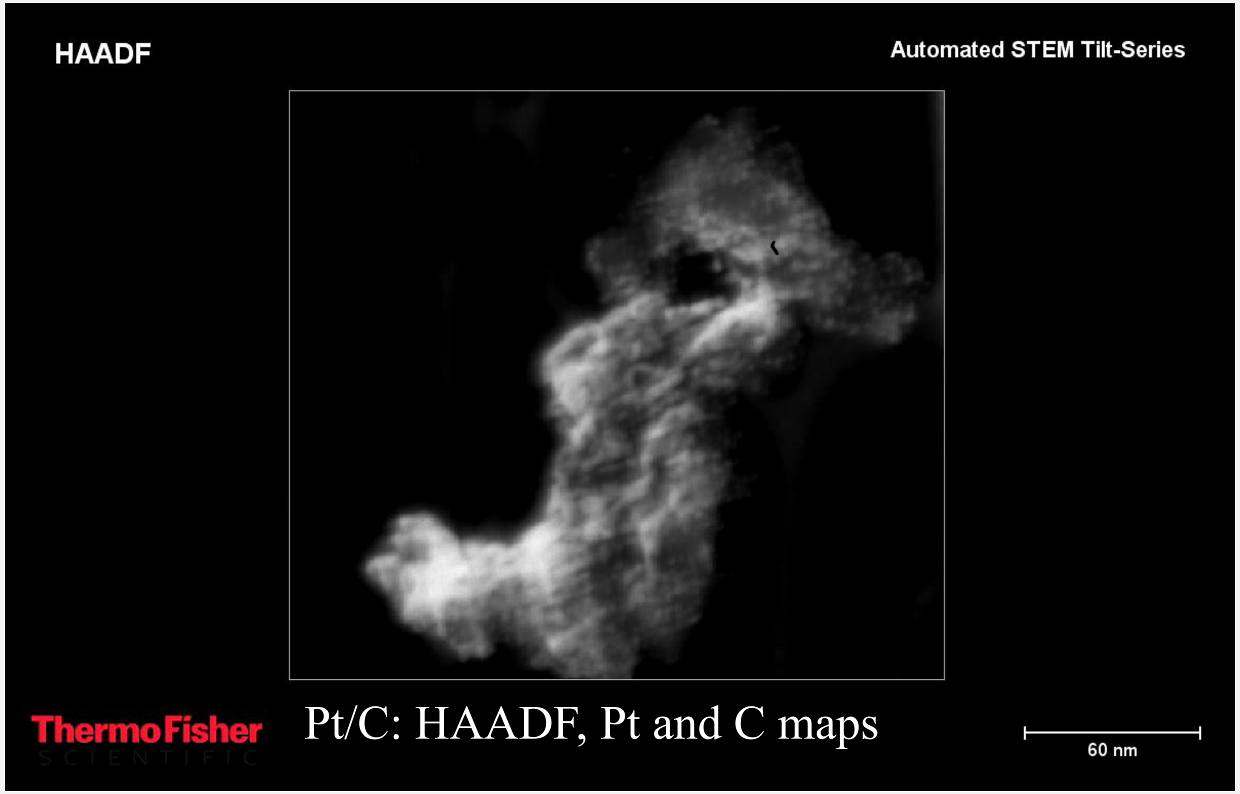






3D Electron Tomography and Stero-Electron Microsvopy

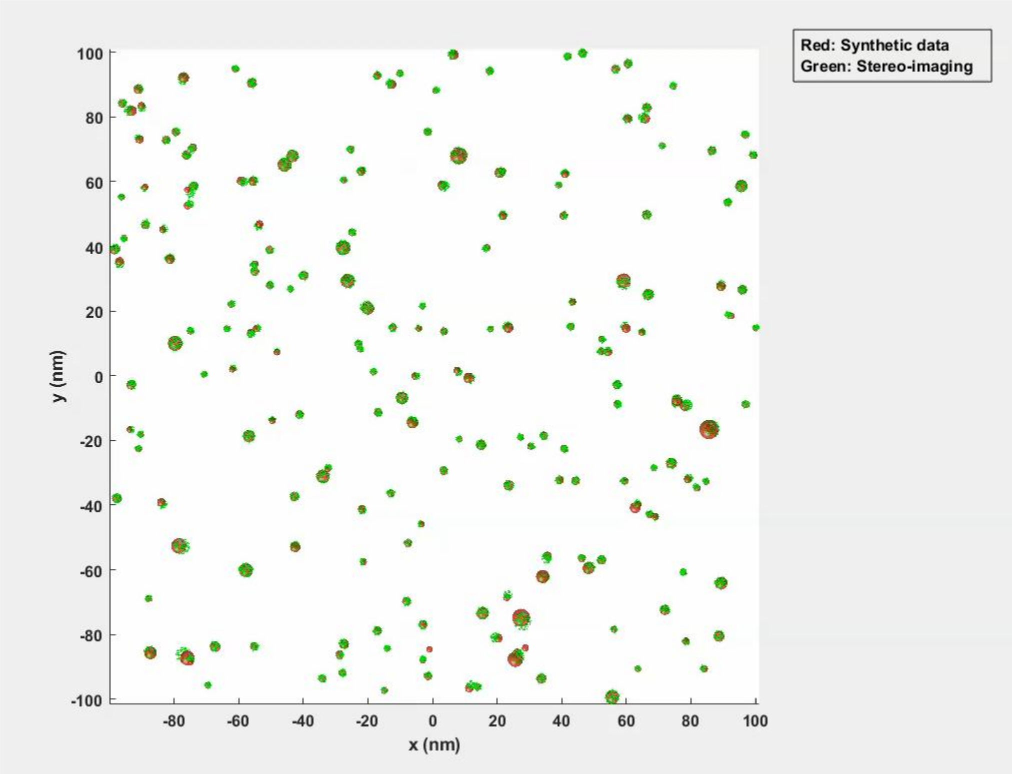
3D electron tomography



Pt NPs on Carbon, using a TF Talos F200 STEM in TF Headquarter

- Higher spatial resolution
- Longer data collection time

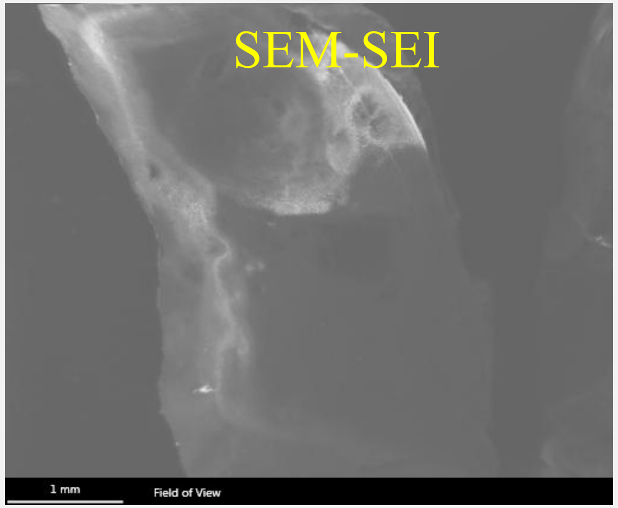
Stereo-TEM imaging of dislocation loops

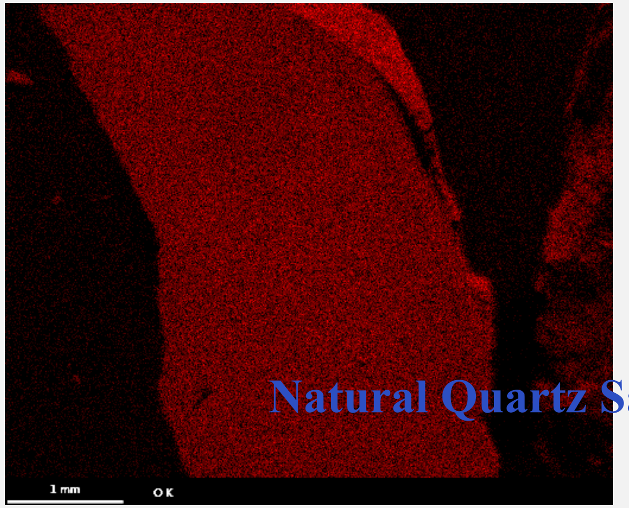


H. Yu et al., 3D reconstruction of the spatial distribution of dislocation loops using an automated stereo-imaging approach, Ultramicroscopy, 195, 58 (2018)

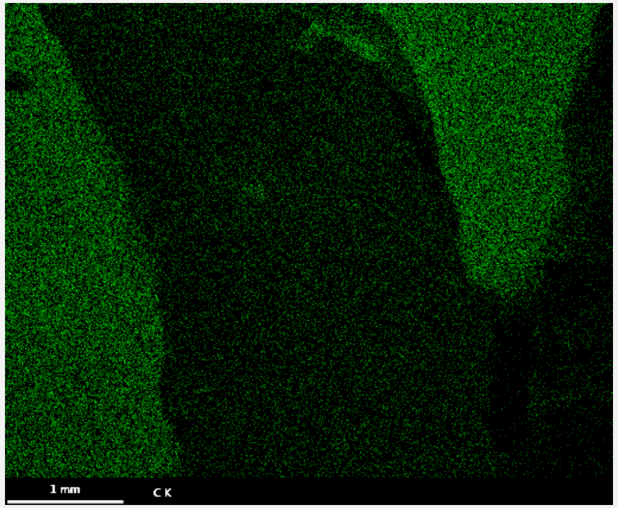
- Lower spatial resolution
- Shorter data collection time

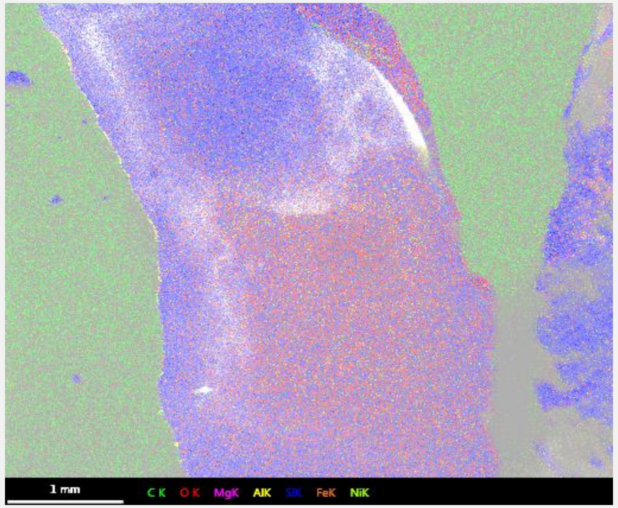
SEM Imaging of Natural Quartz: Sample-1

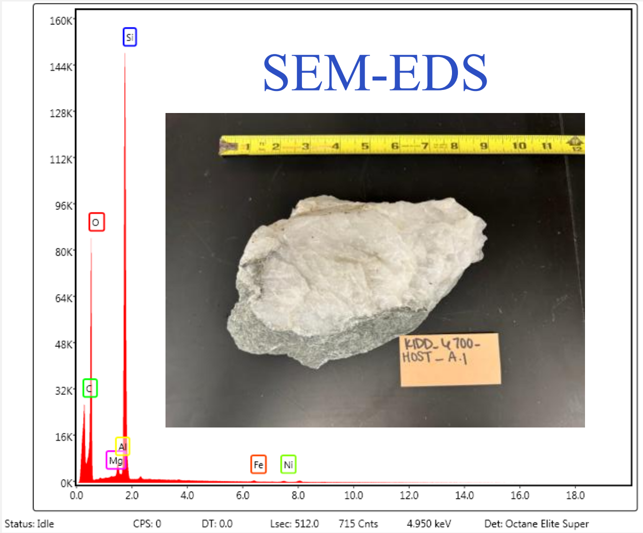




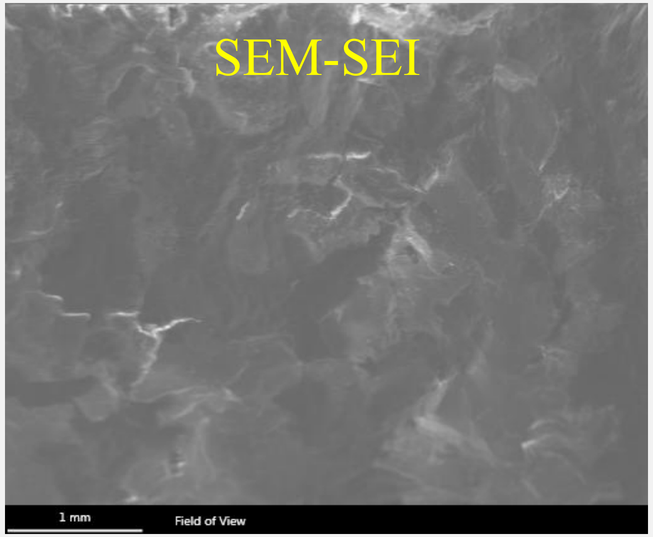
Natural Quartz Samples from the Deep Earth

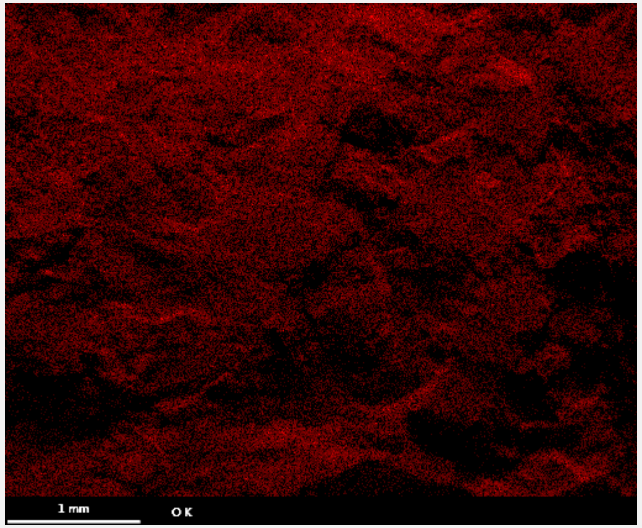


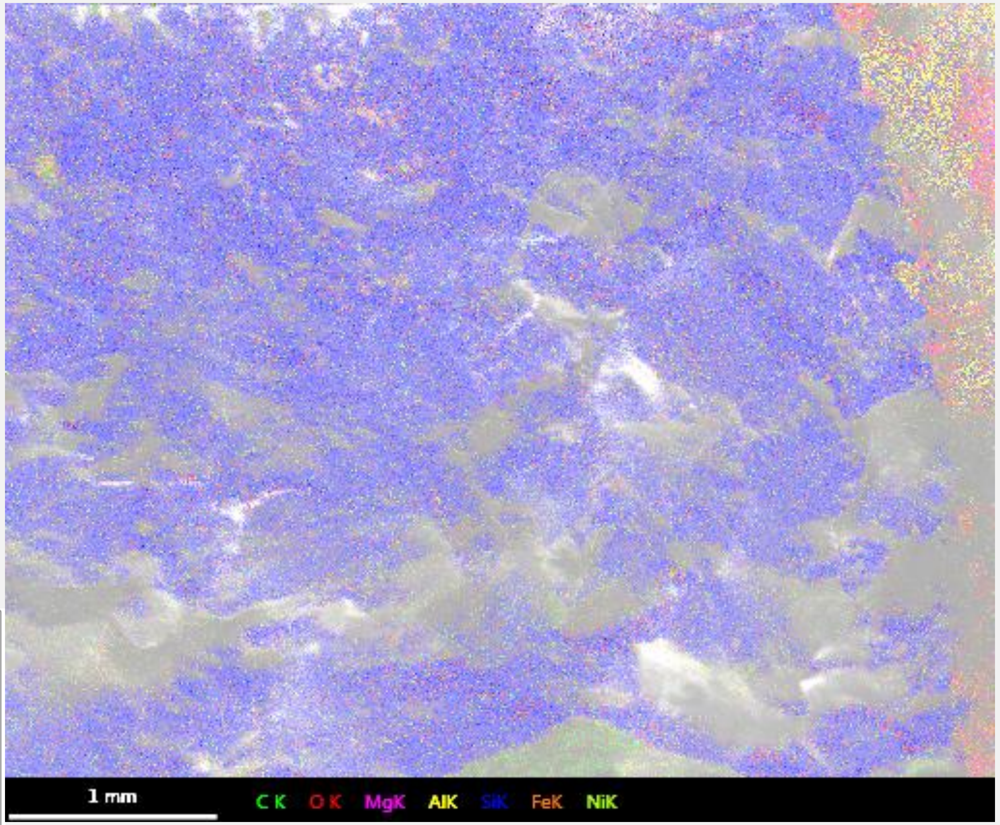


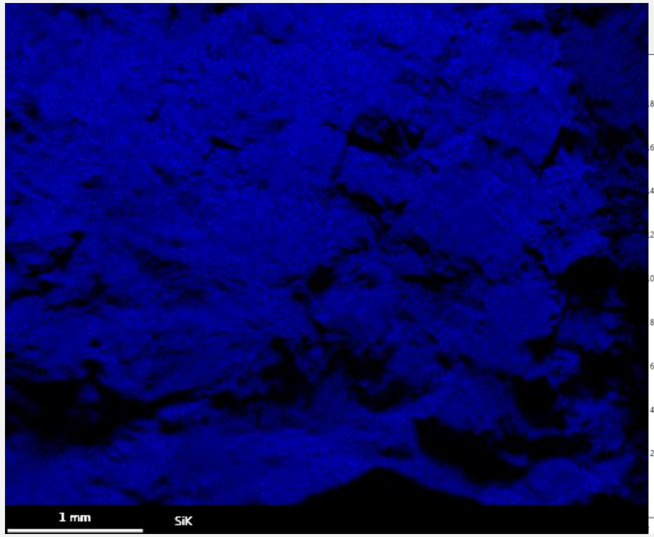


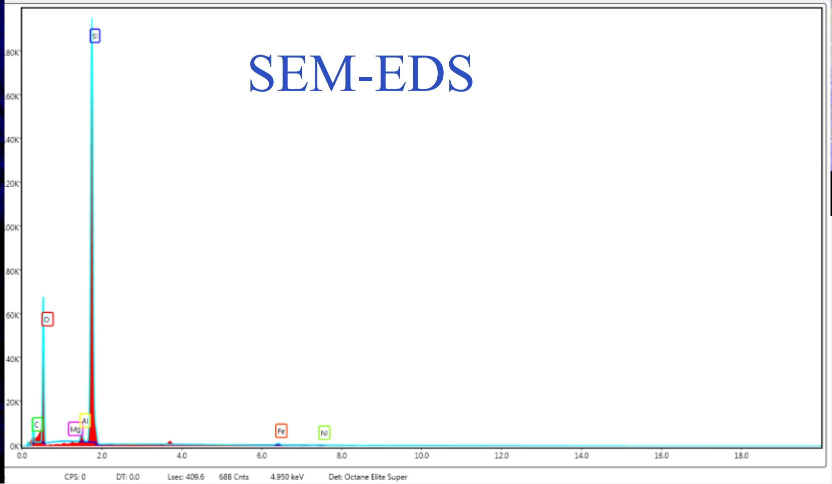
X-STEM Imaging of Natural Quartz: Sample-2

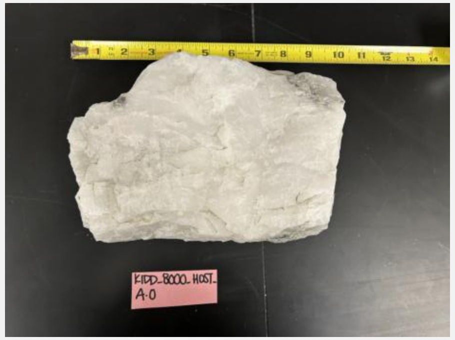




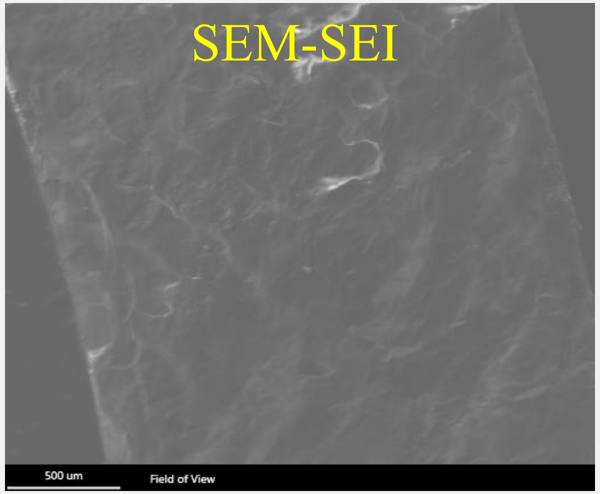


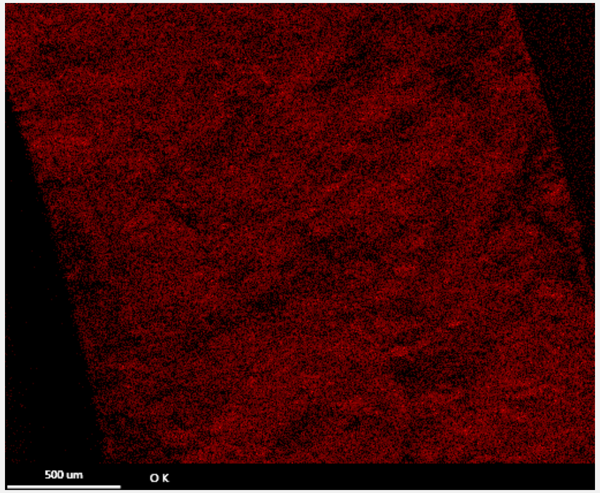


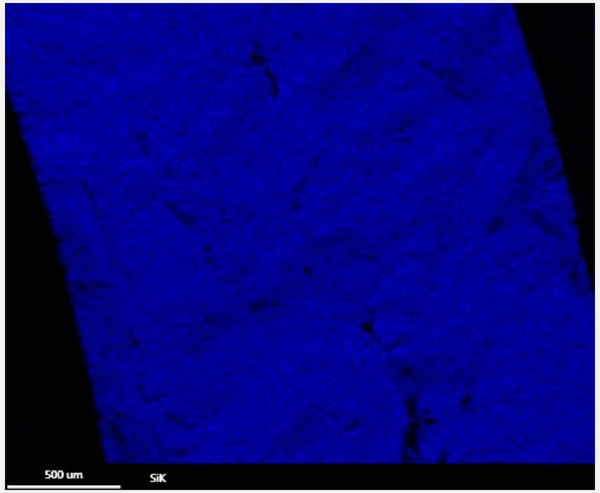


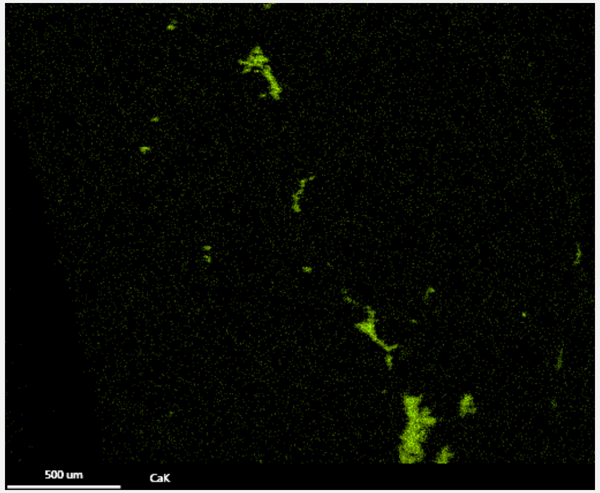


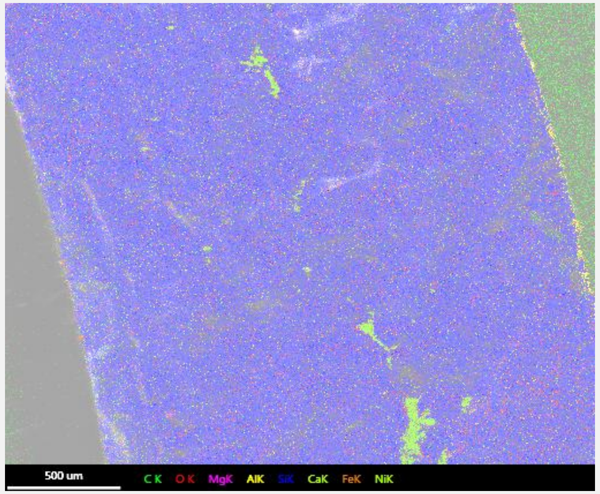
X-STEM Imaging of Natural Quartz: Sample-2

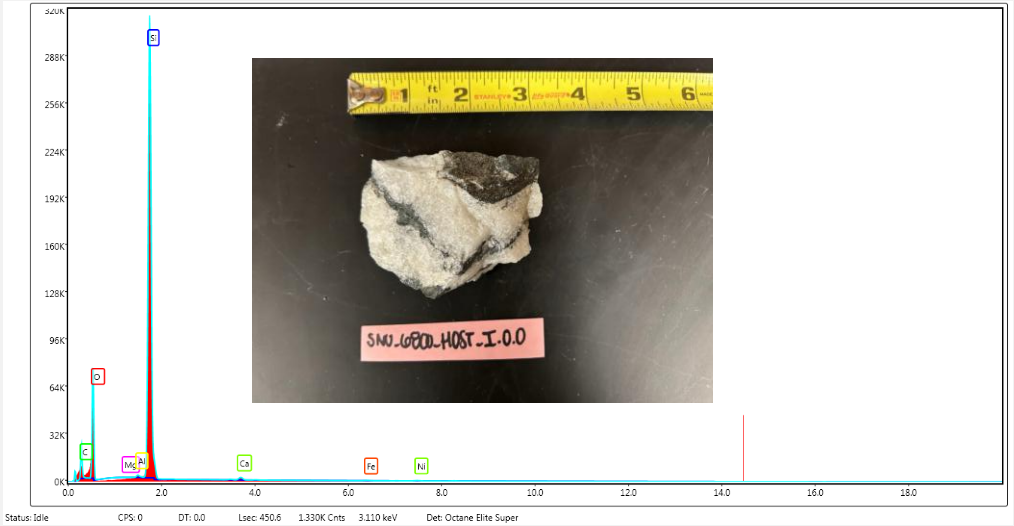




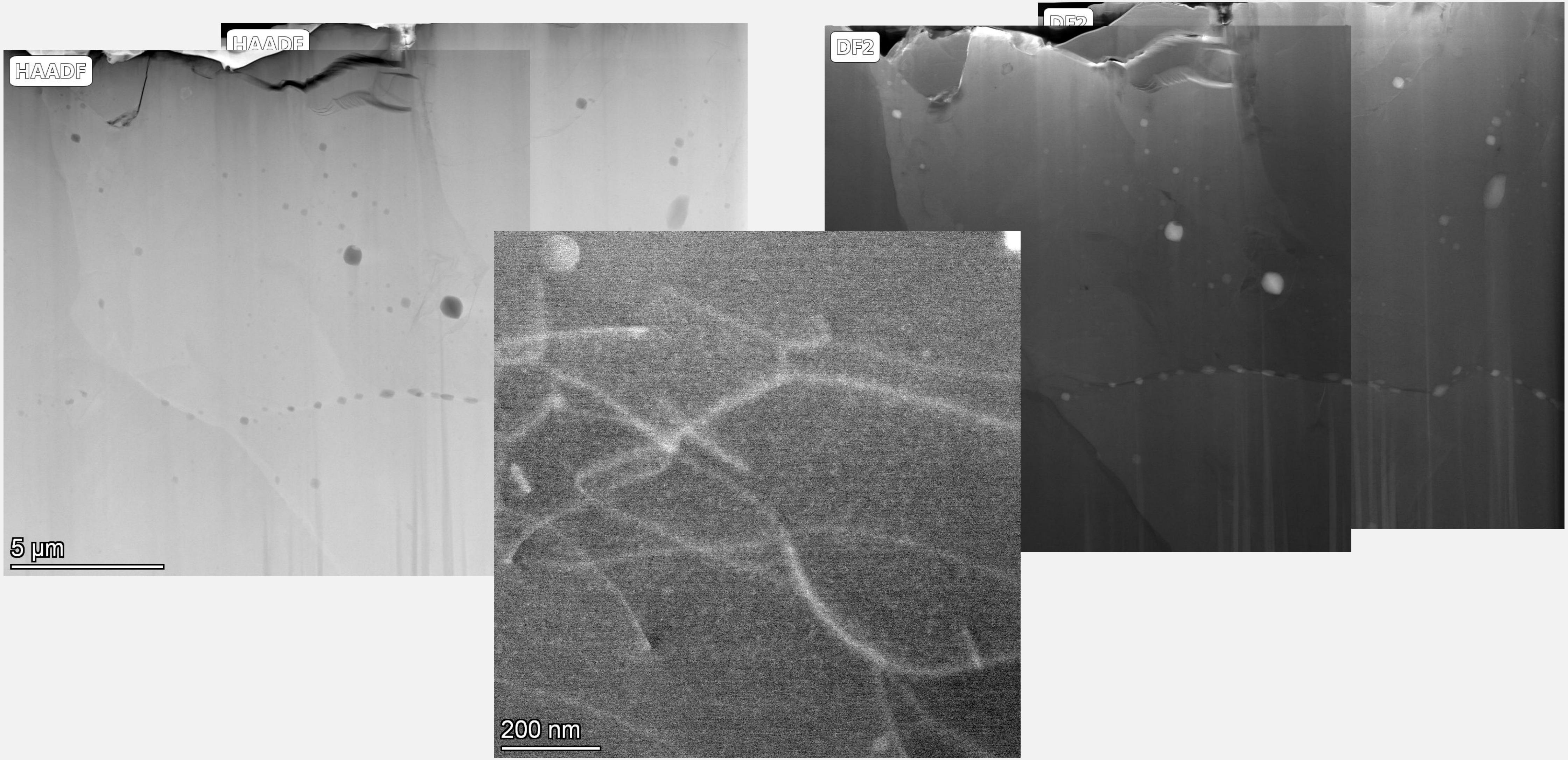




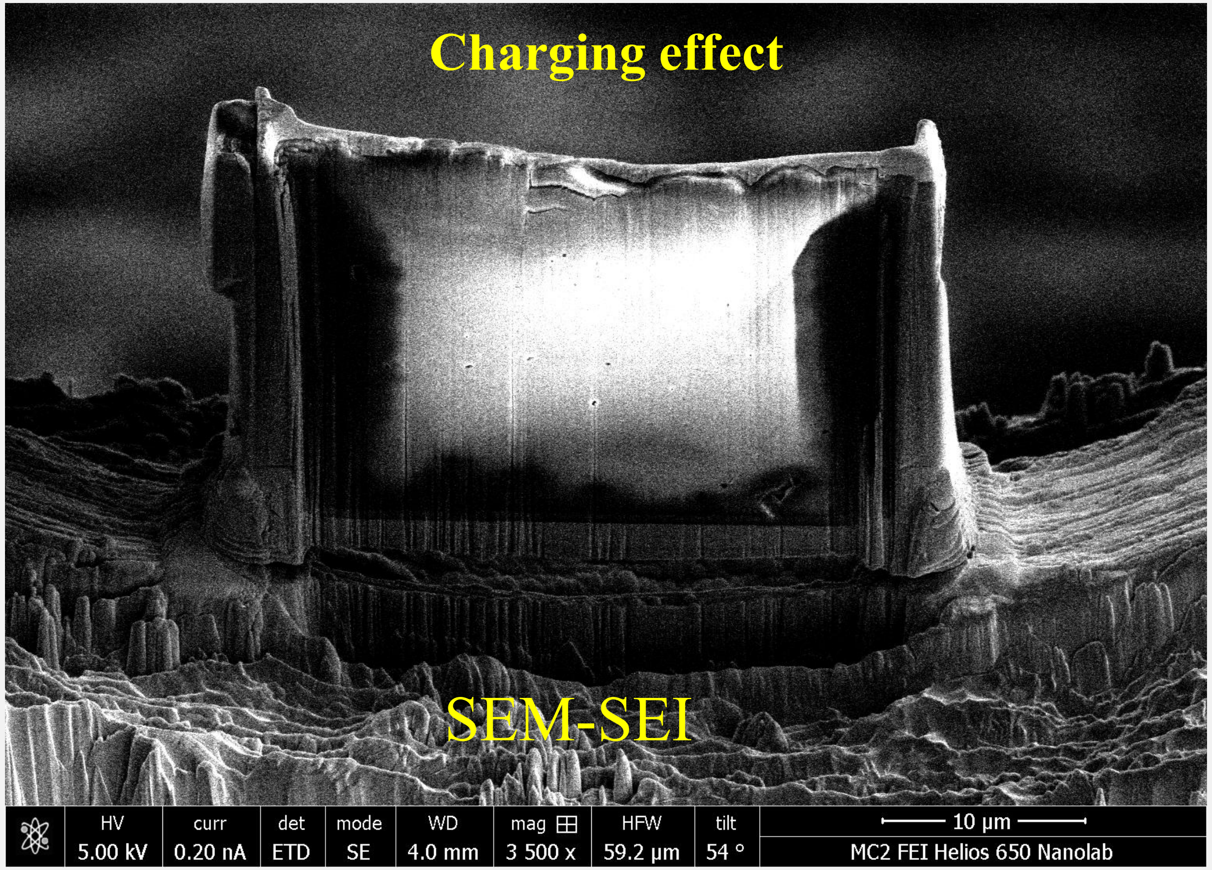


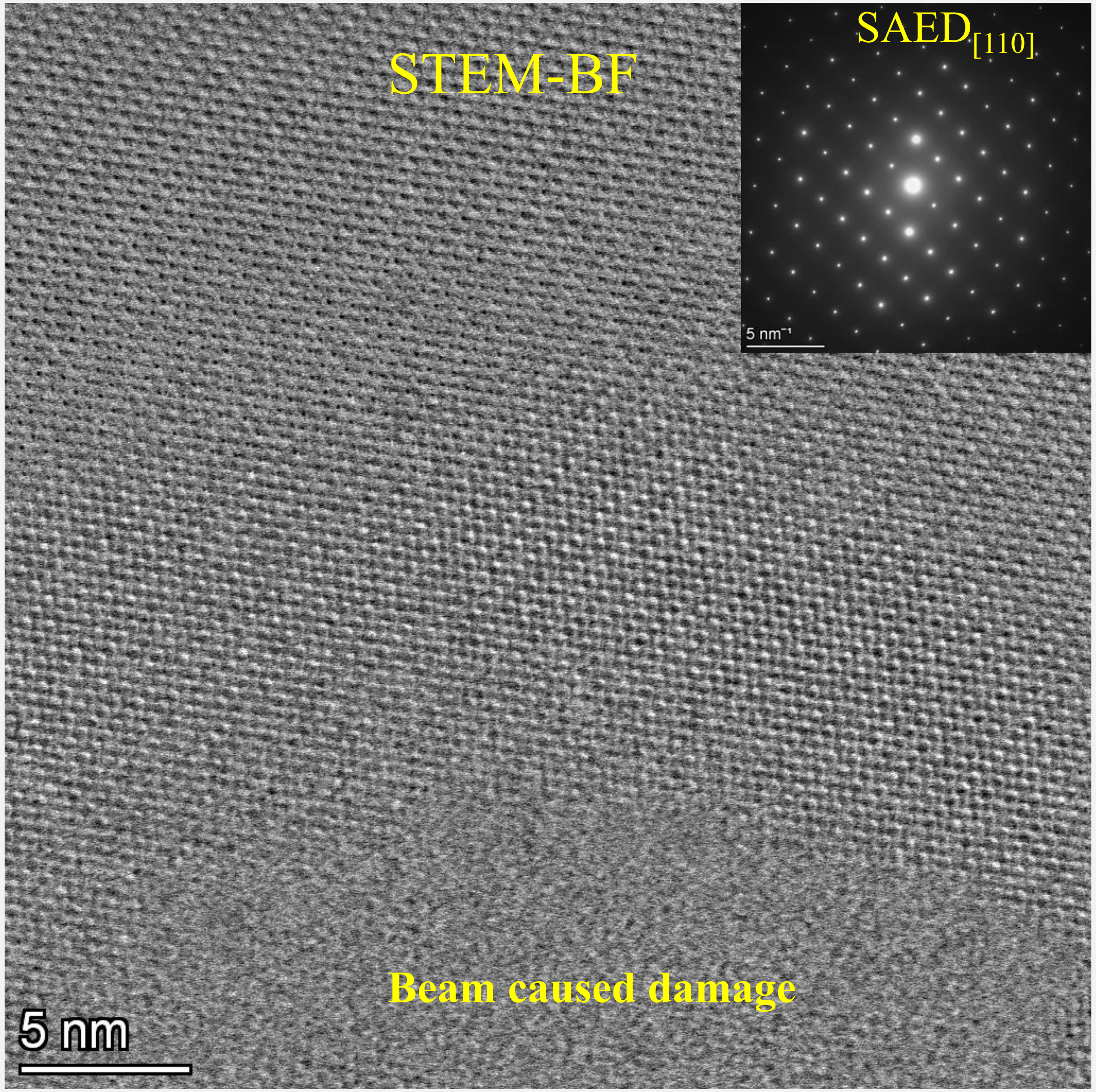


X-STEM Imaging of Natural Quartz: Sample-1



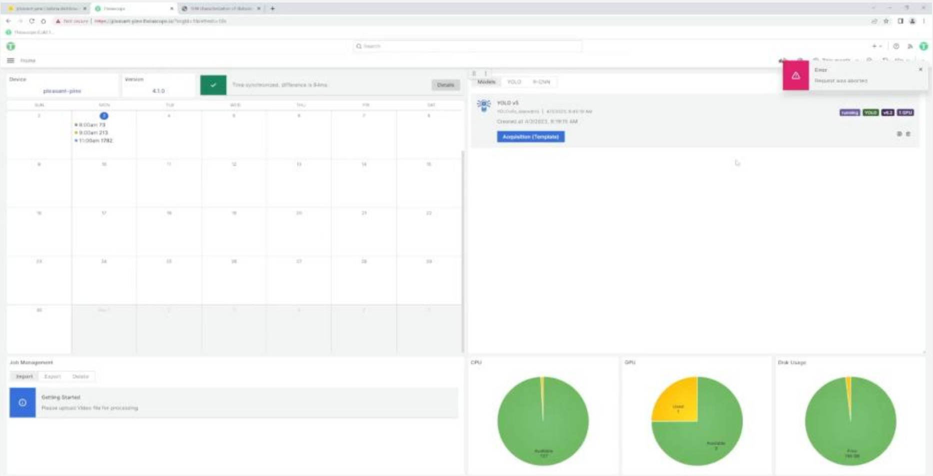
Issues for Electron Microscopy Imaging of Minerals

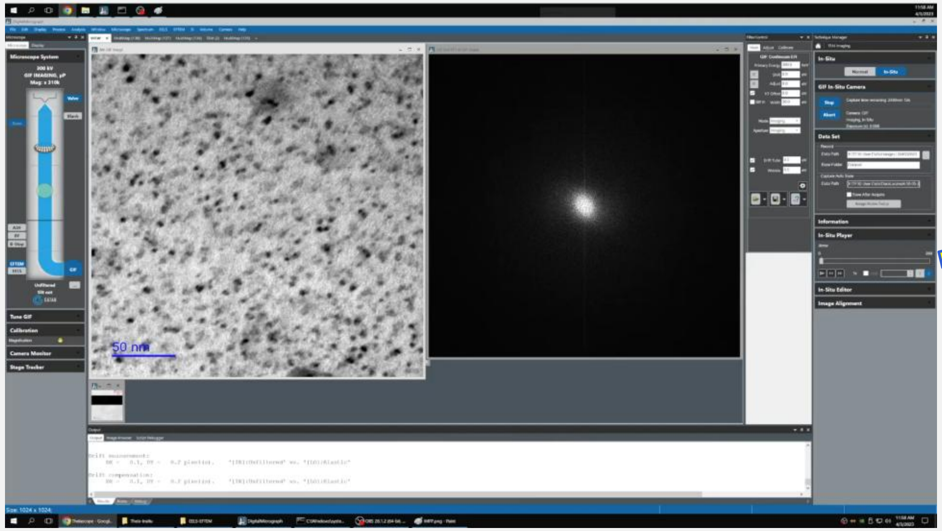


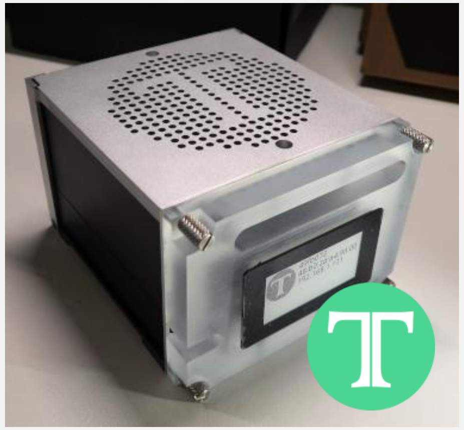


- Badly charging effect in SEM
- Beam sensitive in TEM imaging

ML System: Theiascope™ System for In-situ Data Analysis

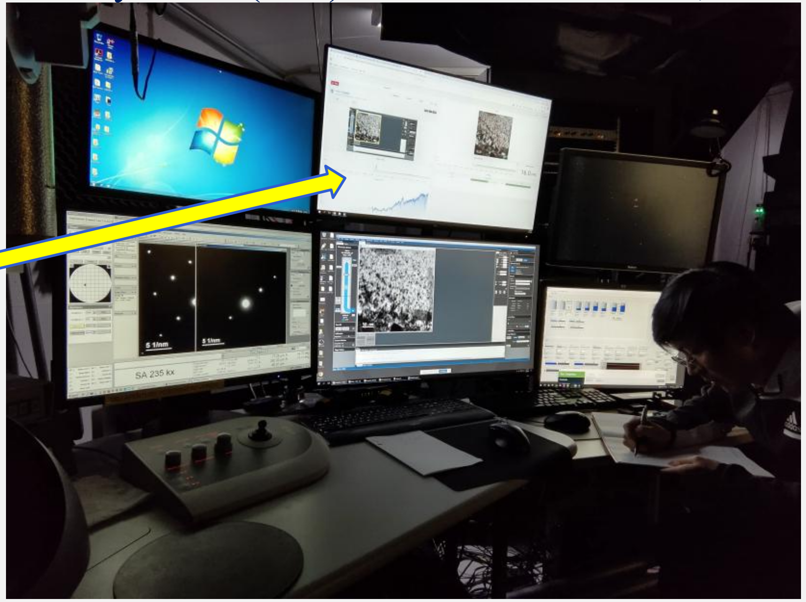






*C.R. Field & K.G. Field, U.S. Patent Application No. 17/718,805

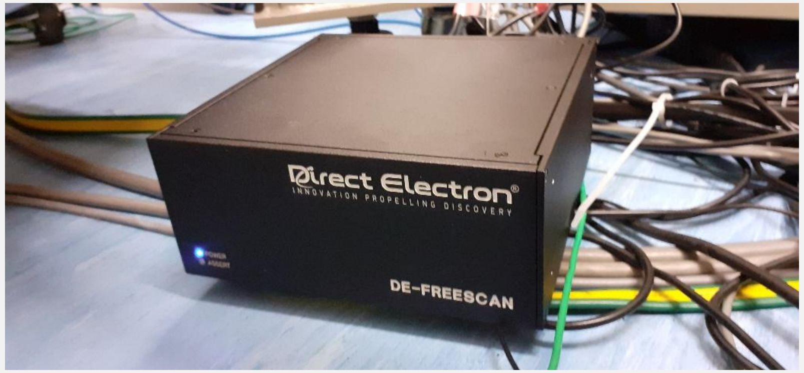
Courtesy H. Li (UM) and Theia Scientific, LLC

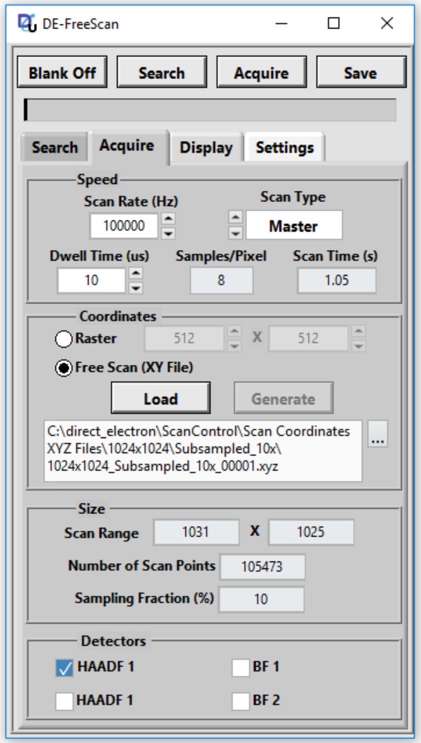


- Real-time quantification of irradiation induced Dislocation loops/voids during an in-situ TEM ion irradiation exposure.
- I am thinking to quantify abundant track data using this machine learning system.

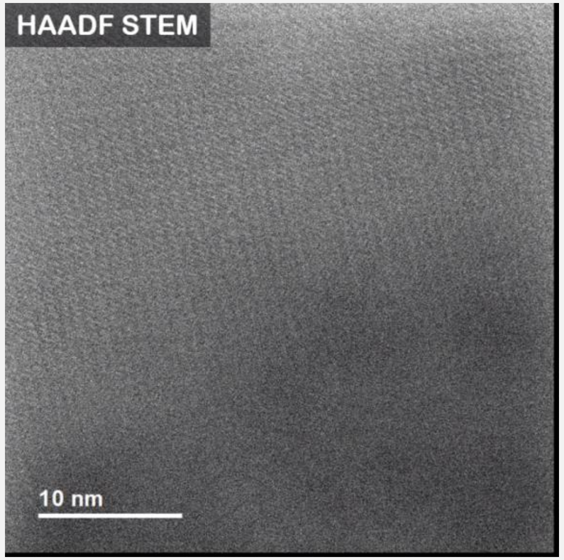
Sense-AI for Fast STEM Imaging: Installed in early January

Modification to the Microscope





$Rb_3NdP_2O_8$- a very e-beam sensitive ionic compound



10% Random Sampling

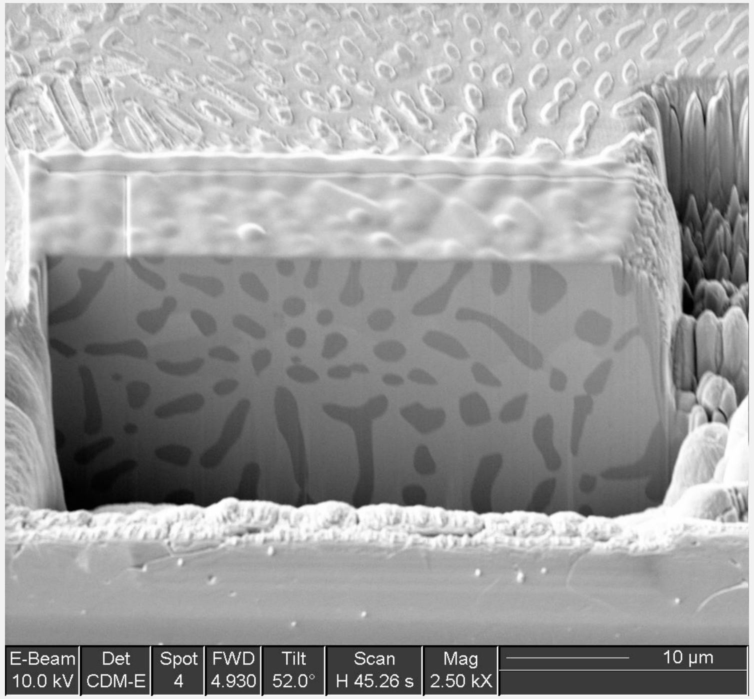
Reconstruction

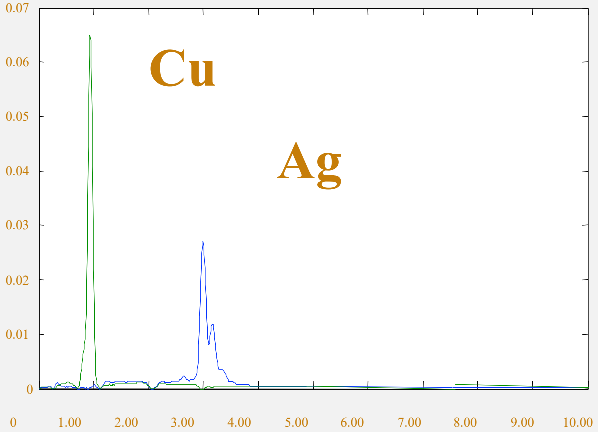
Sub-sampling scan controller plugs into external control and uses pre-generated masks. Images reconstructed currently using Matlab scripts. ~ 10fps scanning rate with good image quality

➢ Possible for imaging ion tricks in electron beam sensitive samples

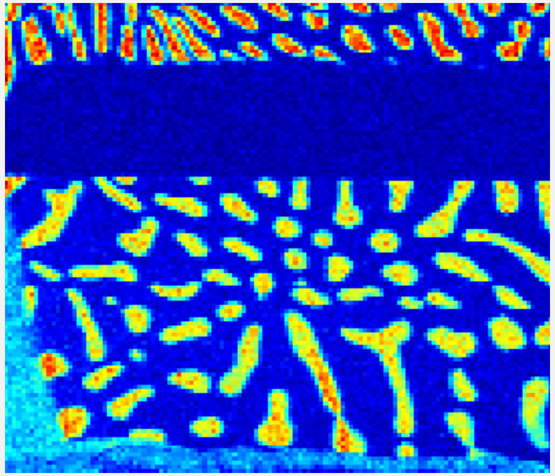
Courtesy SenseAI and University of Liverpool

Volume Electron Microscopy Imaging: SEM-EDS Mapping +FIB

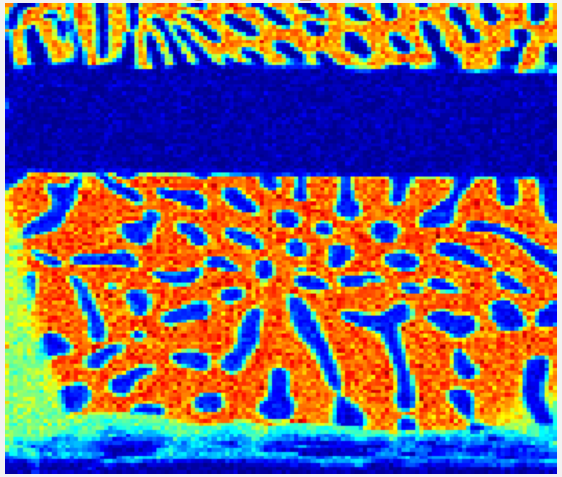


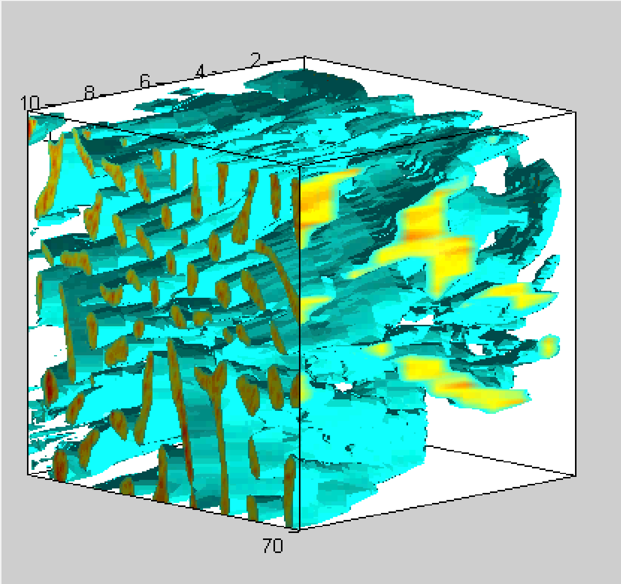


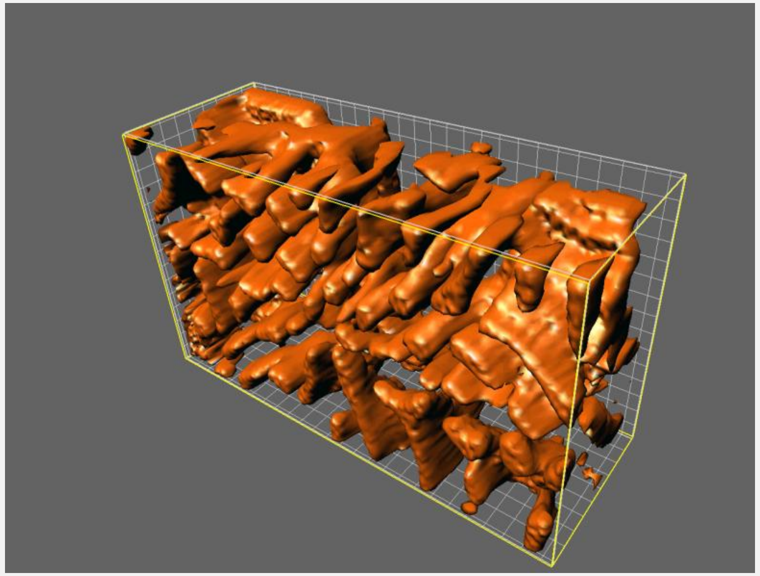
Cu-component



Ag-component





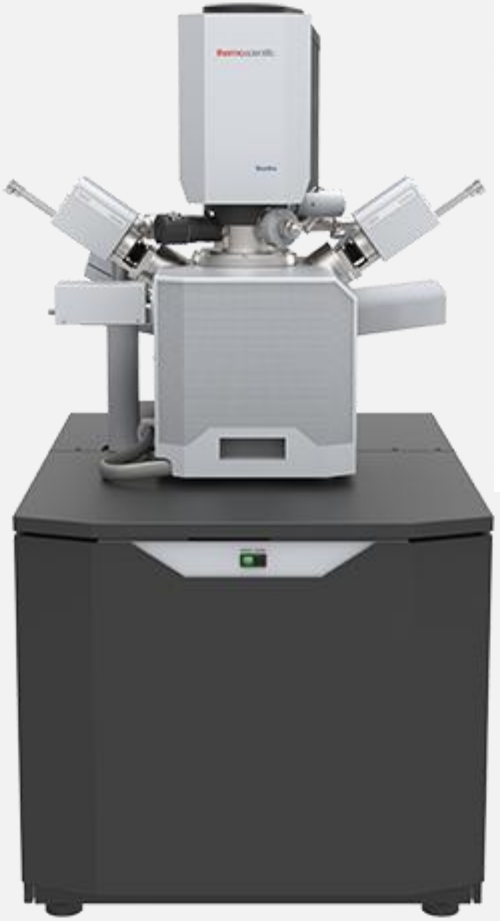


3D reconstruction of multiple spectrum images

Volume Electron Microscopy: Using Helios 5 + Quattro ESEM

Quattro ESEM

Environmental Controlled SEM (ESEM) fo
studying materials in their natural state.



Applications of Quattro ESEM include:

Nano Analysis

- Metals, alloys, fractures, welds, polished surfaces, magnetic materials, superconducting materials
- Ceramics, Composites, Plastics
- Films/Coatings
- Geological cross-section, minerals
- Soft materials: polymer materials, pharmaceuticals, filters, gels, tissues, plant materials
- Particles, porous materials, fibers

In situ analysis

- Crystallization/Phase Transformation
- Oxidation, catalysis
- Material Growth
- Hydration/Dehydration/Wetting/Contact Angle Analysis
- Tensile test (with heating or cooling)

Dynamic *in situ* analysis

--- To be delivered soon

Helios 5 Hydra DualBeam

Preparation of 3D EM and TEM samples using plasma focused ion beam scanning electron microscopy using multiple ion species.

High throughput and high quality

Perform high-throughput, high-quality, statistically relevant 3D analysis, cross-sectioning, and micromachining using next-generation 2.5 μA plasma FIB columns.

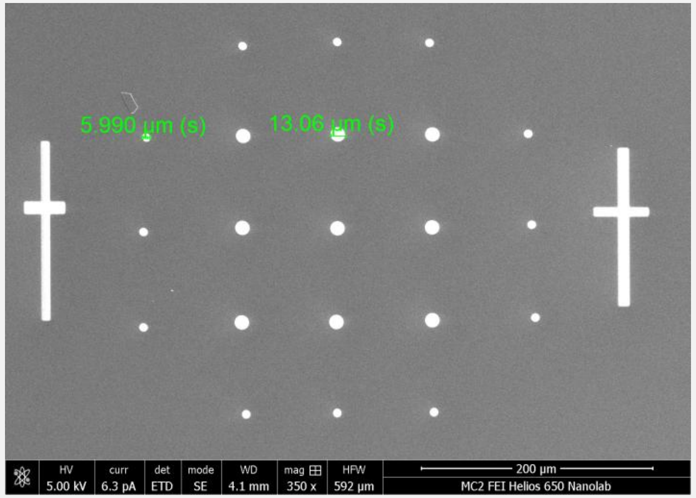
High-quality sample preparation

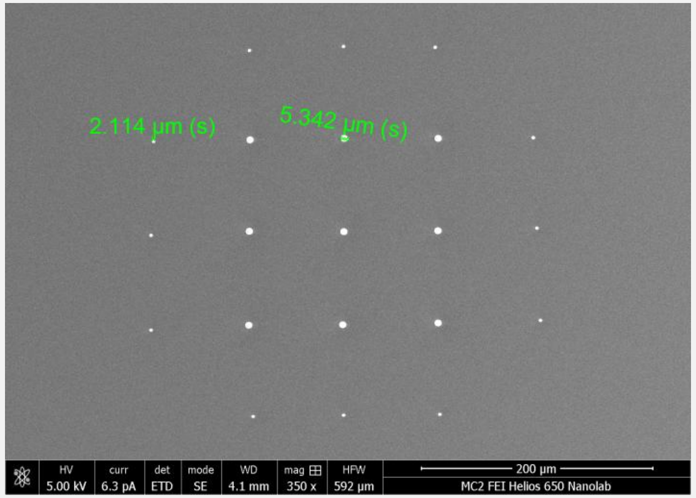
The new PFIB column enables high-quality, gallium-free TEM/APT sample preparation using xenon, argon, and oxygen, and provides superior performance in all operating conditions, including a 500 V final polish.

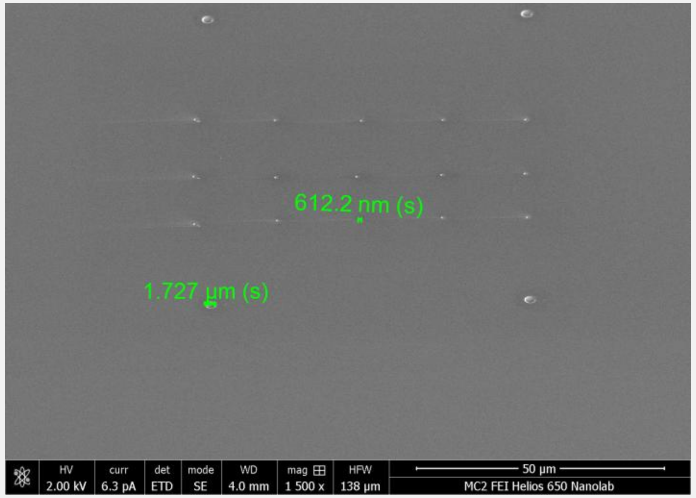
Just installed

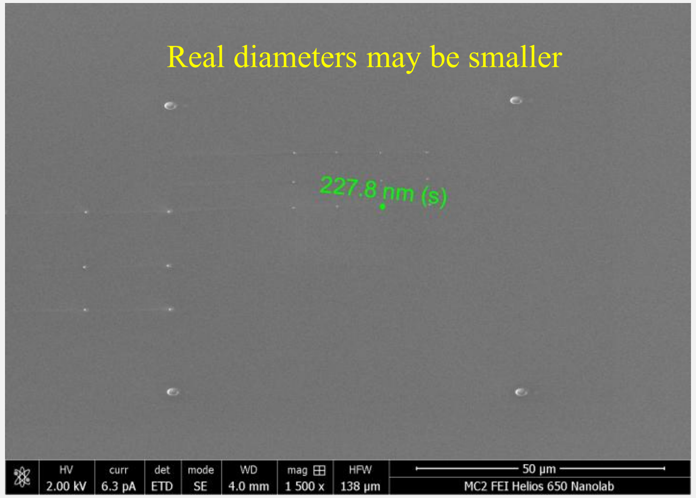
Optimizing X-ray Imaging Using the Zeiss Xradia 810 Ultra

Using FIB to make some patterns in quartz: Control sample for XRM calibration









ZEISS Xradia 810 Ultra

Nanoscale X-ray Imaging

- Nanoscale 3D X-ray imaging at a spatial resolution down to 50 nm and 16 nm voxel sizes
- 3D and 4D *in situ* experiments
- Quantification of nanostructures and using the data for modelling input.

Summary and Future Plan

❑ Summary

- High energy Au ions irradiation has been used for generation of ion tracks in a synthetic quartz single crystal sample.
- We proposed a FIB cutting method for sampling tracks at different depth.
- STEM has been used for imaging Au ion tracks in the synthetic quartz.
- Preliminary data were collected from three natural quartz from deep earth.

❑ Things to do next:

1) Perform self-ion irradiation using either O or Si ions in to both synthetic and natural quartzes with smaller fluences. Also neutron irradiation into those minerals will be conducted.
2) Try other techniques like XRM and AFM
3) Introduce AI and ML systems for data collection and analysis

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