Argonne NATIONAL LABORATORY

FY2013 NEET Award - **Developing Microstructure-Property Correlation in Reactor Materials using *in situ* High-Energy X-rays**

PIs:

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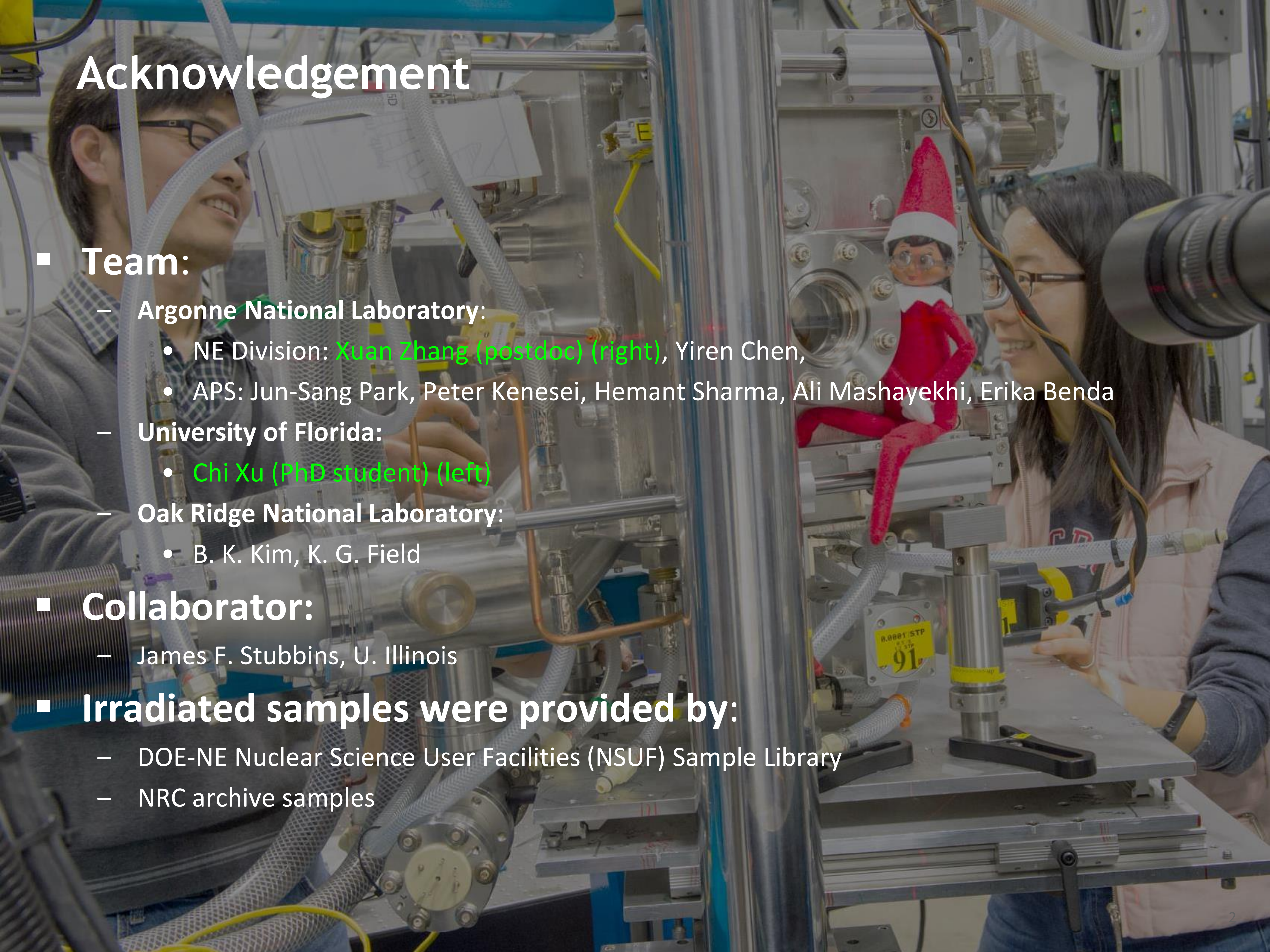
U.S. DEPARTMENT OF ENERGY

Acknowledgement

- **Team**:
  - **Argonne National Laboratory**:
    - NE Division: Xuan Zhang (postdoc) (right), Yiren Chen,
    - APS: Jun-Sang Park, Peter Kenesei, Hemant Sharma, Ali Mashayekhi, Erika Benda
  - **University of Florida:**
    - Chi Xu (PhD student) (left)
  - **Oak Ridge National Laboratory**:
    - B. K. Kim, K. G. Field
- **Collaborator:**
  - James F. Stubbins, U. Illinois
- **Irradiated samples were provided by**:
  - DOE-NE Nuclear Science User Facilities (NSUF) Sample Library
  - NRC archive samples



Outline

- **Introduction**
- **Capability for *in situ* High-Energy X-ray Characterization of Neutron-Irradiated Specimens under Thermal-Mechanical Loading**
  - *In situ* X-ray Radiated Materials (*i*RadMat) Thermal-mechanical Apparatus
  - *In situ* tensile test of neutron-irradiated pure Fe at 300°C in vacuum
- **Research highlights**
  - Plastic instability and strain-induced martensite transformation in neutron-irradiated 316 austenitic stainless steel
  - Radiation hardening mechanisms in low-dose neutron-irradiated Fe-Cr ferritic alloy
- **Synergy of Advanced Characterization Techniques: X-rays/TEM/APT**
- **Summary**

Motivation

Microstructure – Property Correlation

Microstructure

(dislocation loops, extended dislocation structure, voids, He bubbles, phase transformation, etc.)

?

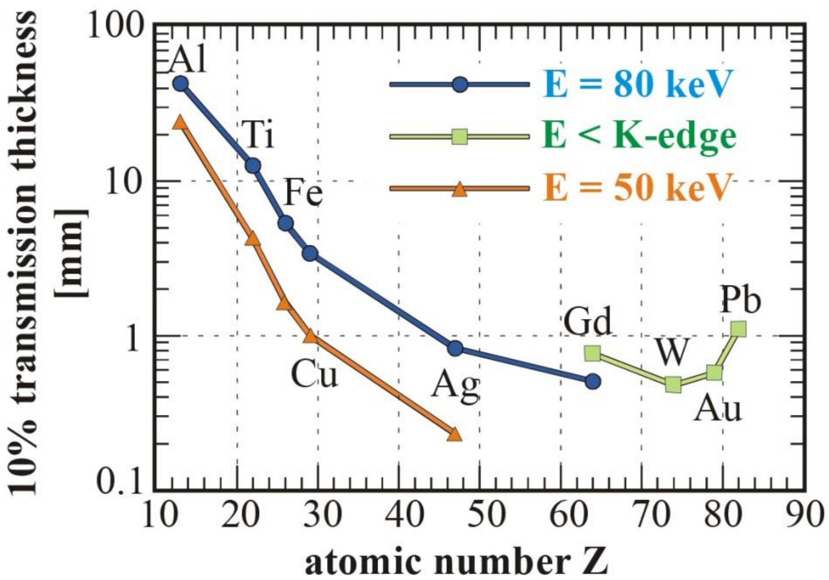
Mechanical Properties

(low-temperature embrittlement, irradiation creep, high-temperature embrittlement, irradiation-assisted stress corrosion cracking)

- Traditionally, microstructure and mechanical properties are measured separately;
- Need **new capability** that measures microstructure and properties **simultaneously**;
  - Existing techniques, e.g. *in situ* straining with electron microscopy of small-scale specimens
  - New capability: *in situ* mechanical-loading of lab-scale specimens with high-energy X-rays

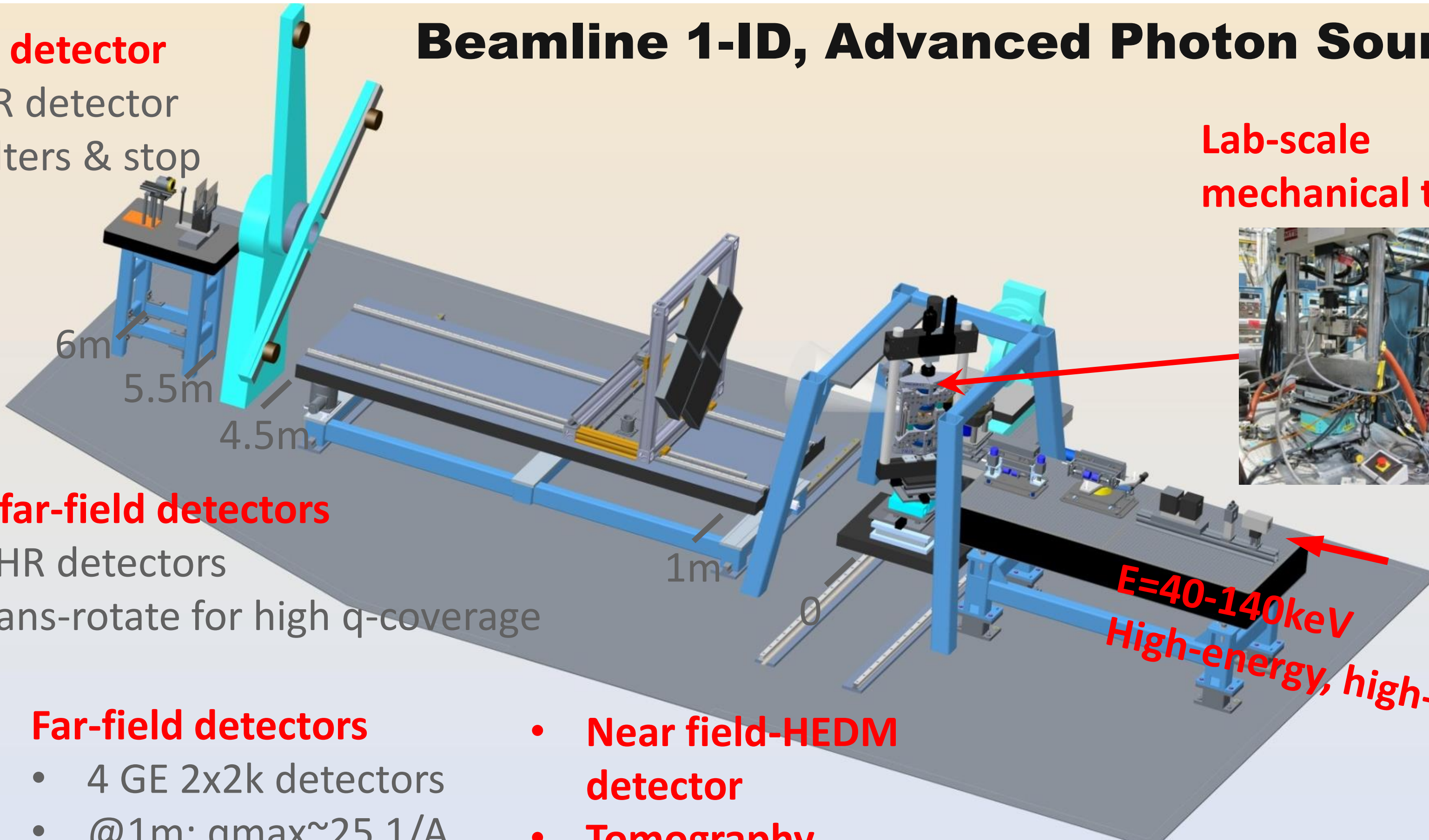
High-Energy, High Brilliance X-rays

- **Deep penetration**
  - mm-thick specimens
  - High-Z materials
  - Bulk properties
  - Environment chambers
    - Loading
    - Heating
    - Corrosion



- **High spatial resolution**
  - Small Bragg angles: forward scattering to access large q-range
  - Kinematical scattering
- **High time resolution**
  - *In situ* real-time studies require high flux
- **Real Material, Real Environment, Real Time**

In situ Thermal-Mechanical Loading with High-Energy X-rays



SAXS detector

- HR detector
- Filters & stop

Very far-field detectors

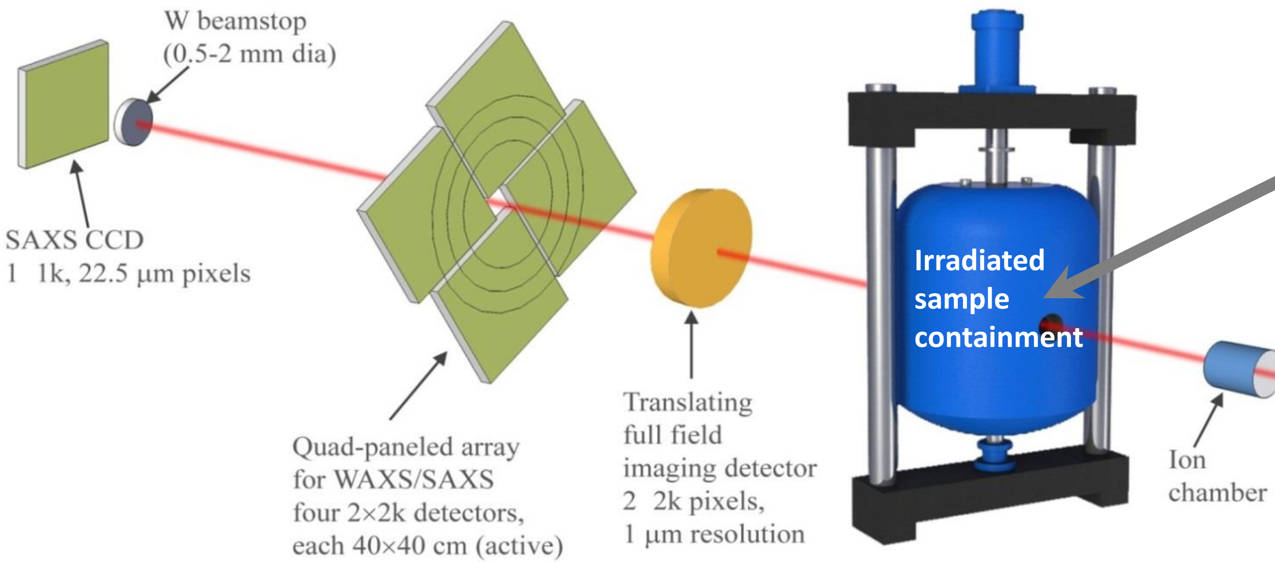
- 3 HR detectors
- Trans-rotate for high q-coverage

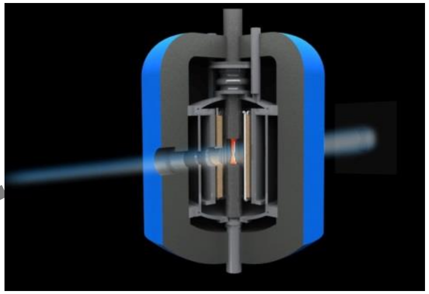
Far-field detectors

- 4 GE 2x2k detectors
- @1m: qmax~25 1/A
- Center-hole (SAXS)

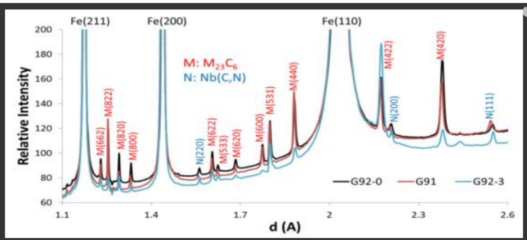
- **Near field-HEDM detector**
- **Tomography**
- Conical slit
- Lasers

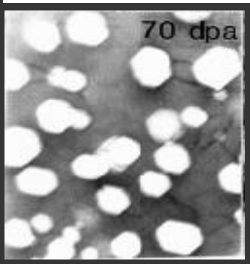
Project Goal -
In situ Characterization of Neutron-Irradiated Specimens under Thermal-Mechanical Loading with High-Energy X-rays

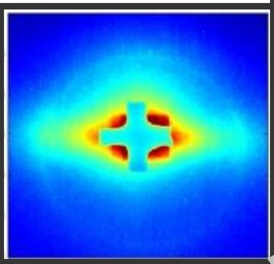




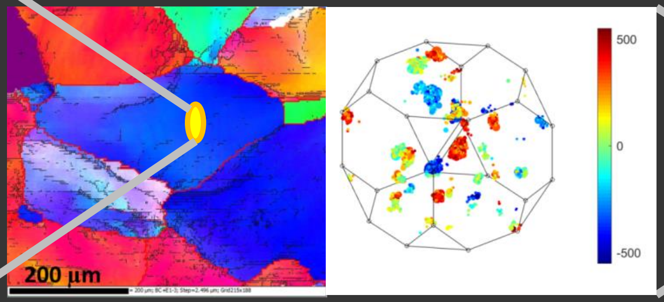
A multiscale experiment combining a suite of techniques



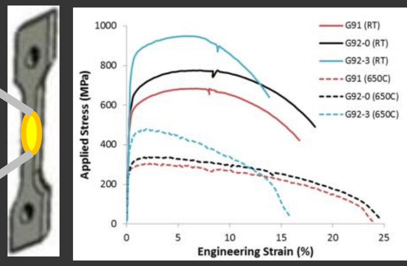




Nanoscale

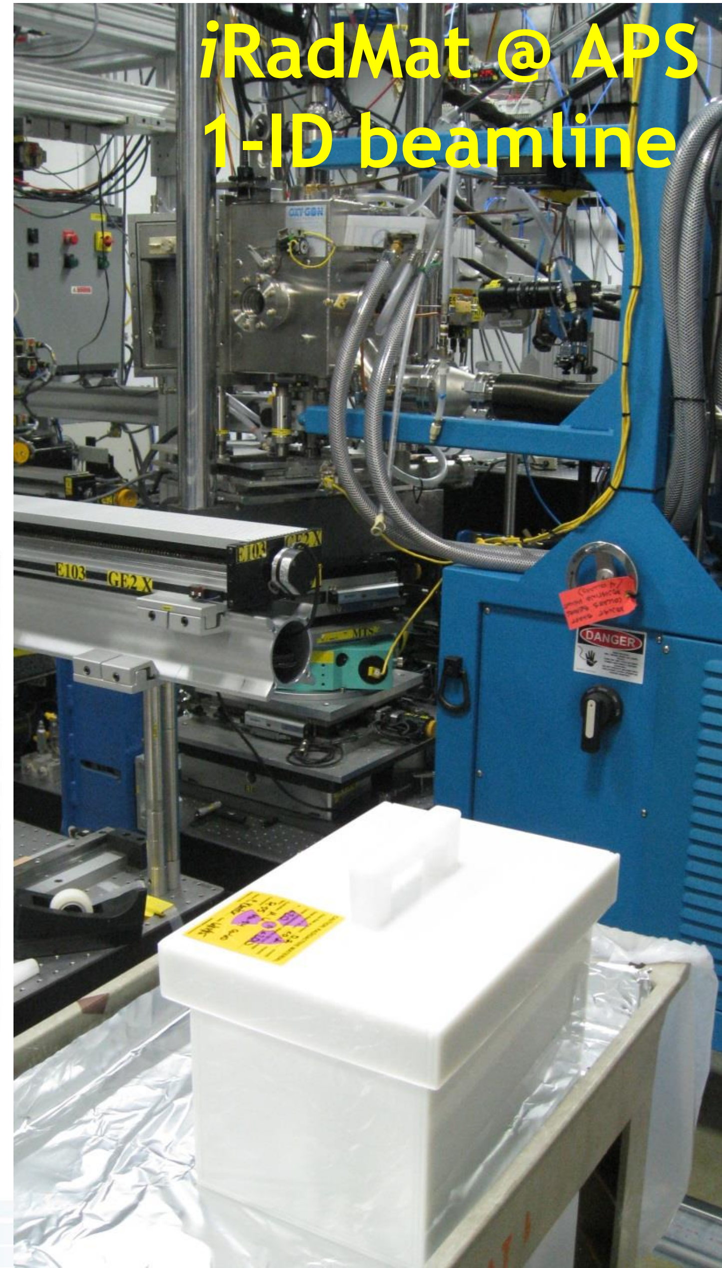


Microscale



Macroscale

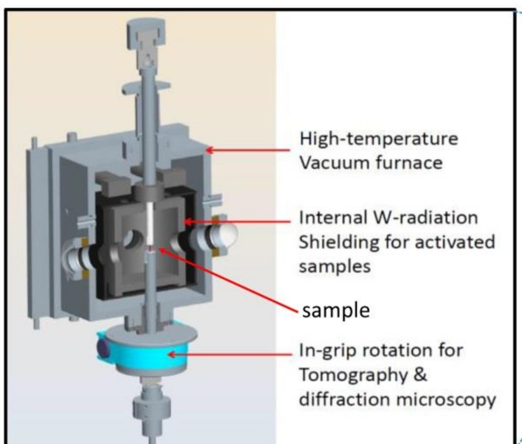
In situ X-ray Radiated Materials (*i*RadMat) Thermal-mechanical Apparatus

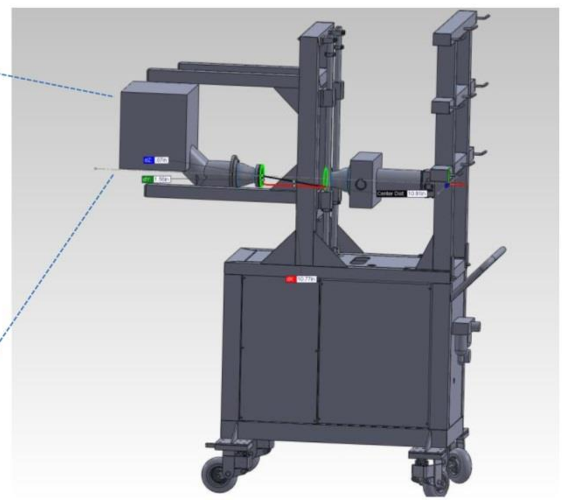


Unique sample environment

- Internal radiation shielding for activated samples
- Temperature: <1200°C
- Vacuum: $1x10^{-5}$ Torr
- Tension, creep, fatigue loading
- In-grip rotation

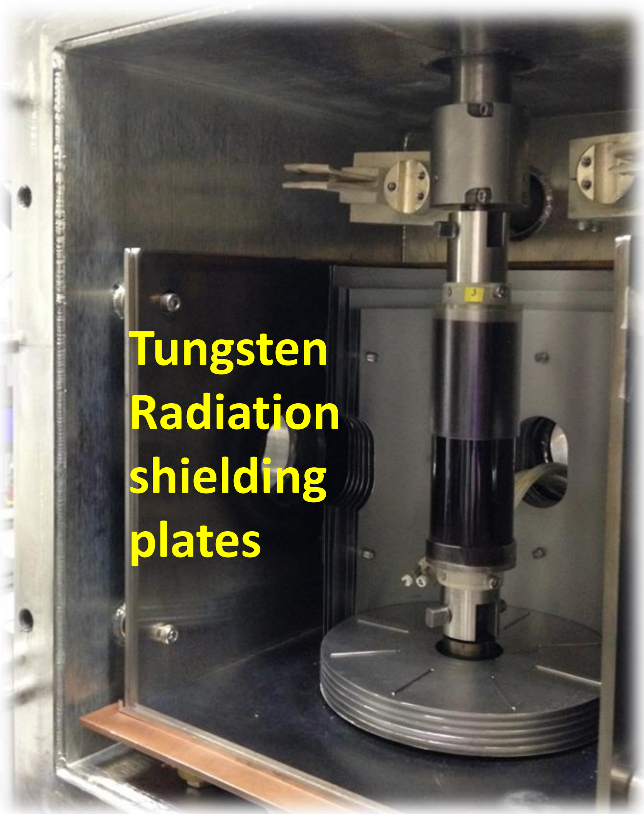
*i*RadMat



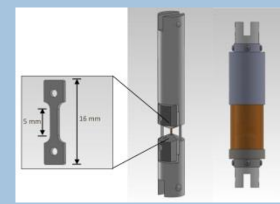


Activated Specimen Loading and Shielding

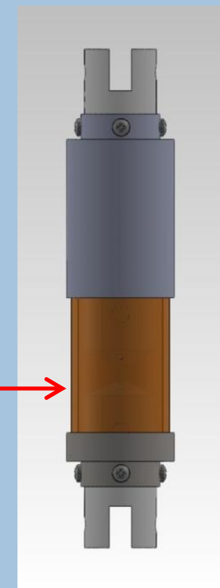
RT tensile test of an irradiated specimen

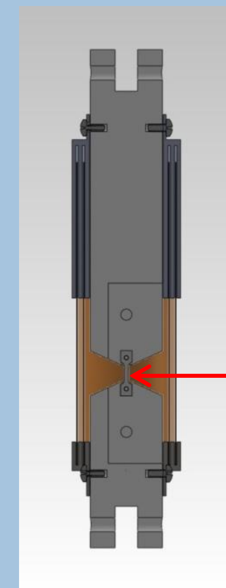


For low-activity specimens



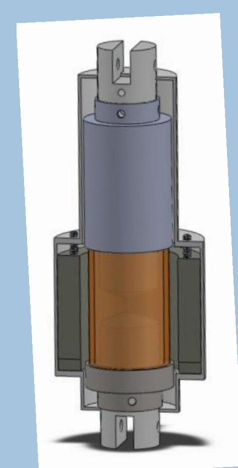
Double-layered Kapton tubes for RT tests

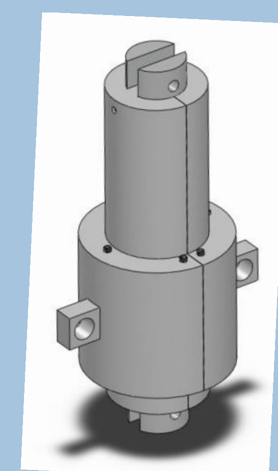




Irradiated Sample

For high-activity specimens: additional sample shielding





Activated Specimen Preparation and Handling

On-site Radiological Facility - Irradiated Materials Lab (IML)

Specimen installation and encapsulation at Irradiated Materials Laboratory (IML) in Bldg. 212, ANL

Survey and transfer

Transfer between IML and APS

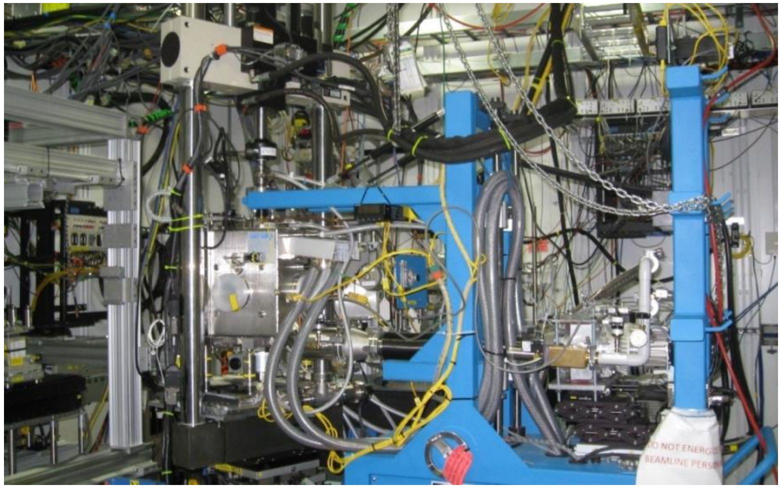
Advanced Photon Source (APS)

Specimen Loading at beamline

In situ Tensile Test of Neutron-Irradiated Pure Fe at 300°C in Vacuum

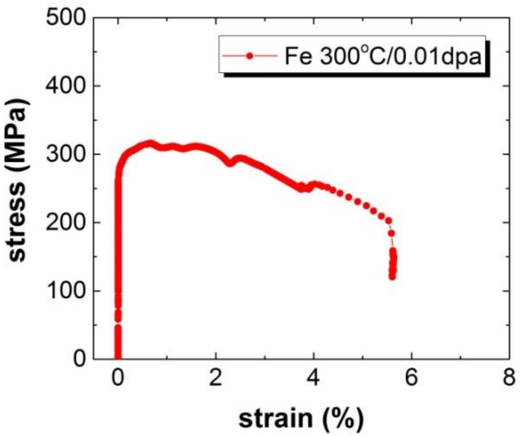
- **Material**:
  - Pure Fe
- **Specimen**:
  - Sheet-type tensile specimen
  - Gauge 5.0x1.2x0.5 mm
- **ATR neutron irradiation (U. Illinois)**:
  - 300°C to 0.01 dpa
- **X-ray measurement**:
  - Energy: 123 keV
  - Beam size: 300x300 μm
  - Sample-detector distance: 2628 mm (2θ coverage of 15°, and 9 Debye-Scherrer rings)
  - WAXS/SAXS/tomography
- ***In situ* tensile test**:
  - Temperature: 300°C
  - Environment: vacuum
  - Strain rate: $1x10^{-5}$/s

Double-contained specimen grips for elevated-temperature tensile tests.

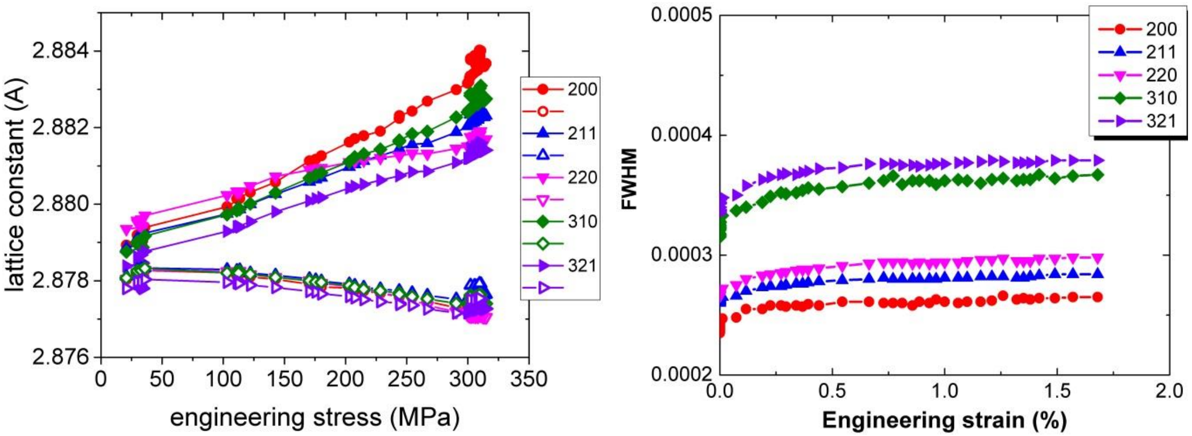


***i*RadMat at APS 1-ID beamline**

Deformation and Failure Mechanisms in Neutron-Irradiated Fe

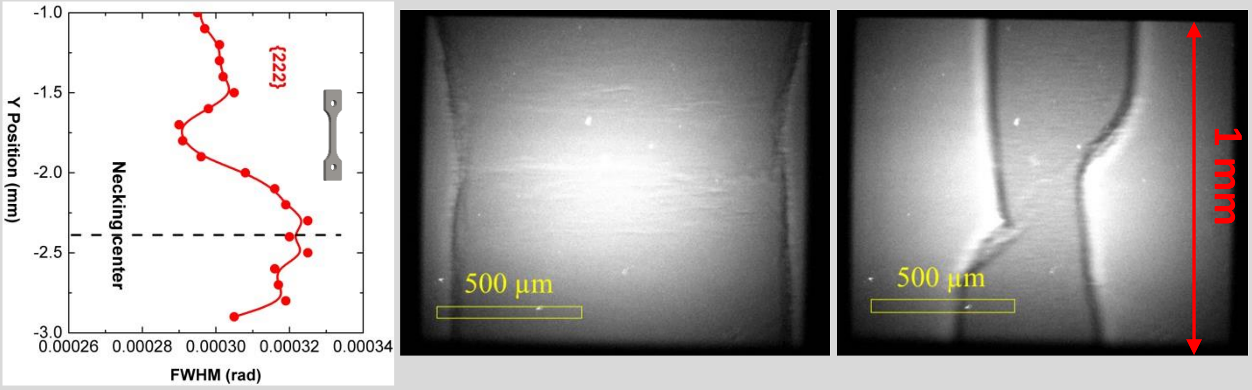


Stress-strain curve recorded during in situ 300°C tensile test.



Lattice constant changes and peak broadening during tensile deformation.

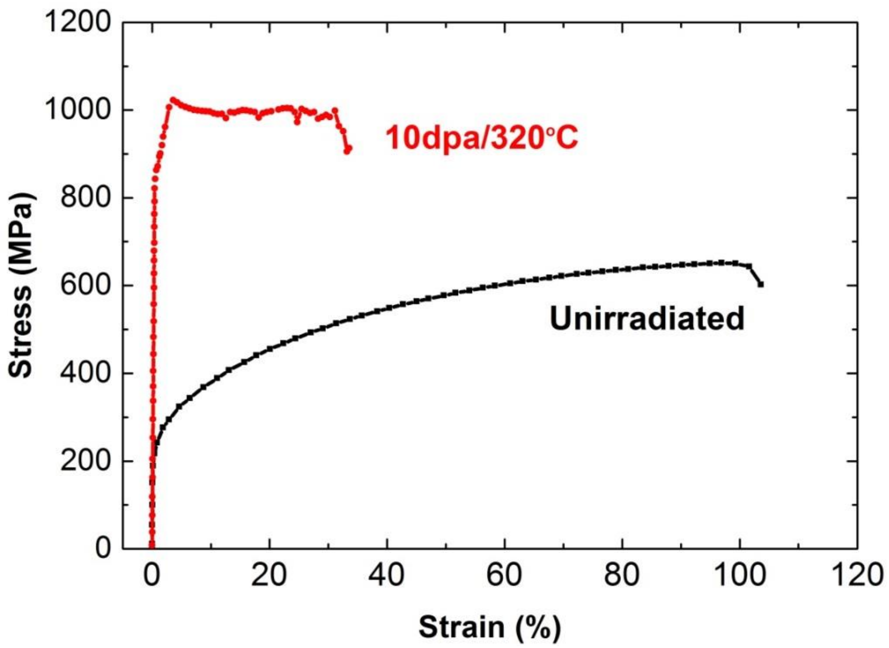
Specimen necking and failure revealed by WAXS and tomography



In situ Tensile Test of Neutron-Irradiated 316 SS at 20°C

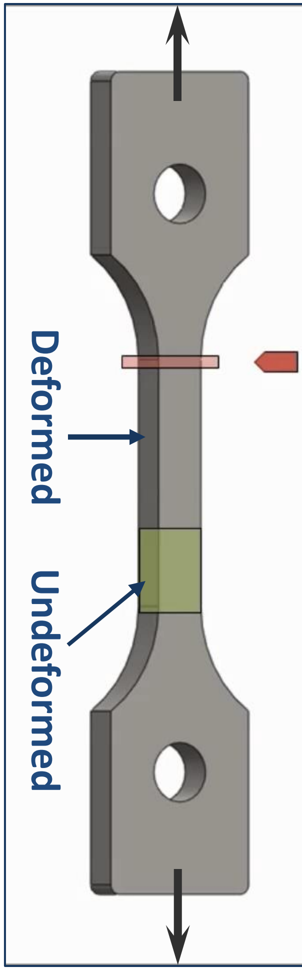
- **Material**:
  - Solution annealed 316 SS
- **Specimen**:
  - Sheet-type tensile specimen (gauge 7.62x1.52x0.76 mm)
- **Neutron irradiation**:
  - 320°C to 10 dpa (NRC archive sample)
- **Microstructure**:
  - Irradiation-induced Frank loops: mean size = 9 nm, density = $6x10^{22}$ /m^3
- **In situ X-ray test**:
  - Energy: 123 keV (0.01008 nm)
  - Beam size: 100x100 μm
  - Strain rate: 1-$3x10^{-5}$/s

- **Deformation behavior of irradiated 316 SS under PWR-relevant irradiation condition**
  - Radiation hardening and ductility loss
  - No strain hardening before necking



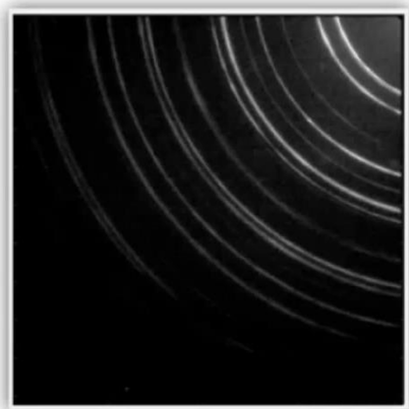
Stress-strain curves recorded during *in situ* room-temperature tensile test.

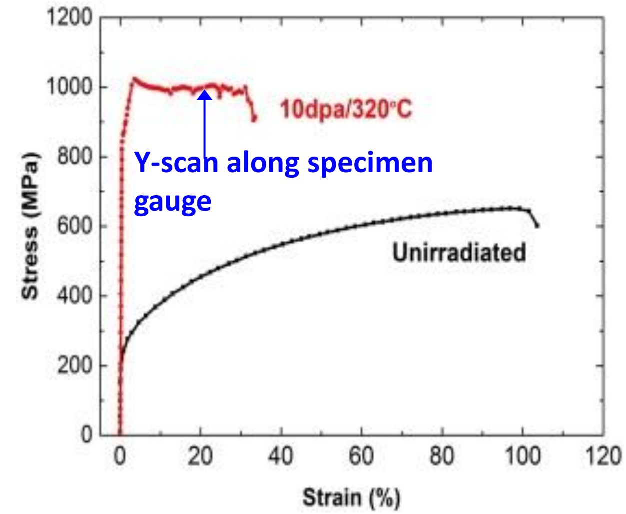
Inhomogeneous Deformation in Neutron Irradiated 316 SS

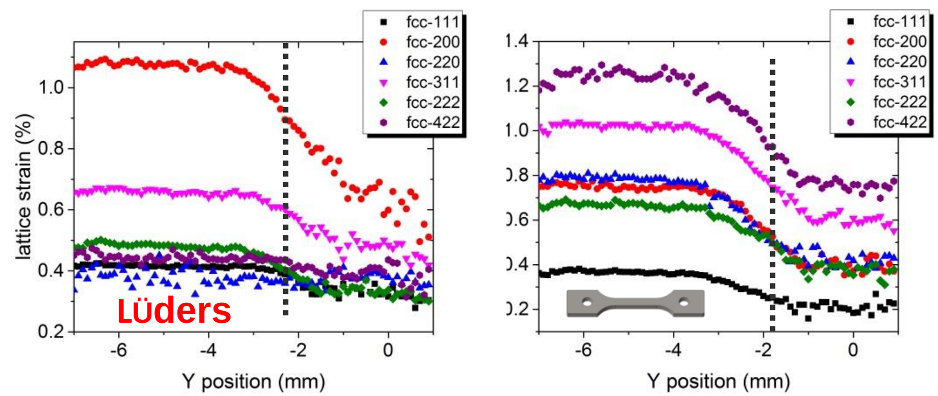


316 SS (320°C/10 dpa)

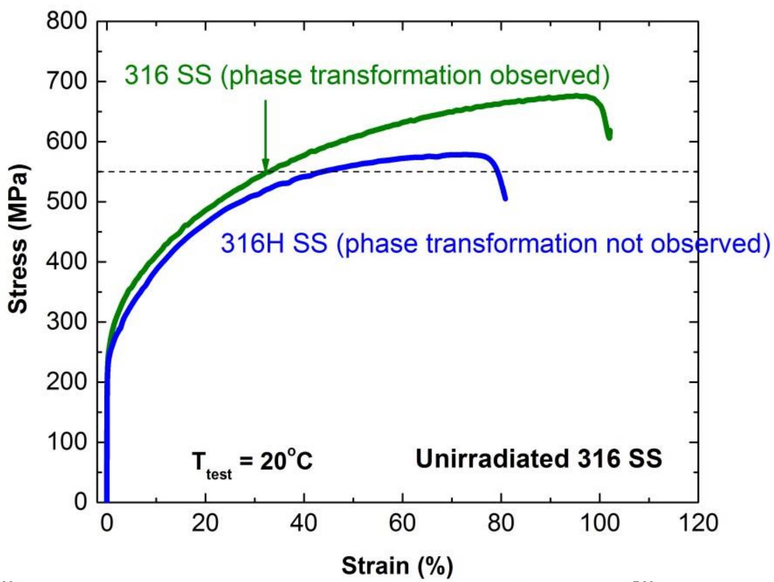
Velocity = 0.2-0.3 μm/s
$\dot{\varepsilon}$ = 0.2 μm/s

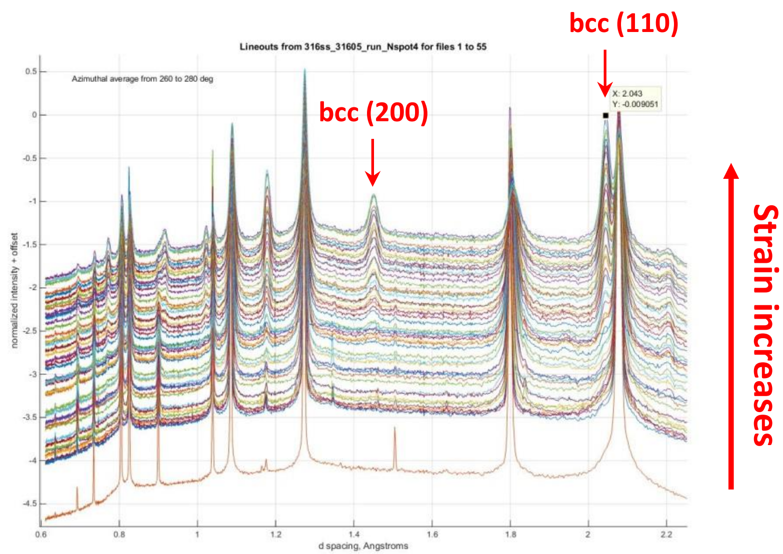


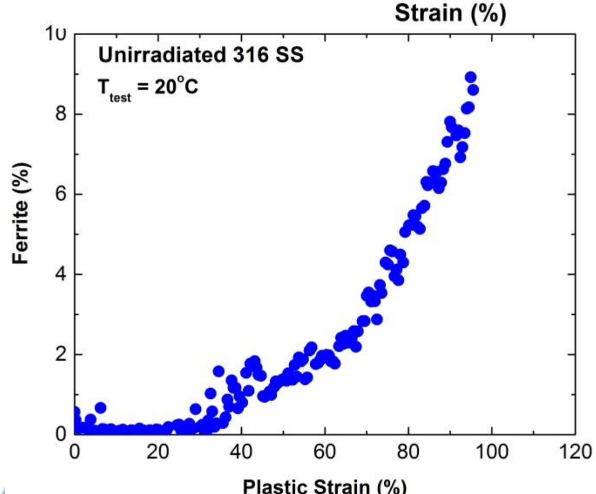


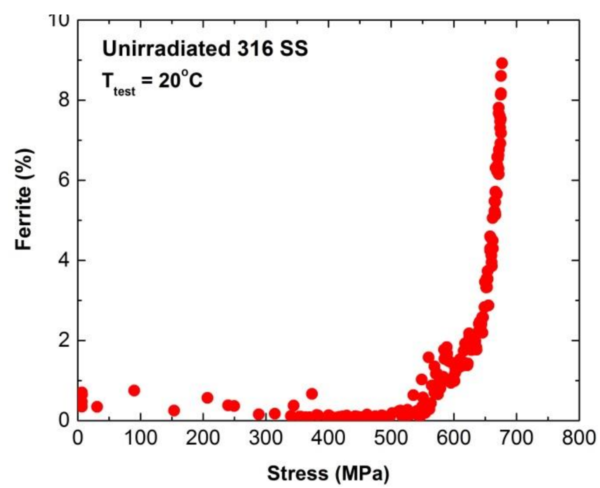


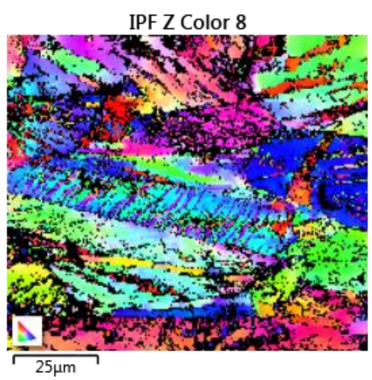
Deformation-Induced Martensite Transformation in Unirradiated 316SS





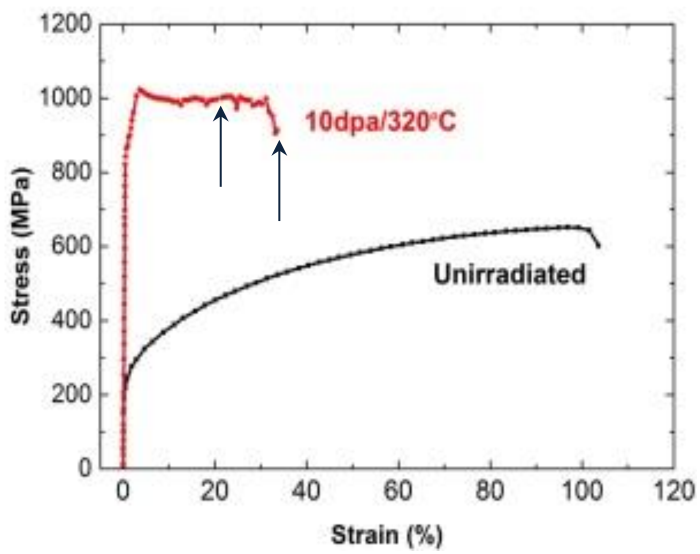


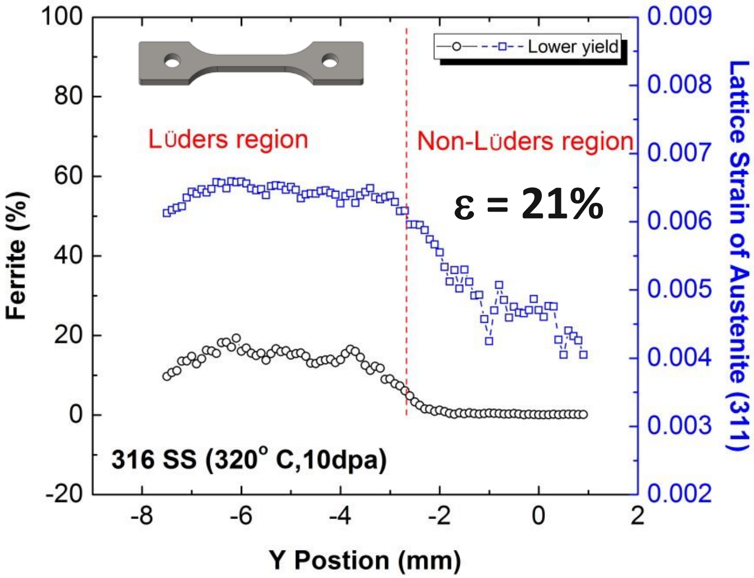


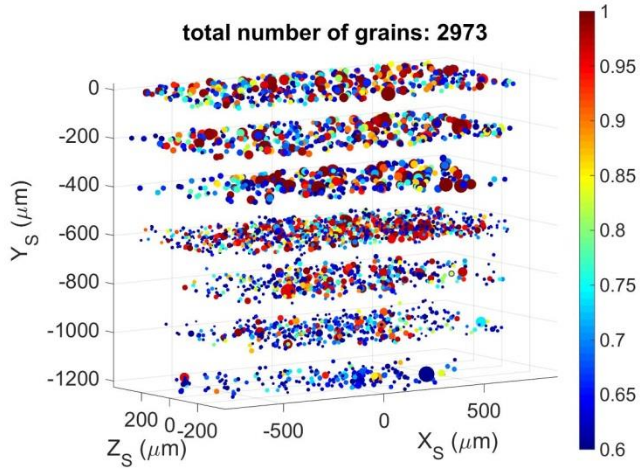


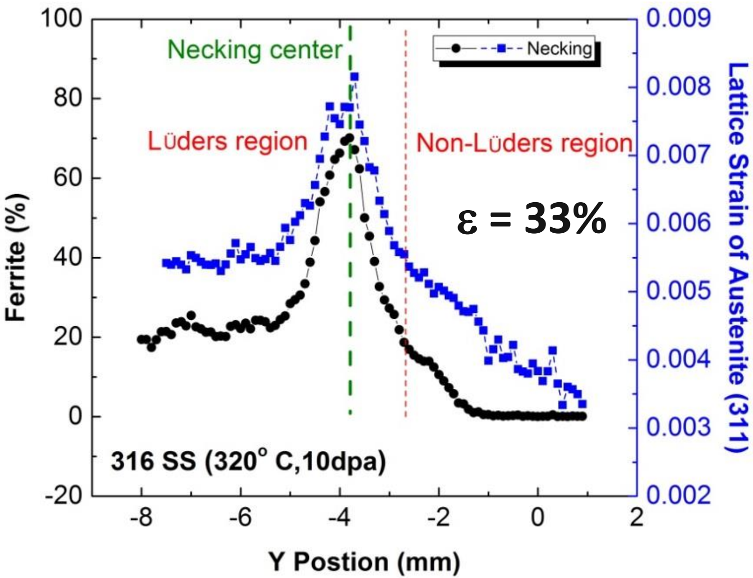
EBDS: Fer = 6%

Deformation-Induced Martensite Transformation in Neutron-Irradiated 316SS



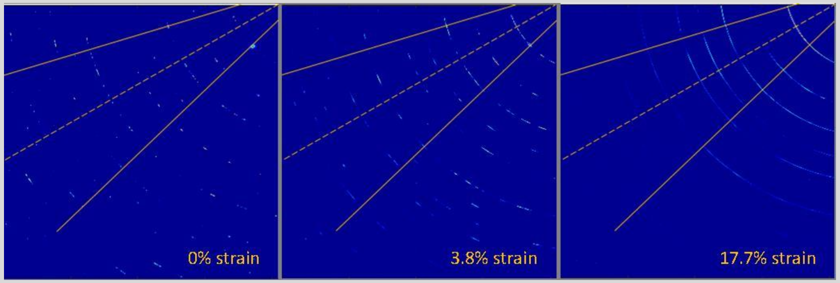




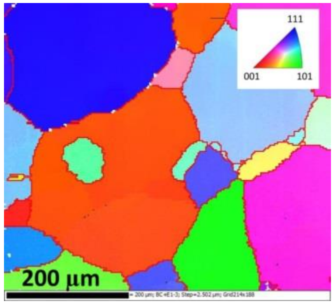


Irradiation Hardening in Low-dose Neutron-irradiated Fe-Cr Model Alloy

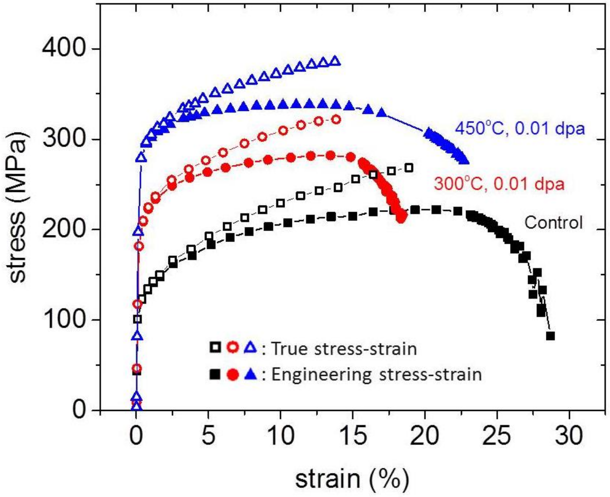
- **Material**:
  - Fe-9Cr model alloy
- **Specimen**:
  - Sheet-type tensile specimen
  - Gauge 5.0x1.2x0.5 mm
- **ATR neutron irradiation (U. Illinois)**:
  - 300°C to 0.01 dpa
  - 450°C to 0.01 dpa
- ***In situ* tensile test**:
  - Temperature: 20°C
  - Strain rate: 1-3x10^{-5}/s
- **X-ray measurement**:
  - Energy: 123 keV
  - Beam size: 100x100 μm
  - Sample-detector distance: 2635 mm
  - WAXS/SAXS
  - Coarse-grain structure, averages over 30 measurements, covering 0.5mm^3 volume.



- **Microstructure**:
  - Large grain size ~200 μm
  - No TEM-visible defects in 300°C-irr specimen
  - ~4 nm loops in 450°C-irr specimen

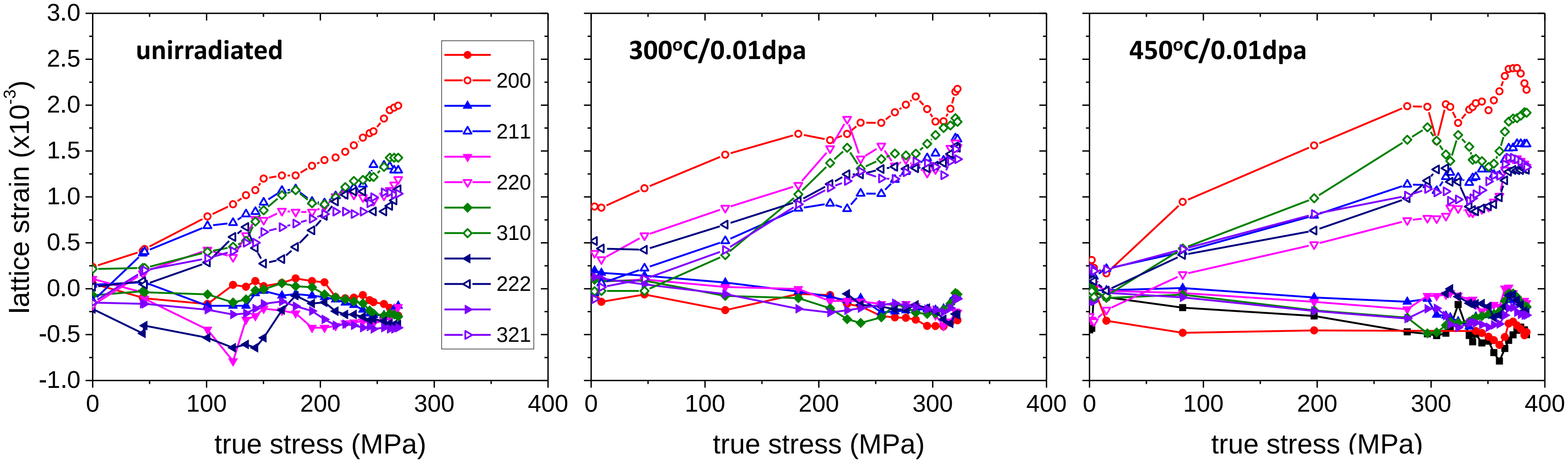


- **Stress-strain curves**:

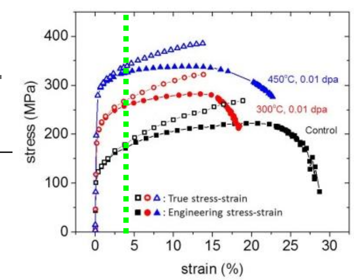


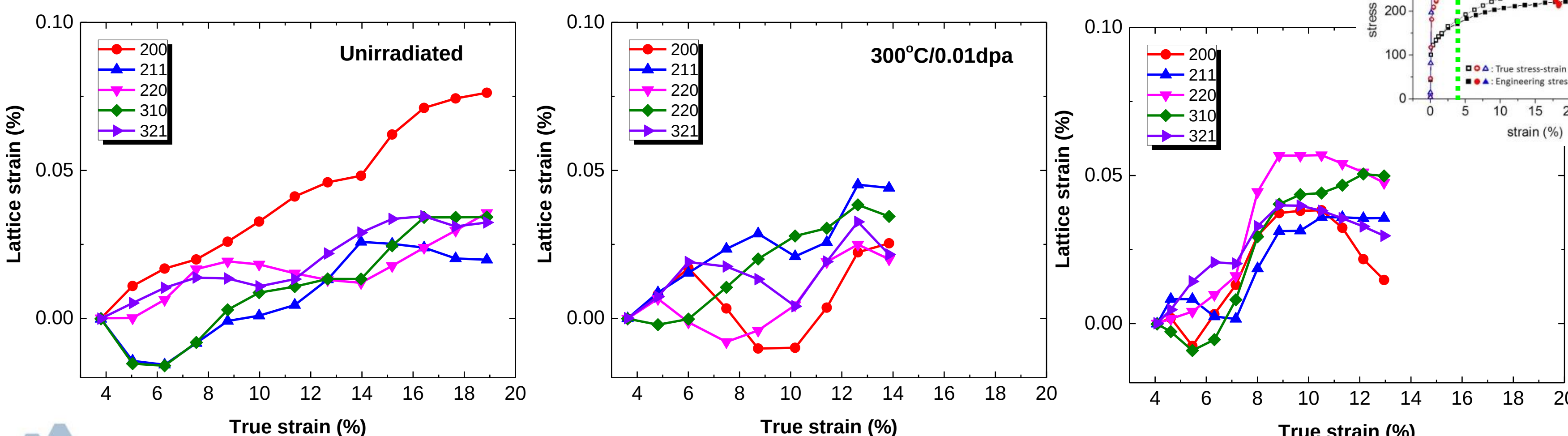
Stress-strain curves recorded during *in situ* RT tensile tests with high-energy X-rays.

Evolution of Lattice Strain during Tensile Deformation

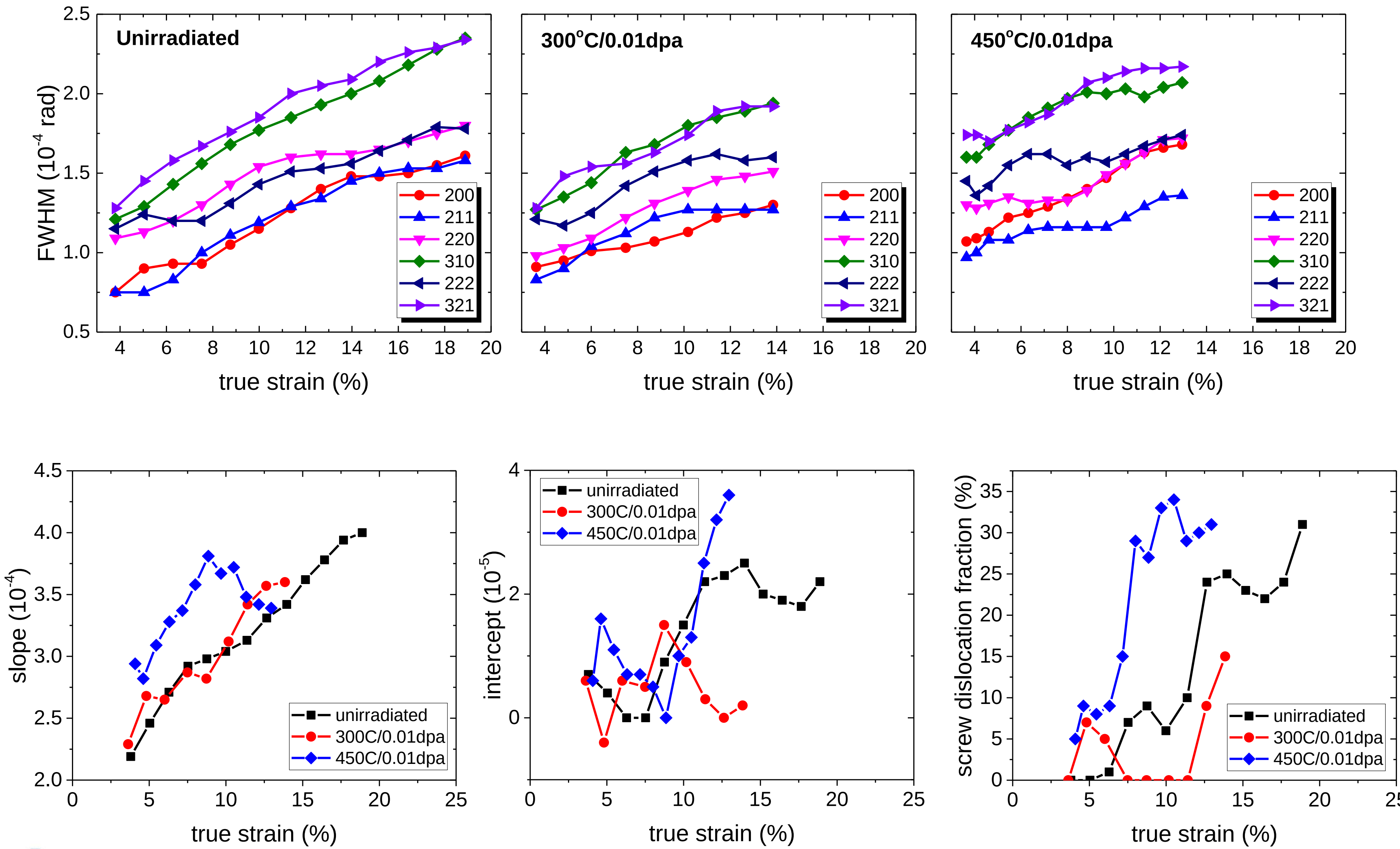


Lattice strain in the loading direction using 4% strain as the reference:



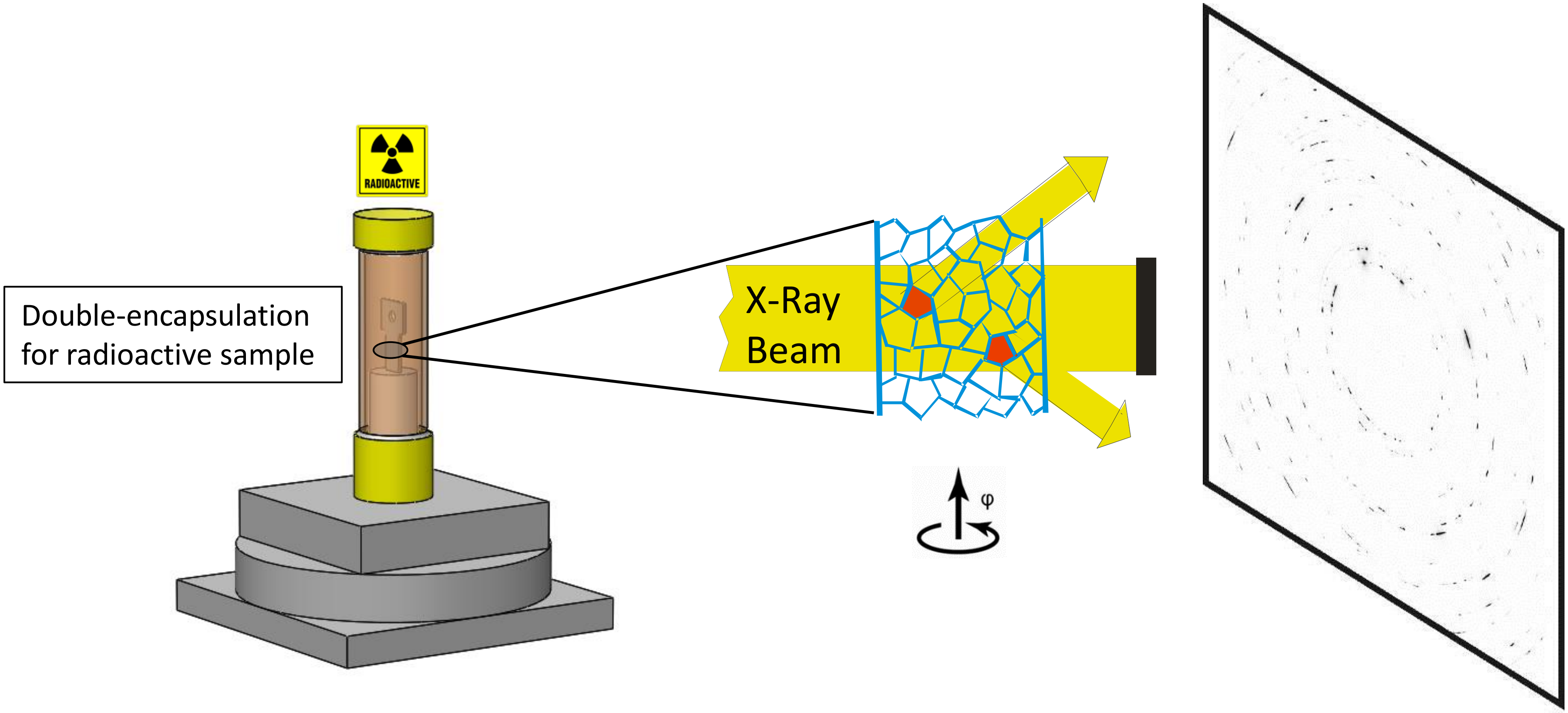


Peak Broadening and Line Profile Analysis

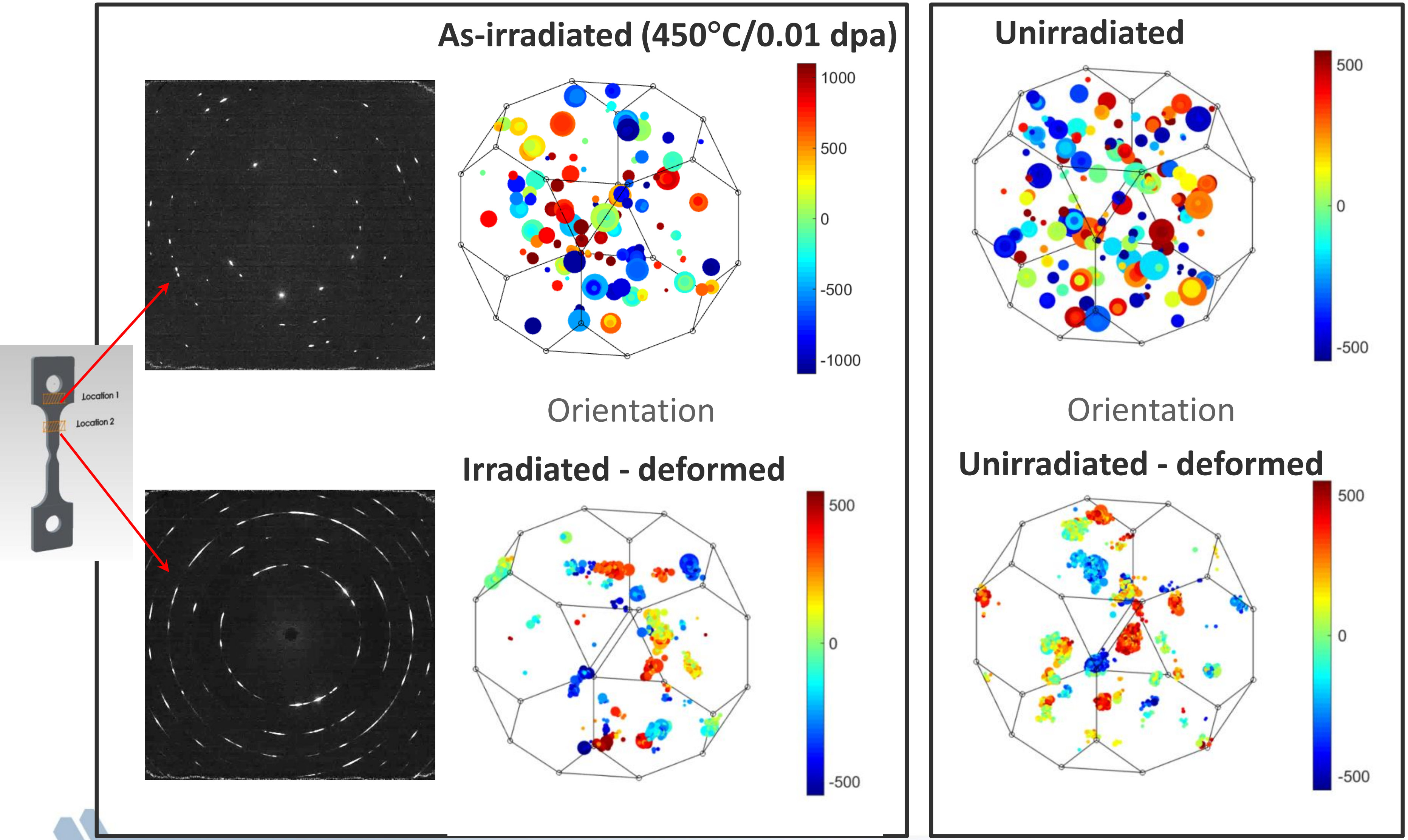


Ex situ Far-field High Energy Diffraction Microscopy (ff-HEDM) of Irradiated Fe-9Cr Alloy

- HEDM also known as 3D-XRD
- **3-dimensional, non-destructive**
- **Statistical significance:** thousands of grains at once
- **Far-field (ff) HEDM:** grain location, volume, orientation, strain

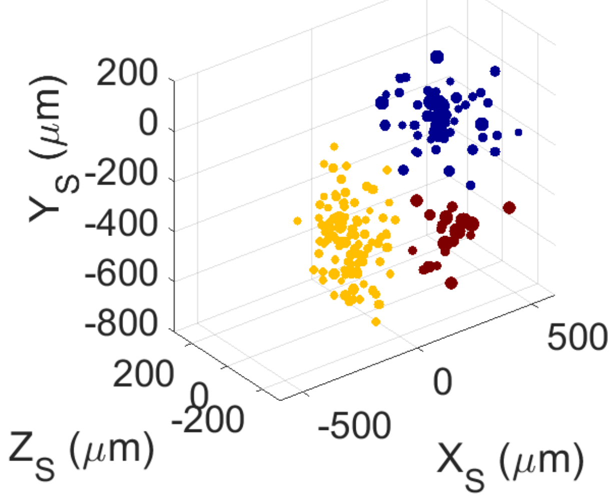


Sub-structure formation in Neutron-Irradiated Fe-9Cr Alloy during Tensile Deformation

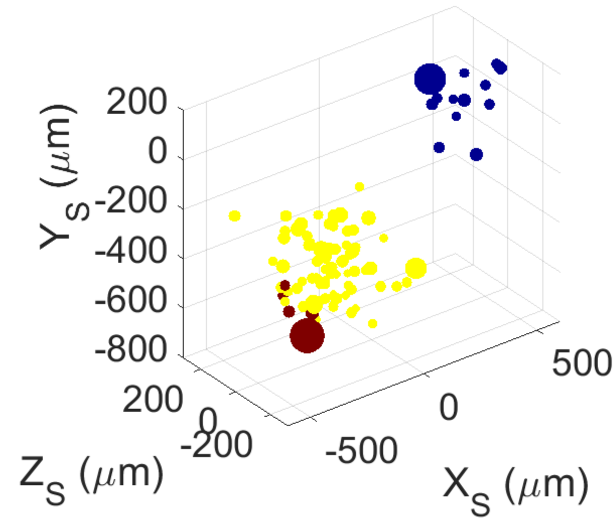


Structural Inhomogeneity in Tensile-Deformed Irradiated Fe-9Cr Alloy

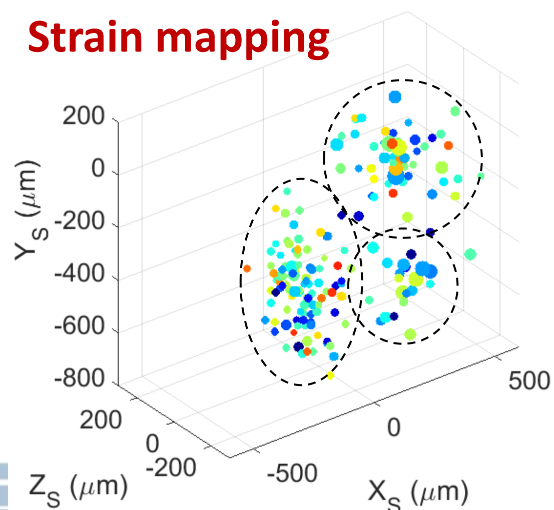
Unirradiated - deformed

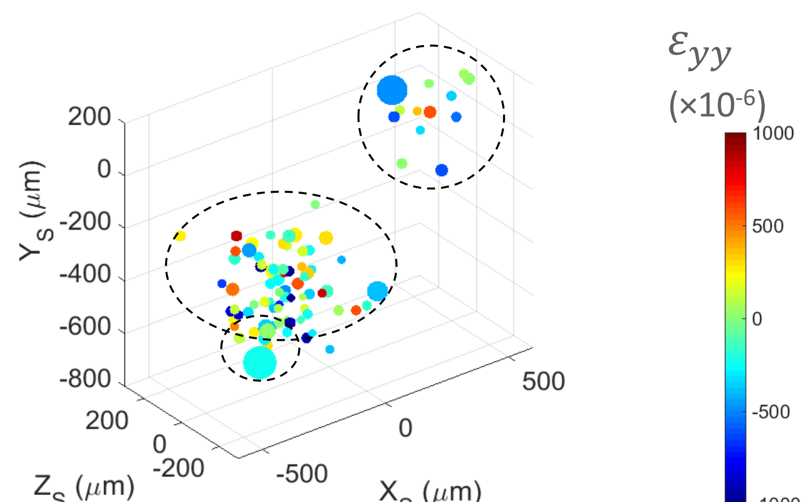


Irradiated - deformed

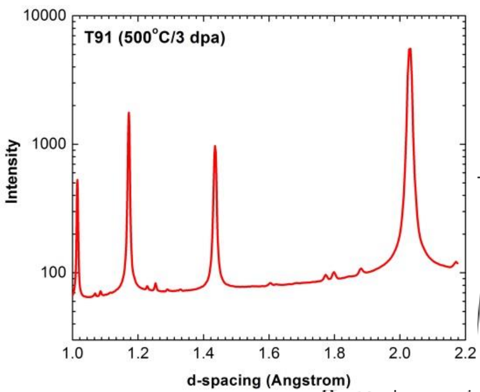


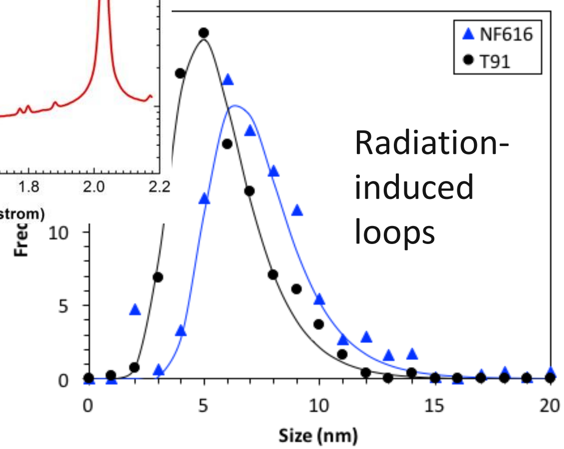
Strain mapping

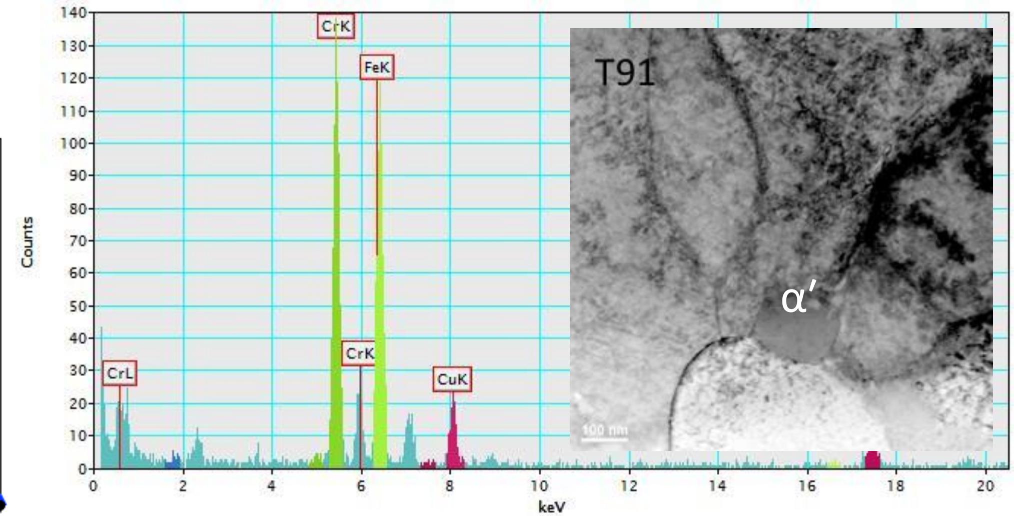


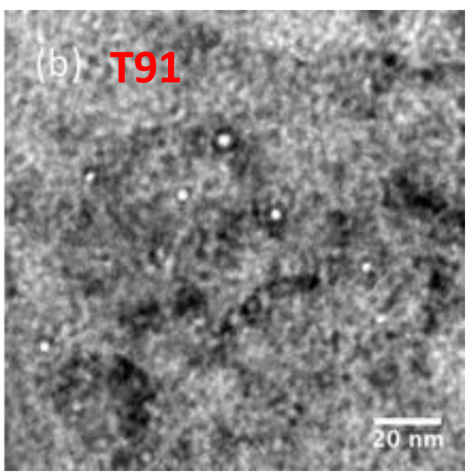


Synergy of Advanced Characterization Techniques

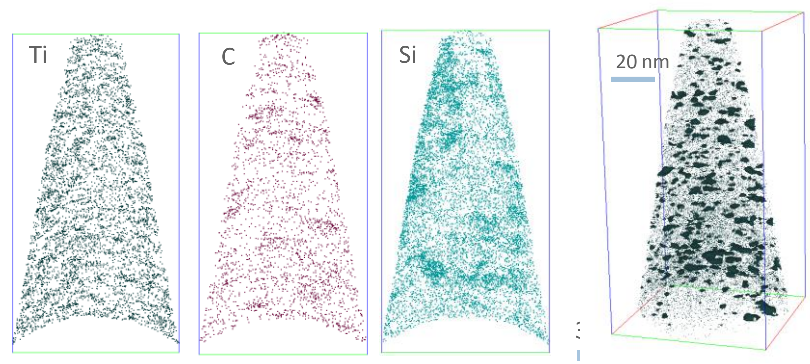








Neutron-irradiated HT-UPS (500°C/3dpa)



Ti 0.7% at. con. isosurface , density: ~$3\times10^{24}/m^3$

Summary

- Established and demonstrated the capability for *in situ* high-energy X-ray characterization of neutron-irradiated specimens under thermal-mechanical loading. Conducted *in situ* tensile test of neutron-irradiated pure Fe at 300°C in vacuum with simultaneous wide-angle X-ray scattering and small-angle X-ray scattering measurements.
- *In situ* tensile tests of neutron-irradiated austenitic stainless and ferritic alloys provide new insight into radiation hardening mechanisms, strain-induced phase transformation, plastic instability, and failure mechanisms.
- Post-mortem *ex situ* 3D characterization of tensile-deformed specimens by far-field high-energy X-ray microscopy revealed the effect of neutron irradiation on substructure formation and strain inhomogeneity within individual grains
- Future effort will focus on 3D characterization with *in situ* thermal-mechanical loading to enable space- and time-resolved 4D characterization under thermal-mechanical loading.


Thank you