Overview of MaCS, CAES and Rules for "Hands-on" Activities

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Outline

- Overview: MaCS
- How to access MaCS
- How MaCS provides services
- MaCS Techniques
- MaCS equipment & capabilities
- Applications
- Rules for “Hands-on” Activities
- Contacts

Overview: MaCS

- Microscopy and Characterization Suite (MaCS)
  - Including Advanced Materials and Characterization Laboratory (AMCL) for sample preparation & Atomic-Scale Imaging Laboratory (ASIL) for HRSTEM
- A service center built by CAES in 2010, managed by BSU
- Houses advanced materials characterization equipment for research down to the atomic level (STEM, FIB, LEAP, SEM, Nanoindenter, XRD, etc.)
- Houses sample fabrication (LPBF 3D printer, etc.) and preparation tools
- Partner of Nuclear Science User Facilities (NSUF)
- Uniquely designed to allow characterizing radiological materials
- Provides Post-Irradiation-Examination (PIE)
- https://caes.org/caes-lab/macs/

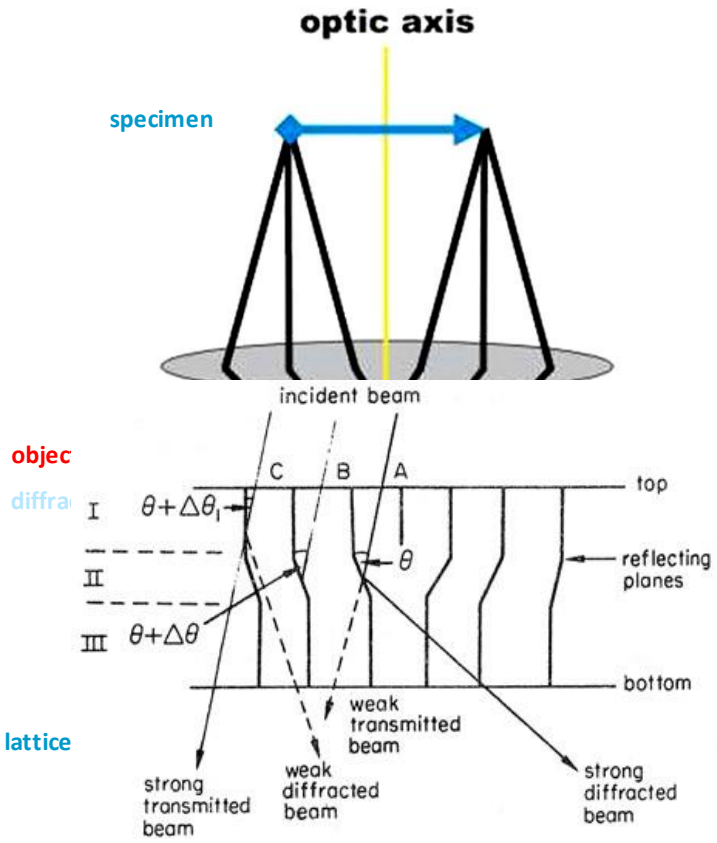
How to Access MaCS

- MaCS is largely accessible to scientists, faculty and students through:
  - Nuclear Science User Facilities (NSUF) Awards, such as the RTE solicitations and the CINR Funding Opportunity Announcements (FOAs); https://nsuf.inl.gov/
  - Non-NSUF Users: contact MaCS -> set up user accounts -> submit service requests -> schedule instrument sessions with MaCS staff -> work with Instrument Leads on experiments & data analyses. https://caesenergy.org/caes-lab/macs/
- Easy access to university faculty, students and Lab scientists.

How MaCS Provides Services

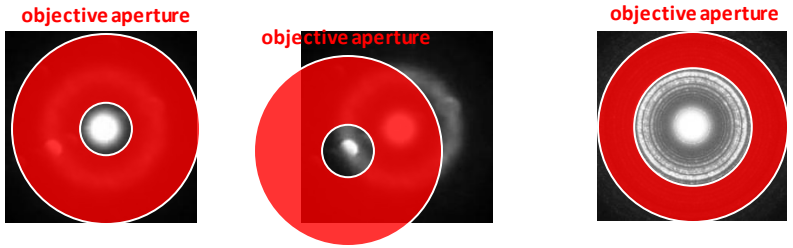
- Full service: MaCS staff do everything for the users;
- MaCS staff + Users: users can be trained to operate the instrument(s) and do data analysis;
- Signed off users work by themselves with limited support from MaCS staff.

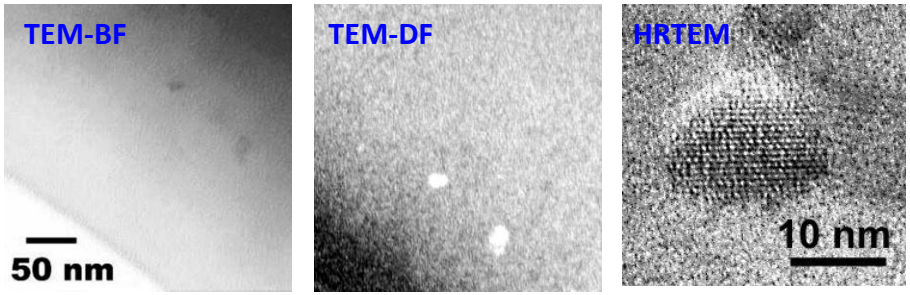
State-of-the-art techniques – Transmission Electron Microscopy (TEM) Imaging



Practical Electron Microscopy in Materials Science, J. W. Edington

Electron diffraction patterns





diffraction contrast

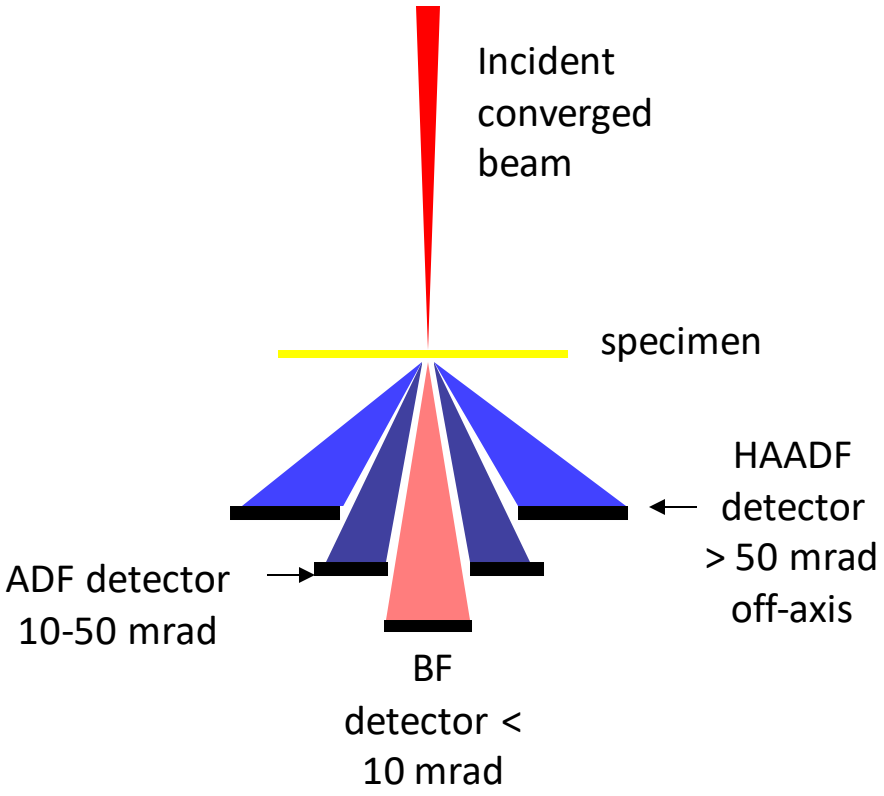
phase contrast

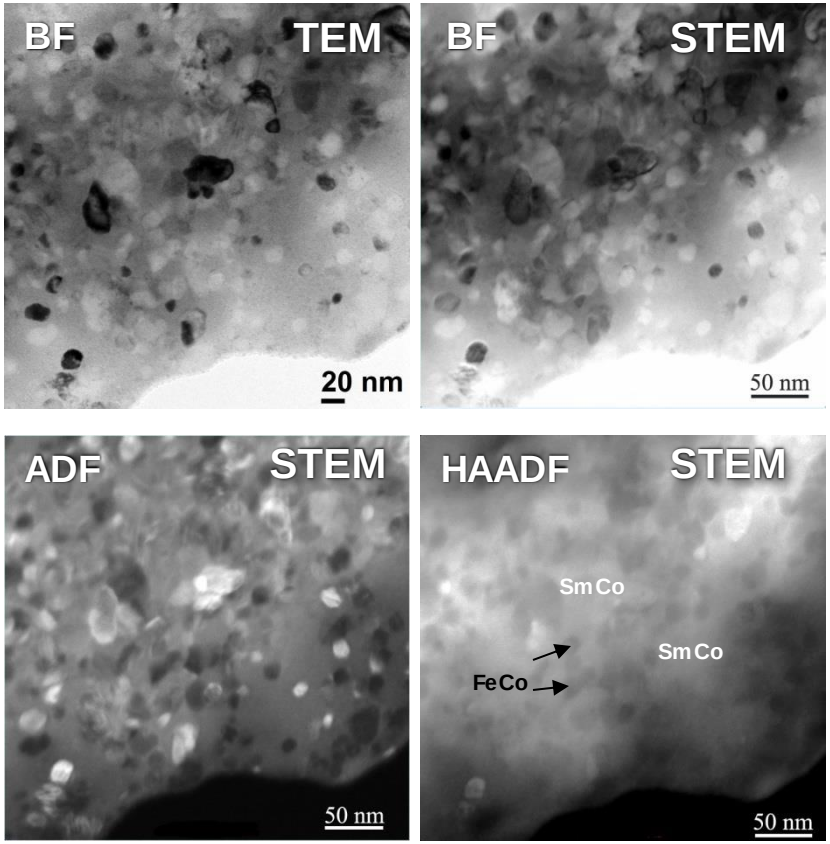
Nanocrystalline phase in amorphous matrix

High Resolution Transmission Electron Microscopy (HRTEM)

Inherent 2D information

State-of-the-art techniques – Scanning TEM (STEM) Imaging





Inherent 2D information

S/TEM – Analytical Techniques

- Energy Dispersive X-ray Spectroscopy (EDS)
  - Chemistry analysis, both qualitative and quantitative.
  - Limitations on light elements.
- Electron Energy Loss Spectrometry (EELS)
  - Chemistry analysis, both qualitative and quantitative.
  - Sample thickness measurement.
  - No limitations on light elements.
- Energy Filtered TEM (EFTEM)
  - Individual elemental mapping.
  - Practically some elements are difficult to map due to their edge energy either too low or too high.
- Electron Tomography
  - Reconstructing three-dimensional (3D) morphology/inner structure information.
  - Combined with other techniques (e.g., EFTEM).

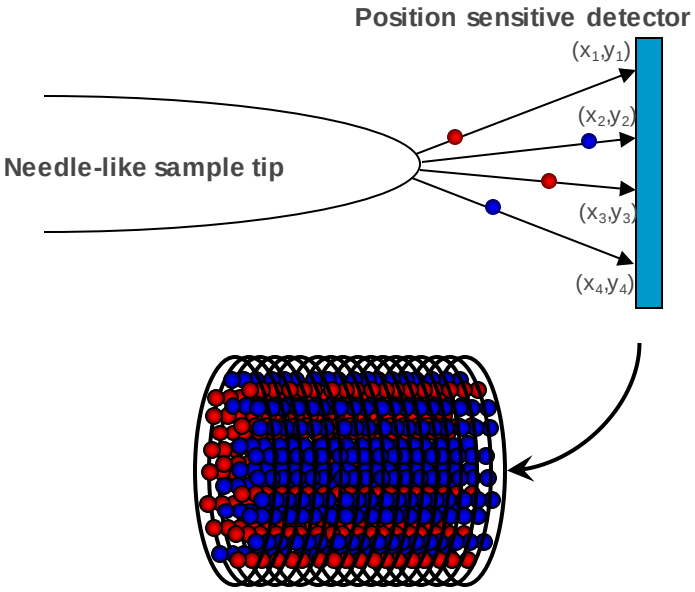
State-of-the-art techniques – Atom Probe Tomography (APT)

What is LEAP?

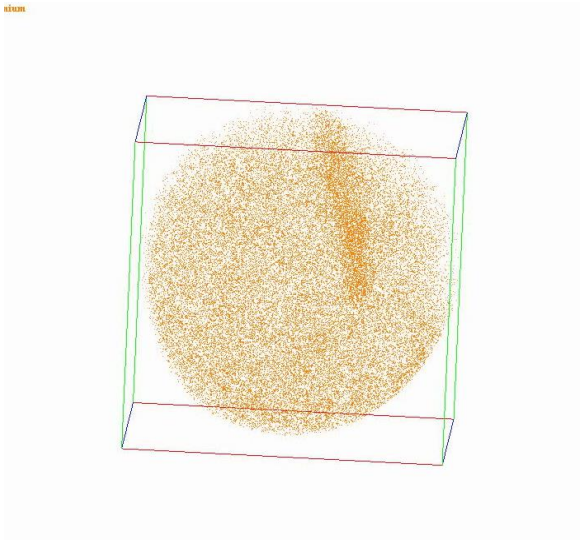
LEAP: Local Electrode Atom Probe, the latest and most advanced 3-dimensional atom probe.

LEAP “provides nano-scale surface, bulk and interfacial materials analysis of simple and complex structures with atom by atom identification and accurate spatial positioning”— CAMECA

State-of-the-art techniques - APT



One dot is one atom.

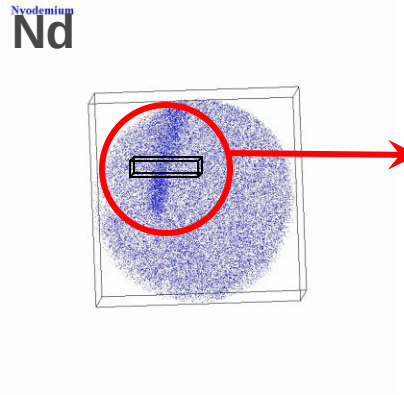


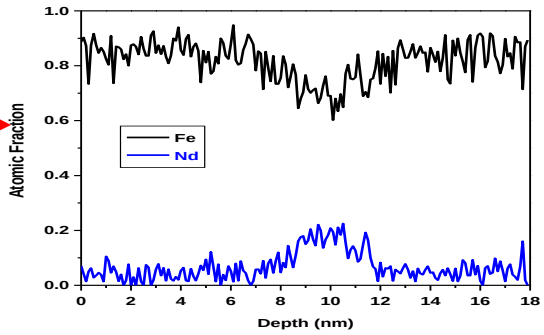
Atom positions:

(x, y) by position sensitive detector;

(z) by calculation.

Atom's identity by time-of-flight mass spectrometry





$(NdYDy)_2Fe_{14}B$

APT is the only technique to obtain atom position/chemistry information in three-dimensional space at atomic level.

MaCS Equipment & Capabilities
- STEM (1): ThermoFisher Spectra 300

- Highlights/Specifications
  - **Monochromated, probe- and image- corrected**
  - **30 – 300 kV HT for various applications**
  - Image and 5th order probe aberration correction (S-CORR).
  - Single electron sensitive STEM detection
  - Integrated differential phase contrast (iDPC) imaging for the study of magnetic and electrical properties and for optimized Z-contrast imaging from hydrogen to uranium
  - **Super-X 4 detector EDS system (≤136 eV for Mn-Kα and 10 kcps (output)), Gatan Continuum super-fast DualEELS (<30 meV) and ASTAR/NanoMEGAS techniques**
  - Drift corrected frame integration (DCFI)
  - **Electron microscope pixel array detector (4D-STEM)**
  - Field-free imaging in TEM Lorentz mode with 2 nm resolution for magnetic property studies
  - Provides quantitative characterization down to sub-angstrom level
  - Brings the highest resolution imaging and spectroscopic capabilities for a wide range of samples
  - Fischione Tomography & Gatan double-tilt Heating Holders

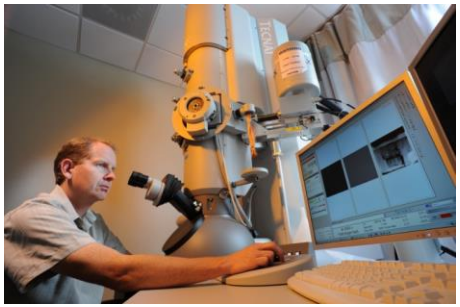
Spectra 300 STEM

Energy Resolution (EELS)	0.2 eV (Mono ON)
Energy Resolution (EELS)	0.025 eV (@30 kV) (UltiMono)
Information limit	60 pm
STEM resolution	50 pm (125 pm @ 30 kV)

Note: All specifications are at 300 kV (unless otherwise noted).

MaCS Equipment & Capabilities
- STEM (2): ThermoFisher Tecnai G² F30 FEG

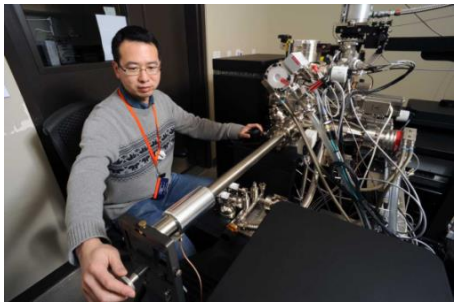
- Highlights/Specifications
  - Analytical Scanning TEM with Field Emission Gun (FEG).
  - 300 kV.
  - STEM HAADF detector (Z-contrast imaging).
  - EDS, EELS, EFTEM and Electron Tomography.
  - TopSpin (ASTAR): EBSD in TEM/Strain Mapping
  - Fischione Tomography holder
  - Humingbird Tomo holder for LEAP samples
  - Bruker (Hysitron) PicoIndenter
  - Humingbird heating stage (1000 ºC, for non-rad)



Tecnai F30 STEM

Energy spread	0.8 eV
Information limit	0.14 nm
STEM resolution	0.19 nm

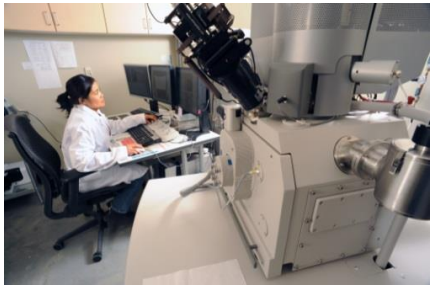
MaCS Instruments & Capabilities - APT: CAMECA LEAP 4000X HR



- 3-dimensional atom probe at near atomic resolution.
- Voltage & UV laser, energy compensated reflectron, provides high mass resolution and better signal-to-noise ratio.
- Field of view > 150 nm.
- Accurate quantitative analysis.
- Statistic analysis.
- Ideal for analyzing phase boundary elemental segregations, nanosized precipitates/second phase/clusters.
- Suitable for wide ranges of materials: metals and advanced materials; semiconductor and microelectronic devices; data storage.

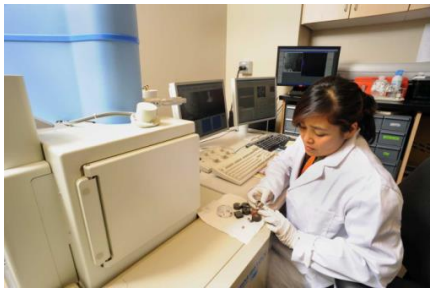
MaCS Instruments & Capabilities
- Dual-beam FIB/SEM: ThermoFisher (FEI) Quanta 3D FEG

- Dual beam FIB with Field Emission Gun (FEG) (FIB + SEM)
- Omniprobe/minipulator – for TEM & LEAP samples
- EDS, EBSD and STEM detectors
- Bruker PicoIndenter



- SEM: JEOL JSM-6610LV

- EDS, EBSD and Cathodoluminescence detectors
- Variable chamber pressures
- For both inorganic and organic materials



- Microhardness Tester: Leco LM247AT

- Micro-indentation hardness tester for non-rad materials
- A variety of objectives from 2.5X to 100X
- Load ranges from 1gf to 2000gf
- Manual or auto turret

BOISE STATE UNIVERSITY

https://caes.org/caes-lab/macs/

CAES Center for Advanced Energy Studies

MaCS Instruments & Capabilities
- NI/AFM/MFM: Bruker (Hysitron) TI-950 TriboIndenter

- Nanoindenter: nanometer scale indentation
- Atomic Force Microscope (AFM)
  22,500 mm^2 testing area
  Resolution: Z-0.7 Å, X/Y-7 Å
  Scan range: Z-4.5 µm, X/Y-40 µm
- Magnetic Force microscope (MFM)



- Rigaku SmartLab XRD

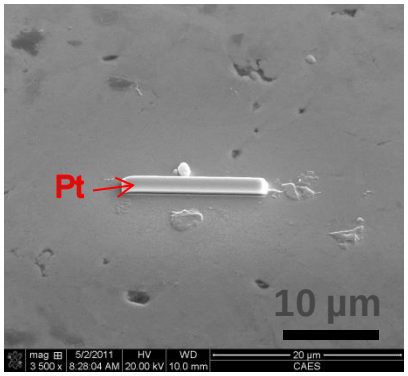
- Focusing and parallel beam geometries
- Cu-Target, 20-60 kV, 2-60 mA
- Detector : NaI (SC) and photomultiplier with preamplifier (D/tex)
- Graphite monochromator: for outstanding resolution
- Mapping stage: 50 mm translation
- Sample types: Powder, bulk, thin film



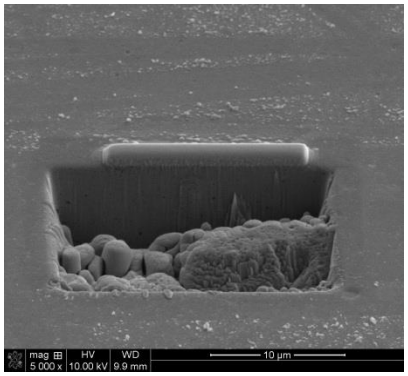
Sample Preparation in MaCS

- Using FIB for making both TEM and LEAP samples
  - No limitation on materials.
  - Site preference.
  - Compared with traditional methods, higher successful rate, safer and cleaner working environment and usually takes shorter time.
  - Small sample size (a few microns), important for RAD materials.
  - TEM sample may be thicker, difficult for HRTEM imaging.
  - Ga damage.
  - May be costly.

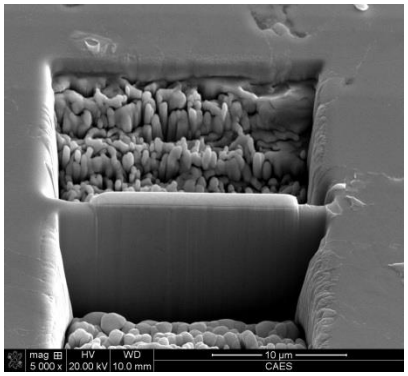
TEM Sample Preparation - FIB



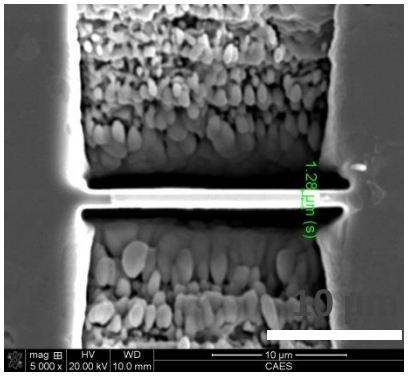
Deposit Pt protection layer



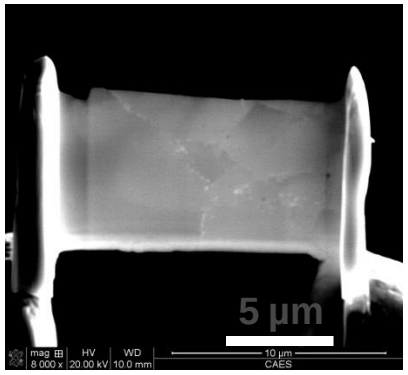
Milling one side



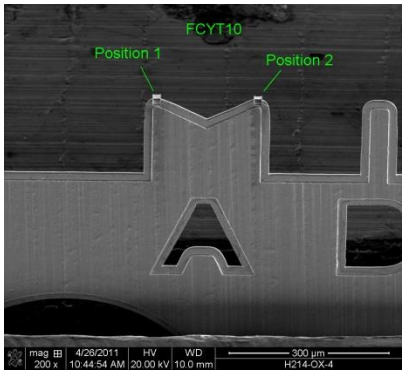
Milling the other side

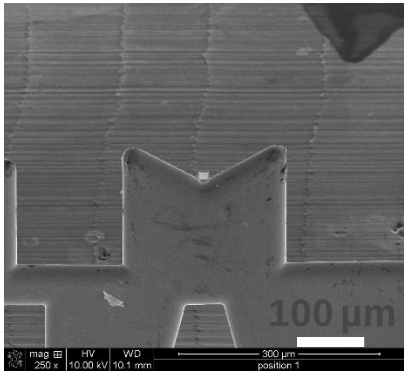


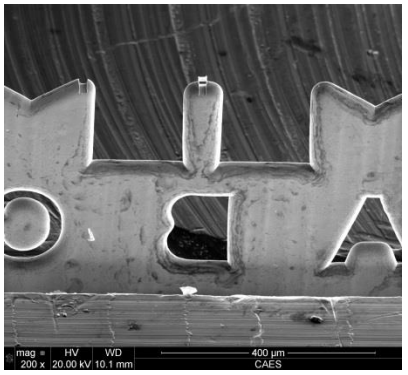
Top view



Lift out with a W wire



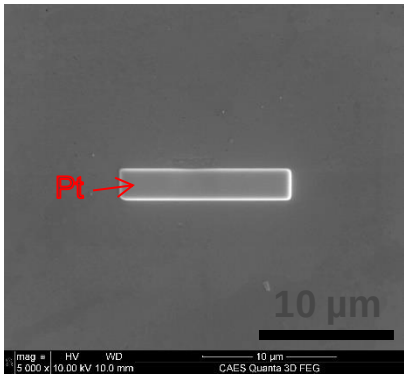




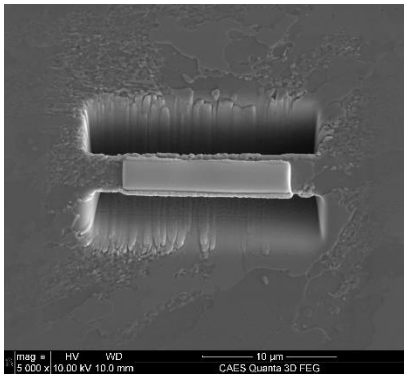
Mounted to TEM Cu supporting grid

ODS FCAY alloy

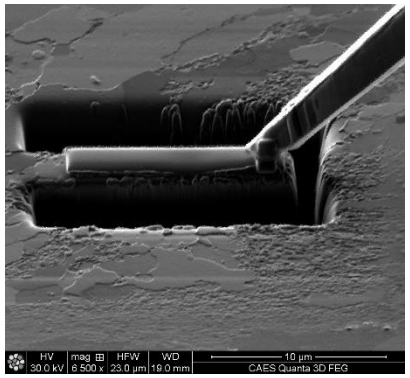
LEAP Sample Preparation - FIB



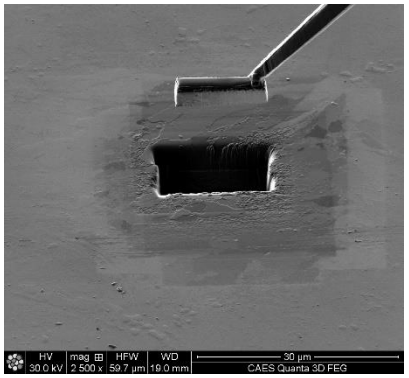
Deposit Pt protection layer



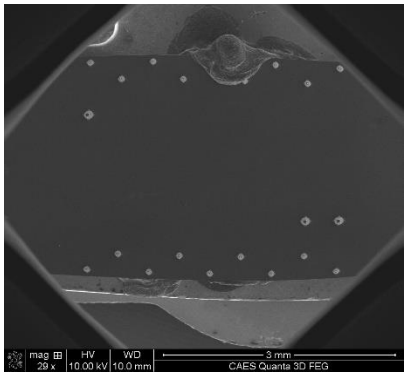
Cut from both sides



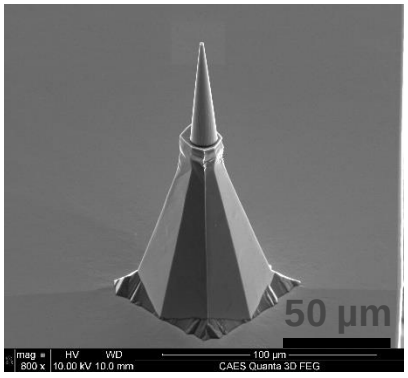
Attached to W wire tip at one end



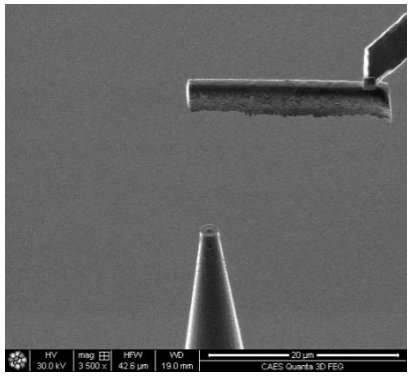
Lift out



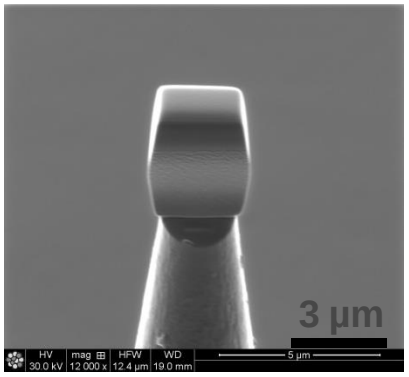
Coupon with micro Si tips



Micro Si tip



Mounted to Si tip

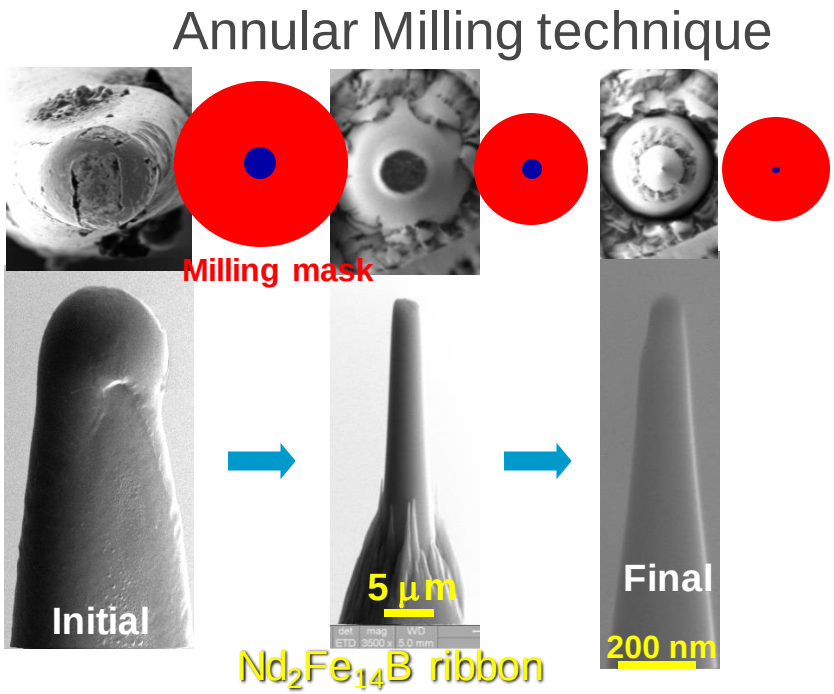


Cut for LEAP tip making

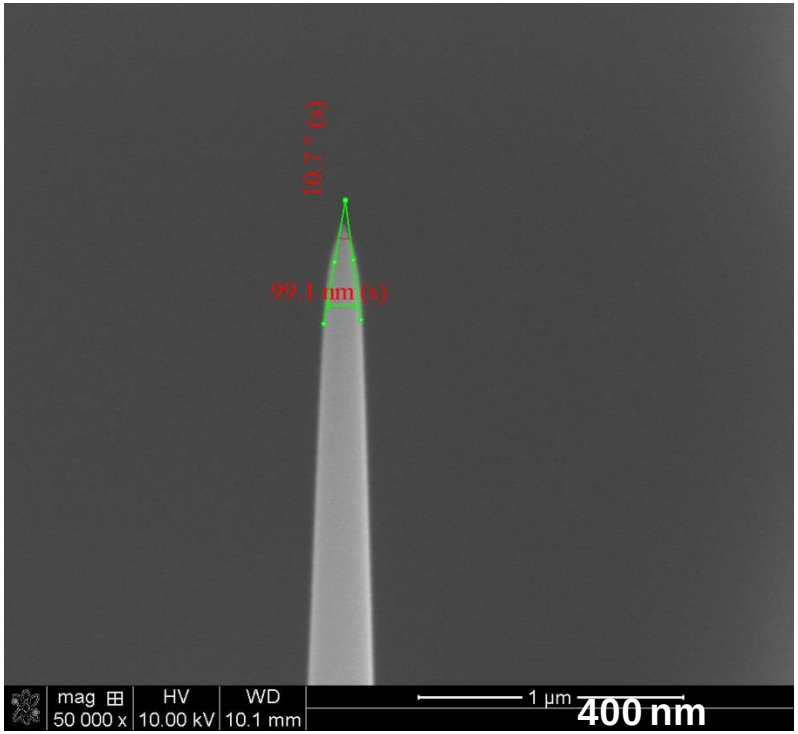
ODS FCAY alloy

LEAP Sample Preparation - FIB

➢ **Final steps:**



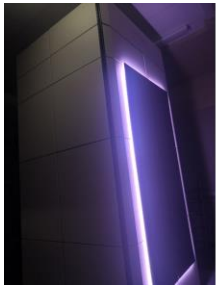
M.K. Miller, et al., Microsc. Microanal. 13, 428, 2007



A final LEAP tip of an ODS FCAY alloy sample. The tip size is much less than 100 nm, which is essential for atom probe run.

MaCS Equipment & Capabilities: Characterizations

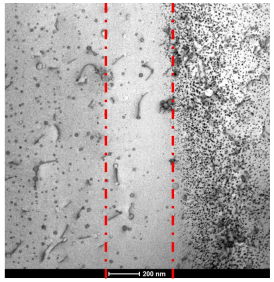
- Characterize non-radiological and low activity radiological materials.
- STEM, EDS, EELS, FIB, LEAP, SEM, XRD, Nanoindentation, etc.
- Advanced microstructure characterization and chemical analysis down to atomic level, sub-Angstrom with the newly installed STEM.
- Mechanical testing down to nanoscale.



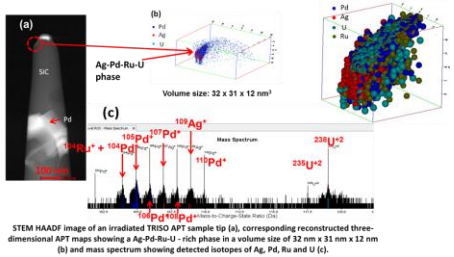


Spectra 300 STEM

Damage-Free Sample Prep. - FIB + Nanomill



Combination of TEM + APT.



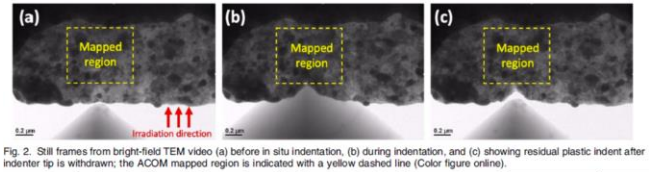
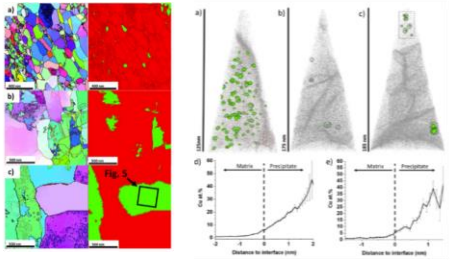


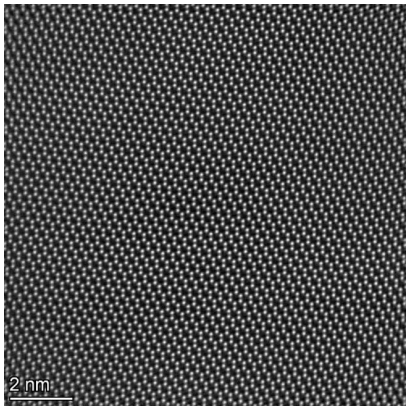
Fig. 2. Still frames from bright-field TEM video (a) before in situ indentation, (b) during indentation, and (c) showing residual plastic indent after indenter tip is withdrawn; the ACOM mapped region is indicated with a yellow dashed line (Color figure online).

Irradiation-Induced Phase Instabilities in Cu-Ta Heterophase Nanocomposite

TEM/PED + APT



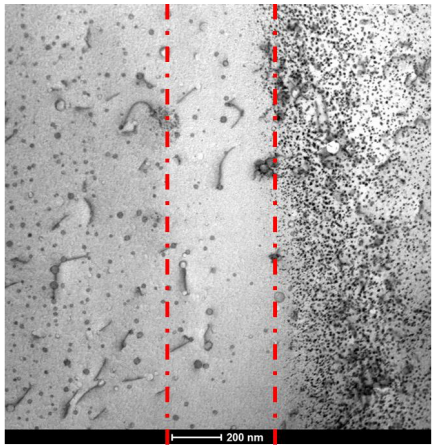
Nano-mechanical in situ Testing in TEM

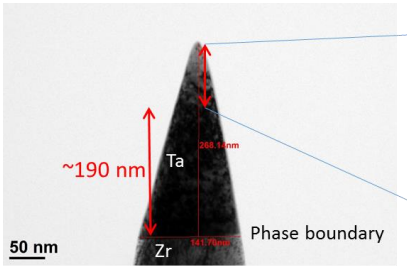


Ga_2O_3 Sensor

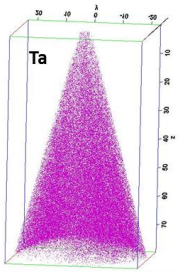
MaCS Instruments & Capabilities - Damage-Free Sample Preparation

- FIB + Nanomill
- Remove the damaged layers on the sample surface from the FIBing process
- Thinning samples for HRTEM
- Apply to both TEM lamellae and APT tips

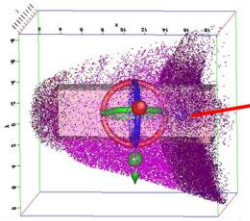




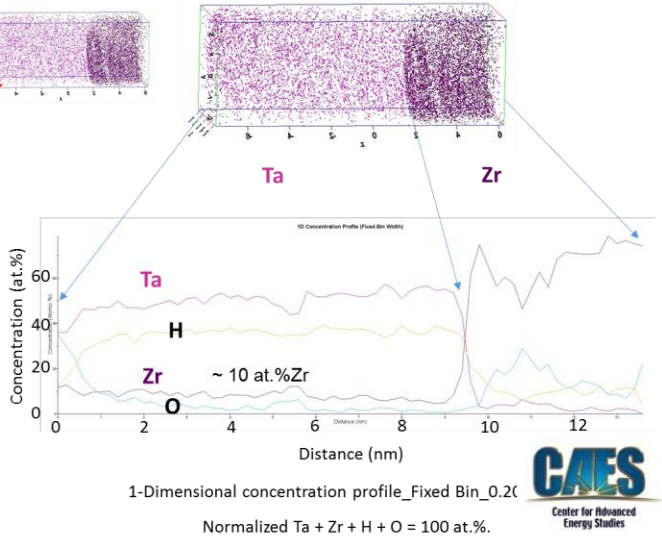
BF-TEM



APT



Selected volume size:
5.0 nm x 5.0 nm x 15.0 nm



volume size: 47.4 nm x 46.5 nm x 79.6 nm

Y.Q. Wu, et al., "APT Composition Profiling for Accurate Evaluation of Diffusion Coefficients in the Zr-Ta Binary System", TMS 2021 Virtual, March 15-18, 2021. Supported by **NSUF RTE 19-1741**.

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BOISE STATE UNIVERSITY

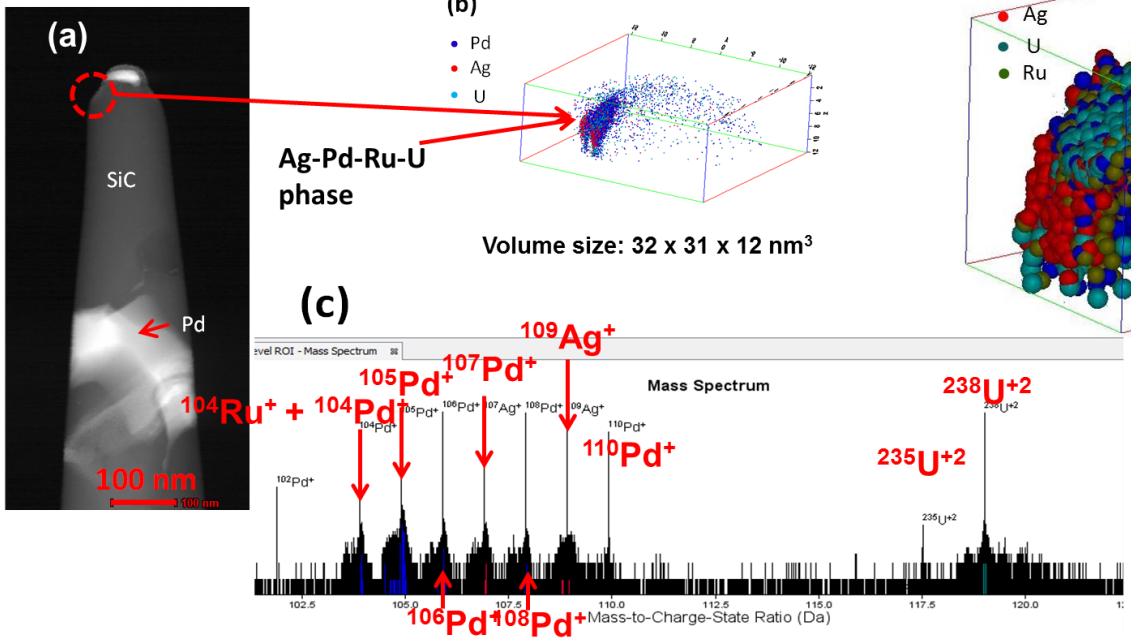
CAES Center for Advanced Energy Studies

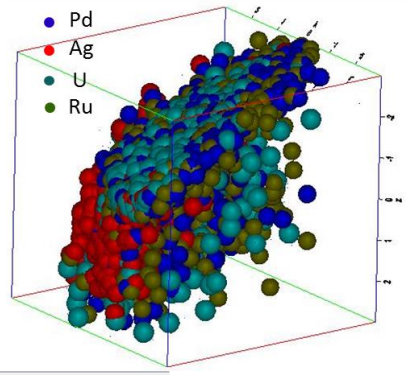
MaCS Instruments & Capabilities
- Correlative Study by Combining Multiple Techniques

- Combination of TEM and APT techniques
  - Hummingbird Tomography TEM holder - to characterize APT sample tips in TEM before running them in the LEAP, to fully utilize the advantages and overcome the shortcomings of the two techniques.

	APT	TEM
Sample form	Needle-shape; conductive (semi-conductor & ceramic under lase-assisted)	ϕ3mm disk or smaller; can be inconductive
Experiment	Destructive; sample can be used only once	Non-destructive; sample is reusable
Data collection condition	Ultra-high vacuum with sample cooled at 20 ~ 100 K	Ultra-high vacuum; sample can be at different temperature condition
Data form/resolution	**3-D/atomic level**	**2-D/ atomic to nano level**
Data field of view	**Nanometers**	**Nanometer to micron**
Data information	**Analytical/chemistry (quantitatively)**	**Micro-/nano-structures/crystallographic information/HRTEM/chemical mapping (STEM EDS, EELS, EFTEM)**
Analytical limitation	No limitation on any element	EDS: light elements limitation/resolution

MaCS Instruments & Capabilities
- Correlative Study by Combining Multiple Techniques





Element	Concentration (at.%)	Error (at.%)
Pd	9.014	0.241
Ag	2.738	0.120
U	2.523	0.126
Ru	0.387	0.059
Si	50.218	0.292
C	31.466	0.218
O	3.654	0.061

3D elemental map of Pd, Ag, U and Ru in a selected small volume of 6 nm x 6 nm x 6 nm, and quantitative data of this phase based on physics predictions of the isotopic inventory, and normalized Si+C+O+Pd+Ag+U+Ru = 100%.

STEM HAADF image of an irradiated TRISO APT sample tip (a), corresponding reconstructed three-dimensional APT maps showing a Ag-Pd-Ru-U - rich phase in a volume size of 32 nm x 31 nm x 12 nm (b) and mass spectrum showing detected isotopes of Ag, Pd, Ru and U (c).

MaCS Instruments & Capabilities
- In-situ Nano-mechanical Testing in TEM and FIB

- Indentation, compression and tensile testing

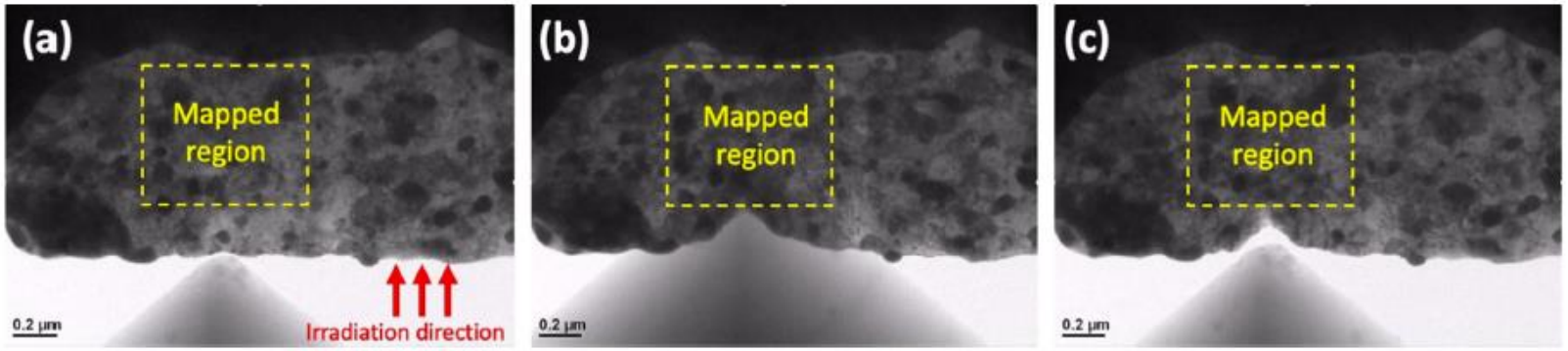


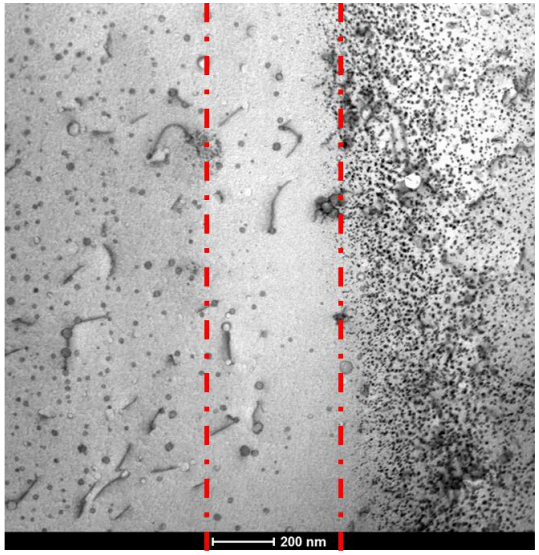
Fig. 2. Still frames from bright-field TEM video (a) before in situ indentation, (b) during indentation, and (c) showing residual plastic indent after indenter tip is withdrawn; the ACOM mapped region is indicated with a yellow dashed line (Color figure online).

Irradiation-Induced Phase Instabilities in Cu-Ta Heterophase Nanocomposite

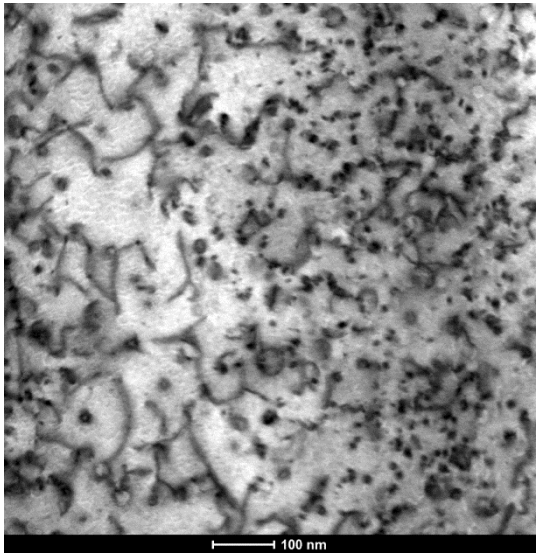
MaCS Instruments & Capabilities
- Improved Defects Imaging through BFSTEM

Ion irradiated ODS MA956 (Fe-Cr-Al-Y_2O_3) at 190C

2.5 dpa



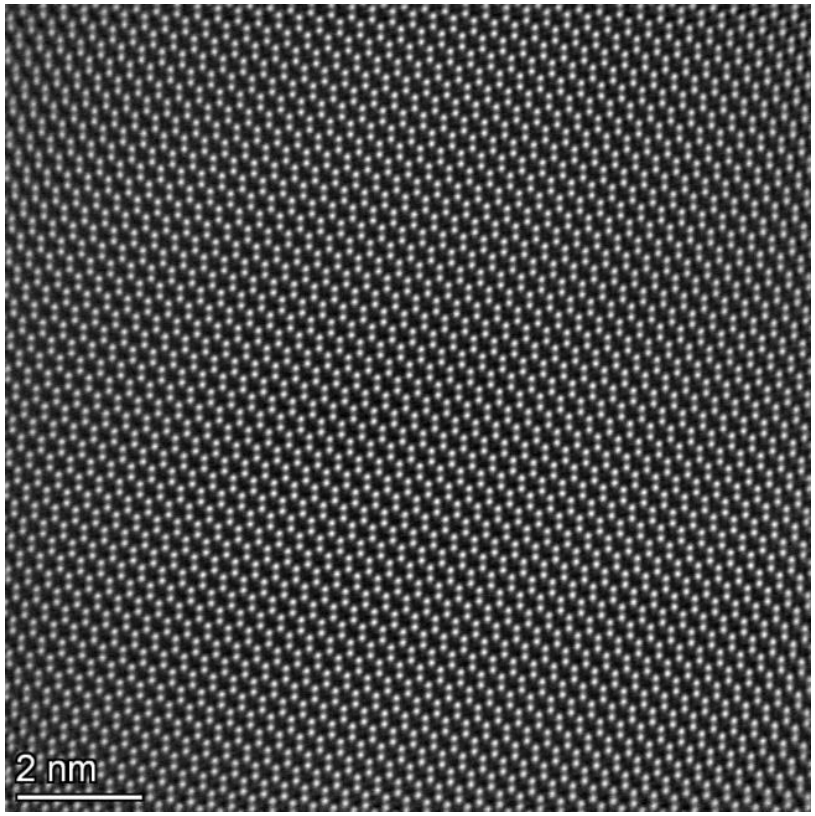
100 dpa



CINR 18-14784, Y. Lu et al., ANS 2021 & TMS2022

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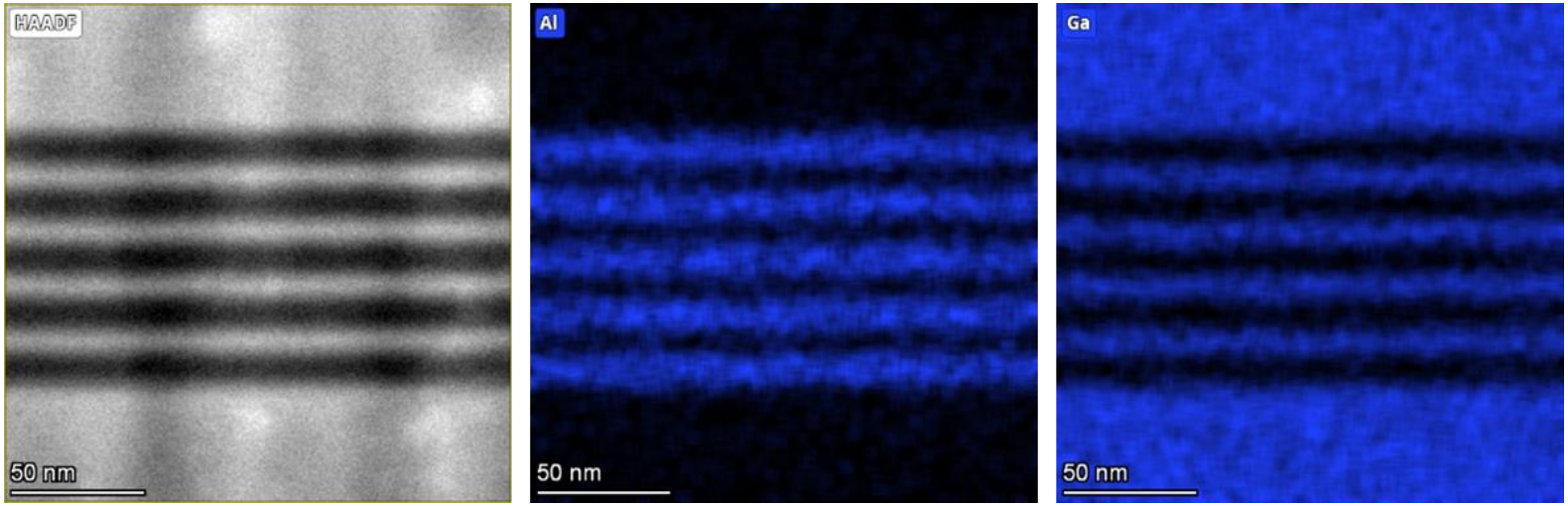
Spectra 300 STEM – Applications - HRSTEM



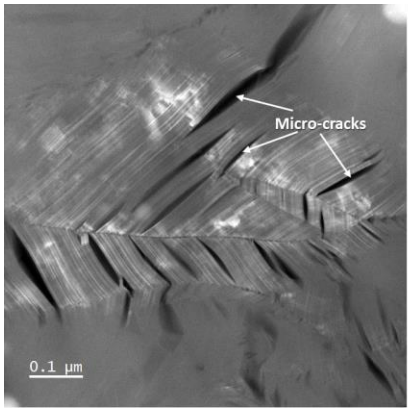
NSUF CINR 21-24020: Ga2O3 Sensor.

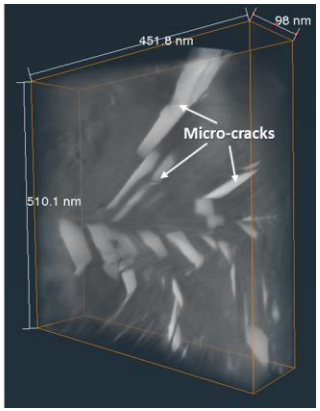
Spectra 300 STEM – Applications - EDS

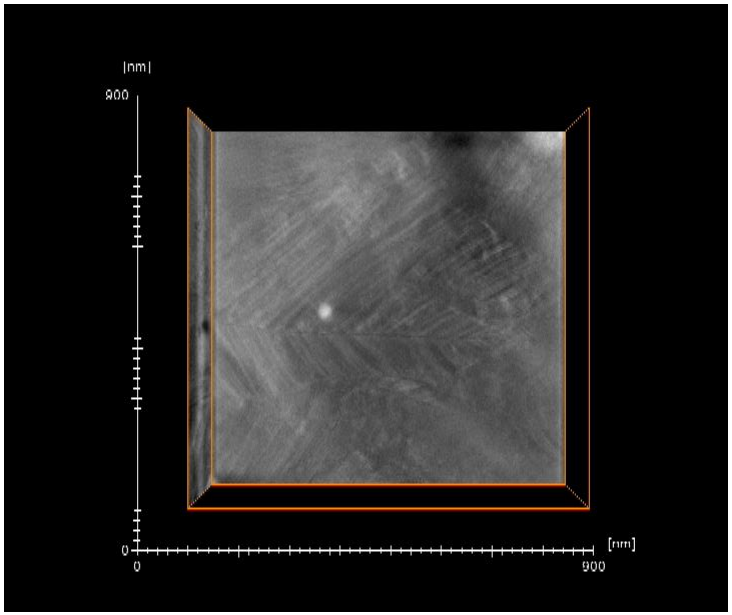
- Super-X EDS mappings



Spectra 300 STEM – Applications – Electron Tomography

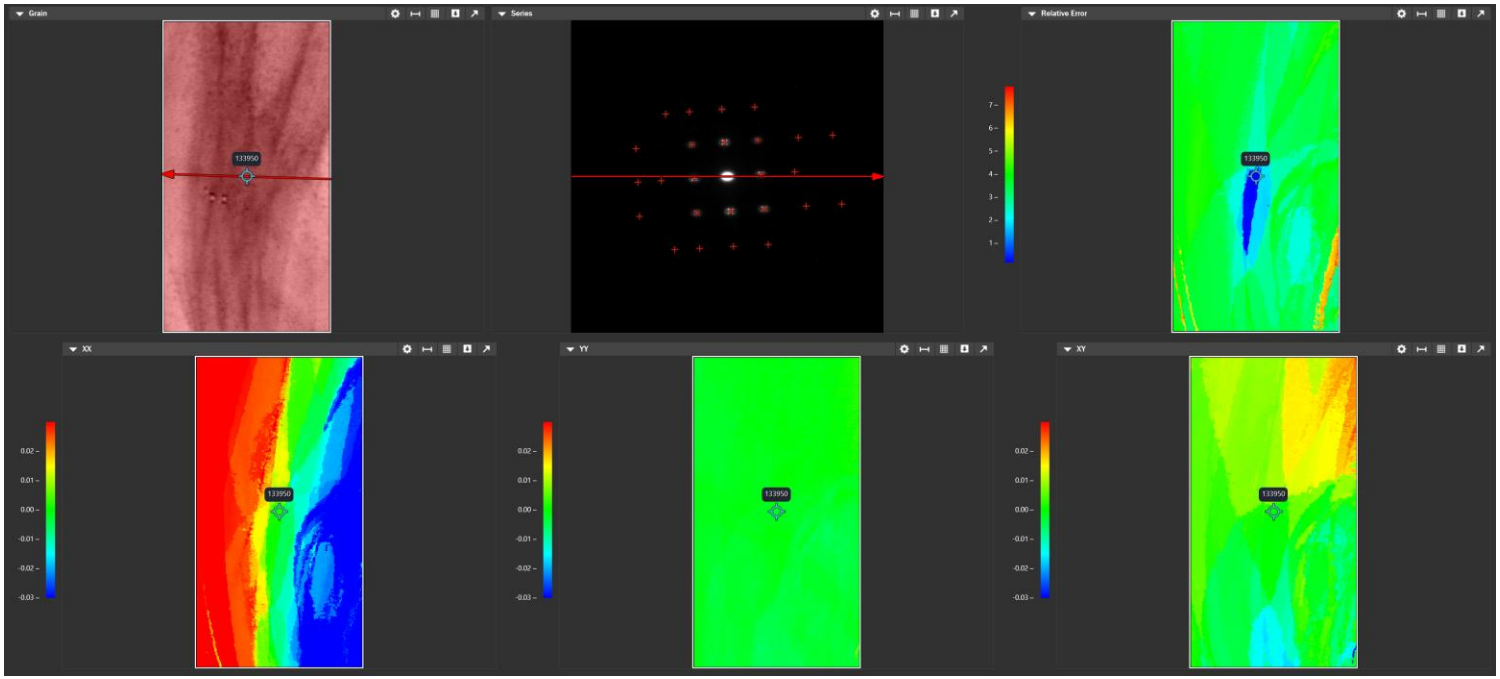






Electron Tomography of nuclear graphite cracks.

Spectra 300 STEM – Applications – ASTAR Strain Mapping

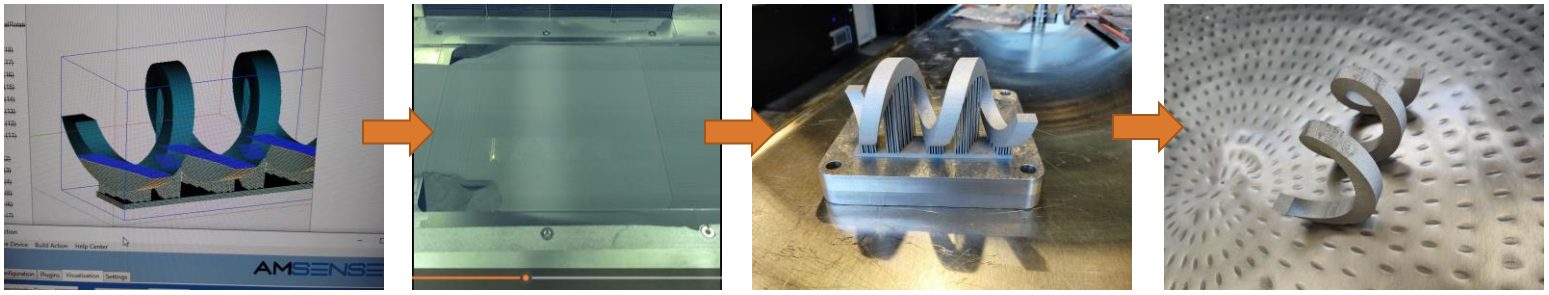


Strain distribution of Ga2O3 acquired by precession electron diffraction

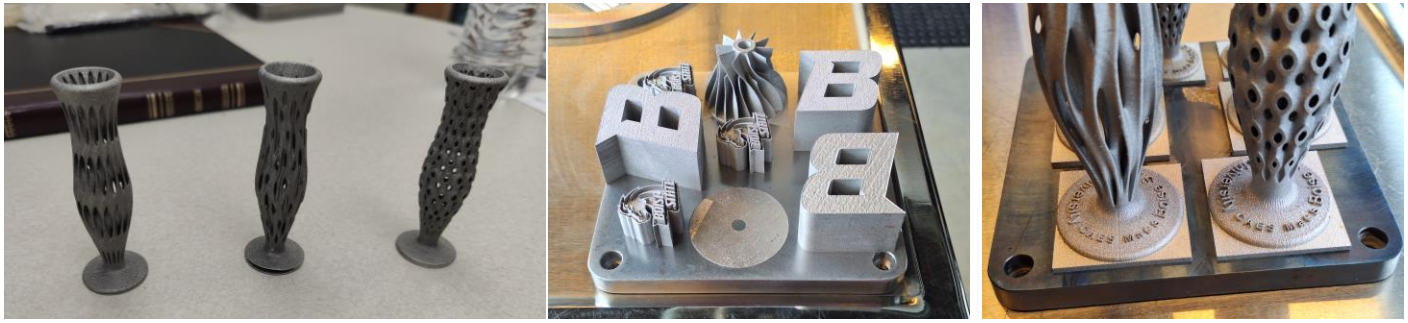
MaCS Equipment & Capabilities: Fabrications - LPBF: Open Additive Panda 3D Metal Printer



- 1 MaCS staff member and 1 INL scientist are trained to operate the printer.
- Materials: 316L only for now.



Design Printing Printed Final



MaCS Equipment & Capabilities: Fabrications
- Dr. Sinter: Spark Plasma Sintering System



- Ball-mill



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MaCS Instruments & Capabilities – Sample Preparation



Non-rad sample prep glovebox



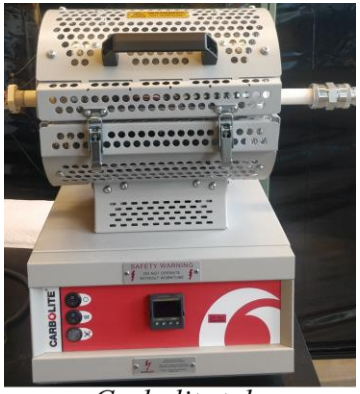
Rad sample prep glovebox



Gatan PIPS II-ion mill



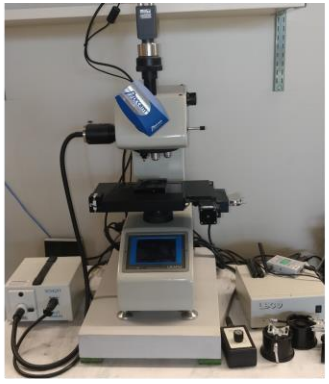
Instron Test Frame Model 5967



Carbolite tube furnace



Keyence VH-250L Microscope



Leco AMH43 Microhardness tester

Rules for “Hands-on” Activities

- Dress codes:
  - Long pants
  - Closed-toe shoes
  - No headphones/earbuds
- Work with MaCS Staff
  - Safety first!
  - Follow MaCS Staff’s instructions
  - Do NOT enter rad area unless guided
  - Clean after yourselves
  - Ask questions!
- Three groups rotate among AMCL, MaCS and ASIL
  - AMCL – hands-on practice: polishing samples; overview tools.
  - MaCS – hands-on practice: operating SEM: sample movement, focusing, correcting Astigmatism, imaging, etc.
  - MaCS and ASIL – observe staff/users working on the instruments: Spectra, FIB, etc.

Contacts

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- https://caesenergy.org/caes-lab/macs/
- https://caesenergy.org/caes-lab/aml/

Thank you for your attention!