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Roundtable : TEM Data Generation/Utilization and Best TEM Practices for Radiation Damage Studies

NSUF Nuclear Science User Facilities

TEM specimen synthesis for radiation damage analysis

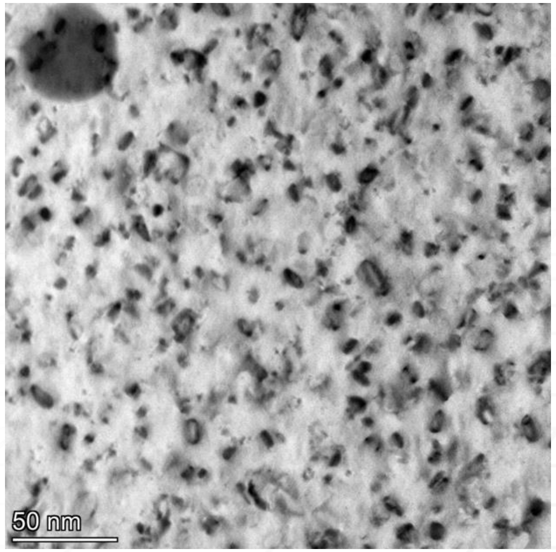
Twin-jet Electropolishing

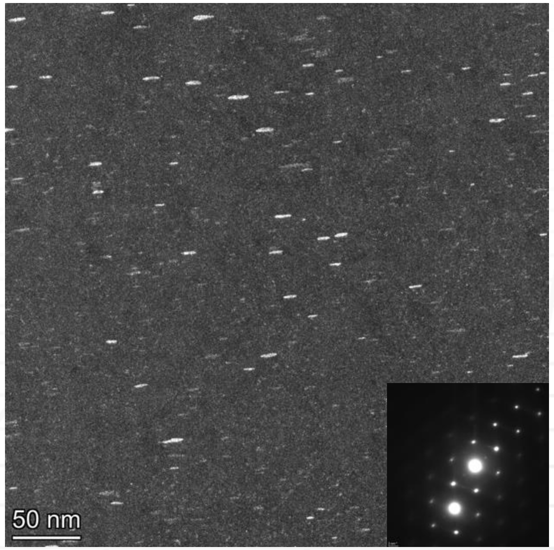
- Perhaps the best way for defect analysis; not possible for surface irradiated alloys

FIB + Nanomill

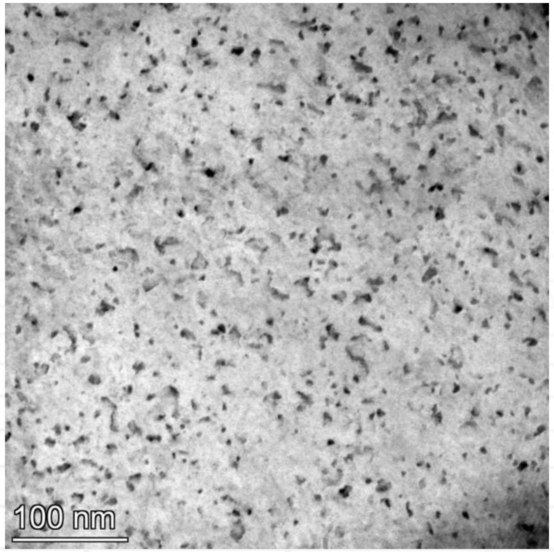
- Choice of FIB milling parameters to reduce the FIB damage + nanomilling

Courtesy: Anshul Kamboj (TETI-EFRC)

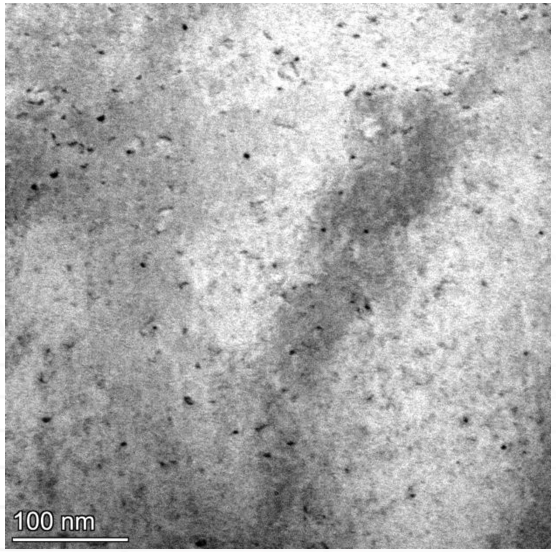




Neutron-irradiated IN718 (NSUF 2.0 CINR)



Nanomill 0 min



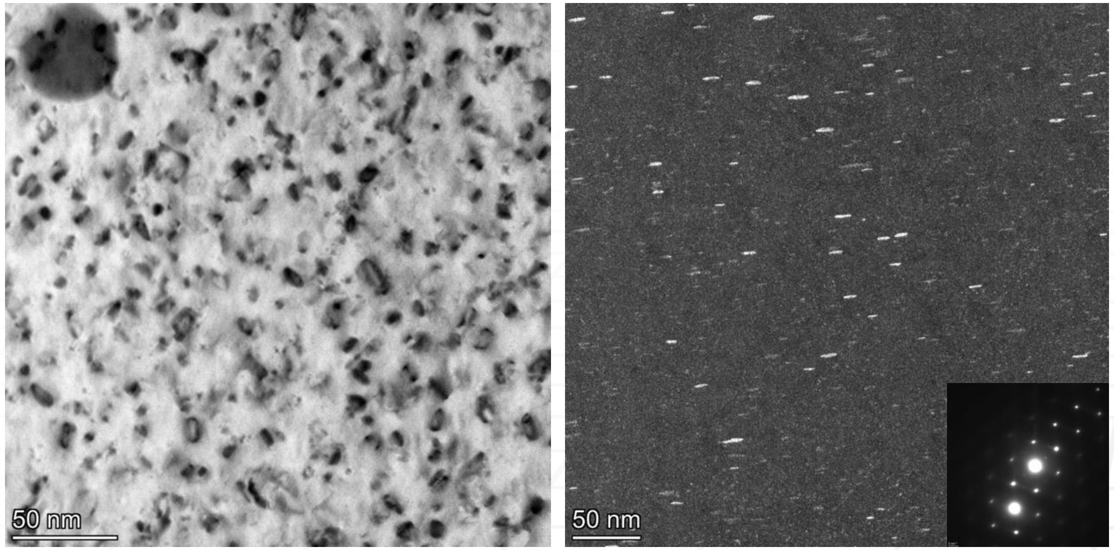
40 mins each side, 700ev, 180uA

Pristine CeO2 specimen

TEM specimen synthesis for radiation damage analysis

Twin-jet Electropolishing

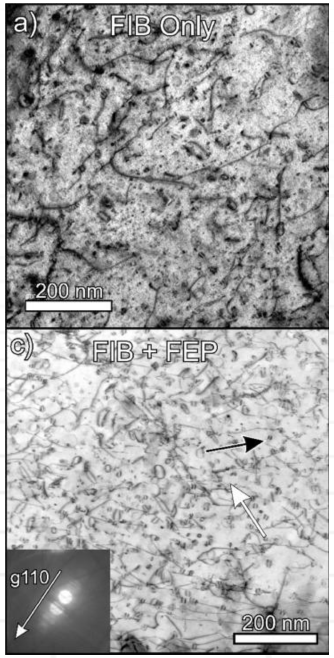
- Perhaps the best way for defect analysis; not possible for surface irradiated alloys

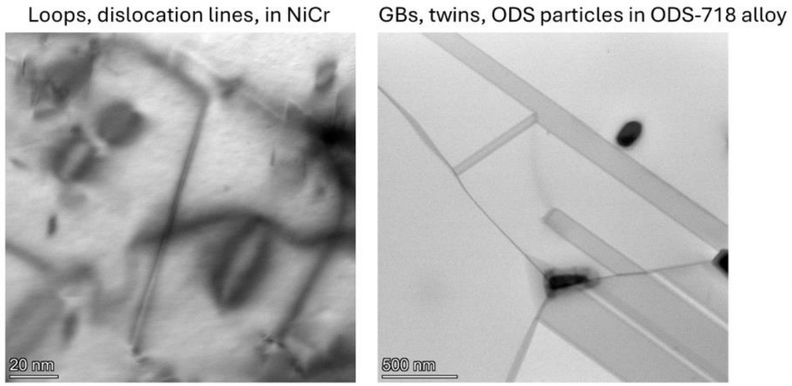


Neutron-irradiated IN718 (NSUF)

FIB + Flash polishing

- Choice of FIB milling parameters to reduce the FIB damage + flash polishing

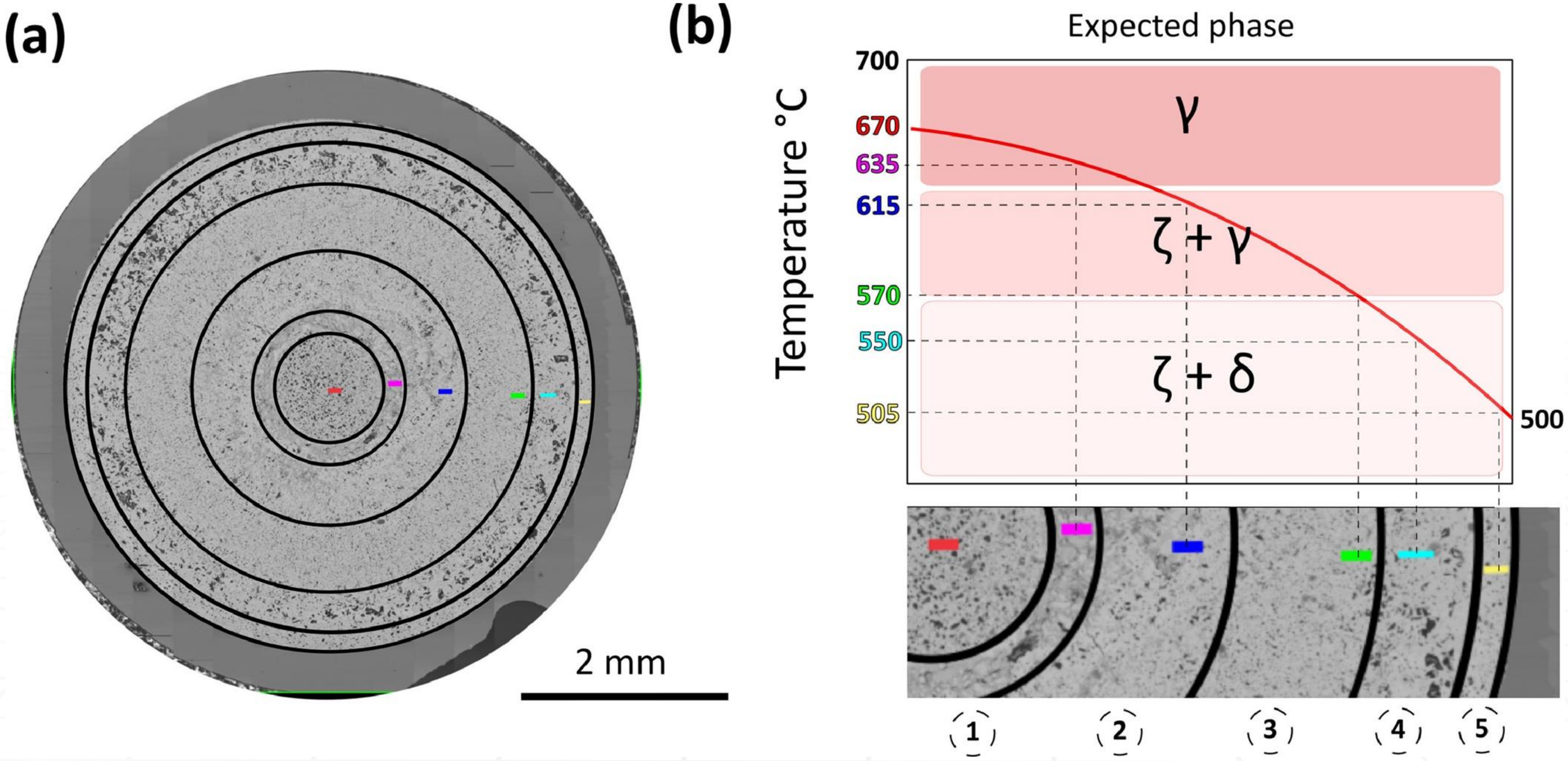




Courtesy: Anshul Kamboj and Sohail Shah

Edwards, Danny J., et al. *Journal of Nuclear Materials* 606 (2025): 155618.

Irradiated Nuclear Fuel sample preparation site specific



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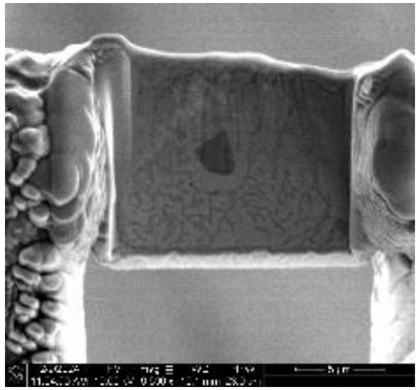
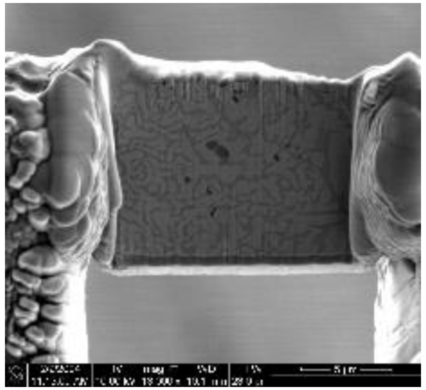
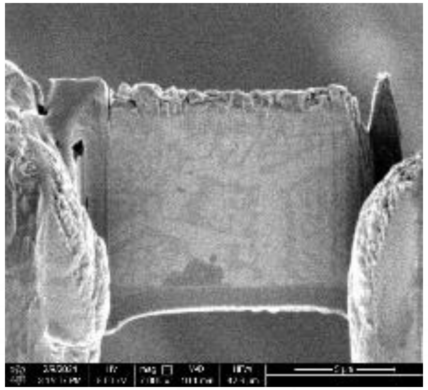
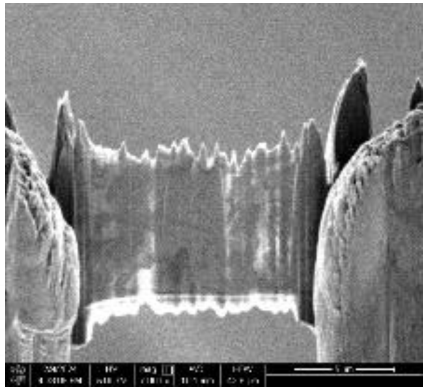
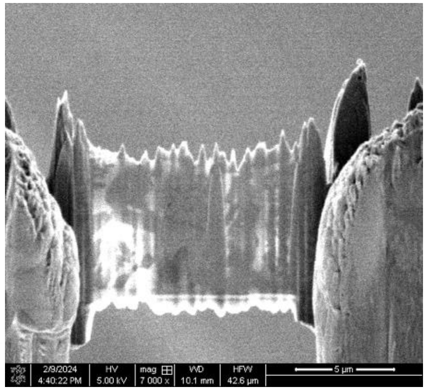
FIB sample preparation
Images taken during thinning matters for TEM characterization


1-1


1-2

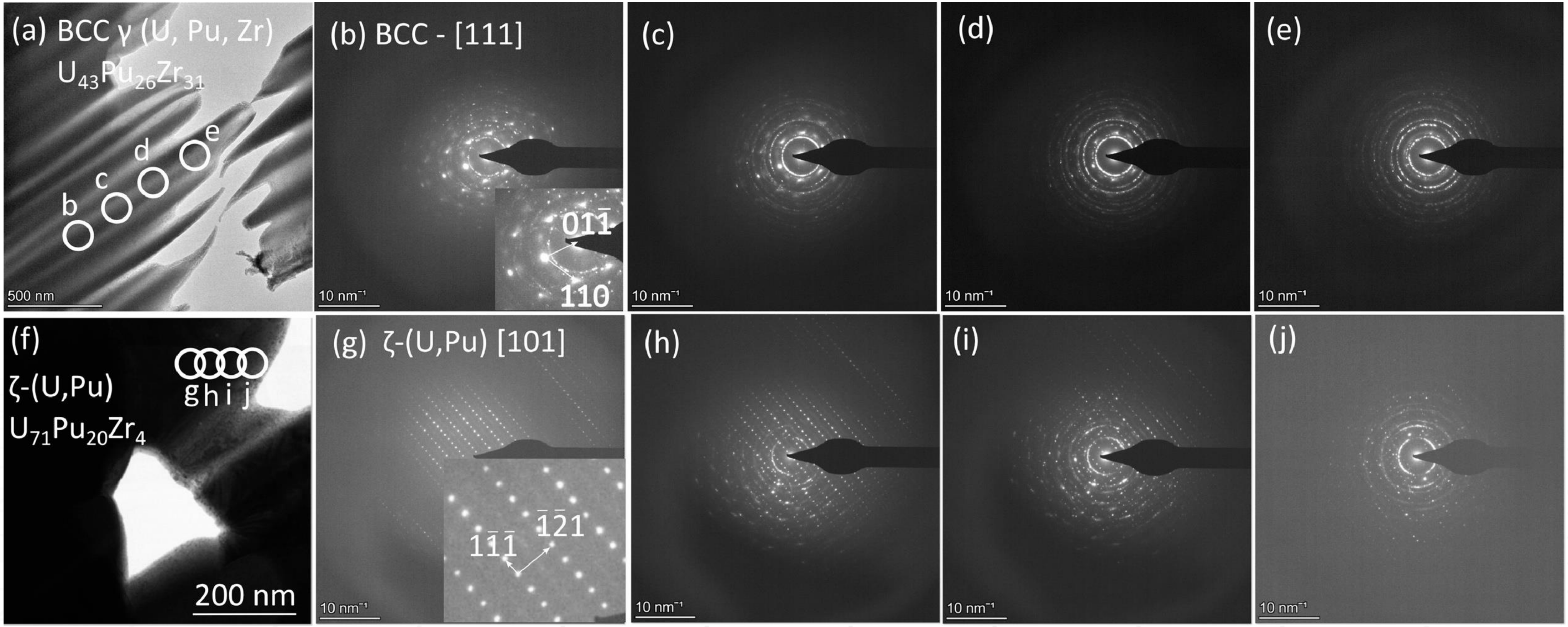

2-1


2-2


2-3

Feature shown in figure 1-1 and 2-1, pre-thinning, is consumed during FIB process.
There is no chance TEM data can inform phase identification as shown in these two images!

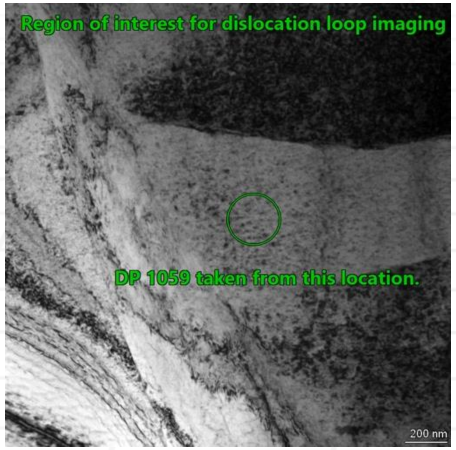
FIB damage may pose a challenging for phase determination

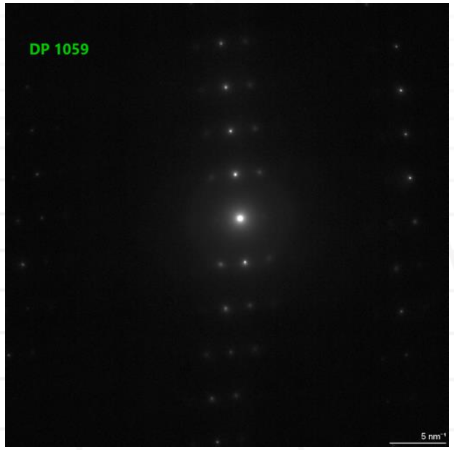


Guidelines to maintain the clarity and traceability of TEM data and linkages over time

Some examples:

- How can we better integrate metadata and data collection workflow?
  - Adding operator details in the metadata
  - Annotations/notes within the data collection software during the data collection stage
  - Saving files with time/date of collection in the file name





Standardization defect analysis for NSUF supported projects

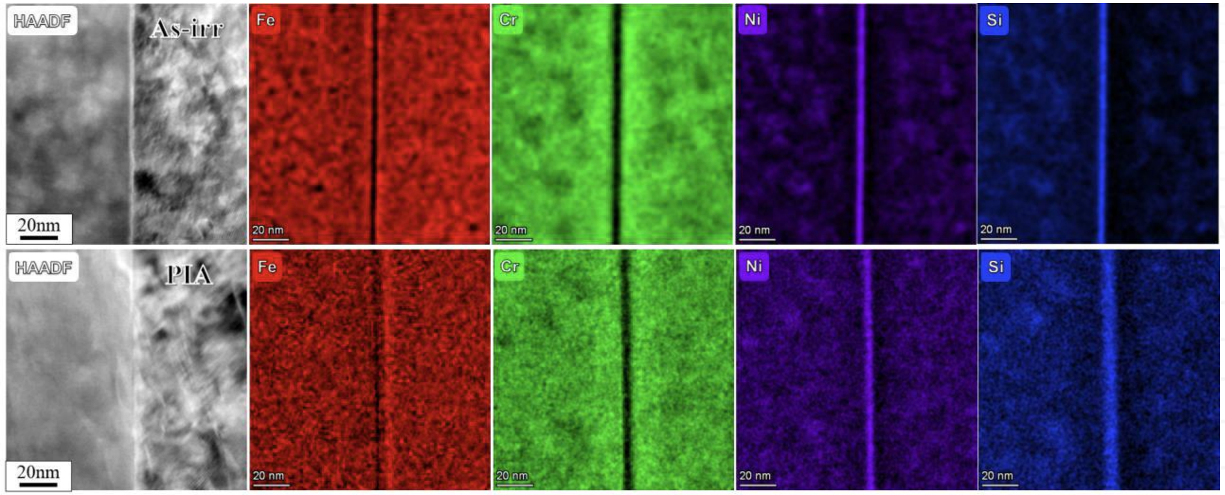
- Reporting the required details to reproduce/utilize the defect data
  - Describing defect imaging conditions (g.b criterion)
  - Statistical information (e.g., how many defects analyzed)
  - Measurement method
  - Defect types (e.g., frank loops versus perfect dislocations)
  - Thickness
  - Scale bar

Guidelines to users to store the underlying data (and metadata) while publishing the results

- E.g., dislocation density as a function of dose produced in published data
  - How can one report the required details (raw data, measurement method, and images etc.) to be transparent and reproduced
    - Sharing via NRDS
    - Data sharing via publication (?)

Guidelines for reporting TEM chemical composition measurements

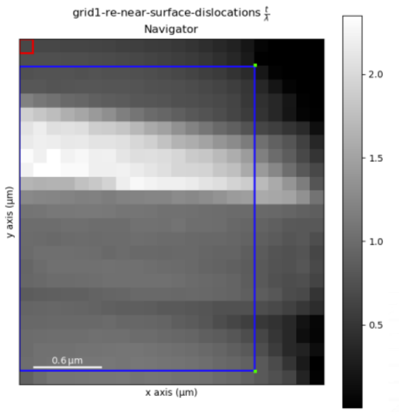
- Best practices for STEM-EDS data collection and storage for traceability
- E.g., RIS: information on GB characteristics and statistical information will be helpful in interpretation
- Any thoughts on STEM-EDS data collection?



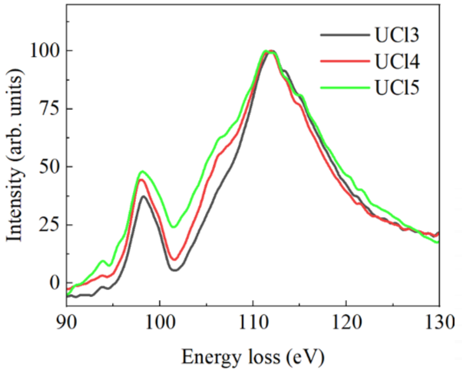
304 SS neutron-irradiated (NSUF RTE)

Python Script Repository on NRDS for Data Processing and Analysis

EELS thickness analysis

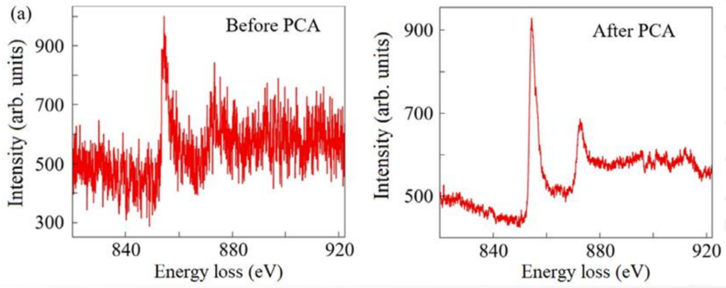


Core loss spectrum data repository for nuclear materials?



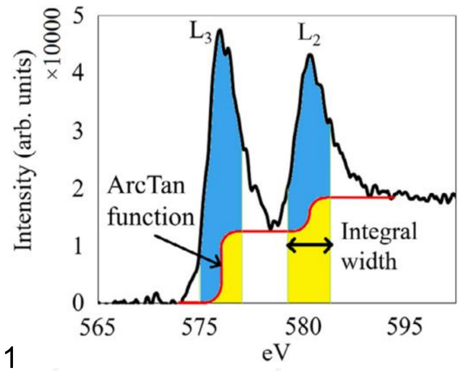
NSUF IS

Denoising EELS spectra



K. Bawane, Scripta Mat., 2021

Oxidation state analysis



4D STEM data analysis

Current workflow at INL

EMPAD on Spectra (.raw)
Merlin on Titan (.mib)

↓

Transfer to HPC to convert to block (.blo) files using Python scripts

↓

Index block files on Nanomegas ASTAR software

↓

Data analysis on EBSD OIM or Oxford Aztec software

4D-STEM using Gatan systems can directly use their new STEMx OIM software for indexing and data analysis

Python scripts for data conversion and analysis can be directly hosted on NRDS

HyperSpy

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TEM characterization request form

Appendix

Table. 1 TEM characterization request form

Check if required	Examination Type	Full name	Description	Requirements	Estimated time for Examination
	STEM-EDS	Scanning Transmission Electron Microscopy-Energy Dispersive X-ray Spectroscopy	EDS is used to analyze the elemental composition of a material by detecting characteristic X-rays emitted when electrons interact with the sample. EDS can provide quantitative information about the distribution and concentration of elements within the sample.	*Example:* *At least three mags for each lamella. Low mag (5-10k), medium mag (50-100k), and high mag (300-500k). At low mag, STEM-EDS for the whole lamella; at medium mag, focus on the feature like grain boundaries and big precipitates; at high mag, focus on the small precipitates around 5-10 nm in the bulk*	
	SAED	Selected Area Electron Diffraction	SAED is a variation of electron diffraction where a selected area of the sample is illuminated with a convergent electron beam.	...	
	EELS	Electron Energy Loss Spectroscopy	EELS is a spectroscopic technique used in TEM to analy the energy loss of electrons as they interact w the sample. Th technique provides information about the electronic structure, chemical bonding, and elemental composition of the material at high spatial resolution.	...	
	EELS-Thickness Map	Electron Energy Loss Spectroscopy-Thickness Map	The function of an EELS Thickness Map to provide nanoscale details about thickness variations withi a sample. It measures the energy loss of electrons passing throug		
			sample.		
	ET	Electron Tomography	Electron tomography involves acquiring a series of TEM images of a tilted sample at different angles and using computational techniques to reconstruct a three-dimensional (3D) representation of the sample's structure. This technique is valuable for studying the morphology and spatial arrangement of nanostructures and complex materials.	...	
	4D-STEM	Four-Dimensional Scanning Transmission Electron Microscopy	4D-STEM combines electron microscopy with diffraction pattern collection at each pixel, offering atomic-scale spatial resolution and	...	

Questions?

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