Sandia National Laboratories

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IN SITU ION IRRADIATION ELECTRON MICROSCOPY CAPABILITIES AT THE SANDIA ION BEAM LAB

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Radiation-Solid Interactions Group

April 18, 2024

NSUF Annual Program Review

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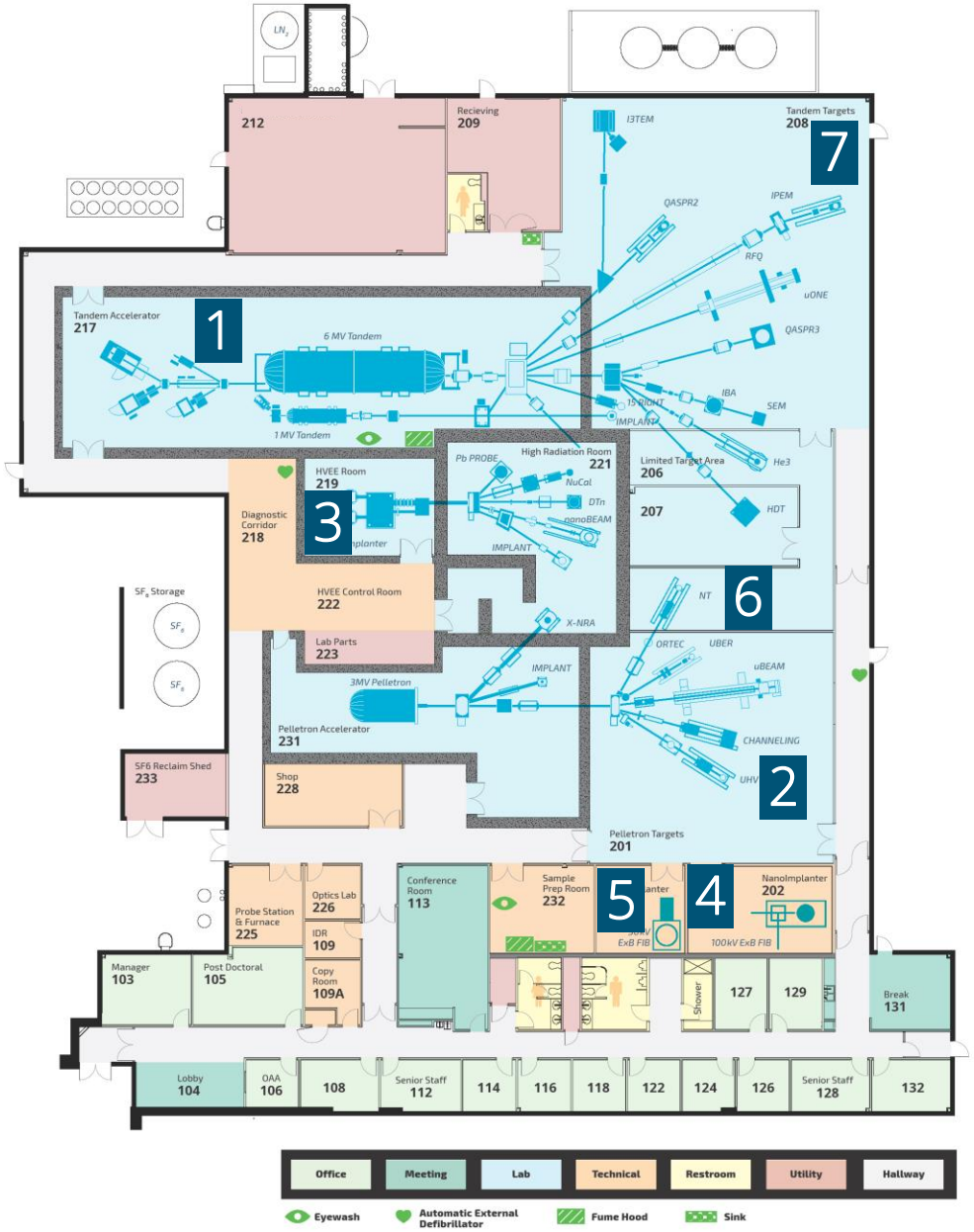
U.S. DEPARTMENT OF ENERGY

NNSA

GENERATING EXTREME ENVIRONMENTS AT THE IBL



Operational

(1) 6 MV Tandem Accelerator
(2) 3 MV Pelletron Accelerator

Ion Beam Analysis

(3) 350 kV HVEE Implanter
DD – 2.45 MeV n
DTn – 14 MeV n

Radiation Effects

(4) 100 kV ExB FIB nanoImplanter
(5) 35 kV ExB FIB Raith Velion
(6) 35 kV Zeiss HeIM

Nanofabrication

Installing

(7) In-Situ X-ray Photoelectron Spectrometer — Radiation Effects

25 end-stations including:
***in-situ* TEM, SEM, PL, LN_2 cooling, ≤1200°C heating**

Virtual tour on Sandia website: https://tours.sandia.gov/ibl_info.html

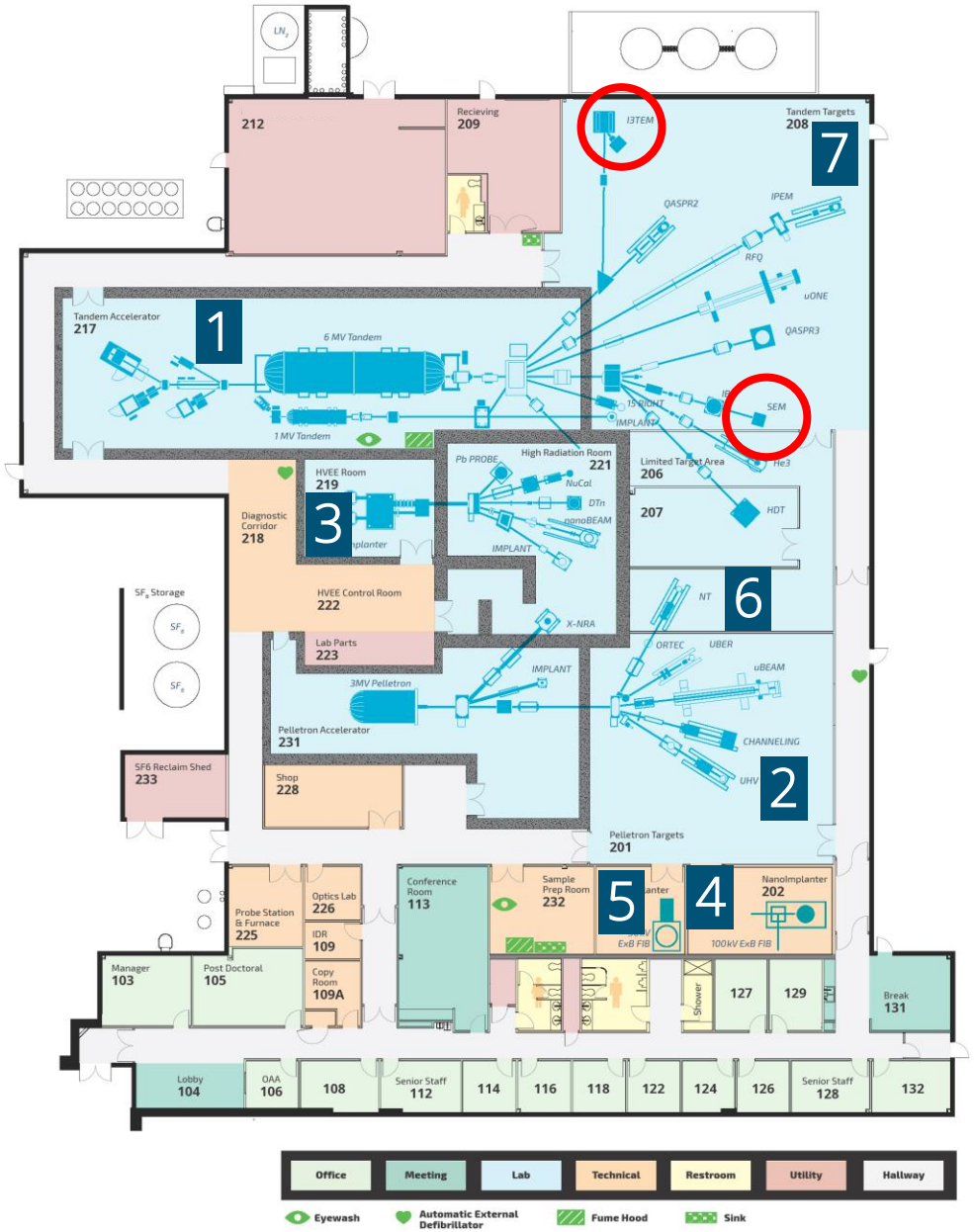
OVERVIEW

JEOL JEM-2100 TEM (I³TEM)

- In-situ *nanoscale* structural characterization under combined extreme environments
- Fundamental mechanisms, localized phenomena

JEOL IT300-hr SEM (I³SEM)

- In-situ *microscale* structural characterization under combined extreme environments
- Application-relevant length scales & sample prep



IN-SITU TEM FOR EXTREME ENVIRONMENTS RESEARCH

Available Holders

1. Heating (1000 °C)
2. Heating + Gas Flow
3. Heating + Straining
4. Cryo (LN_2)
5. Heating + Gas Flow
6. Liquid Cell
7. Liquid Cell + Heating
8. Biasing
9. Tomography
10. Nano-Mechanical*

**Holder operational, redevelopment needed*

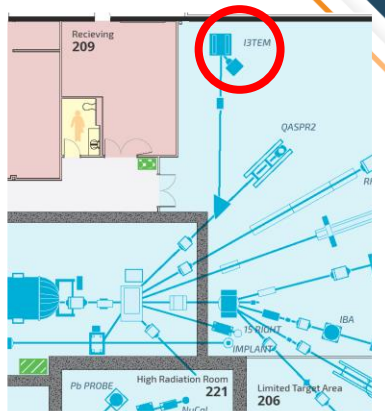
Additional Capabilities

1. User Adjustable Pole Piece
2. Precession Electron Diffraction (PED)
3. Waviks gas manifold

Ion Accelerators

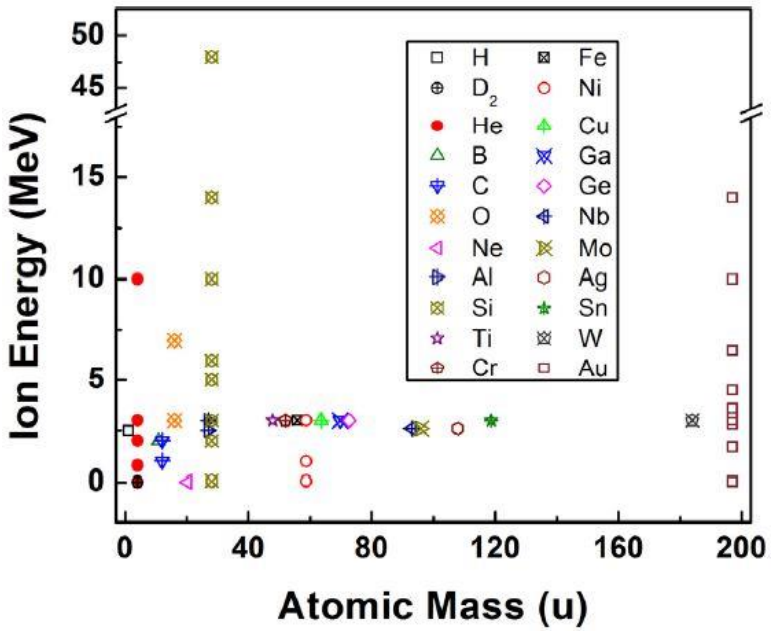
1. 6 MV Tandem / 1 MV Tandem**
2. 0.5 kV – 10 kV Colutron Accelerator

***Redevelopment needed*



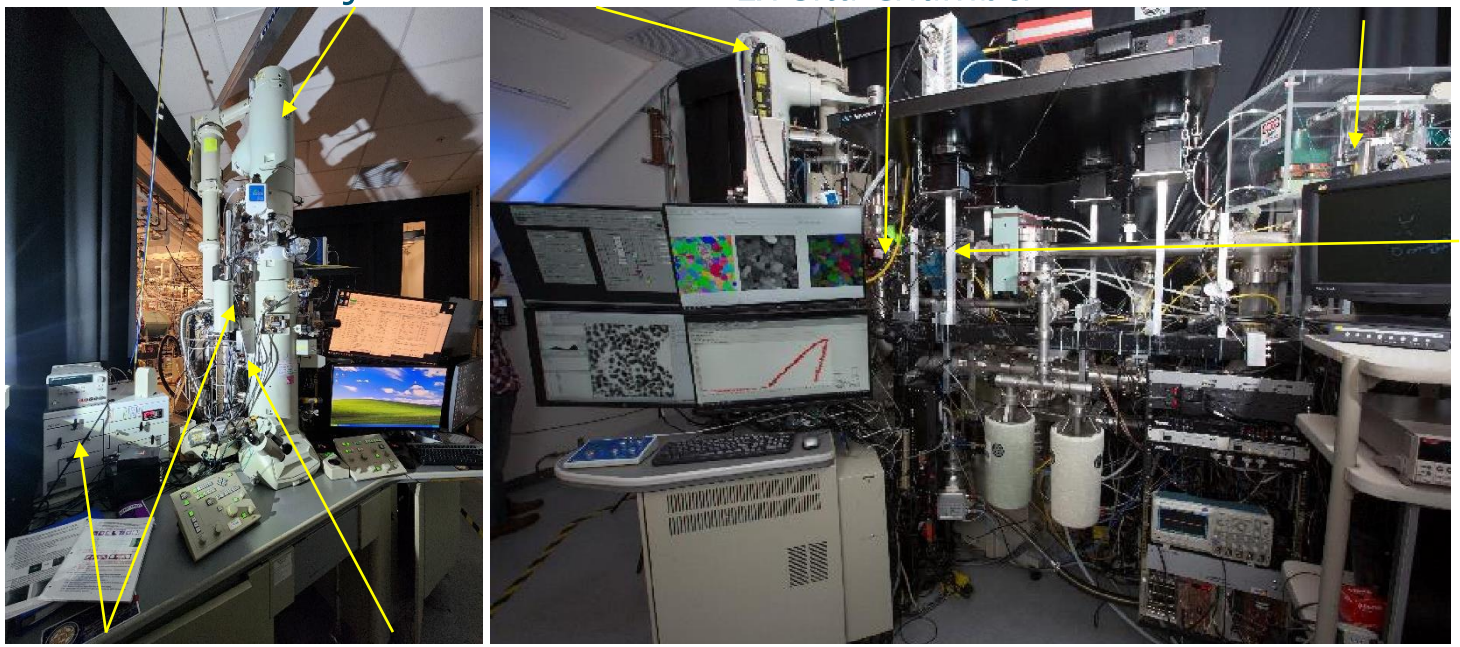
0.5-10 kV Colutron Accelerator

Ion Beams into TEM to Date



JEM-2100 TEM

Ex-Situ Chamber



6 MV Tandem Accelerator Beamline

Waviks Gas Manifold

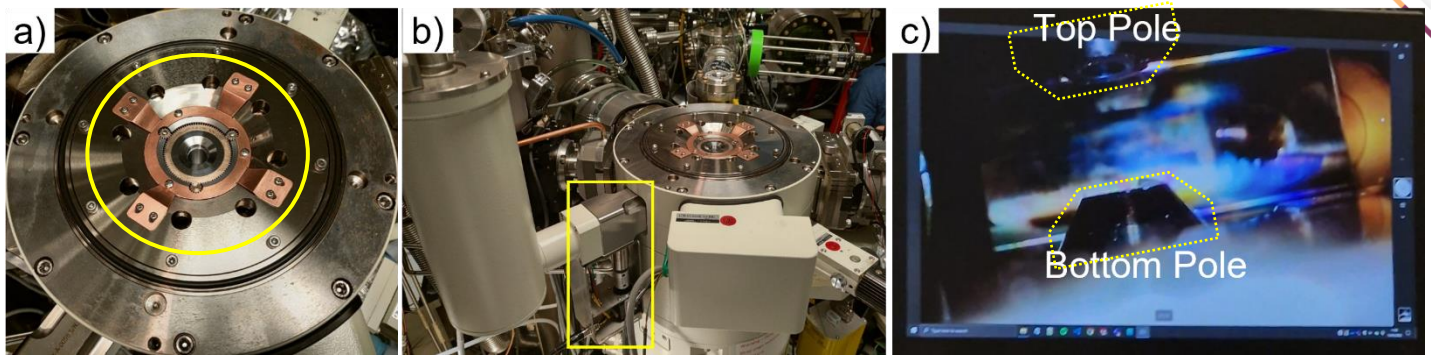
User-Adjustable Pole Piece

In collaboration with S. House (SNL-NM)

CAPABILITY DEVELOPMENT: I³TEM USER-ADJUSTABLE POLE PIECE

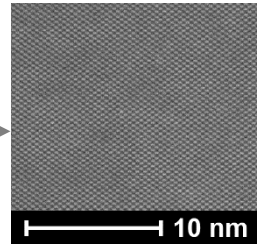
Motivation for UAP Development

- UAP enables unprecedented microscope functionality, making it possible to switch between high resolution imaging (small pole piece gap) and low resolution in-situ imaging (large pole piece gap) within the same experiment on the same TEM
- **Typically would need multiple microscopes to accomplish this**



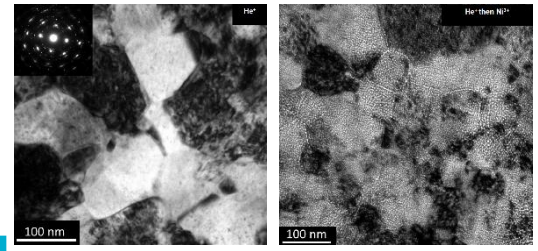
Installed May 2023

Image not representative of current JEM-2100 imaging capability

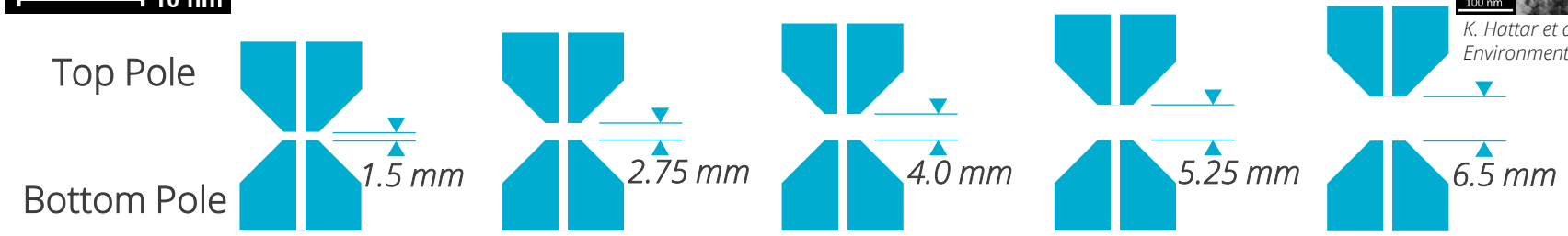


Increasing Contrast

Increasing Resolution



K. Hattar et al., Deconvoluting Mechanisms in Complex Environments via In-Situ TEM, 2021, SAND2021-2542C



Collaborators:

- Kevin McIlwrath (JEOL)
- Doug Medlin (SNL-CA), Stephen House (SNL-NM)
- Lewys Jones (Trinity College Dublin)

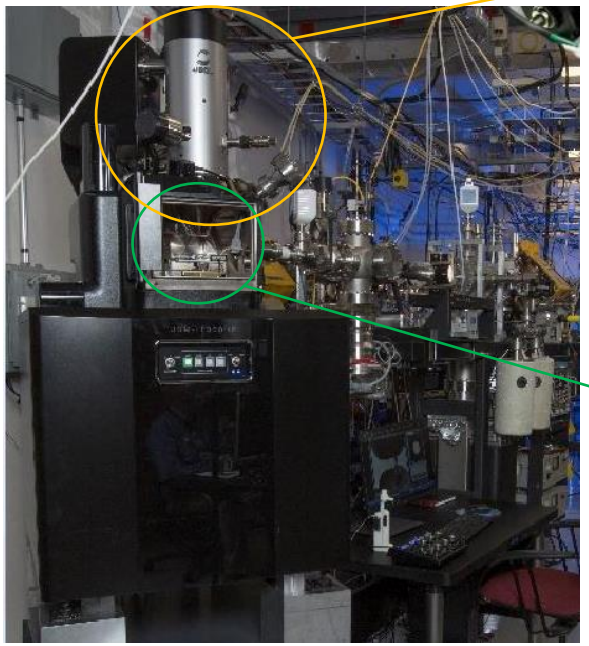
JEOL

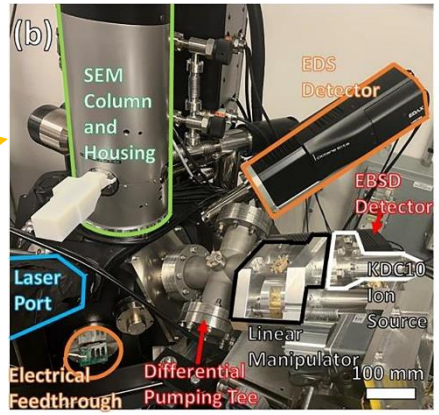
Trinity College Dublin
The University of Dublin

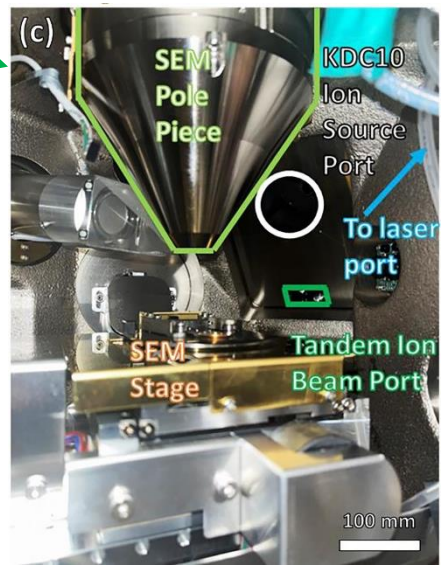
IN-SITU MICROSCOPY FACILITIES AT THE SNL IBL: I³SEM

In collaboration with B. Boyce (SNL-NM)

JEOL IT300hr In-Situ SEM Facility







System Capabilities

- 6 MV Tandem Accelerator
- EDAX APEX EDS Detector
- EDAX APEX EBSD Detector
- Electron Beam Induced Current (EBIC) electrical measurement
- 1064 nm laser (sample heating)*
- KDC10 1 kV Ion Source*

**Modular equipment, must be reinstalled for use*

E. Lang et al., Nuclear Instruments and Methods in Physics Research Section B: Beam Interactions with Materials and Atoms, 2023, 537, 29-37, doi:10.1016/j.nimb.2023.01.016

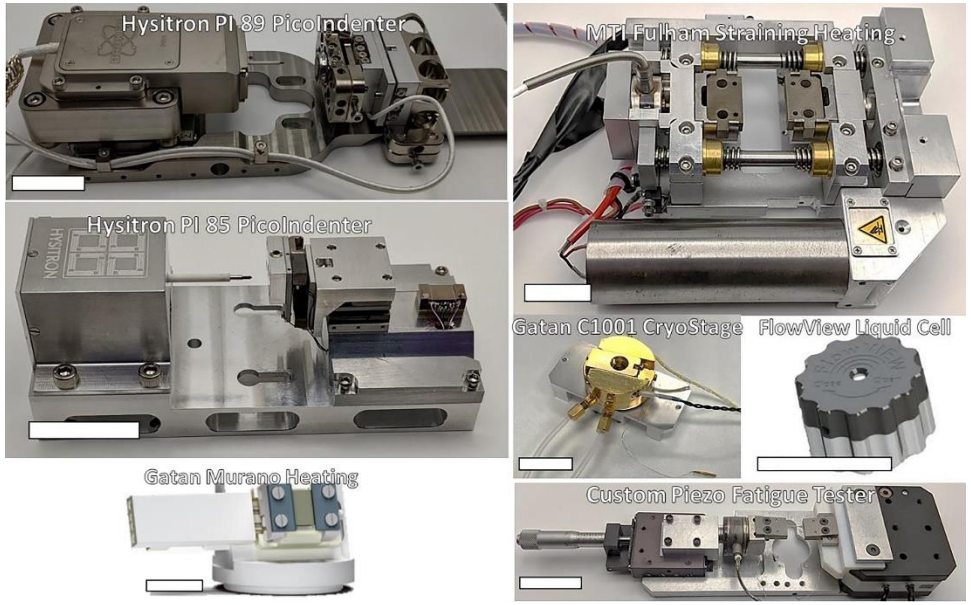
IN-SITU MICROSCOPY FACILITIES AT THE SNL IBL: I³SEM

In collaboration with B. Boyce (SNL-NM)

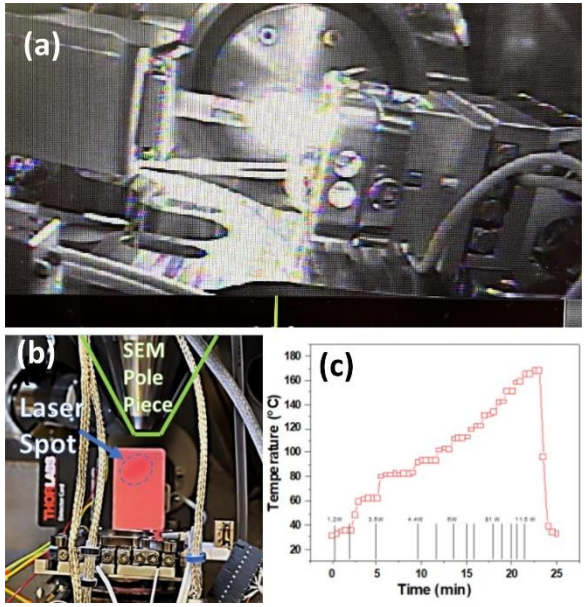
Specialty In-Situ Holders

- Gatan C1001 Cryo
- Gatan Murano Heating
- FlowView Liquid Cell
- Hysitron PI89 PicoIndenter*
- MTI Fulham Straining Heating*

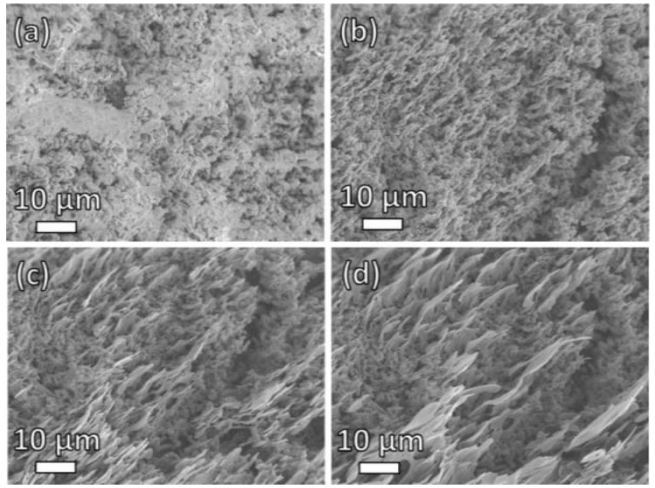
**Holders operational, redevelopment needed*



In-Situ Straining Heating



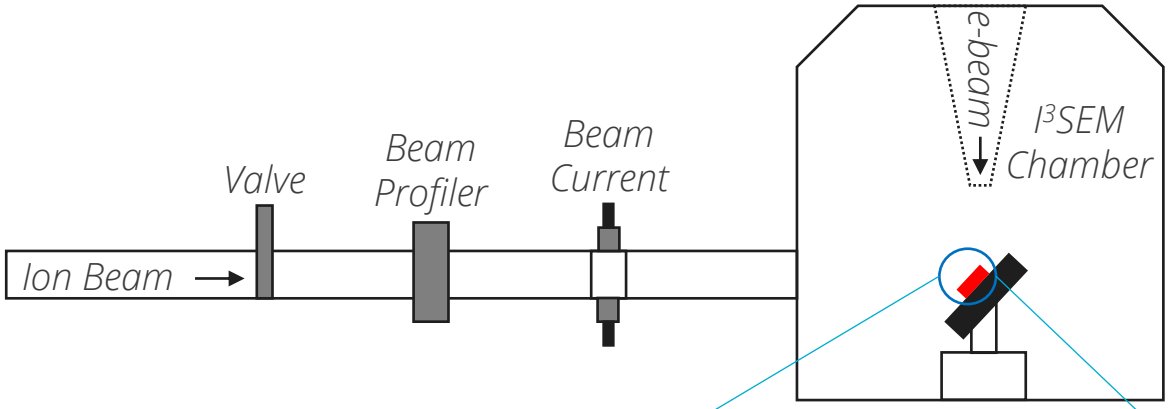
In-Situ Ion Irradiation



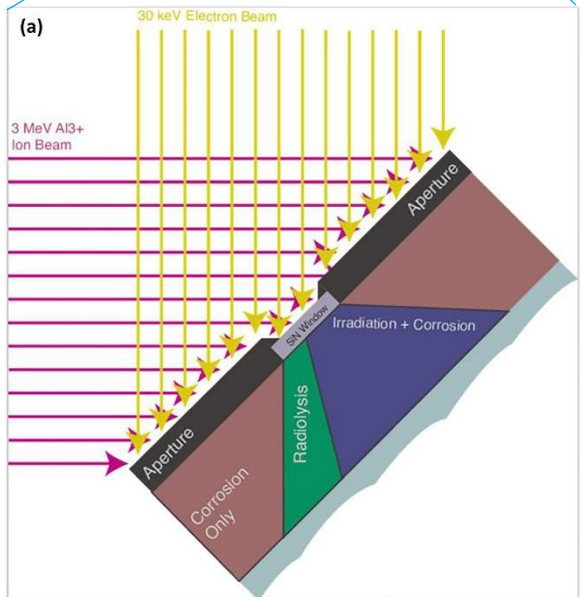
E. Lang et al., Nuclear Instruments and Methods in Physics Research Section B: Beam Interactions with Materials and Atoms, 2023, 537, 29-37, doi:10.1016/j.nimb.2023.01.016

CAPABILITY DEVELOPMENT: IN-SITU LIQUID CELL ION IRRADIATION

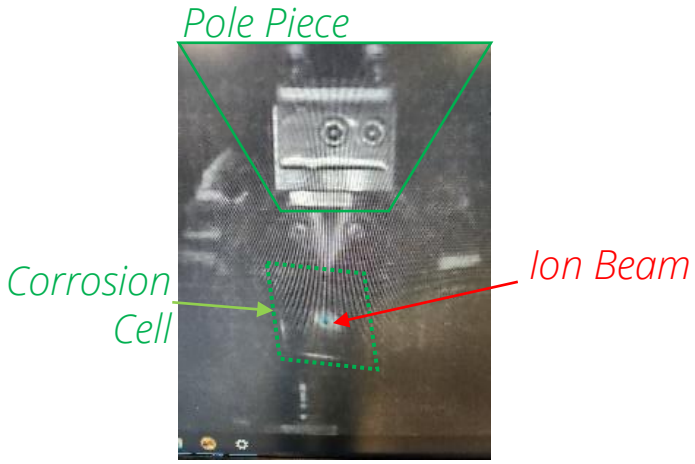
FlowCell Liquid Cell for in-situ ion irradiation and corrosion research



FlowCell Liquid Cell



Aligning ion and electron beams in the SEM



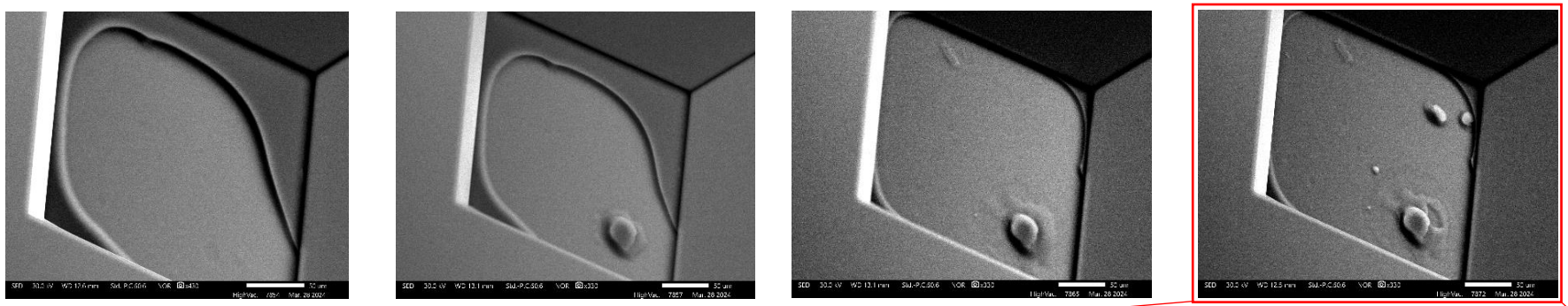
CHARACTERIZING ION BEAM EFFECTS ON IRON CORROSION

In collaboration with B. Derby, T. DeForest (LANL)

In-Situ ion irradiation, imaging, and chemical mapping in a liquid corrosion cell

Initial | *4E14 cm^{-2}* | *8E14 cm^{-2}* | *1.2E15 cm^{-2}*

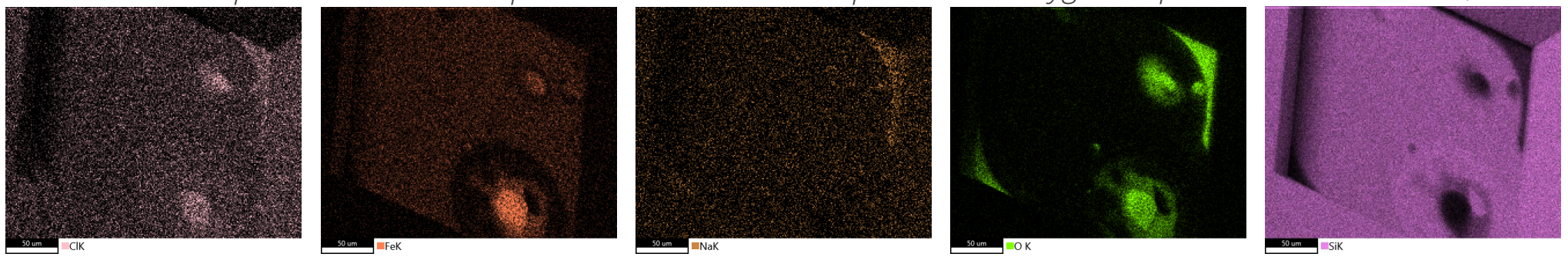
Secondary Electron



After 1.2E15 cm^{-2} irradiation

Chlorine Map | *Iron Map* | *Sodium Map* | *Oxygen Map* | *Silicon Map*

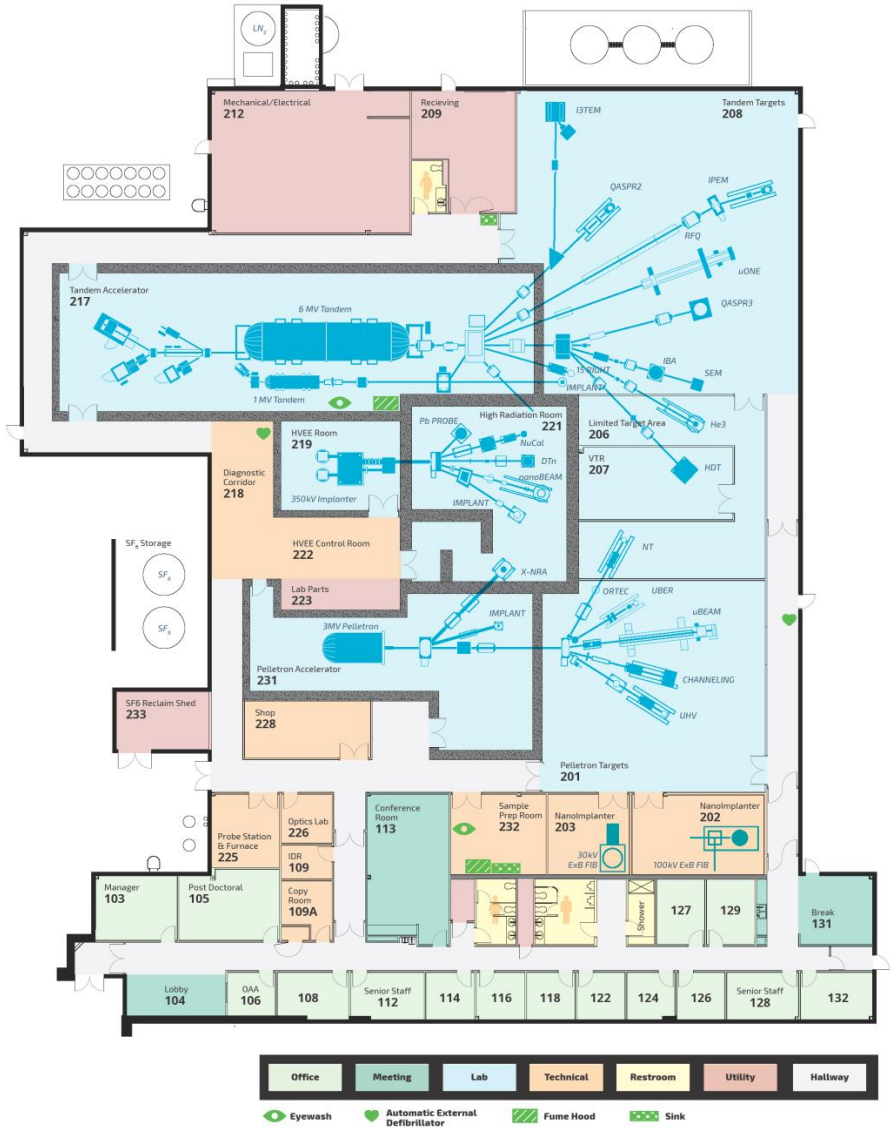
EDS Mapping



Future Development: In-Situ Molten Salt Corrosion and Irradiation in the SEM

Unpublished

THESE AND OTHER IBL CAPABILITIES AVAILABLE THROUGH NSUF



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