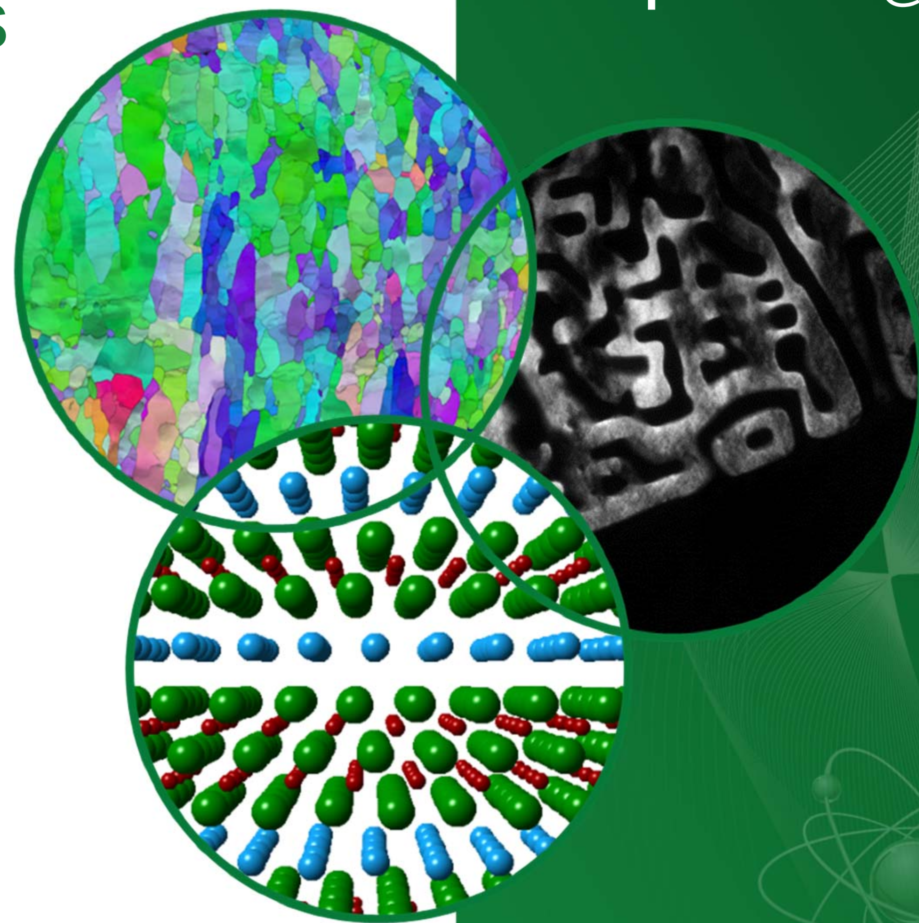
parishcm@ornl.gov

Irradiated materials characterization capabilities at ORNL LAMDA



Chad M. Parish

Collaborators: Kun Wang, Phil Edmondson, Tyler Gerczak, Xunxiang Hu, Josh Schmidlin, Michael McAlister, Kevin Field, Kory Linton, Yutai Katoh, Keith Leonard.

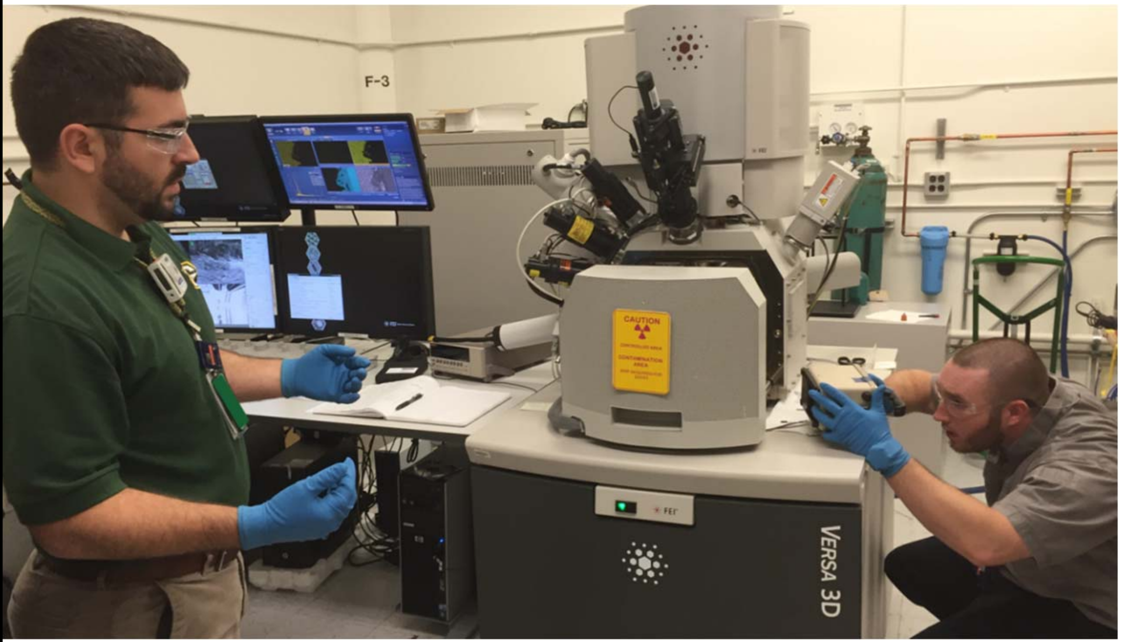
ORNL is managed by UT-Battelle
for the US Department of Energy

OAK RIDGE
National Laboratory

LAMDA: Low Activation Materials Development and Analysis

- ~~LAMBDA~~: there is no "b"
- Capabilities for thermal, mechanical, and microstructural analysis of irradiated materials
- Generally <100 mr/hr @ 30 cm (some exceptions, can occasionally go higher with additional work controls)
- Generally α kept as low as possible, and with additional work controls

Both clean suites and contamination zones



OAK RIDGE National Laboratory

Three LAMDA thrusts

1. **Thermal properties**
2. **Mechanical properties**
3. **Microstructural characterization**

Coefficient of Thermal Expansion

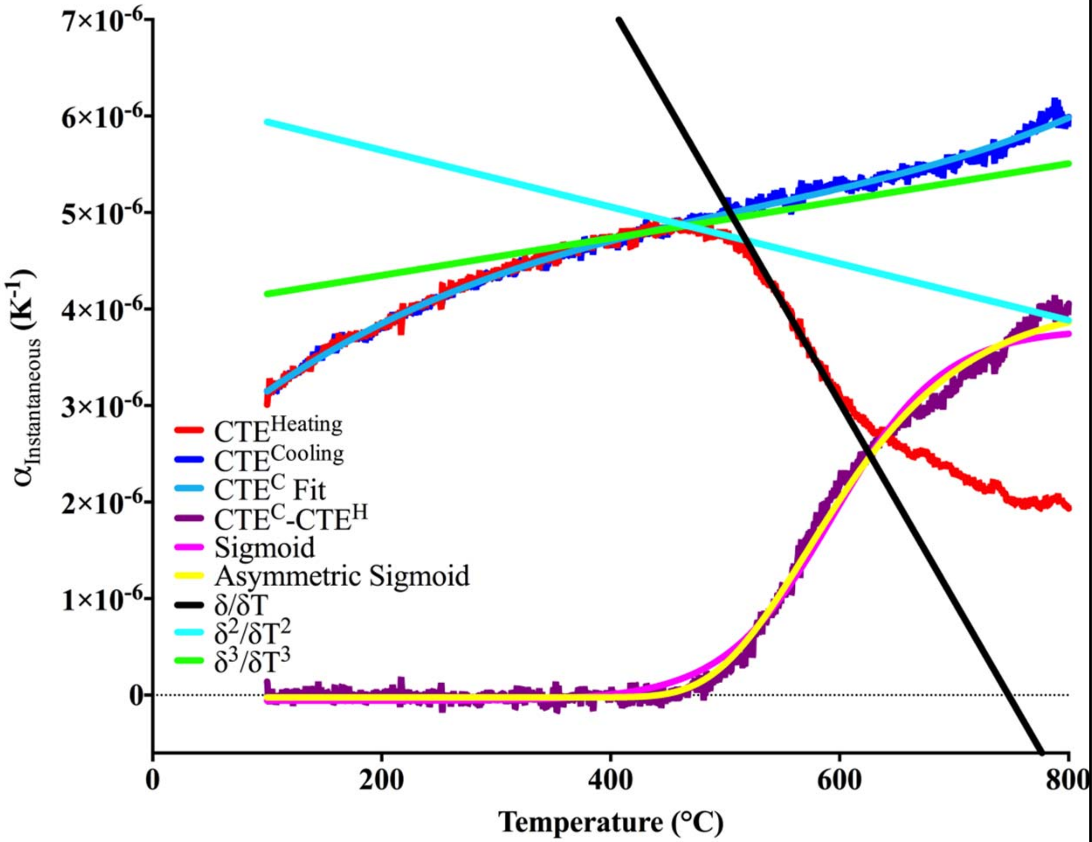
NETZSCH Dilatometer DIL 402 CD

- **Room temperature - 1600ºC**
- **Dual push-rod dilatometer**
  - **2 samples per run**
- **Vacuum tight**
- **Titanium gettered helium flow during measurement**
  - **O_2 partial pressure <10^{-10} ppm**

OAK RIDGE National Laboratory

Thermal Annealing of SiC for Passive Irradiation Temperature Measurement

- **Annealing of radiation defects begins at temperatures just higher than irradiation temperature**
  - **Indicated where the red and blue lines begin to deviate**
- **Difference of the two curves (purple) modeled with an asymmetric sigmoid (yellow)**
  - **Find the first, second, and third derivatives**
  - **Fit lines to heating curve (red) at each derivative**
  - **Intersection of lines and cooling curve (blue) indicates temperatures of interest**

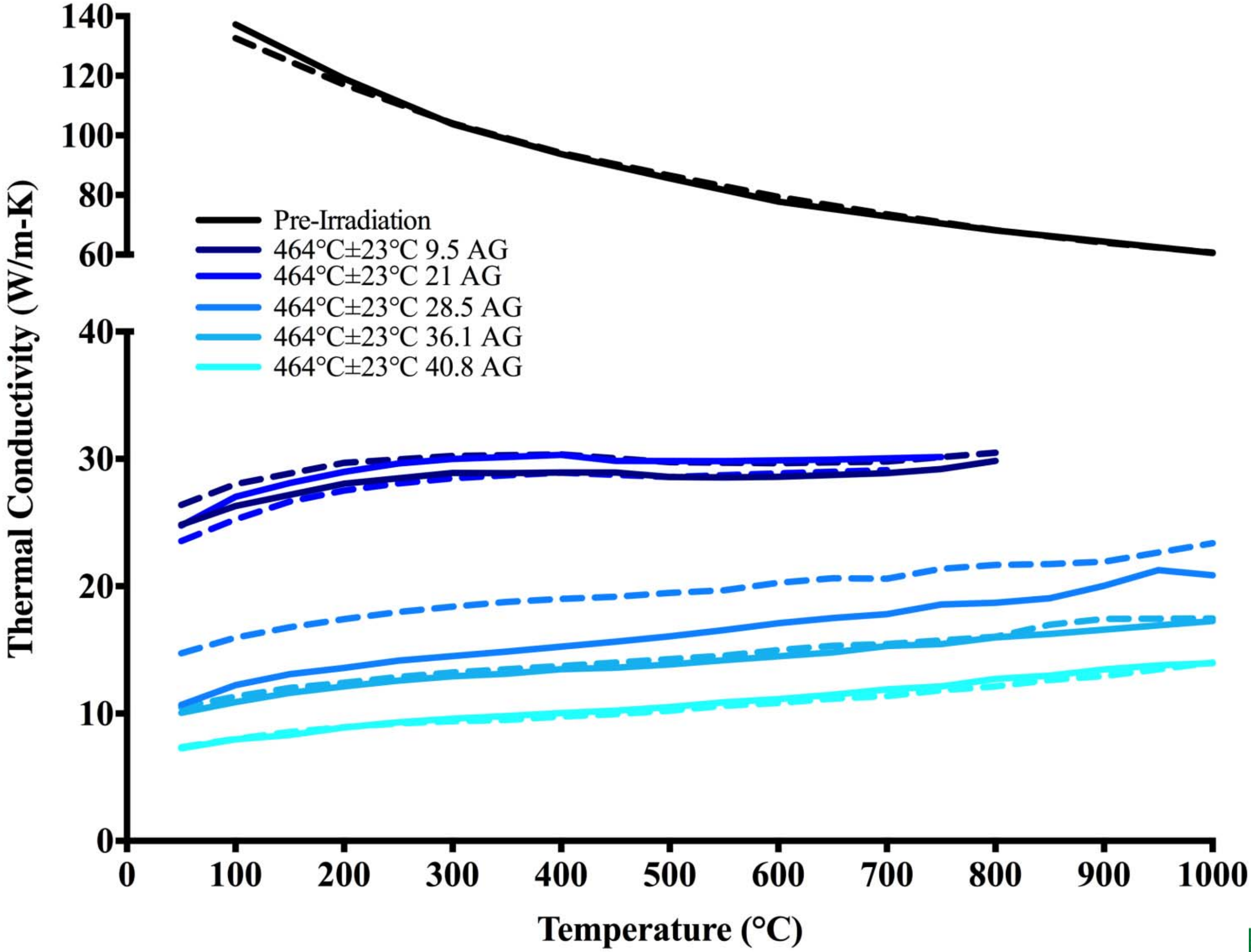


Thermal Diffusivity / Conductivity

- **NETZSCH LFA 447 NanoFlash Xenon Flash Thermal Diffusivity Apparatus**
  - **Room temperature – 300°C**

- **NETZSCH LFA 457 MicroFlash Laser Flash Thermal Diffusivity Apparatus**
  - **50ºC – 1100ºC**

Thermal Conductivity Loss from Neutron Irradiation



A.A. Campbell et al., Carbon, V109, (2016) 860-873

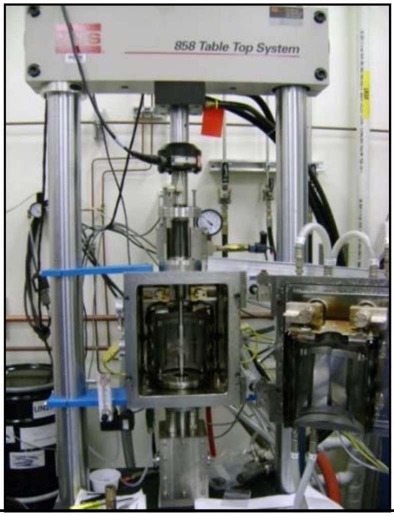
Three LAMDA thrusts

1. Thermal properties
2. **Mechanical properties**
3. Microstructural characterization

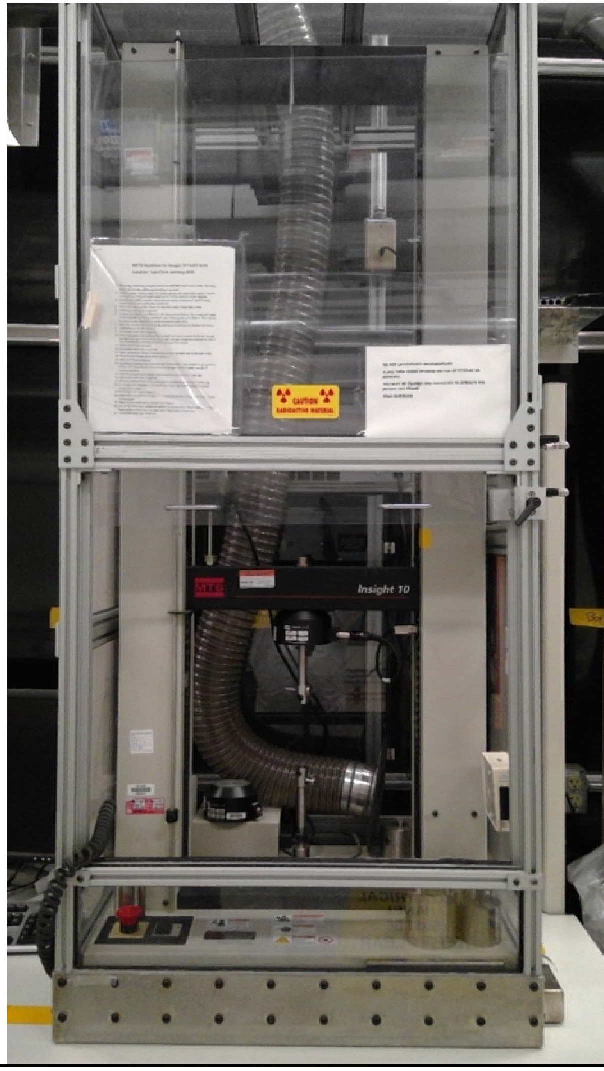
Multiple mechanical test frames

- **MTS Insight 858**
- **MTS Insight 30**
- **Instron 4301**

- **High-temperature, high-vacuum capabilities**
- **Liquid N2 temperature capabilities**
- **Tensile properties and fracture properties**



MTS Insight-10 Mechanical Test Frame



Capabilities

- Electro-mechanical system in ventilated enclosure
- 10kN capacity
- Crosshead Travel: 1100mm
- Test Speed: 0.001mm/min – 500mm/min
- Tension, compression, and flexural testing at room temperature.

Enclosure + HEPA snorkels allow testing of brittle materials

Test Resource 160 Series Torsion Test Machine

Capabilities

- **125Nm Torsion System**
- **Adjustable speed to 8 RPM**
- **Digital Encoder for angular position measurement & control (1 arc min resolution)**
- **24"L x 7"W horizontal table**

Tinius Olsen Impact 104

Capabilities

- **Pendulum impact tester**
- **Charpy or Izod configurations**
- **30J Capacity**
- **Testing temperatures from -196 to 400^{0}C**

Buehler Wilson VH3100 Microhardness Tester

Capabilities

- **Automated Knoop/Vickers Hardness Tester**
- **10kg load capacity**
- **5 Mp camera**

Sonic Velocity Measurement System

Figure 1. Photograph of the analysis computer, pulser, and digitizer.

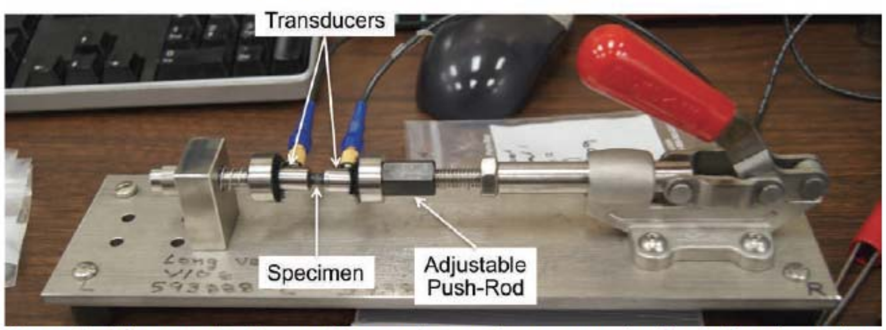


Figure 2. Photograph of a specimen and transducers setup in the designed specimen holder.

Capabilities

- **Measure Young's and shear moduli with the sonic velocity methodology according to ASTM C769 (graphite) and ASTM C1419 (refractories)**

Three LAMDA thrusts

1. Thermal properties
2. Mechanical properties
3. **Microstructural characterization**

ORNL LAMDA lab

nsuf
Nuclear Science
User Facilities

FEI DualBeam FIB-SEMs

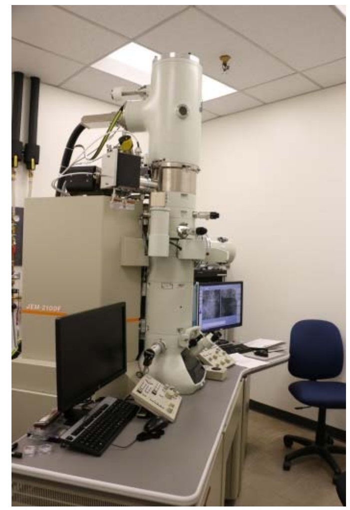
Quanta3D (Cryo)

Quanta3D (Shielded)

Versa3D FEG

Fischione Nanomill

S/TEMs



JEOL JEM2100F

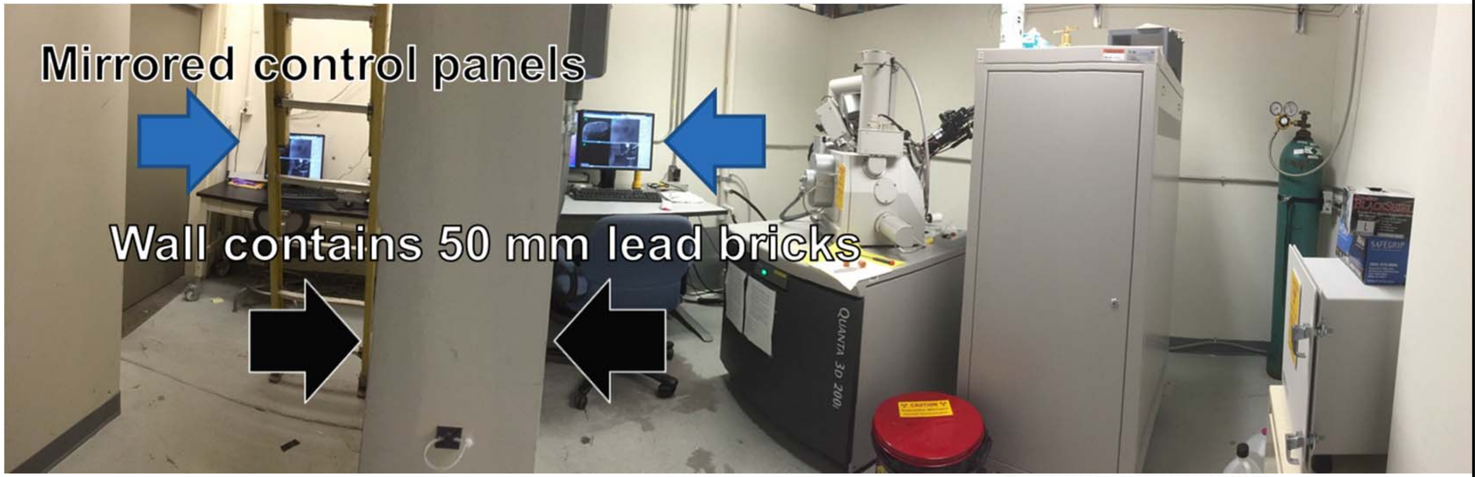
FEI Talos F200X

OAK RIDGE
National Laboratory

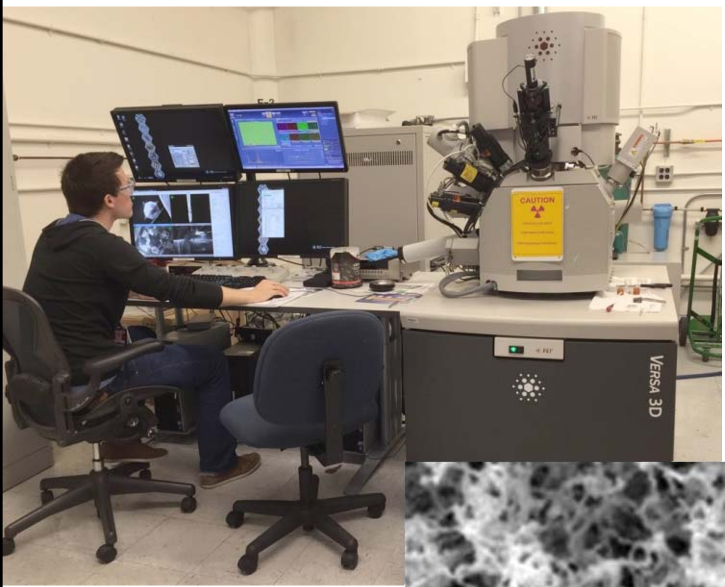
Quanta3D FIBs

FIB with cryo-stage: Good for sensitive materials (i.e., prevent hydriding of Zr alloys)

Shielded FIB: Control panel outside of 50 mm-thick lead envelope. Allows high-dose samples to be milled under ALARA conditions

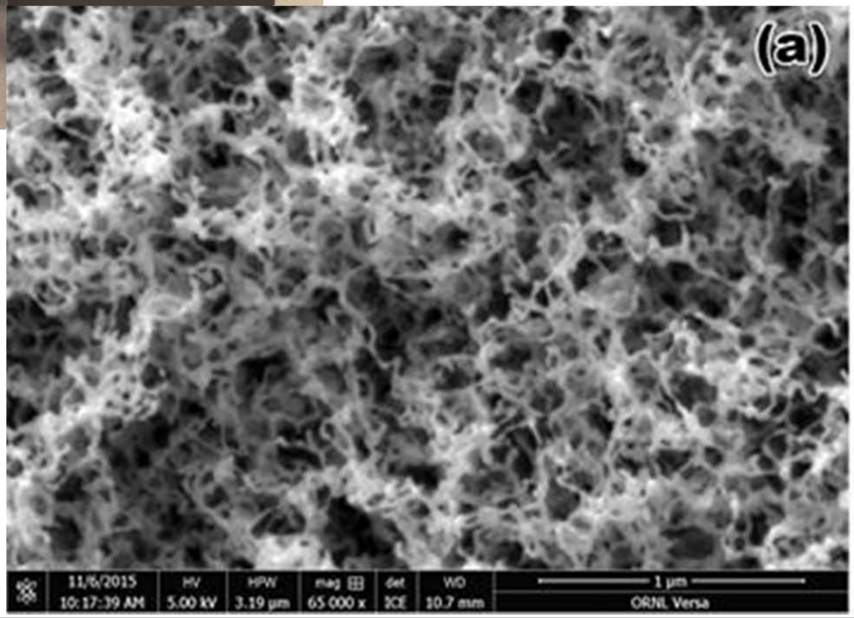


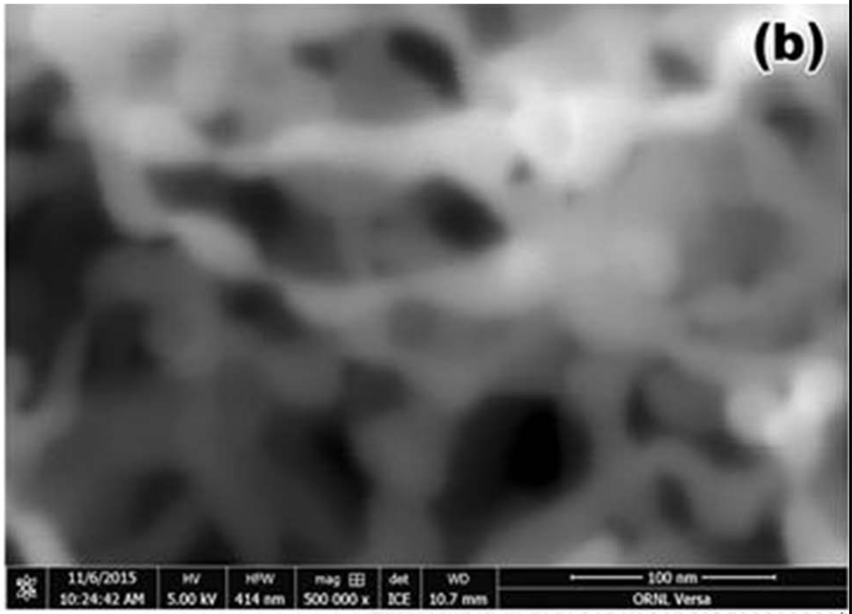
FEI Versa dual beam FIB-SEM



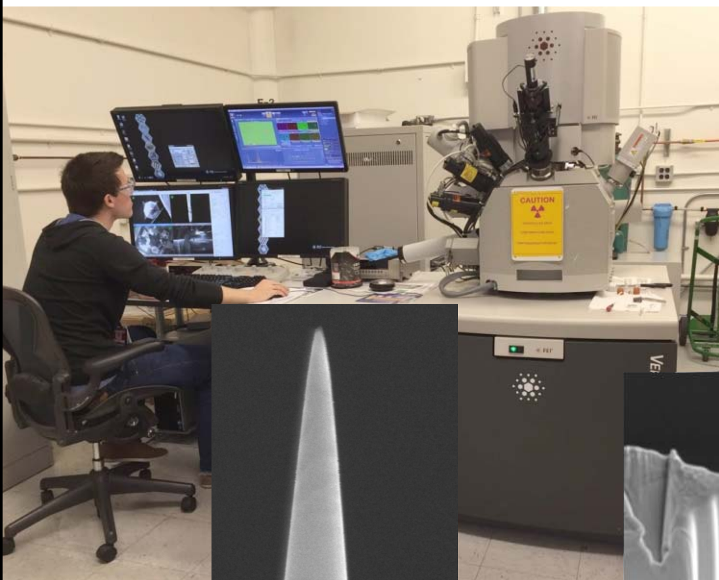
- **High-resolution SEM**

W nanofuzz/UCSD



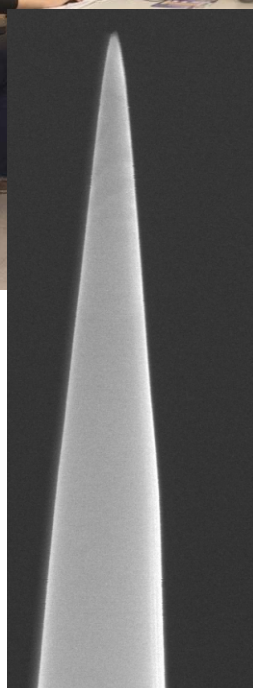


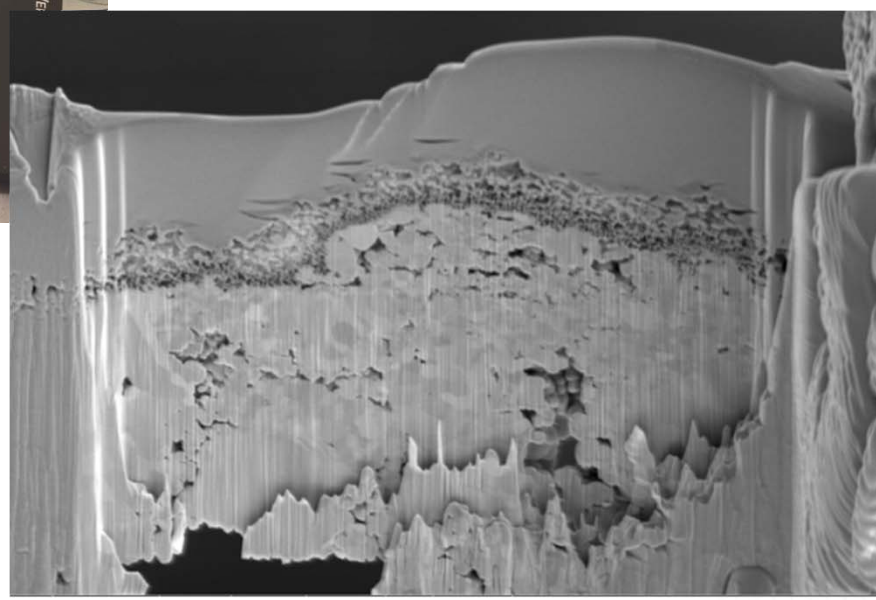
FEI Versa dual beam FIB-SEM

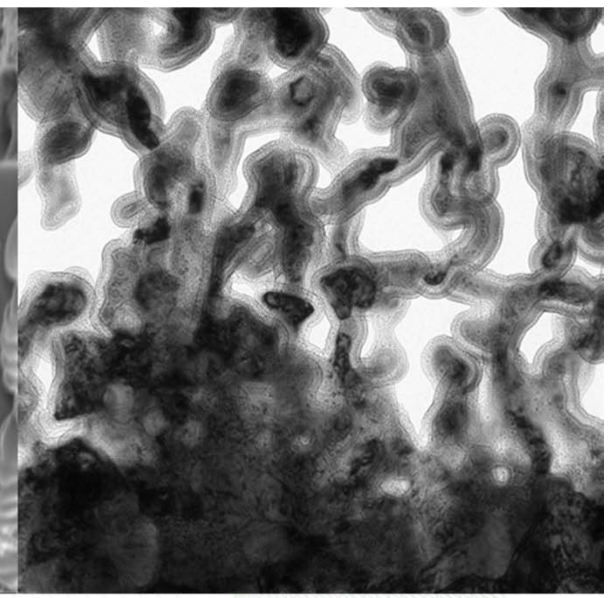


- High-resolution SEM
- **Advanced sample preparation**

TEM: W nanofuzz/UCSD







5 μm

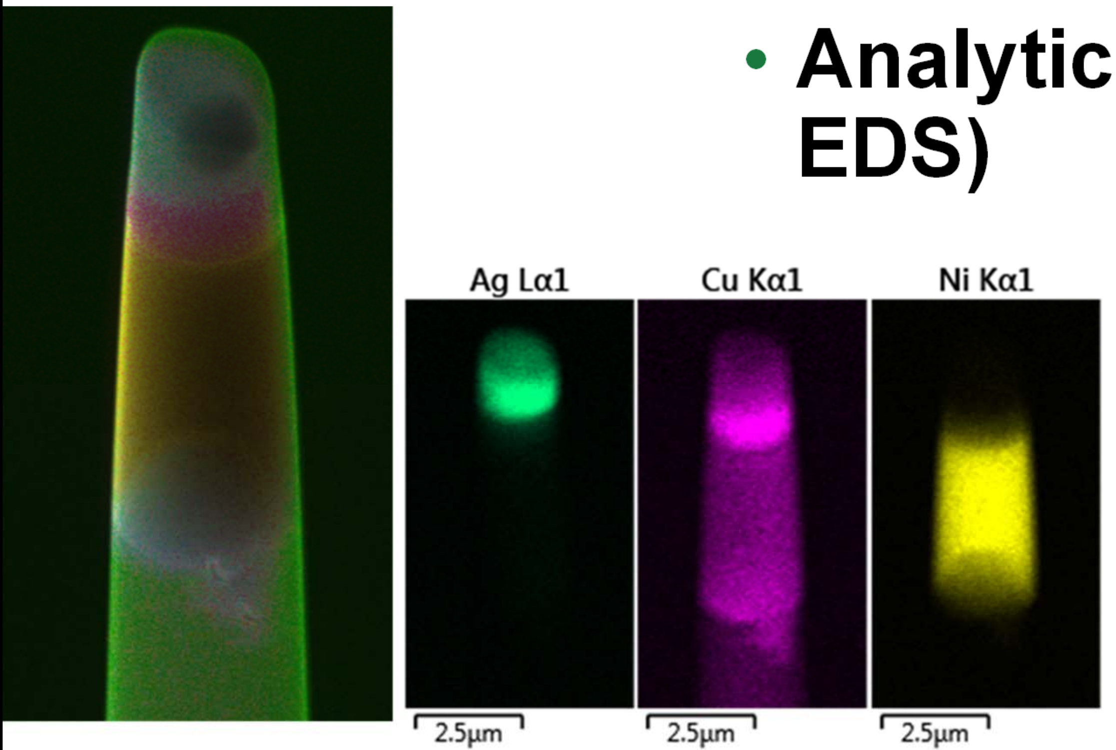
100 nm

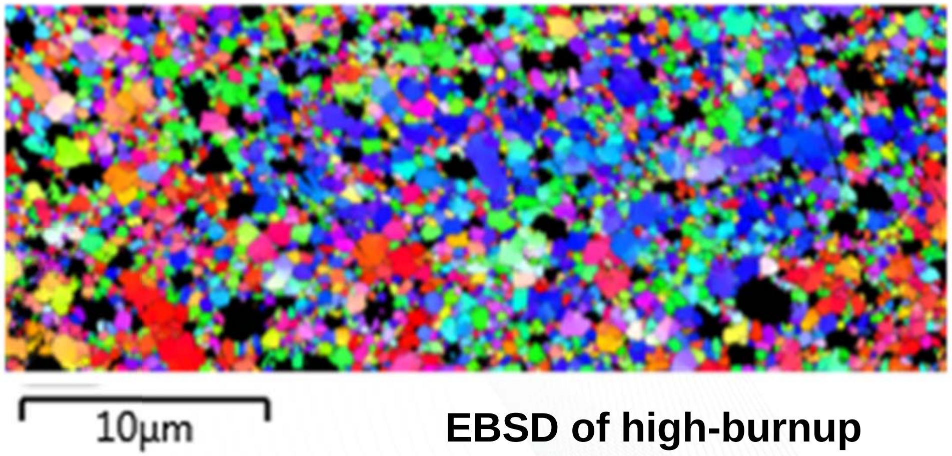
Atom probe: FeCrAl

FEI Versa dual beam FIB-SEM

- High-resolution SEM
- Advanced sample preparation
- **Analytical capabilities (EBSD, EDS)**

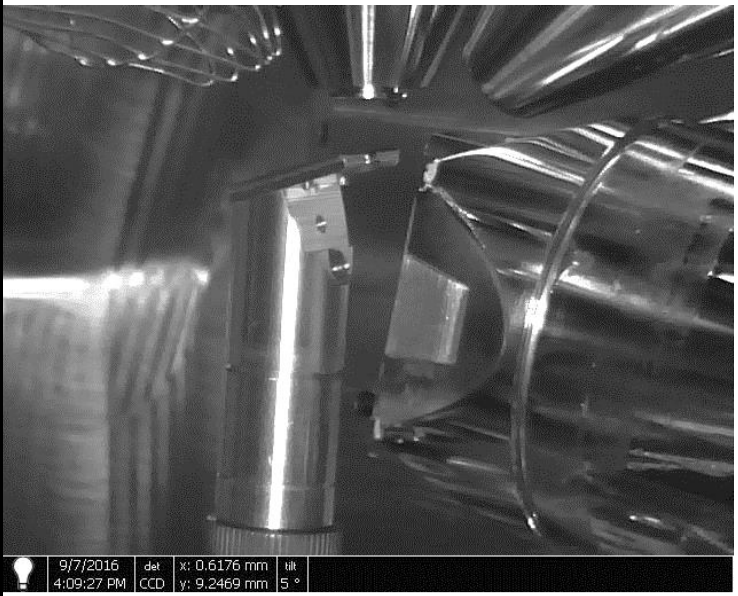
EDS of neutron-irradiated superconductor atom probe needles





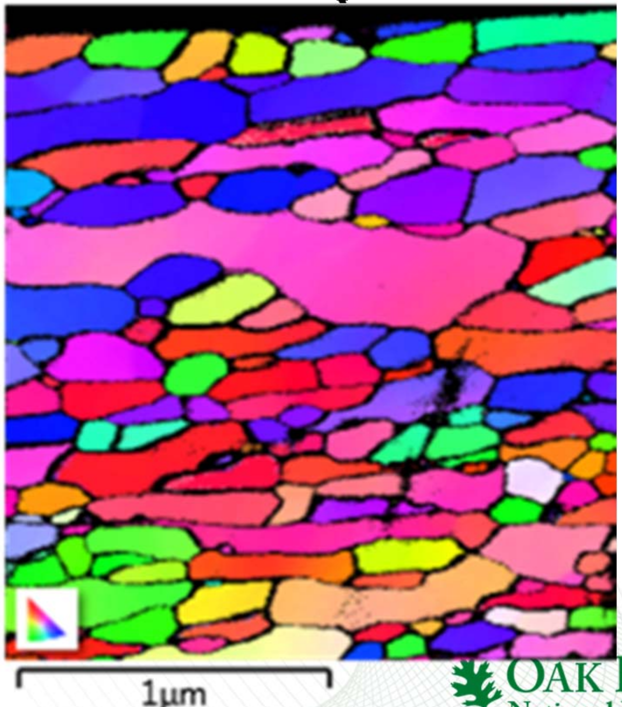
EBSD of high-burnup LWR fuel

FEI Versa dual beam FIB-SEM



- High-resolution SEM
- Advanced sample preparation
- **Analytical capabilities (EBSD, EDS)**

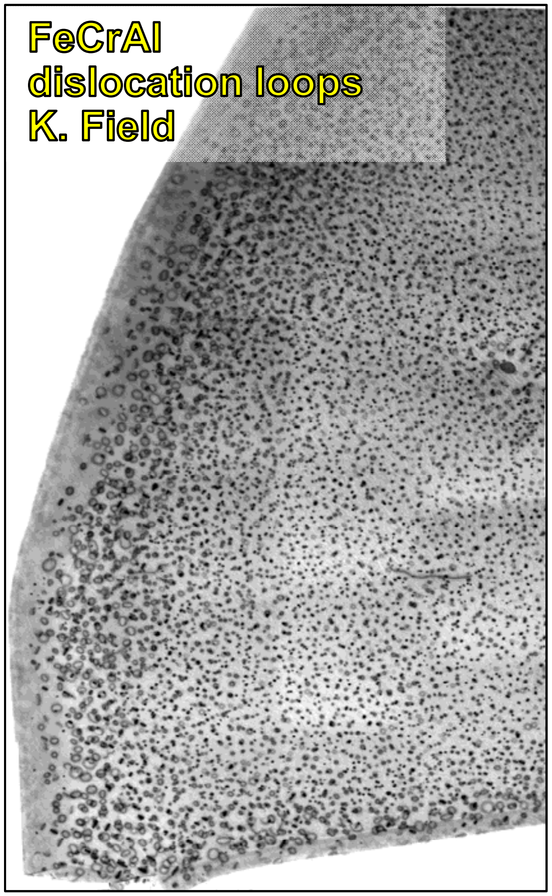
Transmission Kikuchi Diffraction

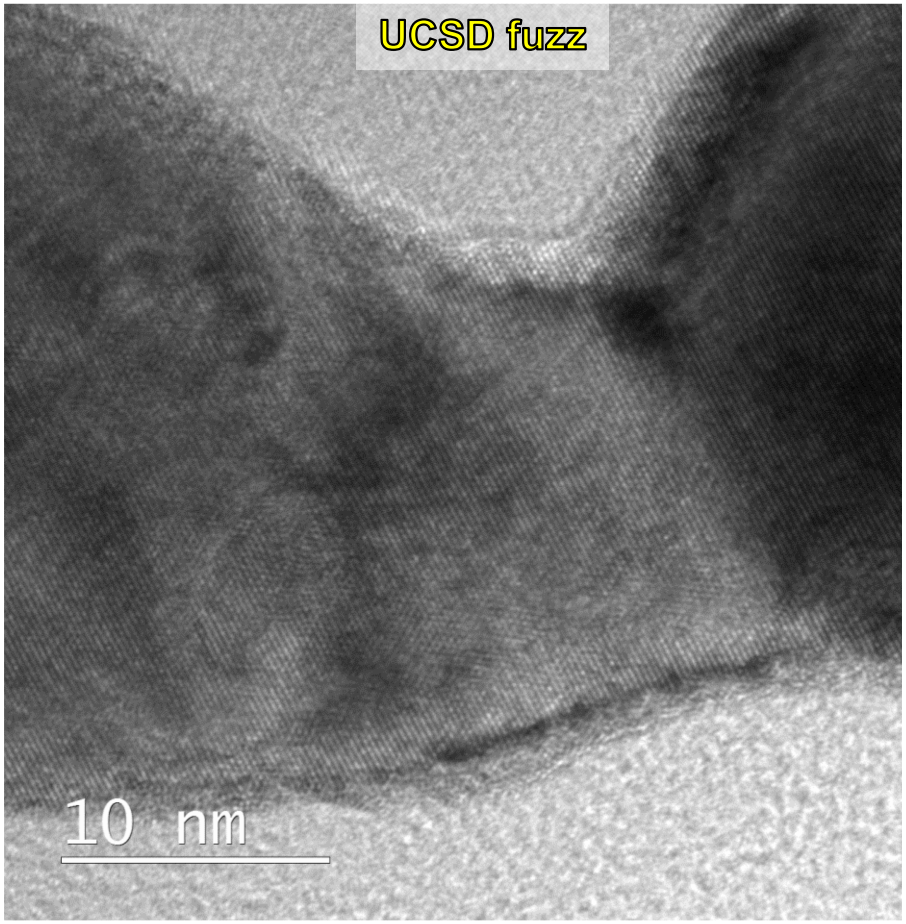


Parish et al. Scripta Mat., submitted

JEOL 2100F S/TEM

- **Defect and high-resolution imaging**

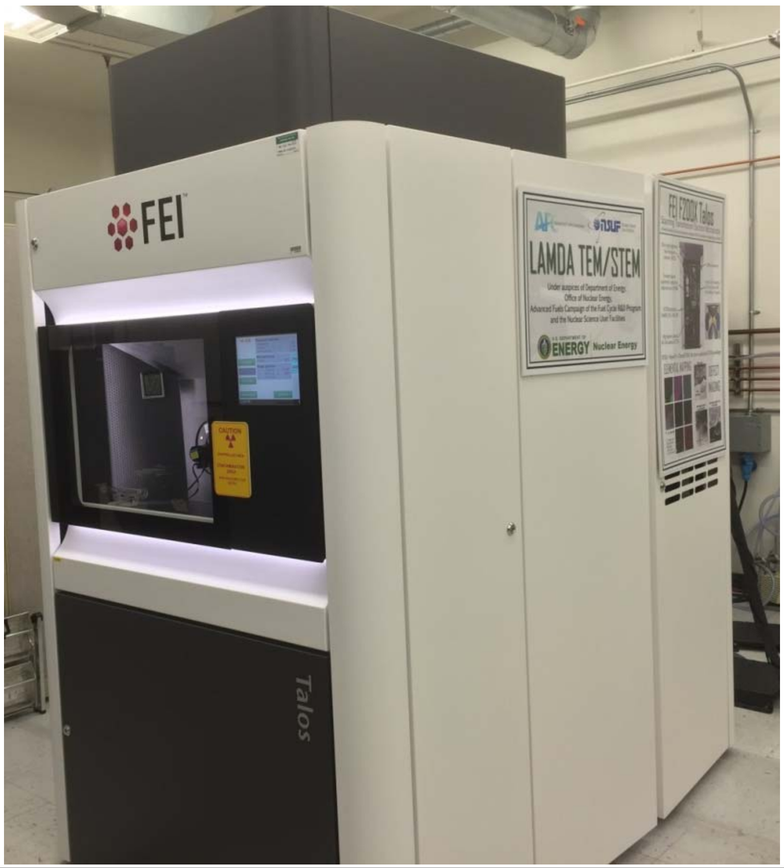




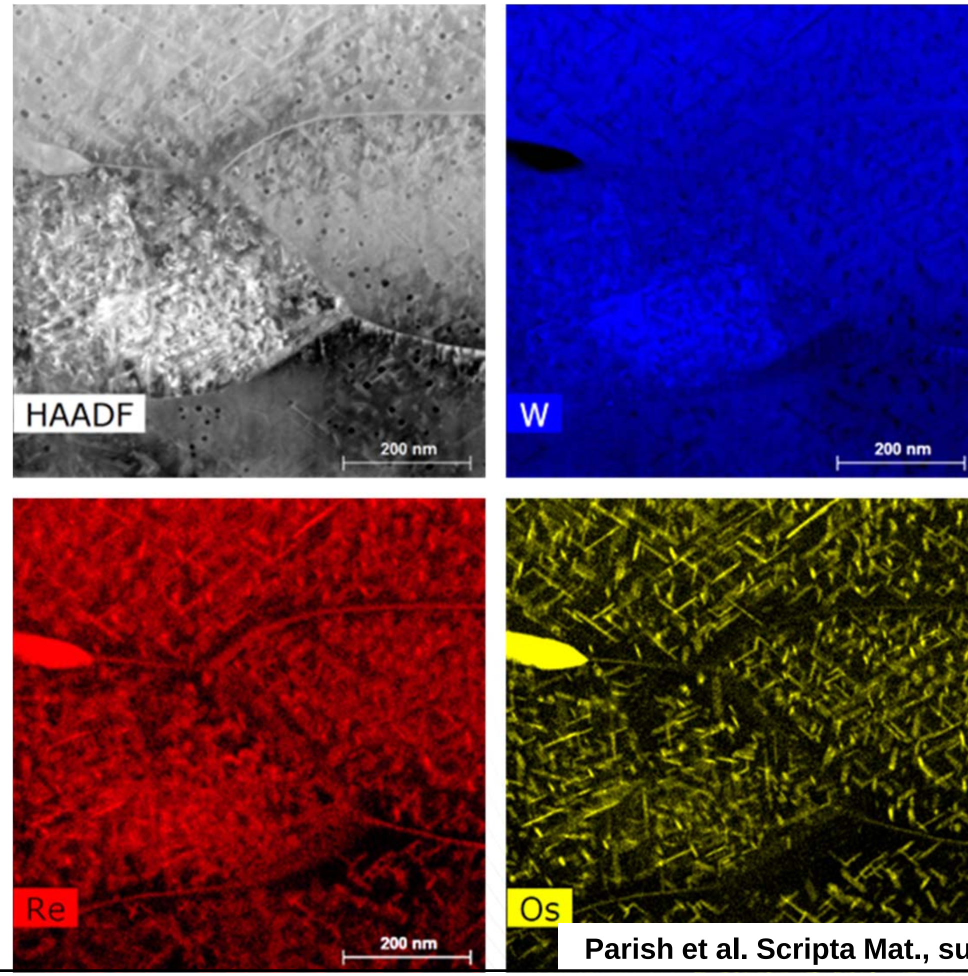
FEI Talos F200X

High-purity W
2.2 dpa, 650°C HFIR

FEI F200X XFEG-STEM (2015)
200 keV, XFEG source
4×SDD, 0.9 srad collection



- **High-resolution X-ray mapping**

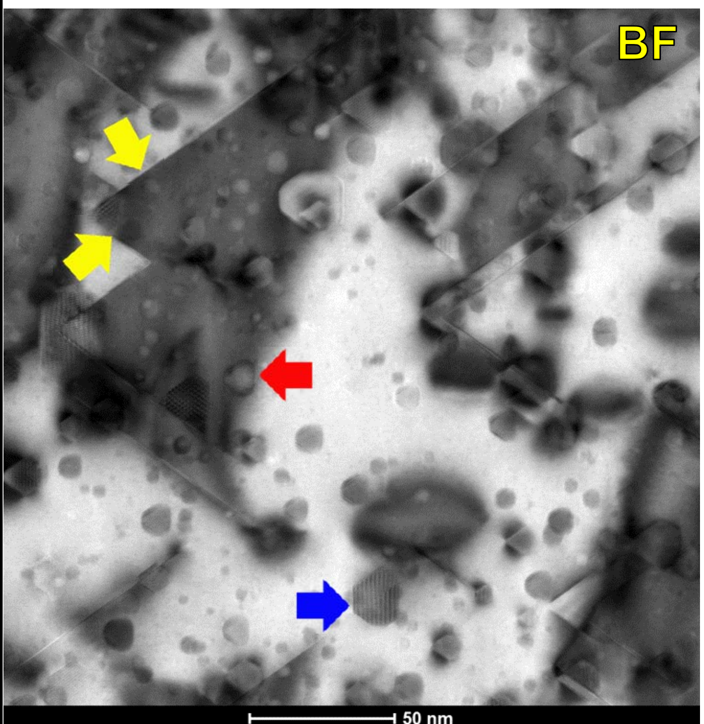


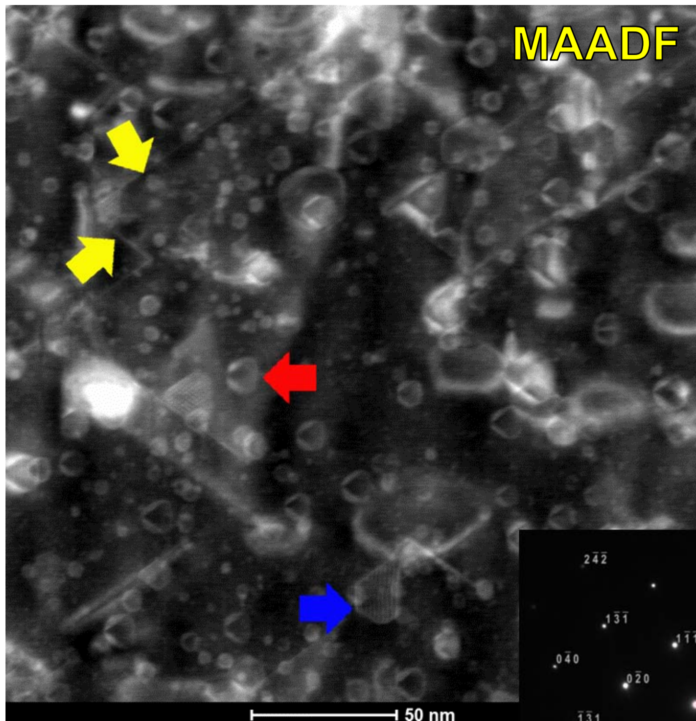
Parish et al. Scripta Mat., submitted

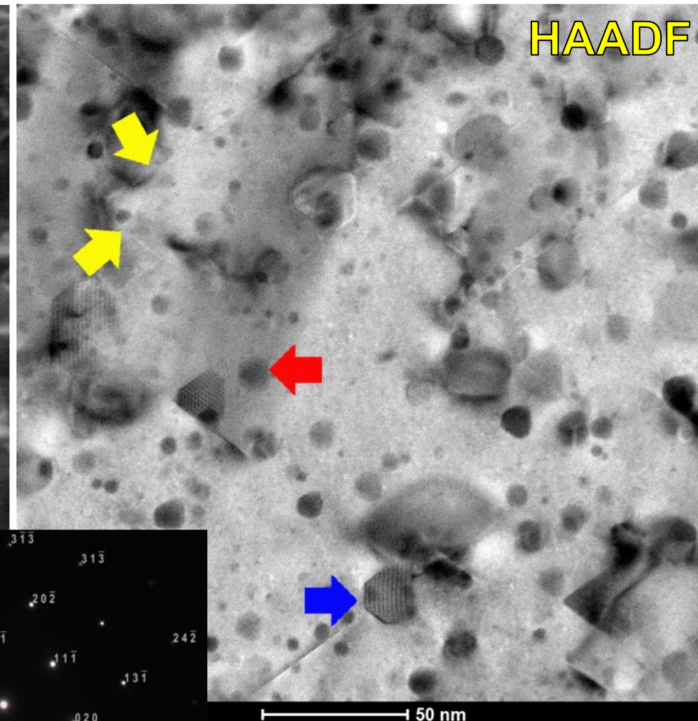
FEI Talos F200X

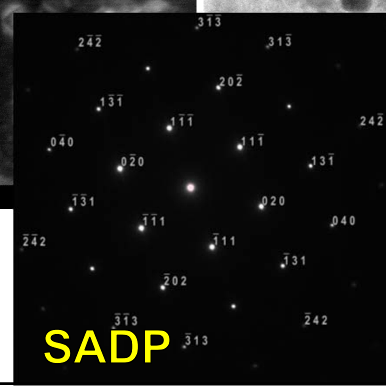
- **High-resolution imaging and defect analysis**

3C-SiC
1440°C
9 dpa









11-1 and -111 Frank loops.
tetragonal voids with strong diffraction contrast
second-phase regions

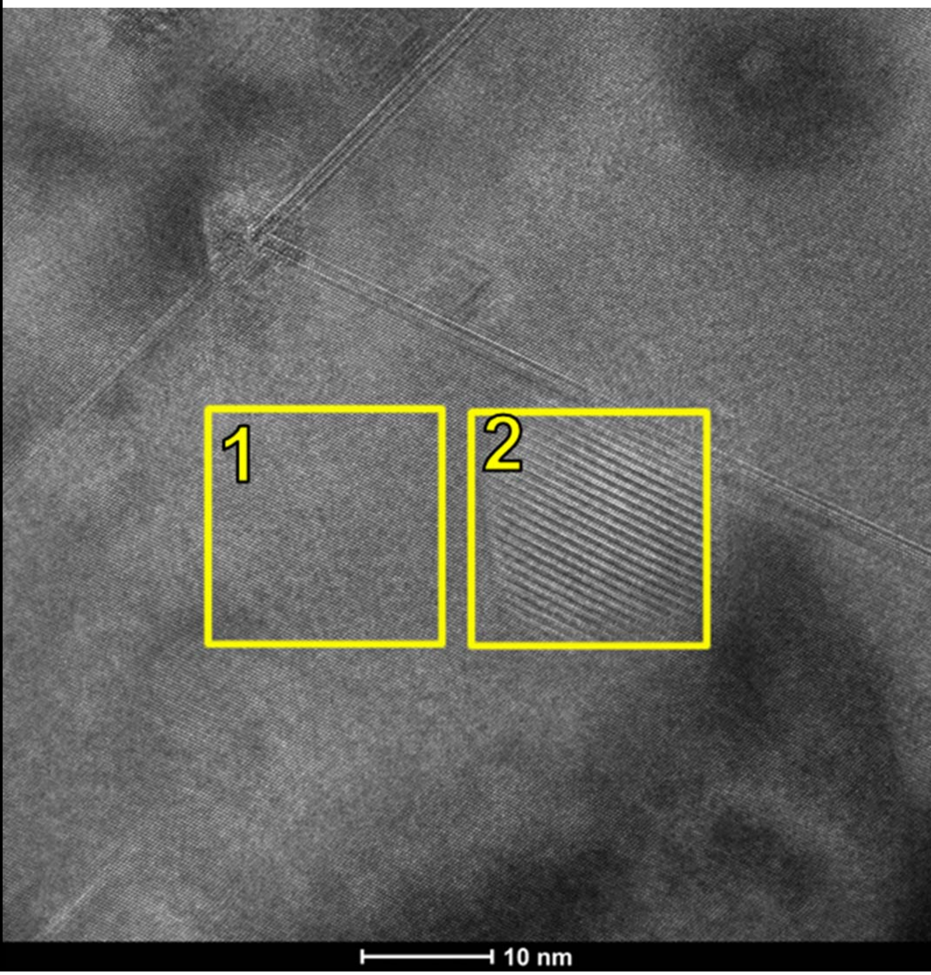
Parish et al. Sci. Reports, submitted

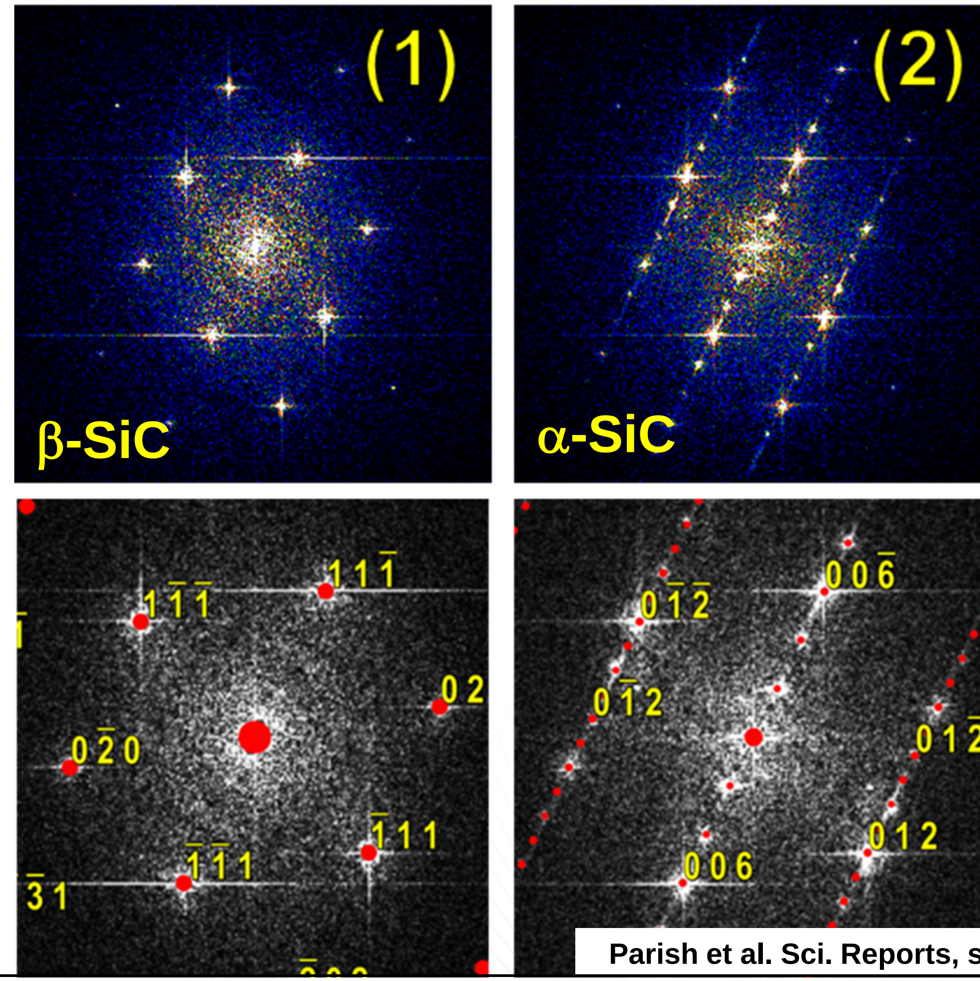
OAK RIDGE National Laboratory

FEI Talos F200X

- **High-resolution imaging and defect analysis**

3C-SiC
1440°C
9 dpa

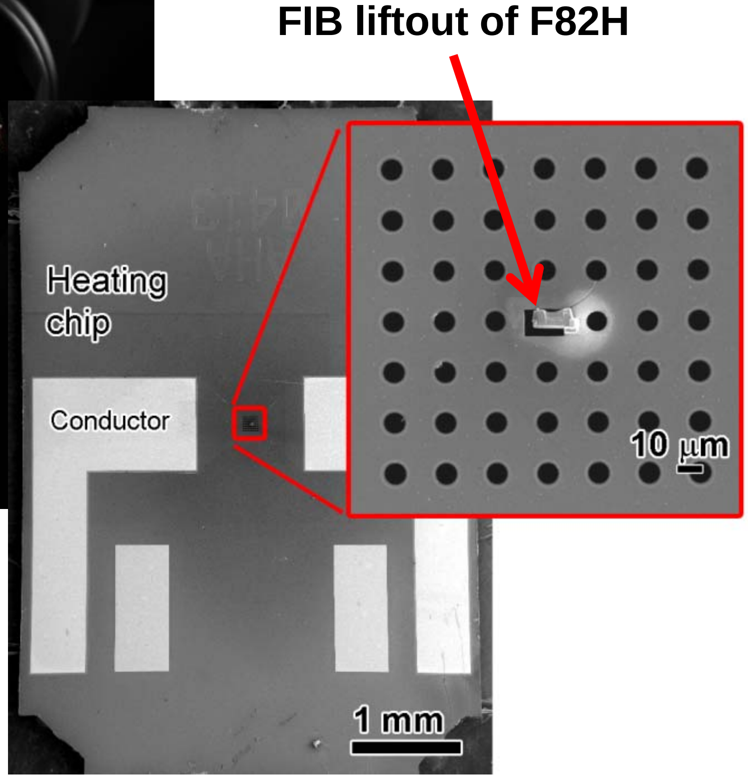


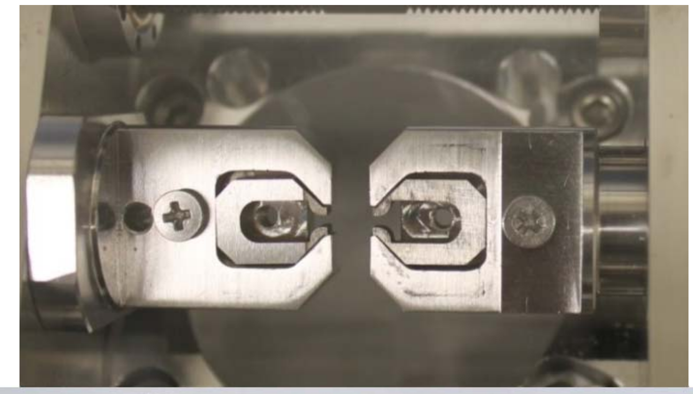


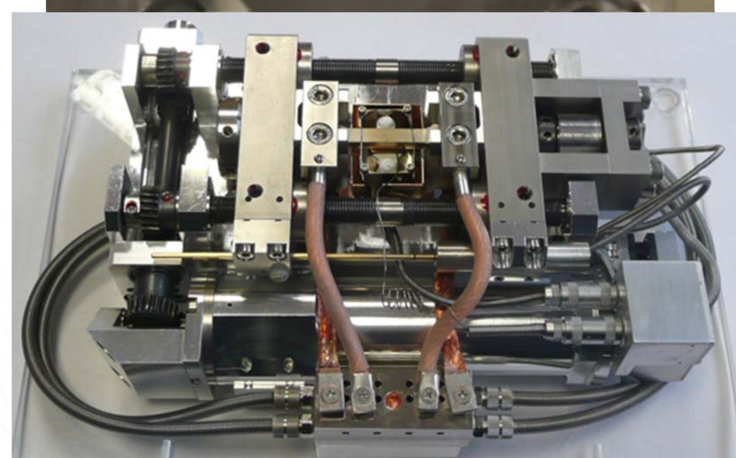
Parish et al. Sci. Reports, submitted

New capabilities (implemented but not debugged)

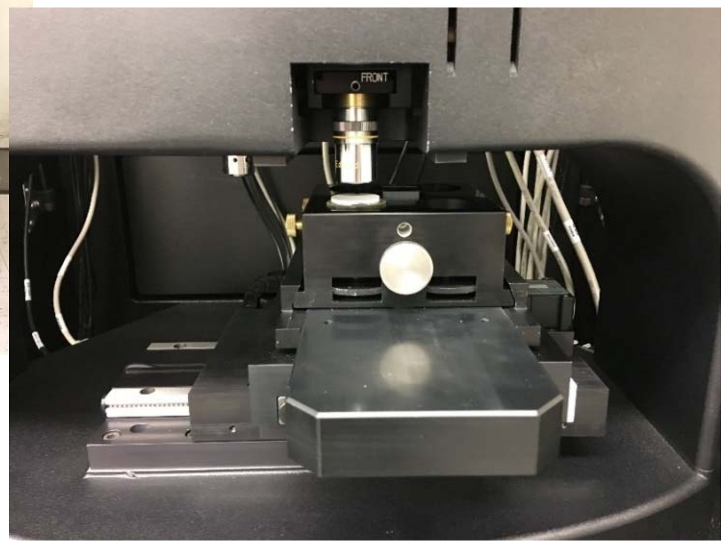
- **In situ heating for TEM (JEOL and FEI)**
- **In situ heating tensile testing for FEI Versa FIB**







Agilent Technologies Nanoindenter G200

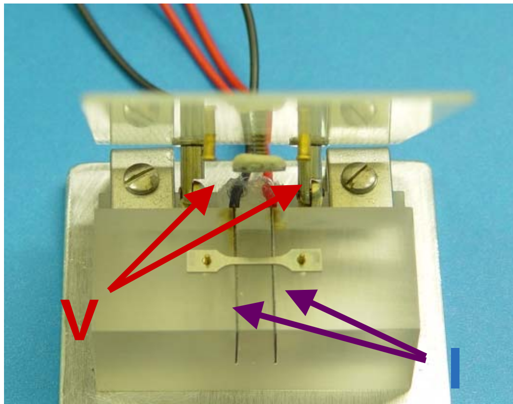


Capabilities

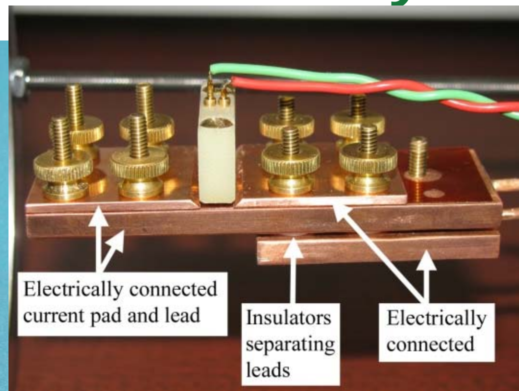
- **Total Indenter Travel:1.5mm**
- **Max Indentation Depth: >500 microns**

Max load with high load option: 10N

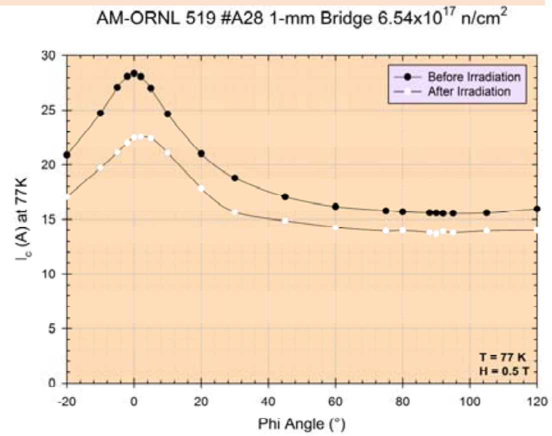
Electrical Resistivity Characterization



Standard electrical resistivity holder with SS3 type sample



Neutron irradiated high temp. superconductor angular dependence testing (at 77K)



Various electrical testing performed in LAMDA.

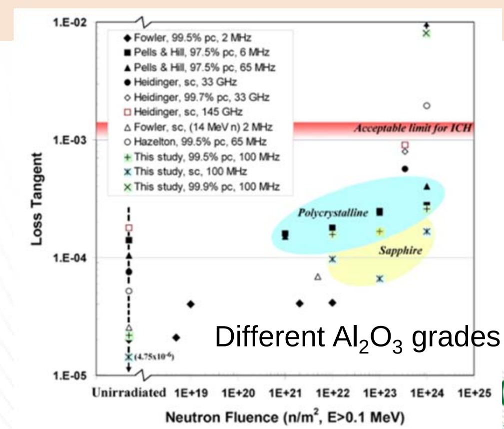
Typical work involves simple electrical resistivity, through 4-point probe.

This testing provides a non-destructive indicator of changes in microstructure.

Additional test stands for liquid nitrogen temperatures.

Specialized instruments can be brought into LAMDA for a campaign.

Loss tangent measurement using a capacitive loaded coaxial resonant cavity



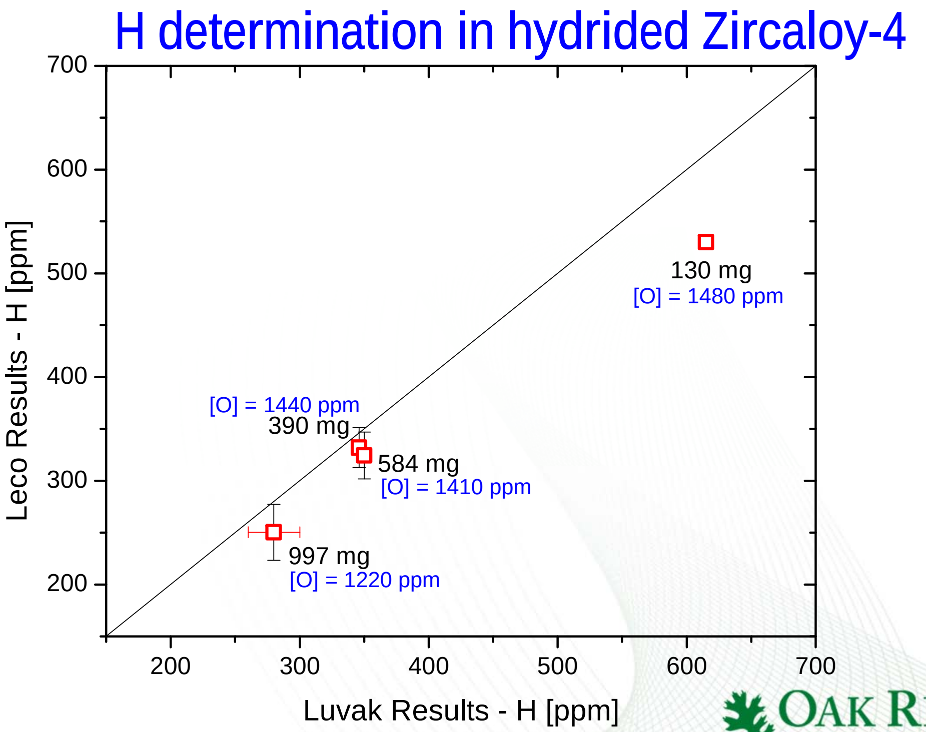
Different Al_2O_3 grades

LECO O&H Analyzer (OH836)

- **LECO OH836 instrument (oxygen and hydrogen analysis only) utilizes the inert gas fusion technique for analysis of O and H contained in the materials of interest.**

Instrument Range:

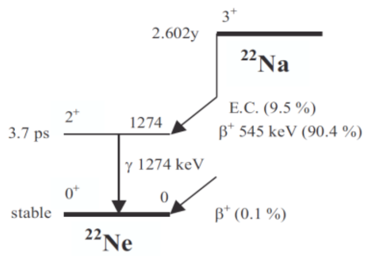
Oxygen: 0.05 ppm to 5.0% for a 1 g sample
Hydrogen: 0.1 ppm to 2500 ppm for a 1 g sample



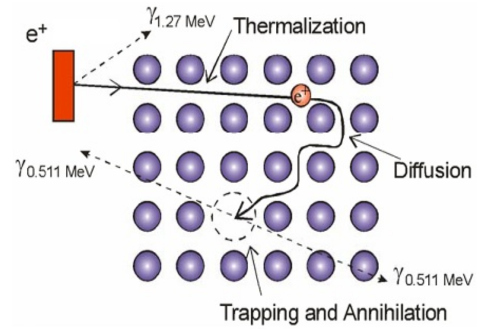
OAK RIDGE National Laboratory

Positron Annihilation Spectroscopy

- **PAS is a well-established technique to study the free volume in condensed materials directly at atomic level and nano-scales.**

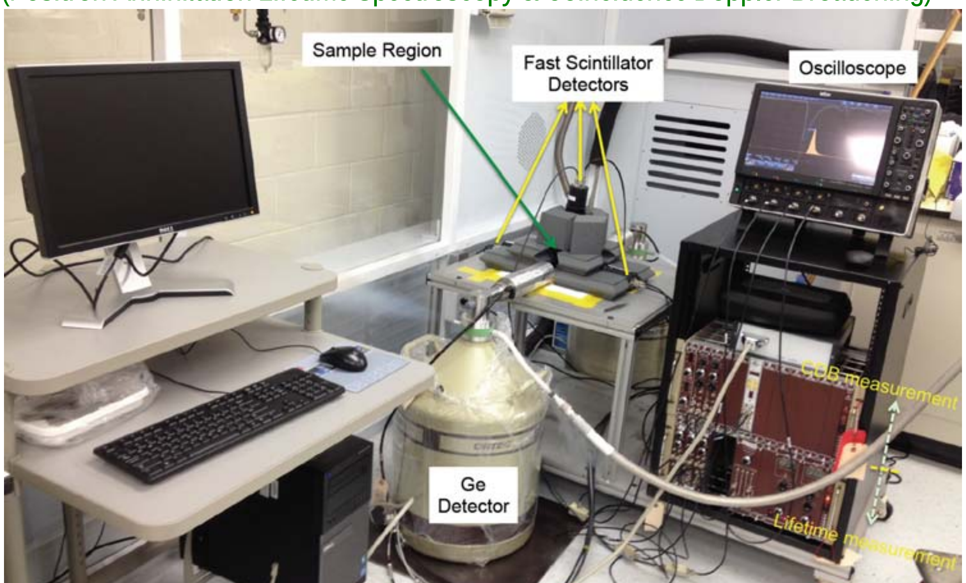


- localize in open-volume regions (vacancies, voids, other defects) due to lack of positively charged atomic nuclei
- localize in regions of higher positron affinity (elemental specific)
  Fe:-3.84 eV Ni:-4.46 eV Al: -4.41 eV W:-1.31 eV



PAS at LAMDA of ORNL

(Positron Annihilation Lifetime Spectroscopy & Coincidence Doppler Broadening)

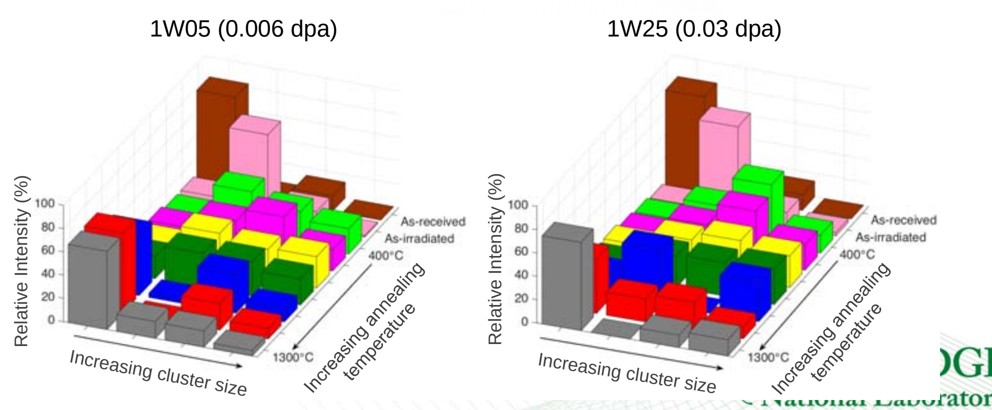


- ➢ Simultaneous measurements of PALS and CDB
- ➢ Calculated system timing resolution: 140 ps

Example:

Damage recovery in neutron-irradiated W

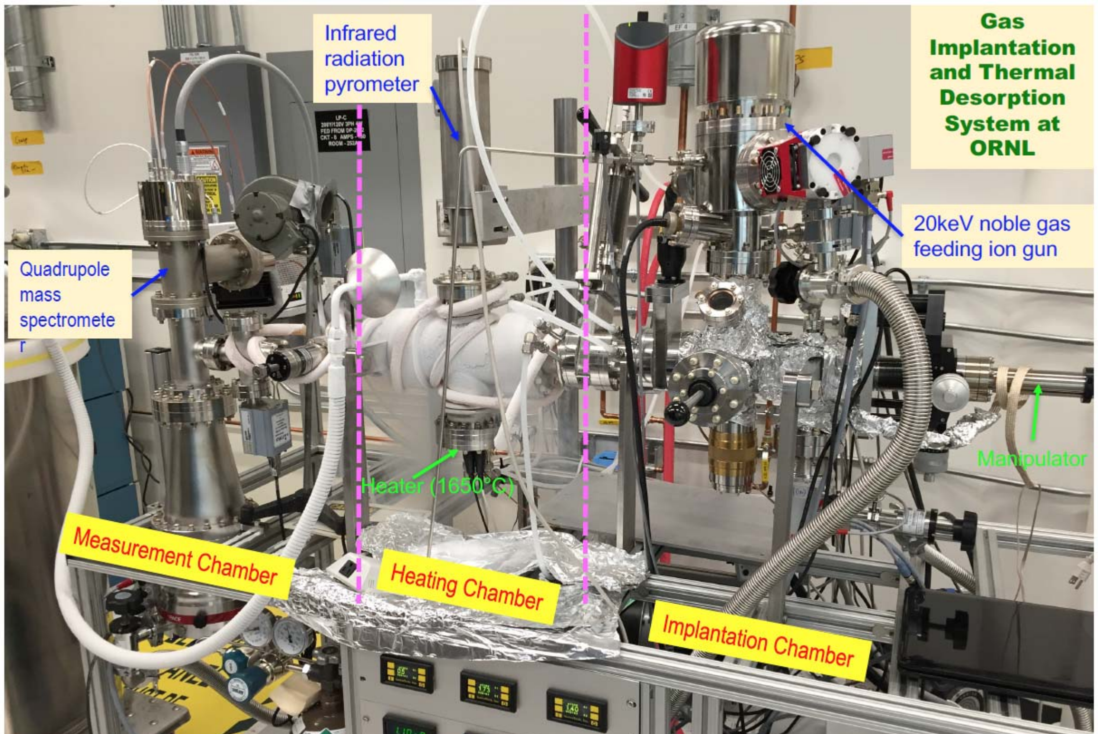
Changes in vacancy defect population distribution are determined as a function of isochronal annealing temperature after 90°C irradiation to 0.006 dpa and 0.03 dpa, respectively.



Thermal Desorption Spectrometry

- **TDS is a powerful tool to investigate gas behavior (e.g., He, H, D) in PFMs by quantifying the retained isotopes and studying the kinetics and energetics of gas-defect interactions.**

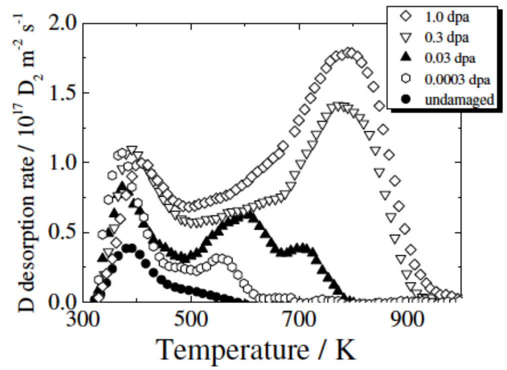
TDS coupled with 20keV ion gun at LAMDA of ORNL



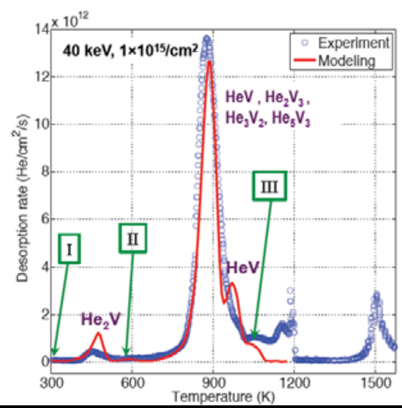
- Ultra high vacuum
- High quality quadrupole mass spectrometer
- Well-controlled heating assembly
- In-situ ion implantation (e.g., $^{4}He^{+}$, $^{3}He^{+}$, D^{+})

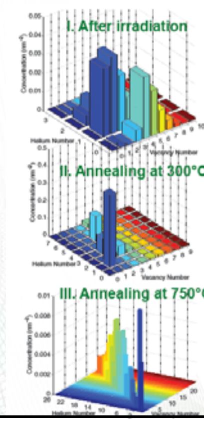
Examples:

D retention in Fe^{+}-irradiated W following 1 keV D_2^{+} implantation to a fluence of 5×10^{21} D m^{-2} *(Y. Oya, et al. JNM (2015))*



Mechanistic interpretation of helium desorption peaks in He-implanted Fe *(X. Hu, et al. JNM (2013))*





~~Three~~ Four LAMDA thrusts

1. Thermal properties
2. Mechanical properties
3. Microstructural characterization
4. **Sample handling:**
   - **Hot-cell receiving and cleaning; inventory and logging; cutting, grinding, and polishing; electropolishing; interim storage vaults; etc.**