## FY2013 NEET Award -

# Developing Microstructure-Property Correlation in Reactor Materials using *in situ* High-Energy X-rays

**PIs**:

Meimei Li (ANL), Jonathan Almer (ANL), Yong Yang (U. Florida), Lizhen Tan (ORNL)

**Contributors**:

Erika Benda, Yiren Chen, Peter Kenesei, Ali Mashayekhi, Jun-Sang Park, Hemant Sharma (ANL), B.K. Kim and K.G. Field (ORNL)

**Postdoc and student**:

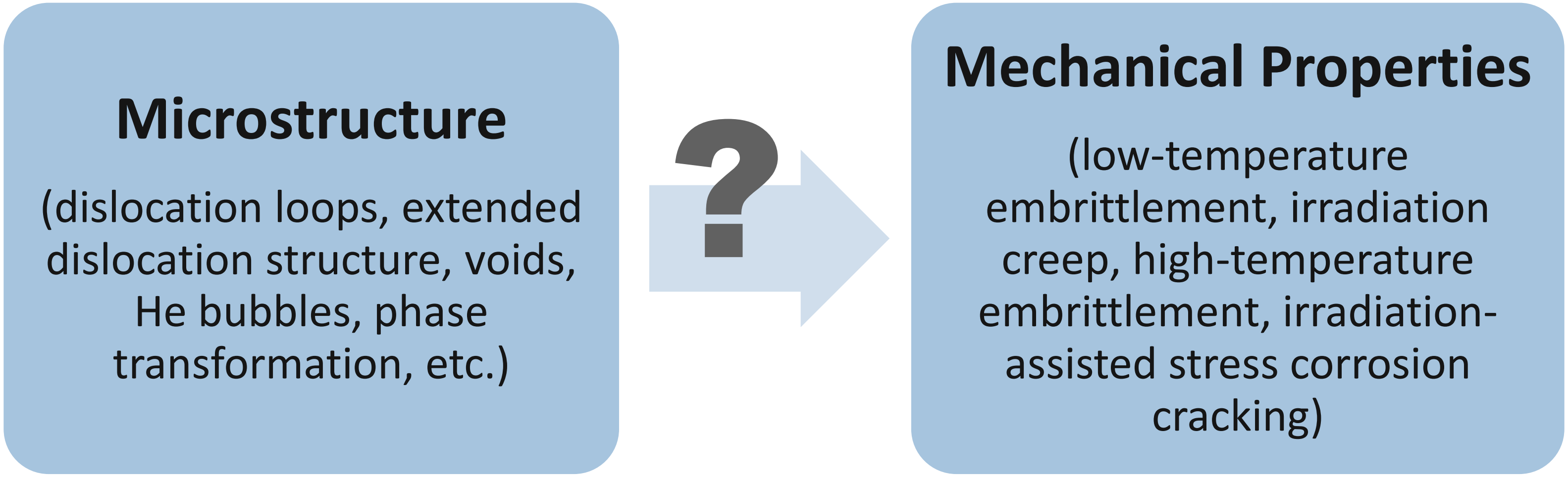
Xuan Zhang (Postdoc, ANL), Chi Xu (PhD student, U. Florida)

Special thanks to NSUF and Prof. Stubbins (U. Illinois) for providing irradiated specimens

FY2015 DOE NE NEET Webinar
September 16, 2015

# Motivation

## Microstructure – Property Correlation



**Microstructure**

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

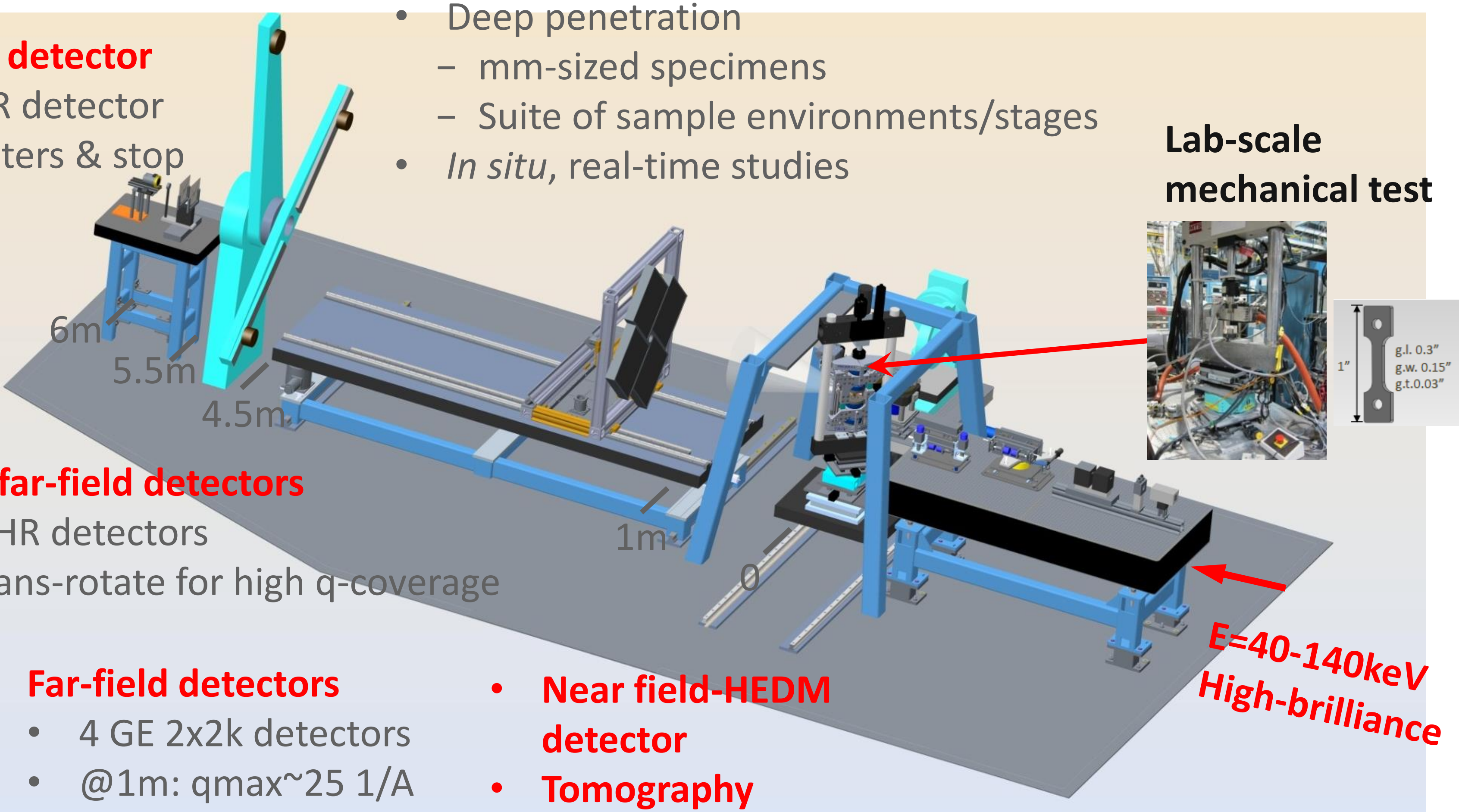
**Mechanical Properties**

(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* straining of lab-scale specimens with multiple probes

# *In situ* Straining with High-Energy X-rays and Multiple Probes

- Beamline 1-ID at Advanced Photon Source



**High-energy, high-brilliance X-rays:**

- Deep penetration
  - mm-sized specimens
  - Suite of sample environments/stages
- *In situ*, real-time studies

**SAXS detector**

- HR detector
- Filters & stop

**Lab-scale mechanical test**

**Very far-field detectors**

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

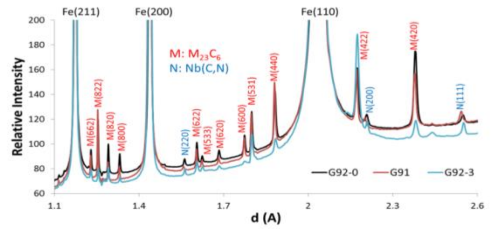
**E=40-140keV High-brilliance**

**Far-field detectors**

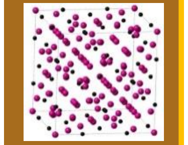
- 4 GE 2x2k detectors
- @1m: qmax~25 1/A
- Center-hole (SAXS)

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

# Wide-angle X-Ray Scattering (WAXS)



**$M_{23}C_6$**



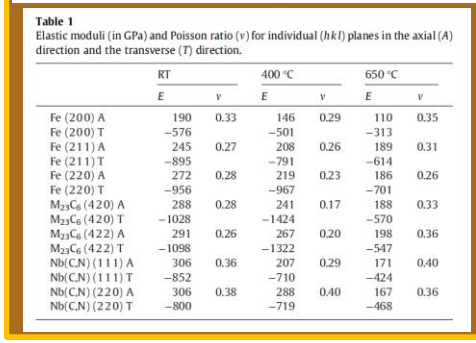
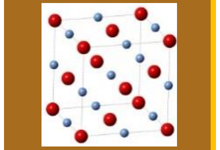
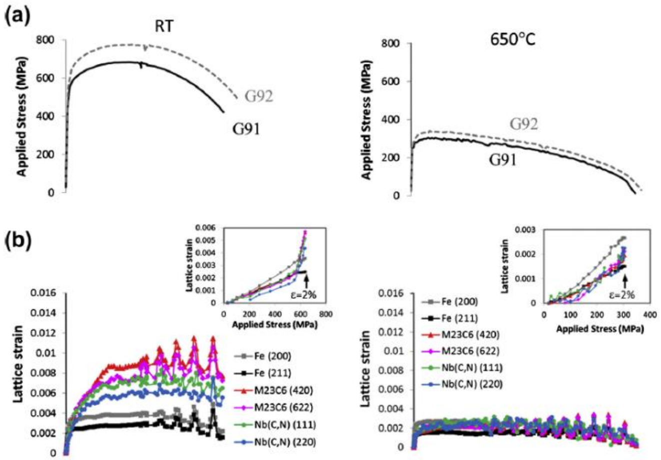


Table 1
Elastic moduli (in GPa) and Poisson ratio (*ν*) for individual (*hkl*) planes in the axial (*A*) direction and the transverse (*T*) direction.

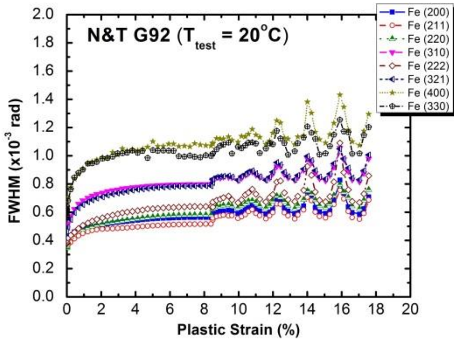
| | RT | | 400 °C | | 650 °C | |
|---|---|---|---|---|---|---|
| | *E* | *ν* | *E* | *ν* | *E* | *ν* |
| Fe (200) A | 190 | 0.33 | 146 | 0.29 | 110 | 0.35 |
| Fe (200) T | −576 | | −501 | | −313 | |
| Fe (211) A | 245 | 0.27 | 208 | 0.26 | 189 | 0.31 |
| Fe (211) T | −895 | | −791 | | −614 | |
| Fe (220) A | 272 | 0.28 | 219 | 0.23 | 186 | 0.26 |
| Fe (220) T | −956 | | −967 | | −701 | |
| $M_{23}C_6$ (420) A | 288 | 0.28 | 241 | 0.17 | 188 | 0.33 |
| $M_{23}C_6$ (420) T | −1028 | | −1424 | | −570 | |
| $M_{23}C_6$ (422) A | 291 | 0.26 | 267 | 0.20 | 198 | 0.36 |
| $M_{23}C_6$ (422) T | −1098 | | −1322 | | −547 | |
| Nb(C,N) (111) A | 306 | 0.36 | 207 | 0.29 | 171 | 0.40 |
| Nb(C,N) (111) T | −852 | | −710 | | −424 | |
| Nb(C,N) (220) A | 306 | 0.38 | 288 | 0.40 | 167 | 0.36 |
| Nb(C,N) (220) T | −800 | | −719 | | −468 | |

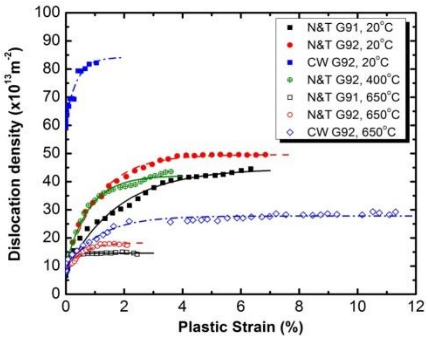
**MX**





- Identify phases
- Measure elastic moduli for individual (hkl) planes for each phase and temperature dependence
- Measure lattice strain evolution, load partitioning among different phases during deformation to quantify strengthening effects
- Measure dislocation density and subgrain structure evolution as a function of stress/strain to understand deformation mechanisms

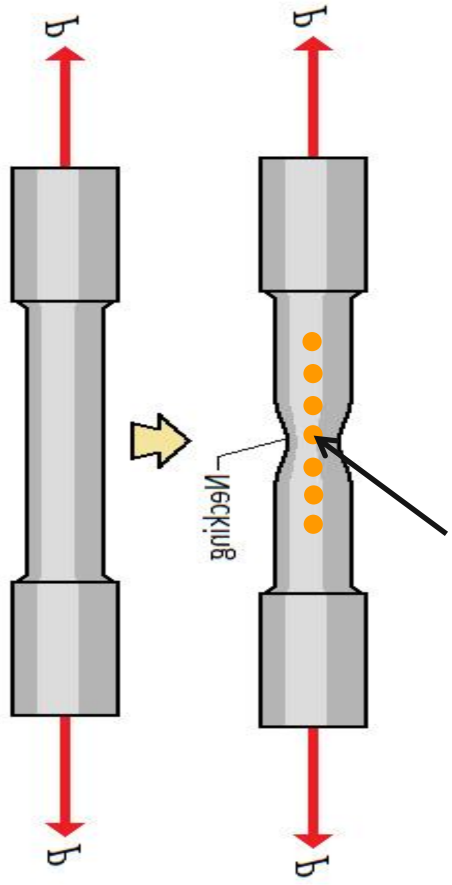




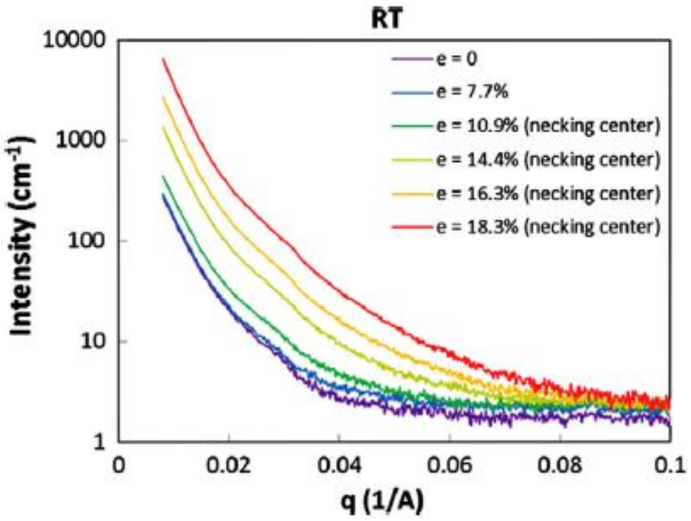
Wang, et al. Acta Mat. 62 (2014) 239; Li, et al. Acta Mat. 76 (2014) 381.

# Small-angle X-ray Scattering (SAXS)

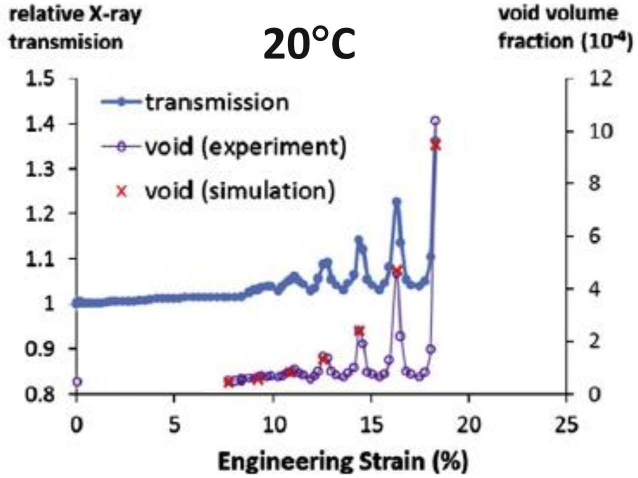
## Measure void formation and evolution



Vertical scans along the gauge



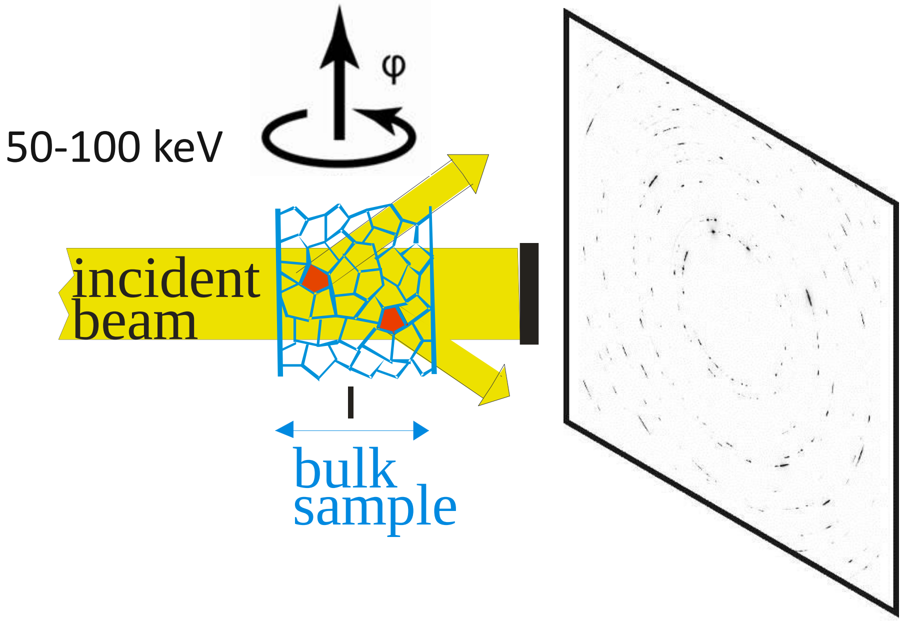
Void formation and grow during necking captured by SAXS

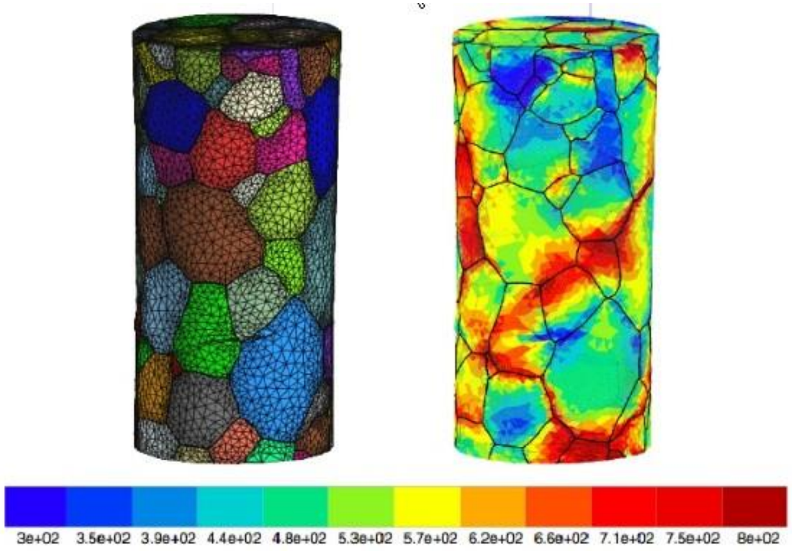


Wang, et al. JNM 440 (2013) 81.

# High Energy Diffraction Microscopy (HEDM)

- **Three-dimensional**, **grain-scale**, **non-destructive** characterization of microstructural and micromechanical response of individual grains within the bulk of a polycrystalline specimen.



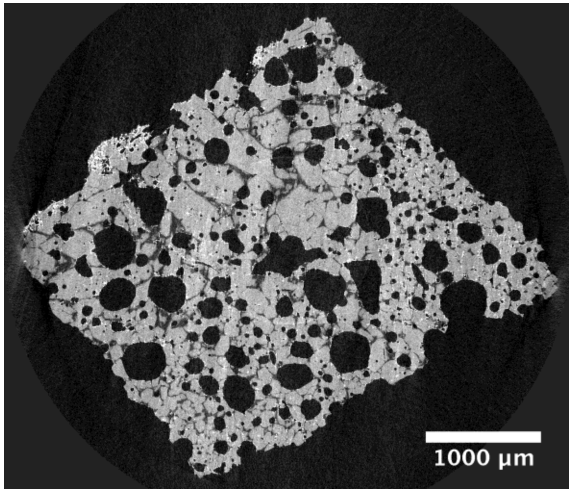


FEM simulation of von Mises stress in a Ti alloy sample loaded to 500 MPa. (Ludwig, et al, MSE A524 (2009))

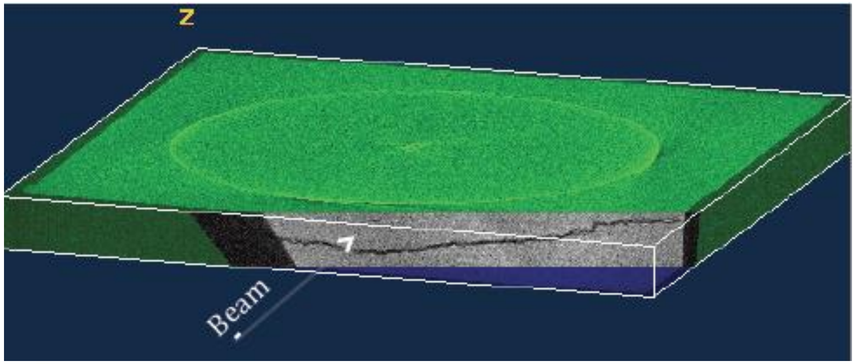
- Thousands of grains in mm-size samples
- Near-field HEDM: grain shape, orientation
- Far-field HEDM: strain, orientation

# X-ray Tomography

- Nondestructive technique for visualizing internal microstructure within a material
- Provide 3D images of the internal structure (pores, voids, cracks, etc.) in a material

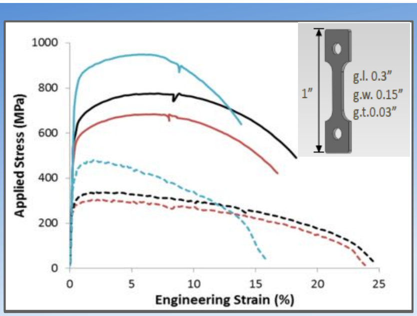


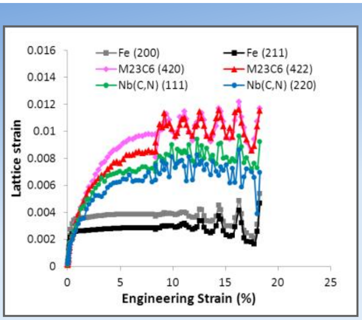
Absorption Tomography provides information due to electron density, revealing presence of voids, cracks, etc.
(by AFRL, unpulished)

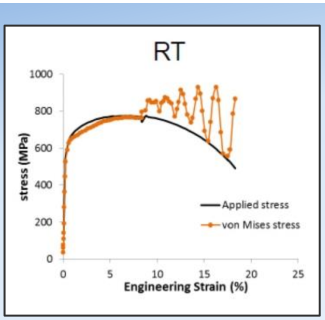


X-ray tomography of thermally-fatigue GlidCop specimen measured at APS beamline 1-ID.
(A. Khounsary et al. J. Phys 425 (2013) 212015)

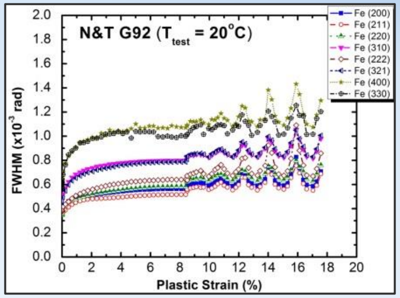
# *In situ* Characterization of F-M G92 Steel during Tensile Deformation by WAXS/SAXS/Radiography



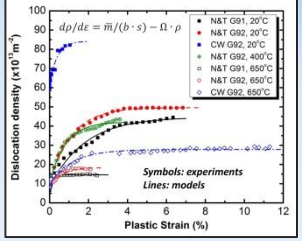




**Diffraction peak shifts revealed load partitioning among phases during deformation**

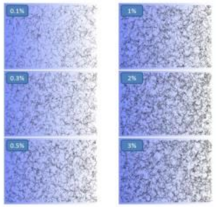


Evolution of the dislocation density during deformation is described by dislocation creation/storage and dislocation annihilation



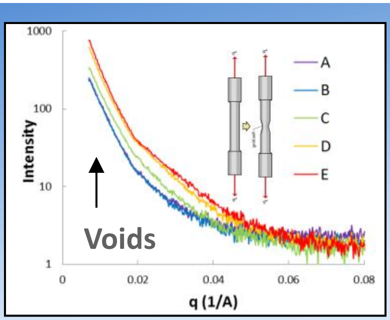
*Symbols: experiments*
*Lines: models*

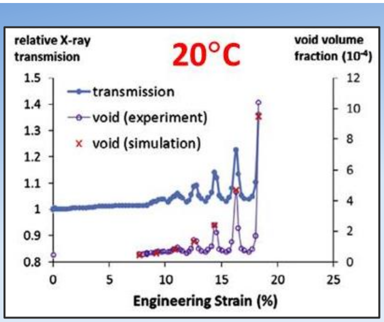
Dislocation dynamic modeling of dislocation density change as a function of strain that may be validated by in situ X-ray measurements.

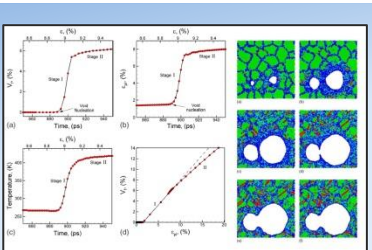


*(Liu et al., 2012)*

**Diffraction peak broadening revealed dislocation evolution during deformation**



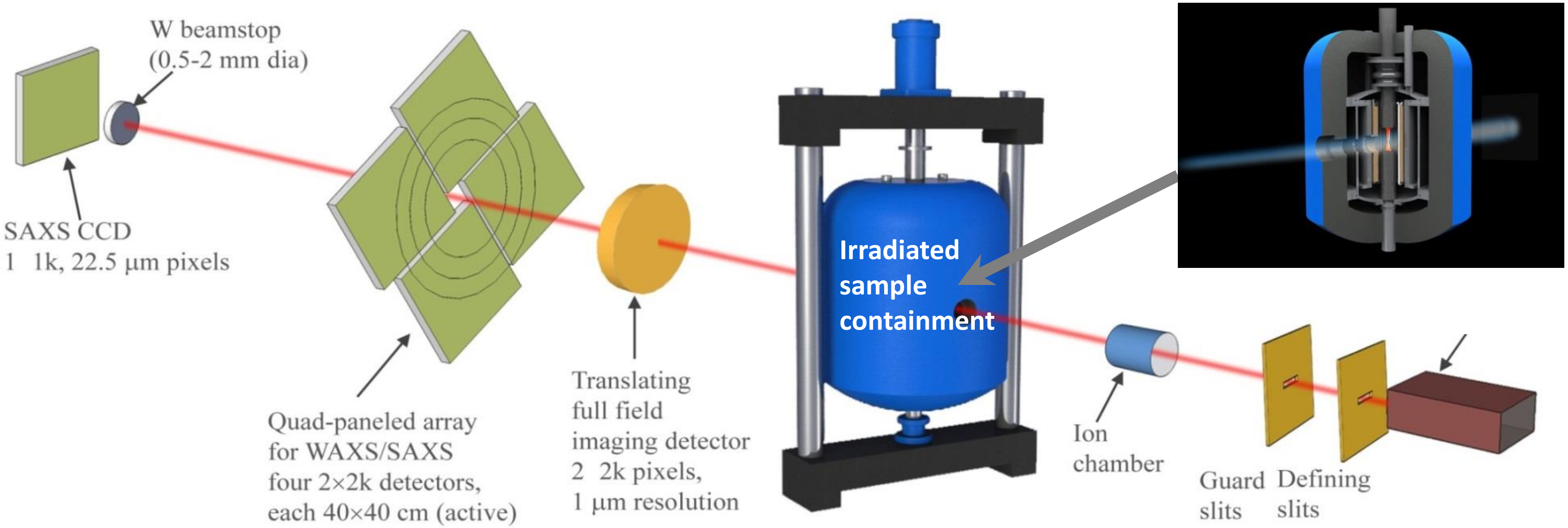




Molecular Dynamics (MD) simulations showed void evolution during tensile deformation
(Dongare et al (2009))

**SAXS captured void formation and evolution during necking**

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



W beamstop
(0.5-2 mm dia)

SAXS CCD
1 1k, 22.5 μm pixels

Quad-paneled array
for WAXS/SAXS
four 2×2k detectors,
each 40×40 cm (active)

Translating
full field
imaging detector
2 2k pixels,
1 μm resolution

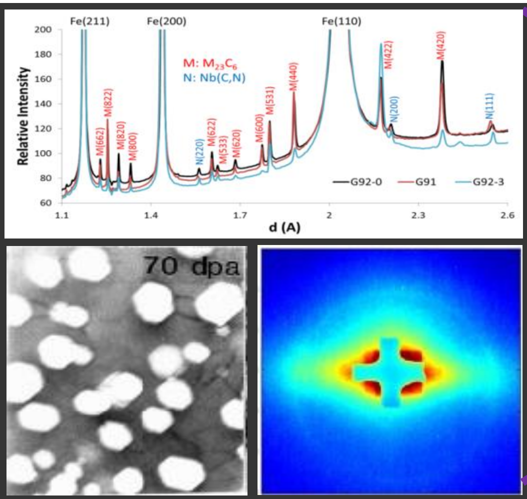
Irradiated
sample
containment

Ion
chamber

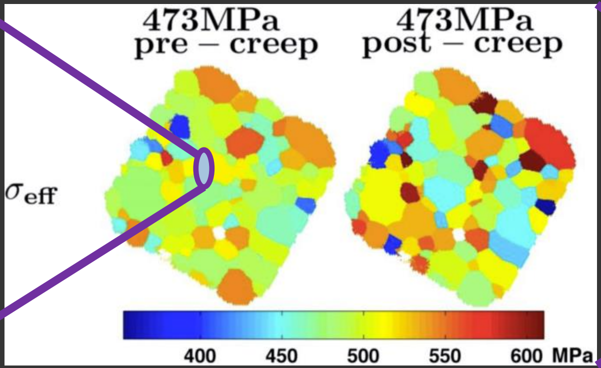
Guard
slits

Defining
slits

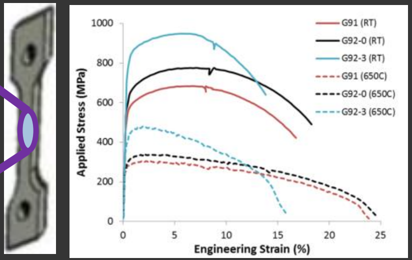
(Schuren, et al 2014, pre-publication)



**Nanoscale:** WAXS and SAXS



**Mesoscale:** diffraction microscopy & tomography



**Macroscale:** stress-strain behavior

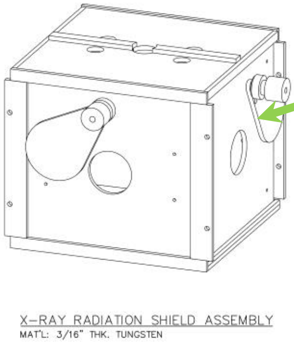
# *In situ* X-ray Radiated Materials Straining/Annealing (*i*RadMat) Apparatus

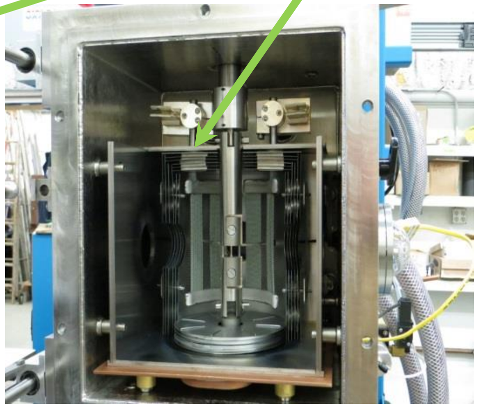
**Unique x-ray sample environment**

- Internal radiation shielding for activated samples
- Temperature: <1000°C
- Vacuum: $1 \times 10^{-5}$ Torr
- Tension, creep, fatigue loading
- In-grip rotation for tomography & diffraction microscopy

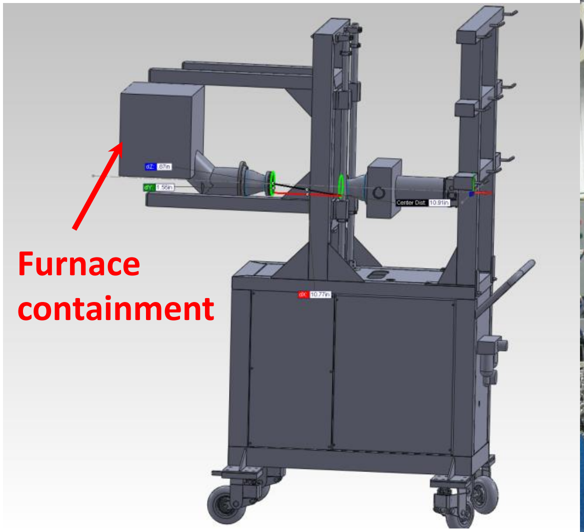
**Vacuum furnace with Integrated Radiation Shielding**

**W plates & shutters**



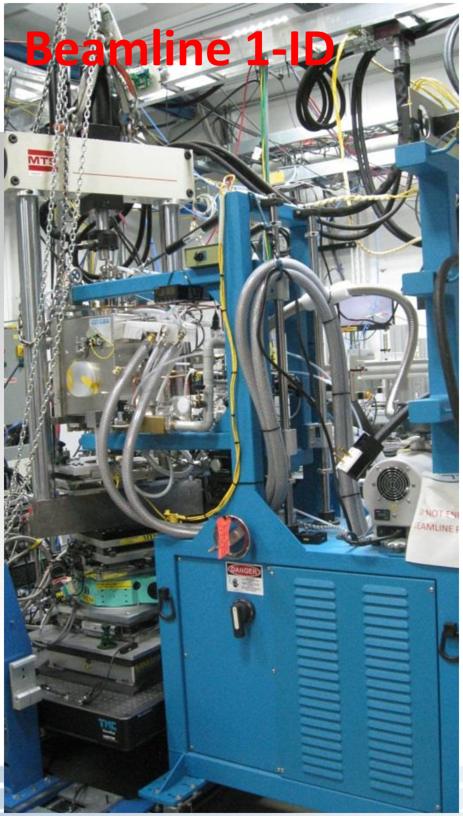


## *i*RadMat

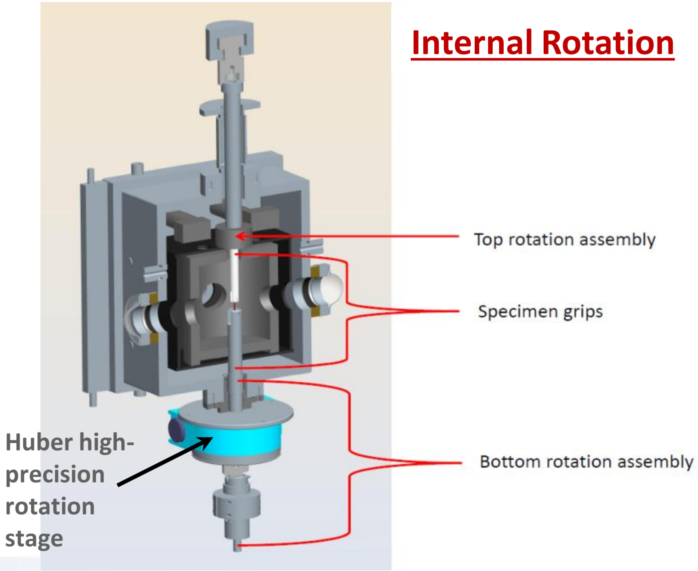


**Furnace containment**

**Beamline 1-ID**



**Internal Rotation**



# Challenge - Handling Activated Specimen

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

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

Pack into a shielded containment and survey.

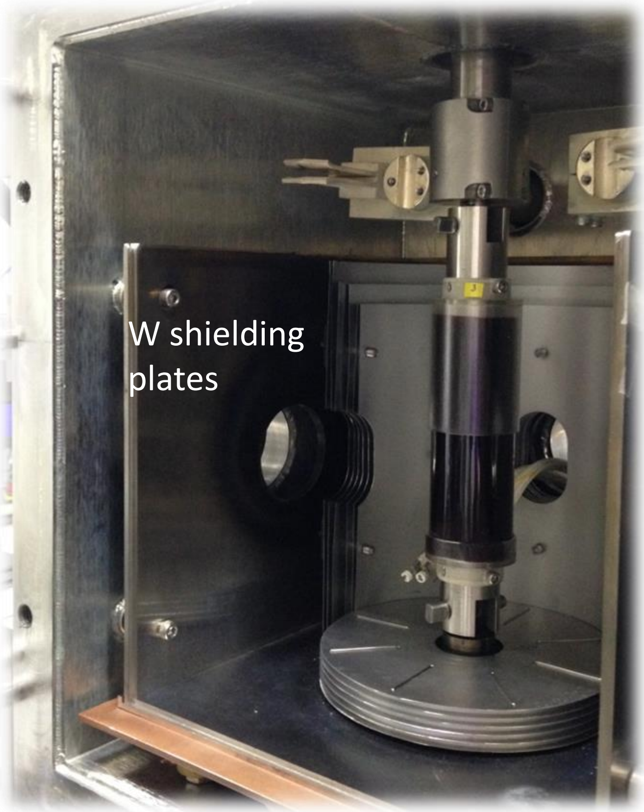
Transfer between IML and APS

## Advanced Photon Source (APS)

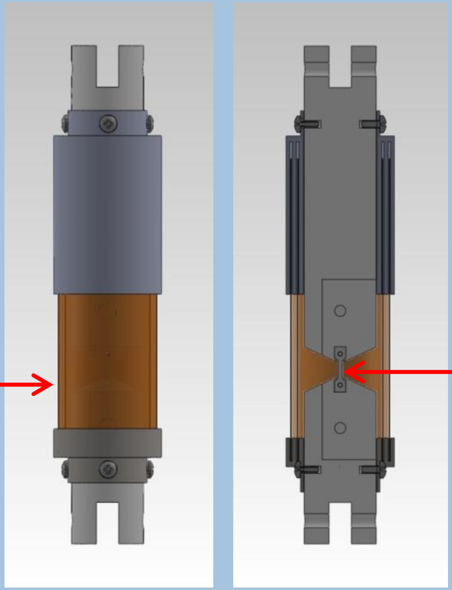
Unpacking and loading at 1-ID beamline

# Encapsulation for Activated Tensile Specimen

## RT tensile test of an irradiated specimen



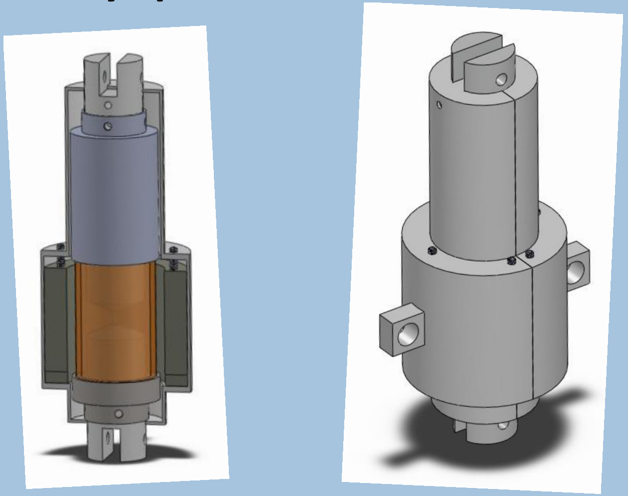
## For low-activity specimens



**Double-layered Kapton tubes**

**Irradiated Sample**

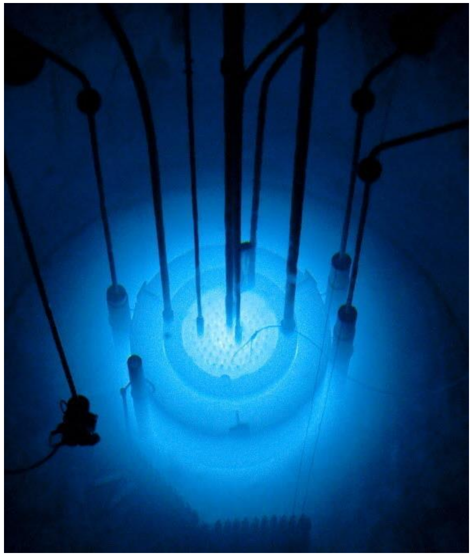
## For high-activity specimens – additional local shielding



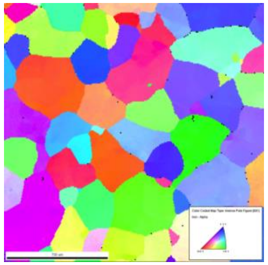
# *In situ* Straining of Neutron-Irradiated Fe-9Cr Alloy

| Samples | Non-irradiated | Irradiated | Irradiated |
|---|---|---|---|
| $T_{irr}$ ($^{o}$C) | N/A | 300 | 450 |
| dose (dpa) | N/A | 0.01 | 0.01 |

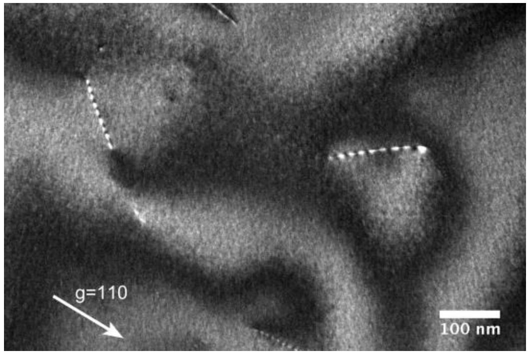
**U. Illinois Irradiation Experiment at ATR**



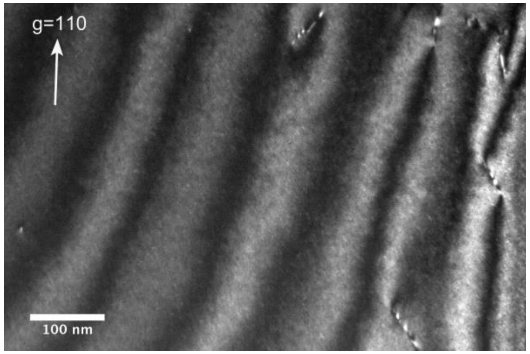
- EBSD mapping of control sample shows an average grain size of 180 μm.



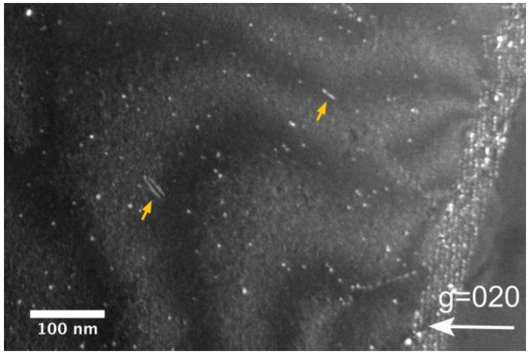
- TEM characterization of defect structures shows:
  - no visible irr-induced defects in 300$^{o}$C-0.01dpa sample;
  - nano-sized loops in 450$^{o}$C-0.01dpa specimen sample.



Unirradiated



300$^{o}$C, 0.01 dpa

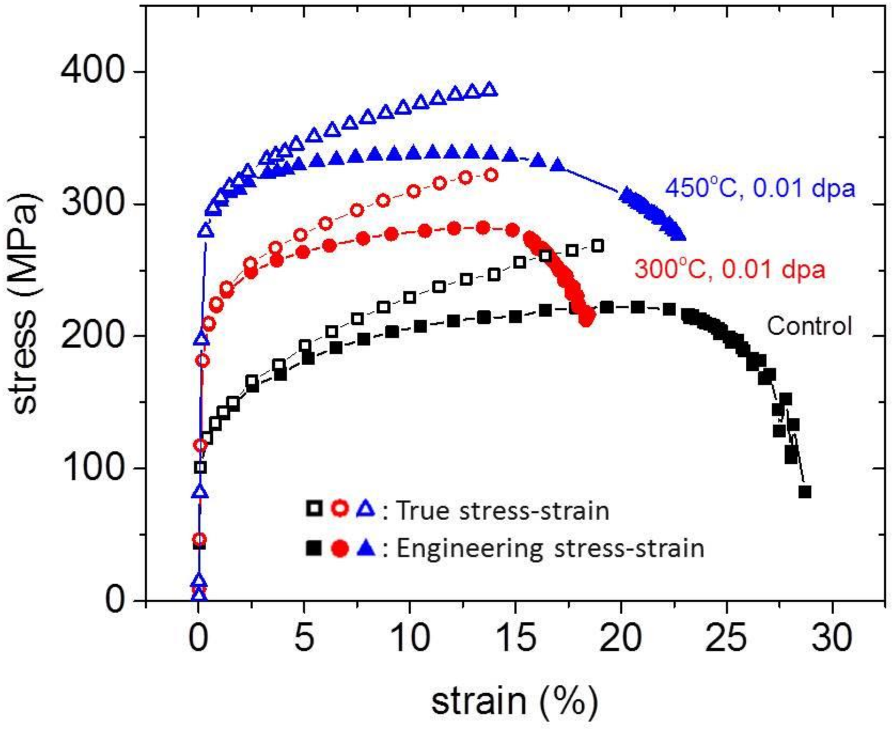


450$^{o}$C, 0.01 dpa

W-Y Chen, Ph.D. thesis, 2014

# Stress-Strain Behavior of Neutron-Irradiated Fe-9Cr

- Stress-strain curves recorded during *in-situ* X-ray measurement



Work-hardening:

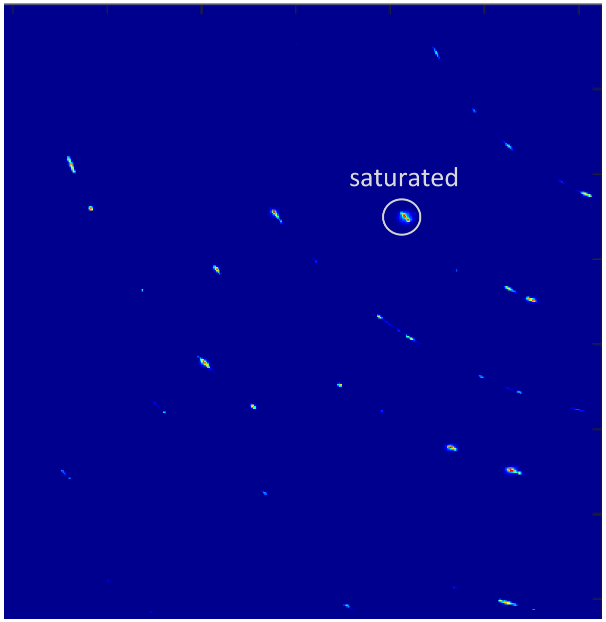
$$\sigma = 76.82 + 63.02\varepsilon^{0.380}$$

$$\sigma = 128.43 + 100.20\varepsilon^{0.254}$$

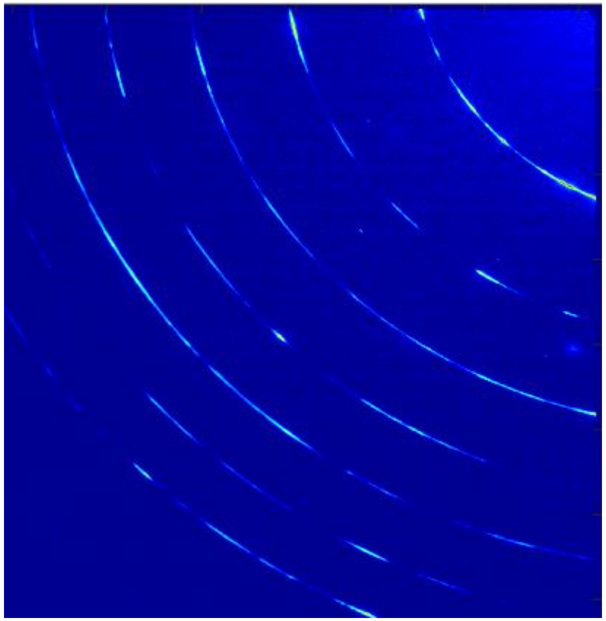
$$\sigma = 214.86 + 88.06\varepsilon^{0.255}$$

# Wide-angle X-ray Scattering during Deformation

300$^{o}$C irr, as received



300$^{o}$C irr, after deformation



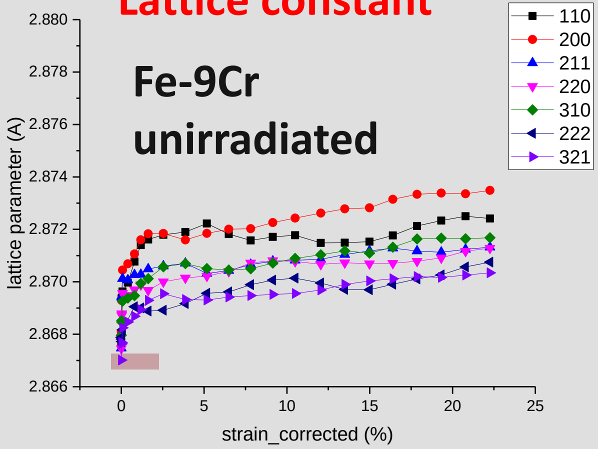
X-ray energy: E = 122keV
X-ray beam size=0.2x0.2mm$^2$

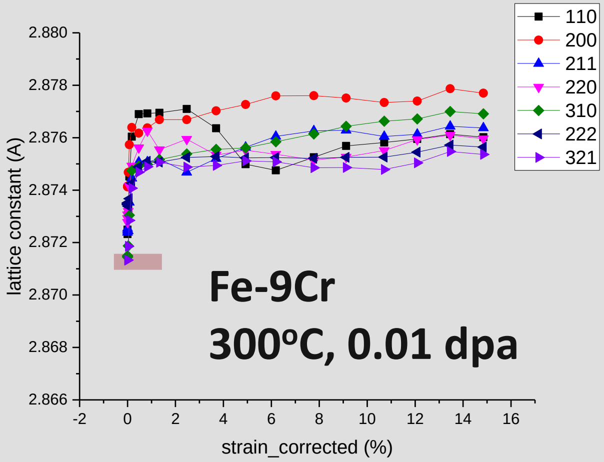
**Strain rate ~ 1-3x10$^{-5}$ /sec**
-> duration for 1 test: ~5h

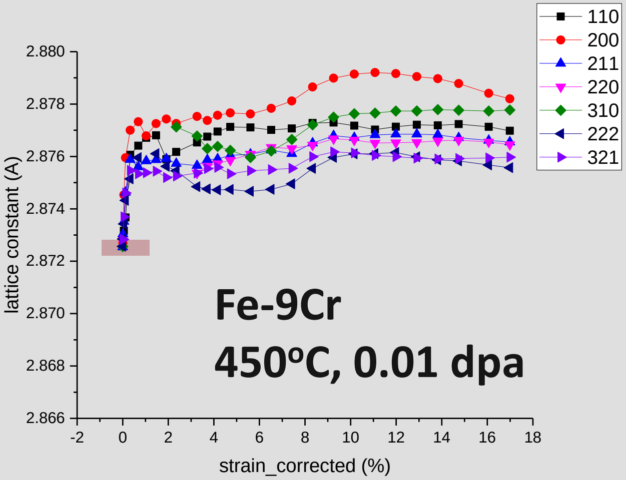
1 data point averages over 30 measurements, covering 0.5mm$^3$ volume of about 100 grains.

# Lattice Strain Evolution during Tensile Deformation

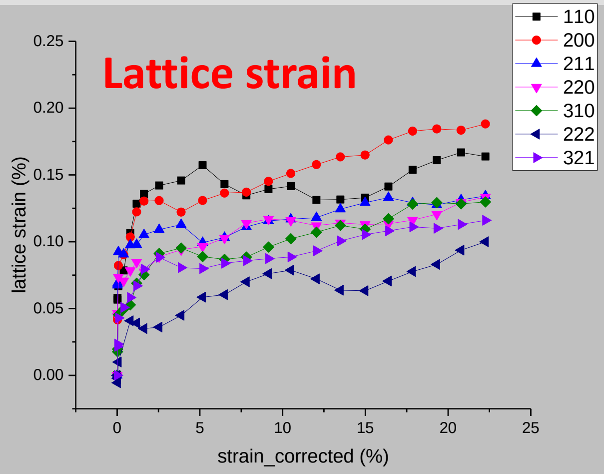
**Lattice constant**

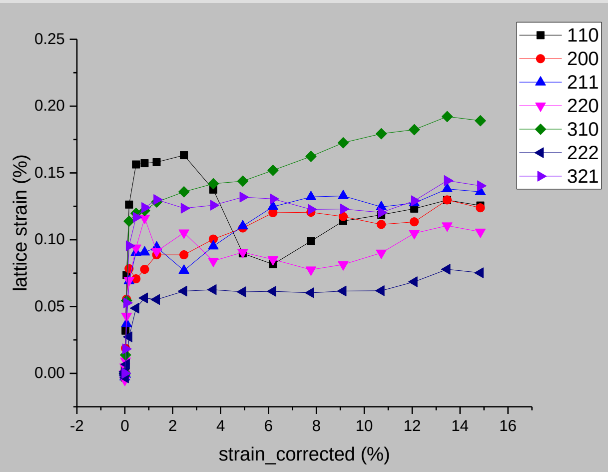

**Fe-9Cr unirradiated**

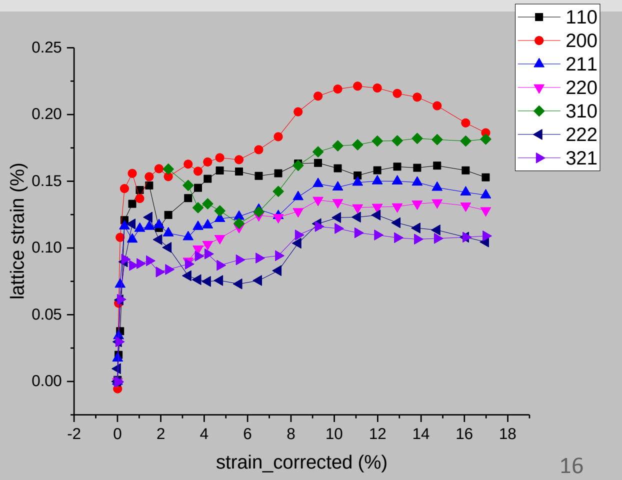

**Fe-9Cr 300ºC, 0.01 dpa**


**Fe-9Cr 450ºC, 0.01 dpa**

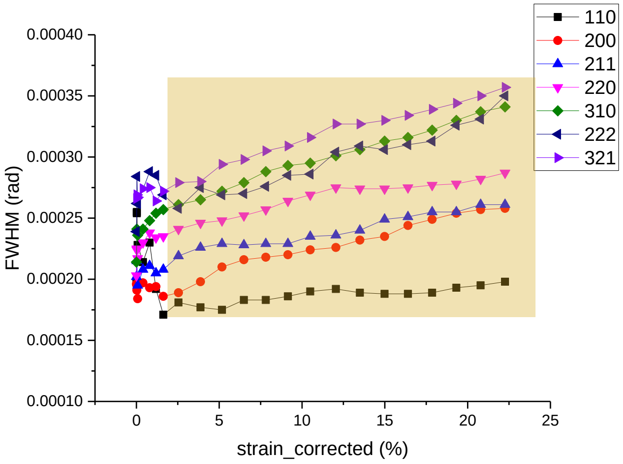
**Lattice strain**

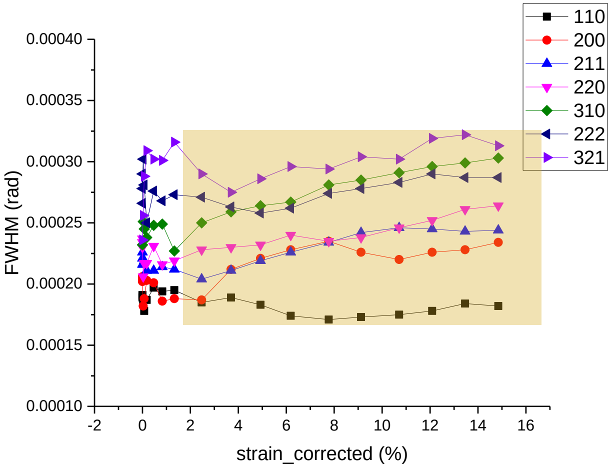


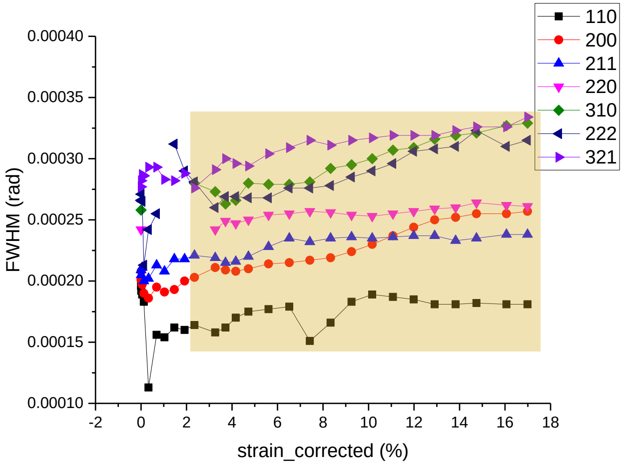




# Peak Broadening during Tensile Deformation



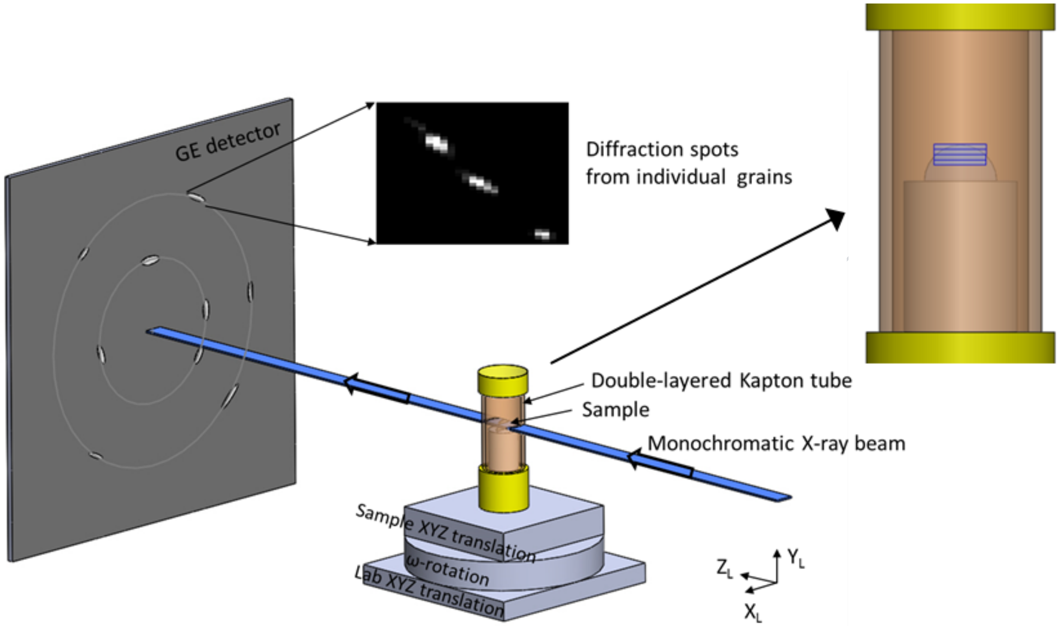




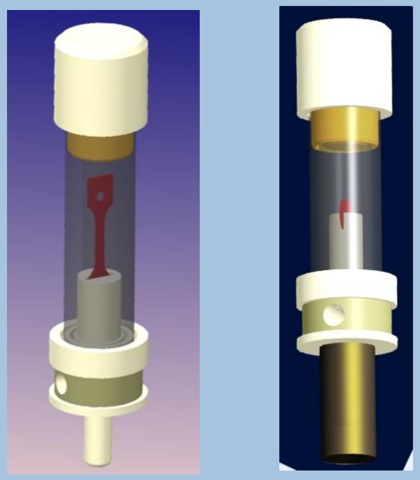
- **Peak broadening data are being analyzed to obtain dislocation density and dislocation structure as a function of strain.**
- **Small-angle X-ray scattering data are to be analyzed.**

# *Ex situ* 3D Characterization of Irradiated Specimens

## Far-field High-Energy Diffraction Microscopy (ff-HEDM)

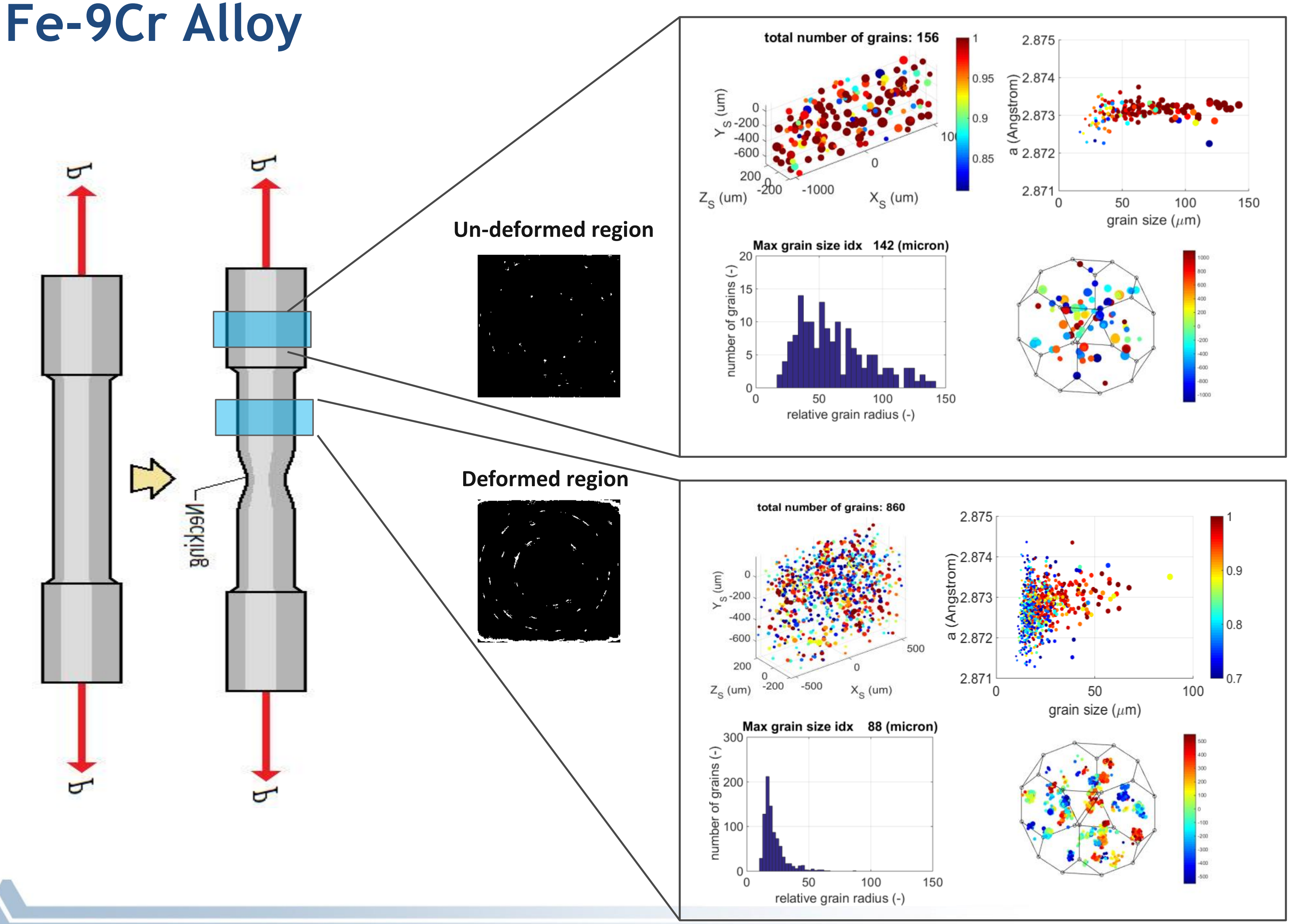


**X-ray Energy: E=70keV**
**Beam size=2x0.2mm$^2$**
**4 layers measured**



Specimen holder for encapsulated tensile specimen (left) and for encapsulated TEM specimen (right)

# ff-HEDM on Deformed, 300$^o$C/0.01 dpa n-irradiated Fe-9Cr Alloy

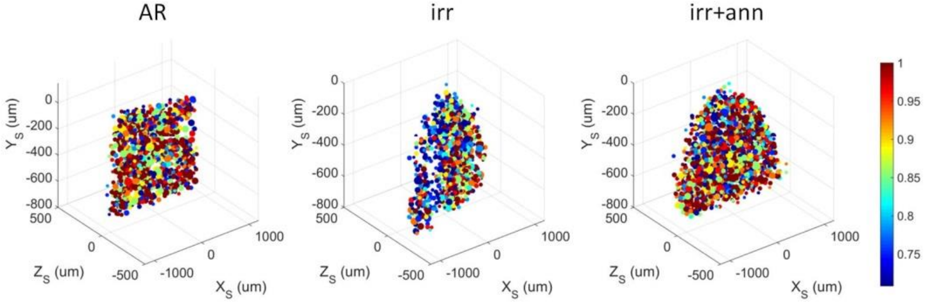


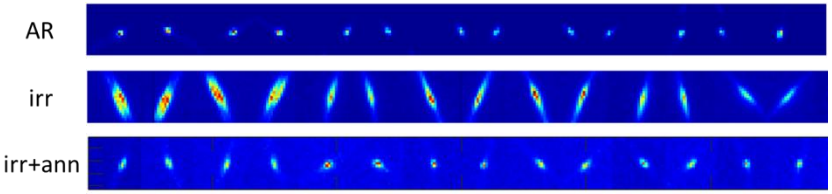
**Un-deformed region**

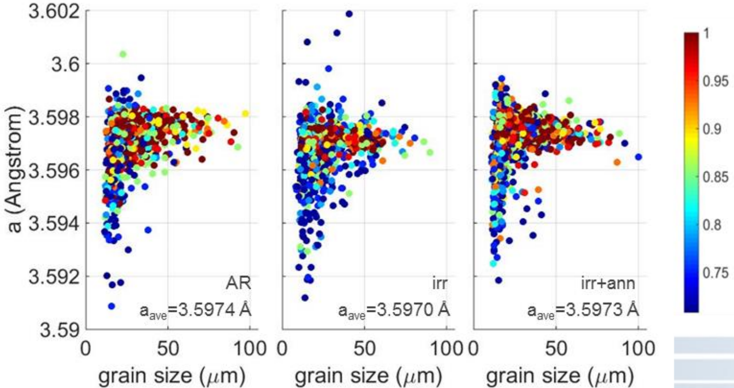
**Deformed region**

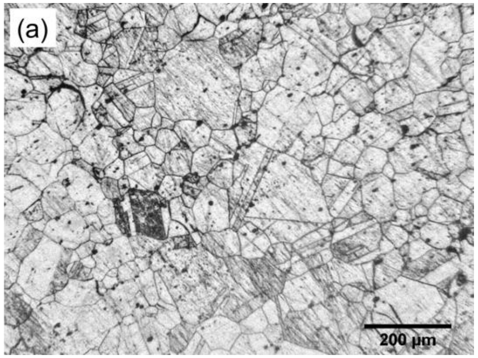
# ff-HEDM of Neutron-Irradiated HT-UPS Austenitic Steel

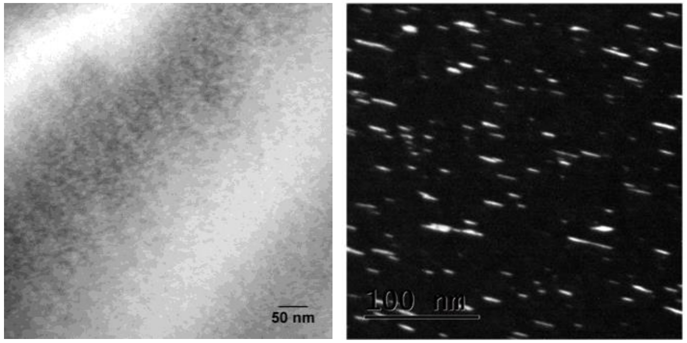
| Sample | Condition | Dimensions |
|---|---|---|
| AR | As-received. | 1.7 ×2 ×0.15 mm |
| irr | 3 dpa, 500ºC | 3 mm dia × 0.16 mm t |
| irr+ann | 3 dpa, 500ºC + annealing at 600ºC for 1 h | 3 mm dia × 0.2 mm t |

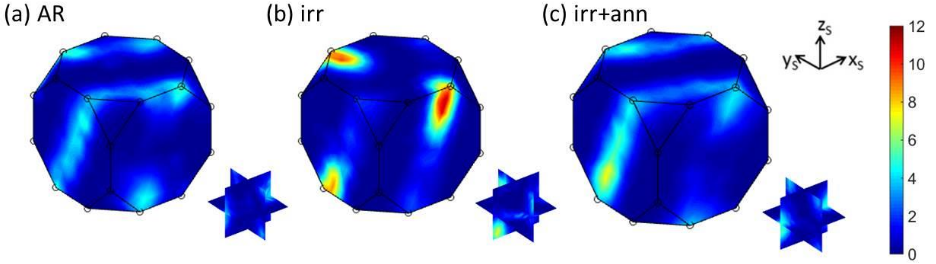






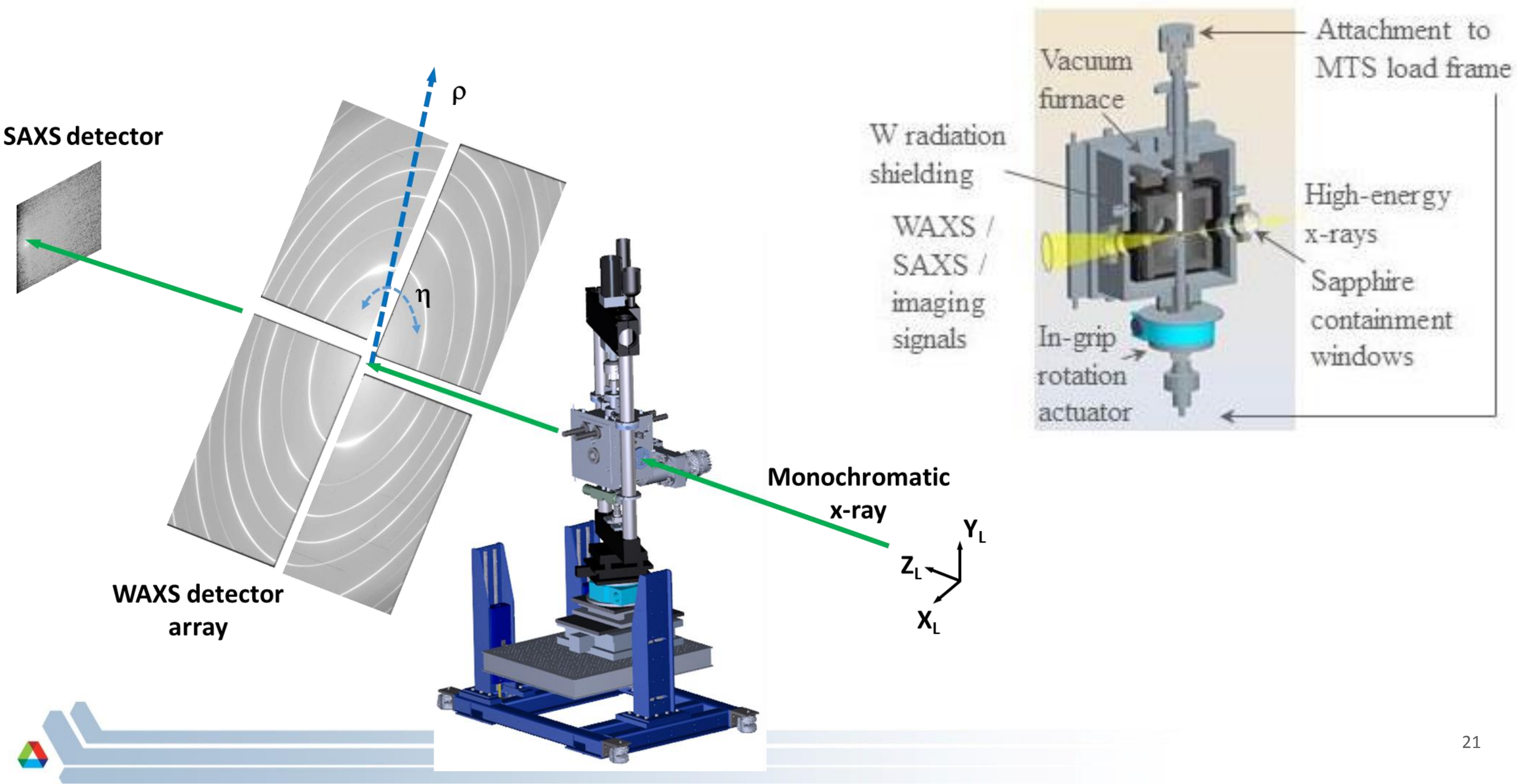




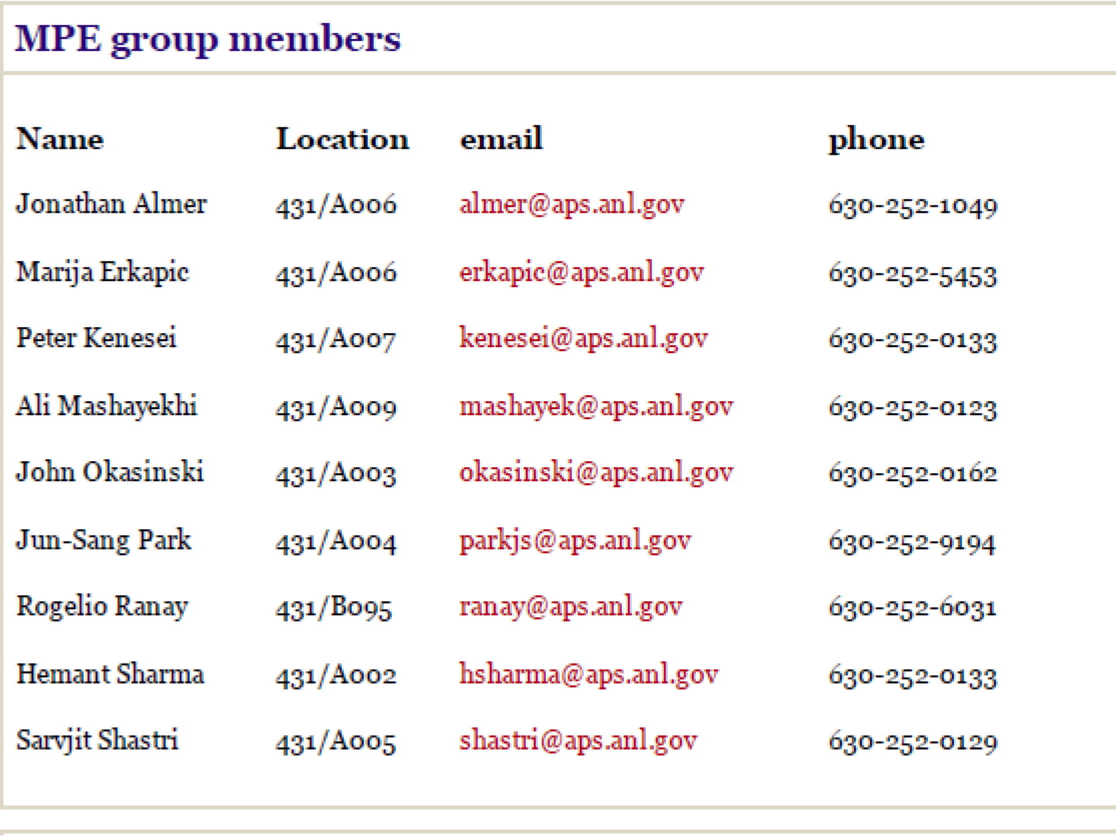


# Outlook – *in situ* 4D Characterization

- Integrate *in situ* straining/annealing capability with 3D characterization techniques for 4D (time- and spatial-resolved) characterization of neutron-irradiated specimens under thermal-mechanical loading.



# Special Thanks to APS Beamline 1-ID



## MPE group members

| Name | Location | email | phone |
|---|---|---|---|
| Jonathan Almer | 431/A006 | almer@aps.anl.gov | 630-252-1049 |
| Marija Erkapic | 431/A006 | erkapic@aps.anl.gov | 630-252-5453 |
| Peter Kenesei | 431/A007 | kenesei@aps.anl.gov | 630-252-0133 |
| Ali Mashayekhi | 431/A009 | mashayek@aps.anl.gov | 630-252-0123 |
| John Okasinski | 431/A003 | okasinski@aps.anl.gov | 630-252-0162 |
| Jun-Sang Park | 431/A004 | parkjs@aps.anl.gov | 630-252-9194 |
| Rogelio Ranay | 431/B095 | ranay@aps.anl.gov | 630-252-6031 |
| Hemant Sharma | 431/A002 | hsharma@aps.anl.gov | 630-252-0133 |
| Sarvjit Shastri | 431/A005 | shastri@aps.anl.gov | 630-252-0129 |