# *In situ* X-ray Study of Materials in Nuclear Environments: The Proposed XMAT Beamline

Meimei Li (ANL/NE)
On behalf of the XMAT Team

APS/Users Monthly Meeting
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# XMAT Team

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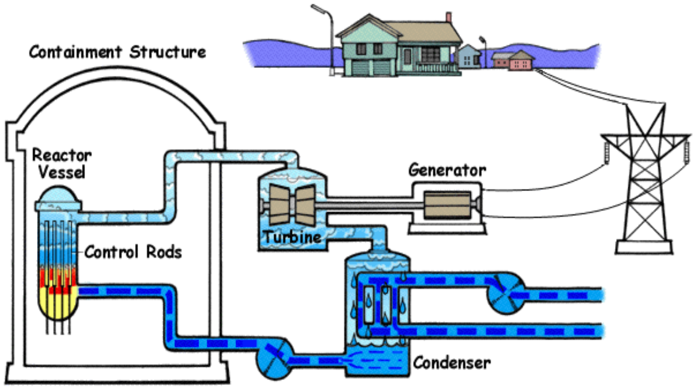
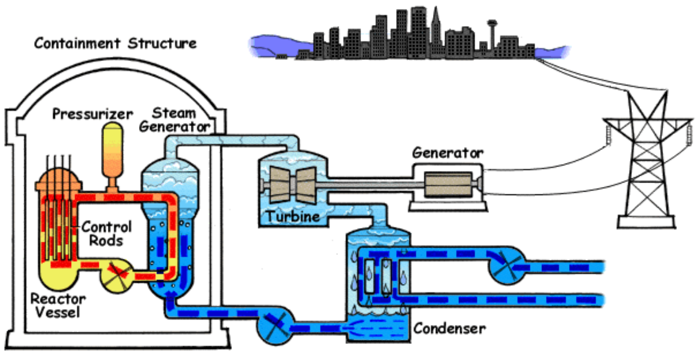
# Overview of Nuclear Energy Systems

## Current Nuclear Power Plants

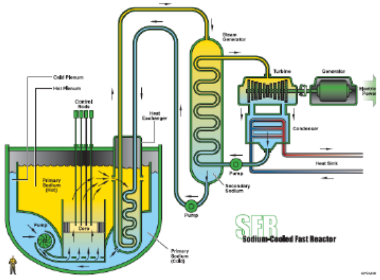


*Boiling Water Reactor (BWR)*

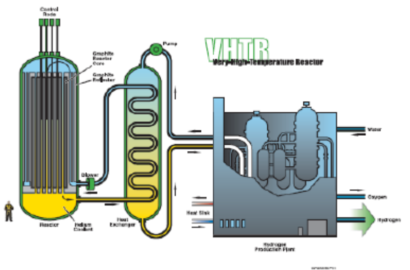


*Pressurized Water Reactor (PWR)*

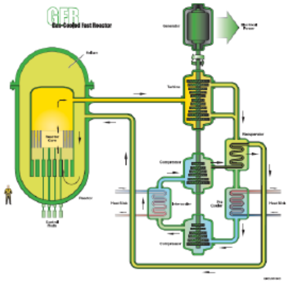
## Advanced Reactor Concepts



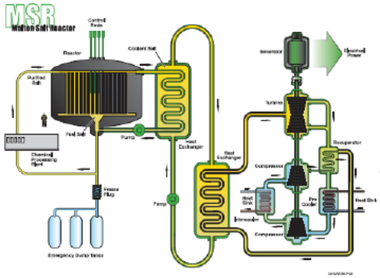
**Sodium-cooled fast reactor**



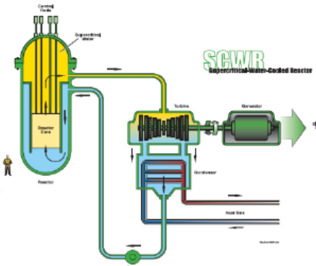
**Very high temperature reactor**



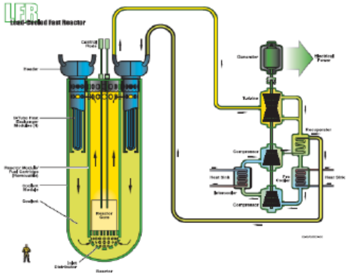
**Gas-cooled fast reactor**



**Molten salt reactor**

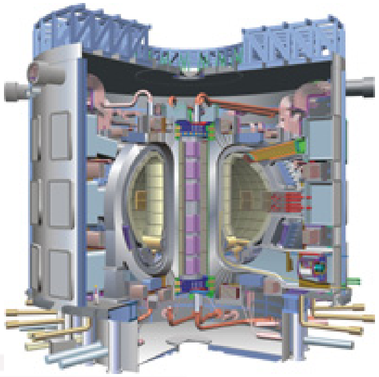


**Supercritical water reactor**



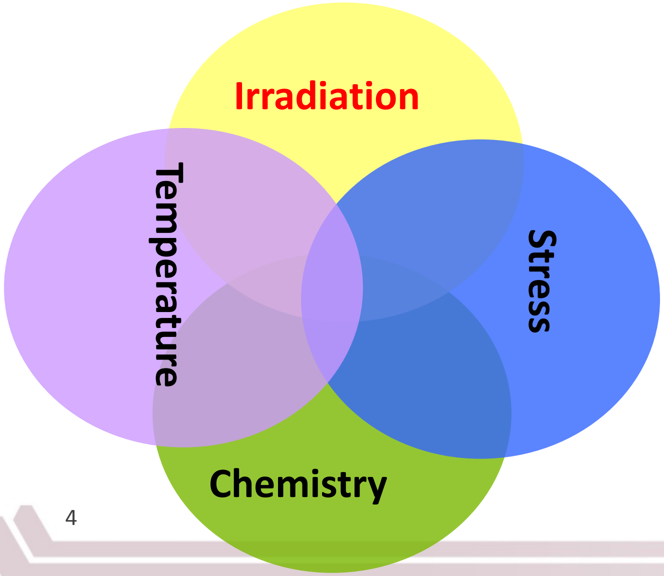
**Lead-cooled fast reactor**

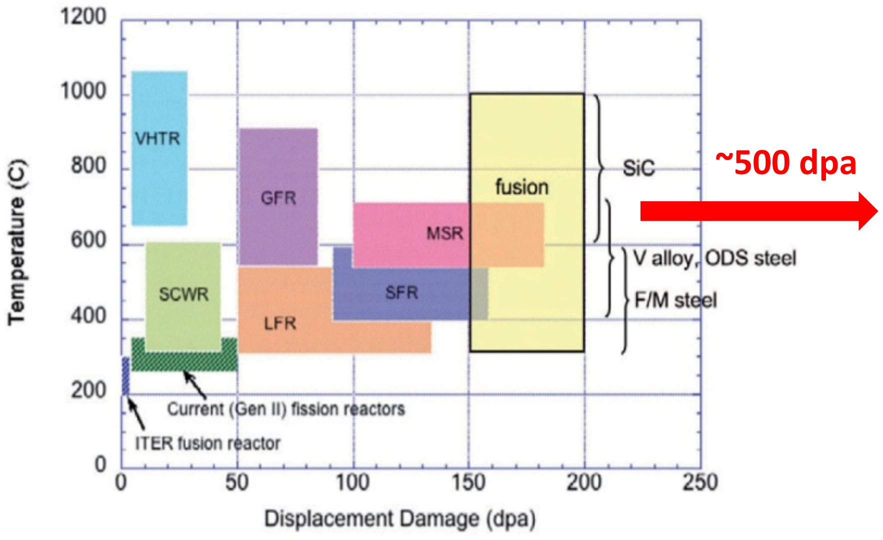
## Fusion Reactors



# Nuclear Reactor Environments

| Reactor Type | Coolant | Temperature (°C) | Pressure | Lifetime irradiation damage (dpa – displacement per atom) | Transmutation |
|---|---|---|---|---|---|
| **PWR, BWR** | Water | 200-300 | 6-15 MPa | up to 80 dpa | ~0.1 appm He |
| **LFR** | Pb or LBE | 550 – 800 | ~ 1 MPa | 150 - 200 | 3-10 appm He |
| **SFR** | Sodium | 550 | ~ 1 MPa | 150 – 200 | 3-10 appm He |
| **GFR** | Helium | 850 | - | 60 | 3-10 appm He |
| **VHTR, NGNP** | Helium | 700 – 1000 | 20 MPa | ~10 | ~0.1 appm He |
| **SCWR** | Water | 510 | 25 MPa | 10 – 30 | ~0.1 appm He |
| **MSR** | Molten salt | 700 – 850 | 1 MPa | 100 – 150 | ~0.1 appm He |
| **Fusion** | Li/Pb alloy | 300-1000 | 50 MPa | ~150 (DEMO) | ~1500 appm He max |



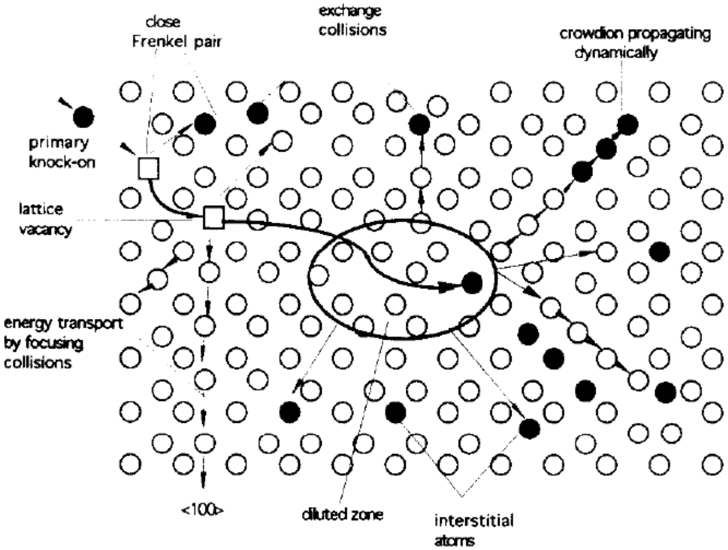


*Zinkle and Busby (2009)*

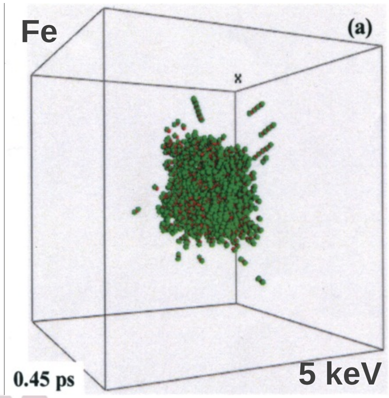
# Radiation Damage

## Atomic Displacement Cascades

**Radiation damage unit:**
**dpa (displacements per atom)**

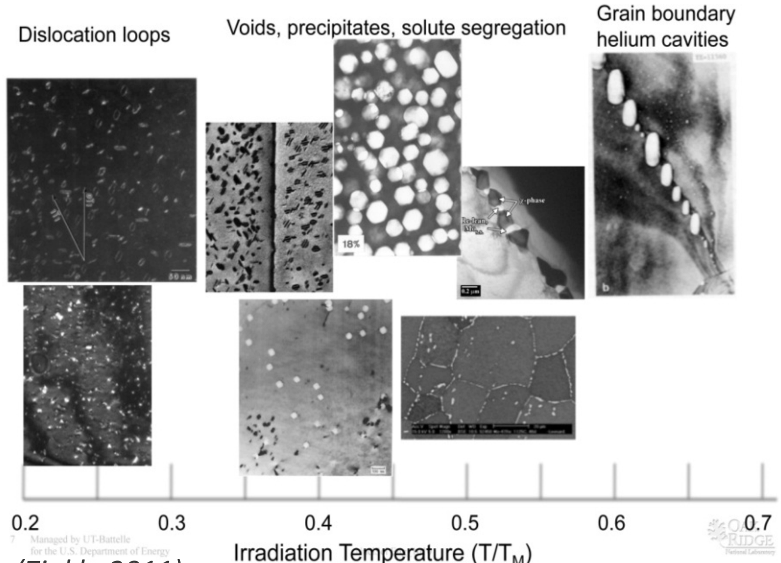


A schematic of a cascade (Seeger 1958)



Molecular dynamic simulation of displacement cascade: Peak damage < 1 ps; (Stoller)

## Radiation-induced defects

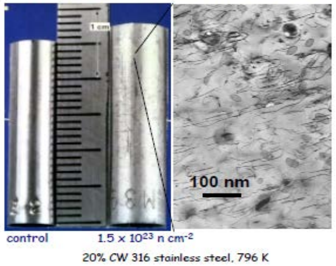


(Zinkle 2011)

## Radiation-induced degradation

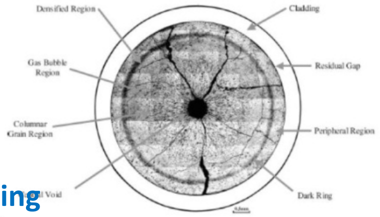
- Radiation embrittlement
- Void swelling
- Irradiation creep
- Irradiation-assisted stress corrosion cracking
- High temperature helium embrittlement

*Neutron Damage on Stainless Steel*

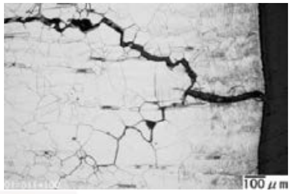


**Void swelling**

(*Straalsund et al, 1982*).



**Fuel cracking**



**Stress Corrosion Cracking**

(*T. Shoji, 11th Env. Deg. Conf.*)

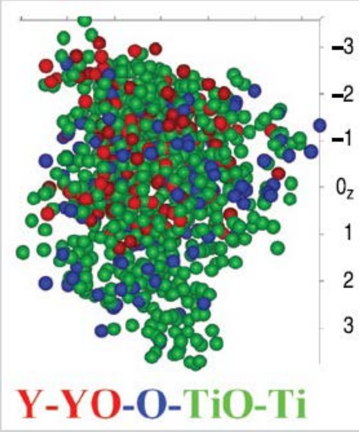
# Control Radiation Damage

- **Interfaces (grain boundaries, particle interfaces, etc.) provide defect absorption and recombination centers, enhancing radiation resistance.**

## *Roles of nano-sized particles*

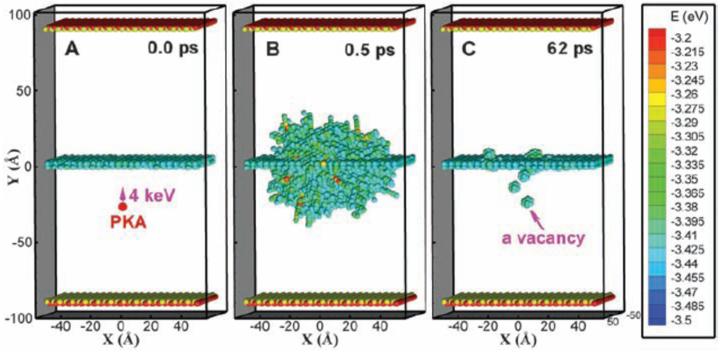
High density, very fine dispersion of Y-Ti-O particles in steels may provide better radiation resistance due to a high volume fraction of particle-matrix interfaces

*(Odette et al 2008, Odette and Hoelzer 2010)*



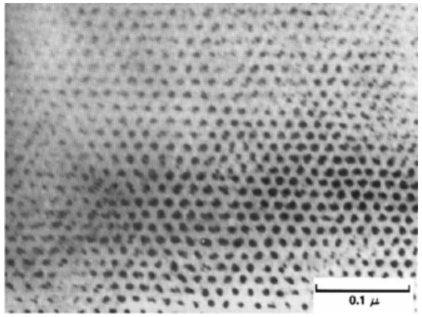
## *Effects of grain boundaries*

Molecular dynamic simulations showed a “self-healing” effect due to presence of grain boundaries (Bai et al. 2010)



## *Radiation-induced self-organization*

Irradiation-induced defects can self-organize into ordered nanostructure, e.g. void lattice in irradiated Mo (Evans et al 1972).



# Hard X-rays Critical to Applied Research

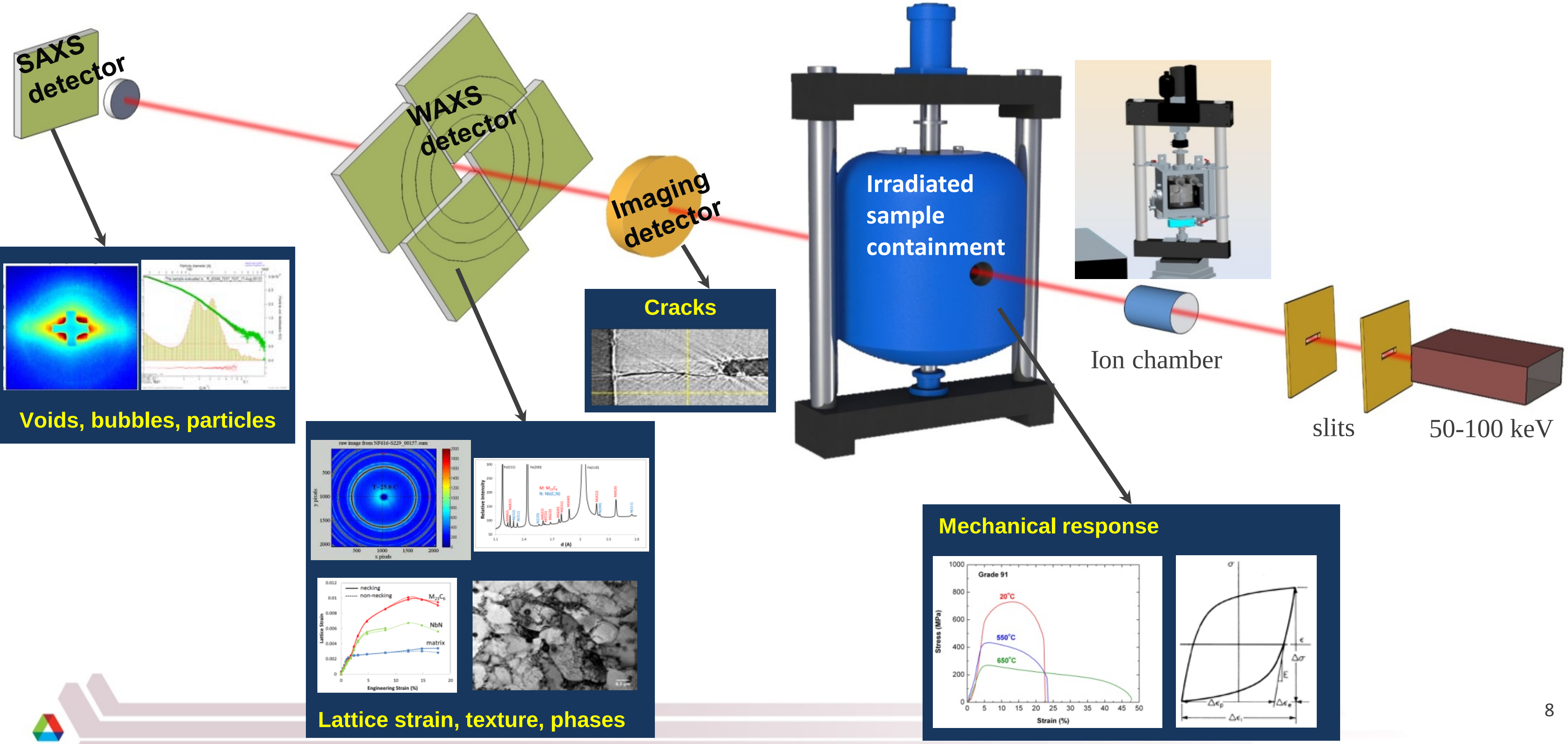
## - Real material, real environment, real time

- Deep penetration
  - Bulk behavior
  - Environmental chambers
- Spatially resolved (inhomogeneity)
  - Resolve complex structures
  - Direct comparison with simulations on same length scales
- In situ, real-time studies
  - Dynamics
  - structrual evolution
- Ideal for complex engineering materials
  - Deformation and failure mechanisms
  - Phase-specific
  - Chemistry-specific
  - Multiple probes for concurrent, multi-scale characterization

# Characterization of Neutron-irradiated Materials – Concurrent, Multi-scale, and Real-time

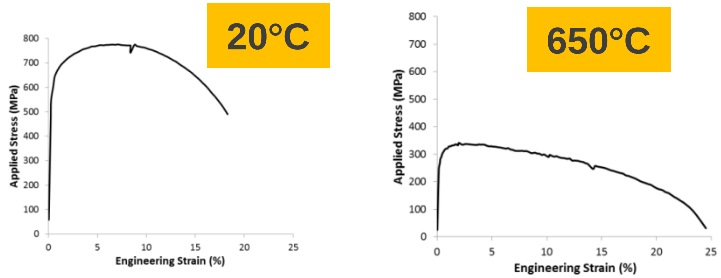
*M. Li (NE), J. Almer (XSD), E. Benda (AES), Y. Chen (NE), A. Mashayekhi (XSD), K. Natesan (NE), D. Singh (NE), L. Wang (NE), F. Westferro (AES)*

Combination of multiple probes
(WAXS/SAXS/imaging + 3D tomography)

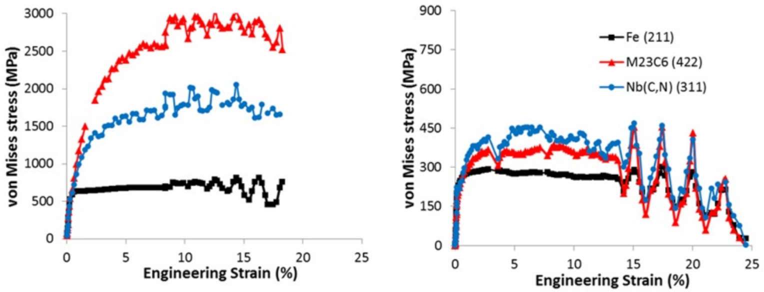


# Deformation and Fracture Mechanisms in Ferritic-Martensitic Steel

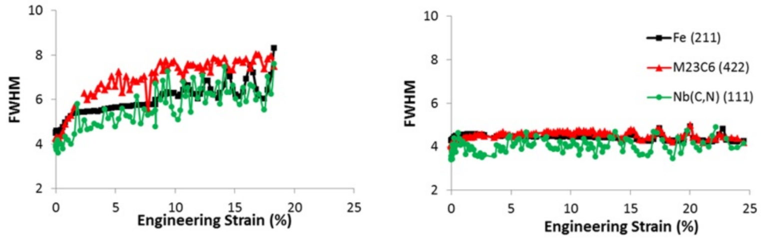
*Meimei Li (NE/ANL), Leyun Wang (NE/ANL), and Jon Almer (XSD/ANL)*



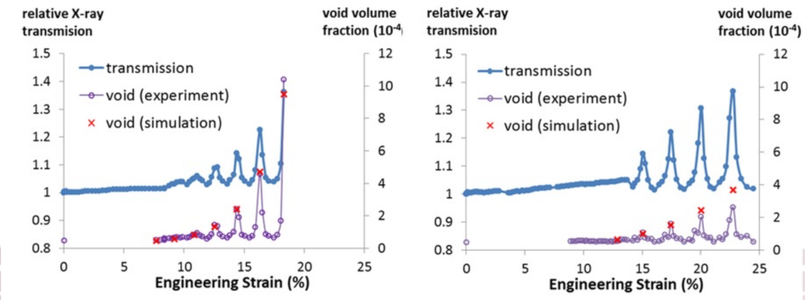
**Macroscopic stress-strain measurements showed significant strength reduction at high temperature.**



**Changes in strengthening mechanisms of $M_{23}C_6$ and MX precipitates resulted in strength reduction.**



**Peak broadening showed strain hardening at 20°C, and sharpening showed strain softening at 650°C.**



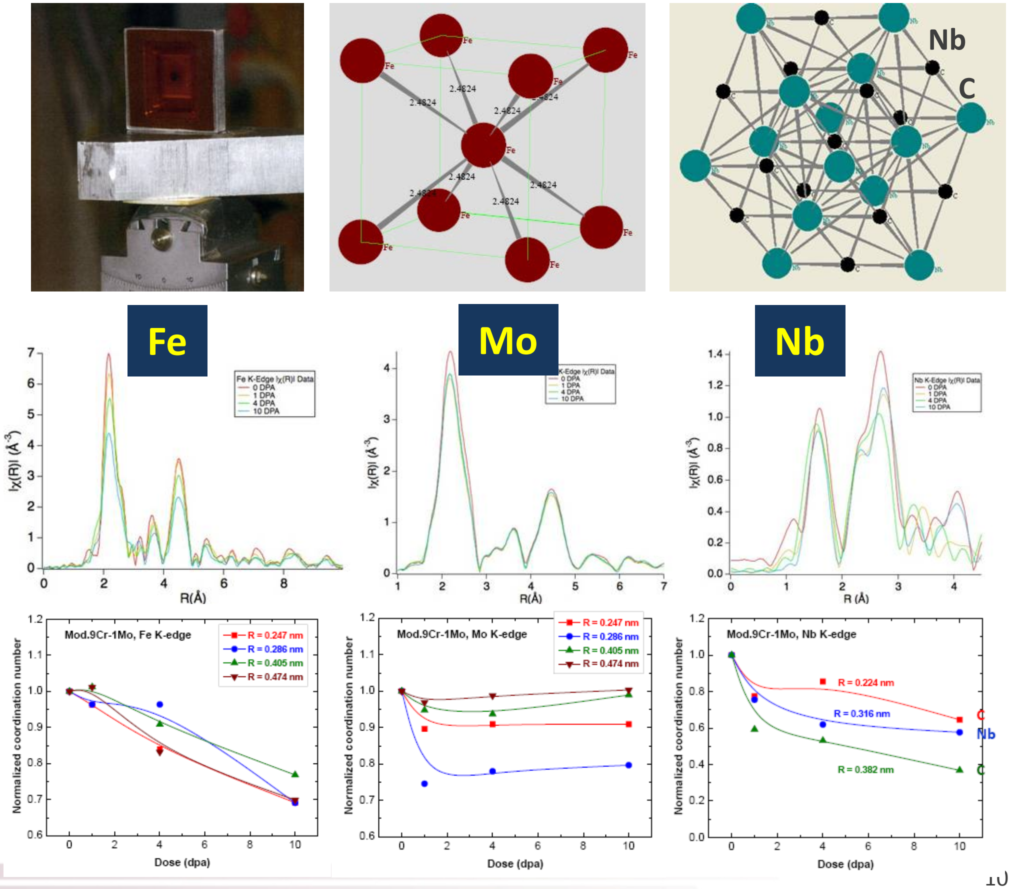
**Significant void formation and growth led to specimen failure.**

# Chemistry-specific study of radiation defects

*Meimei Li (ANL), Jeff Terry (APS MRCAT/IIT), Stuart A. Maloy (LANL)*

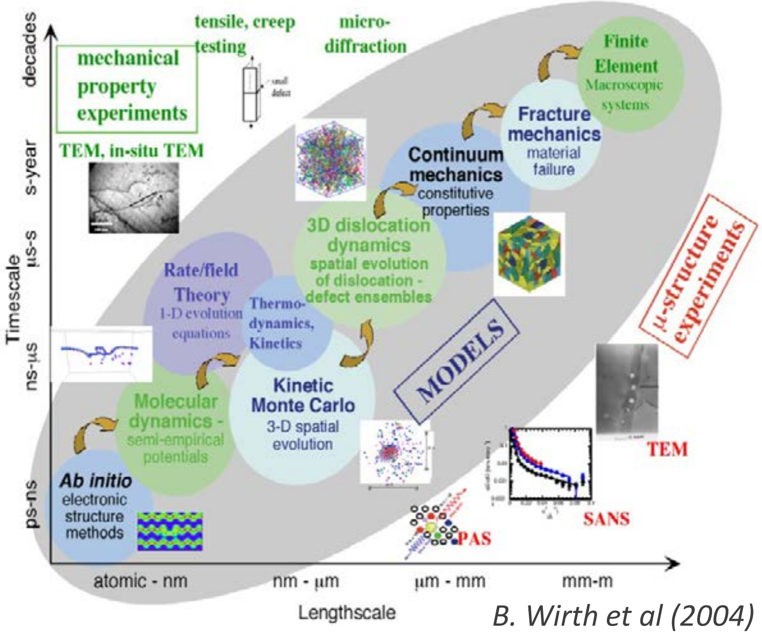
Synchrotron extended x-ray absorption fine structure (EXAFS) technique allows detection of defect interactions with each individual alloying element in irradiated steels at the atomic level, providing new insight into the design of radiation tolerant materials.

- Detect irradiation defects at the atomic level – local changes of atomic environments within 6 Angstroms
- Element-specific studies – defects associated with each alloying element
- Useful in multi-component complex engineering alloys
- Findings are important in understanding the roles of alloying in radiation-induced segregation and void swelling.



# Why *in situ*?

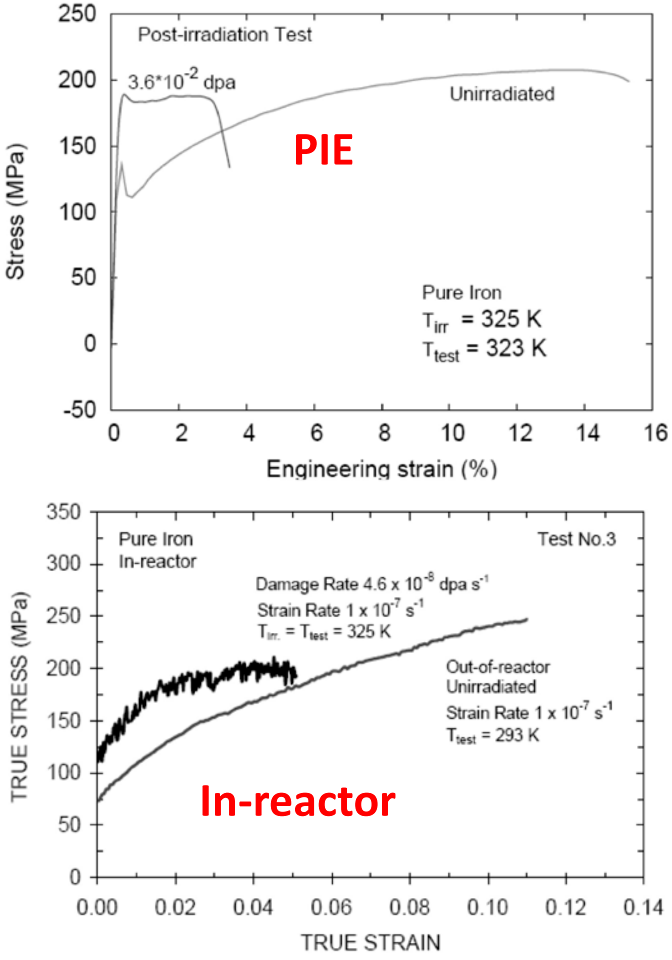
**Irradiation produces atomic defects at picoseconds that impact properties of materials and fuels for many years.**



*B. Wirth et al (2004)*

**Many competing processes are directly affected by radiation field, temperature, stress, and environment.**

**Post-irradiation examination (PIE) vs. In-reactor experiment**



*B. N. Singh (2007).*

# IVEM – *in situ* Ion Irradiation Facility at ANL

- Real-time observation of defect formation and evolution during irradiation
- High doses (e.g. 100 dpa) can be achieved in hours; irradiation dose rates can be varied over several orders of magnitude
- Well-controlled conditions (temperature, ion, ion energy, dose rate, dose)
- Studies of single-parameter effects and synergistic effects of irradiation, temperature and stress
- A wide range of techniques including imaging, electron diffraction, and spectroscopy

## ARGONNE'S CORE CAPABILITIES

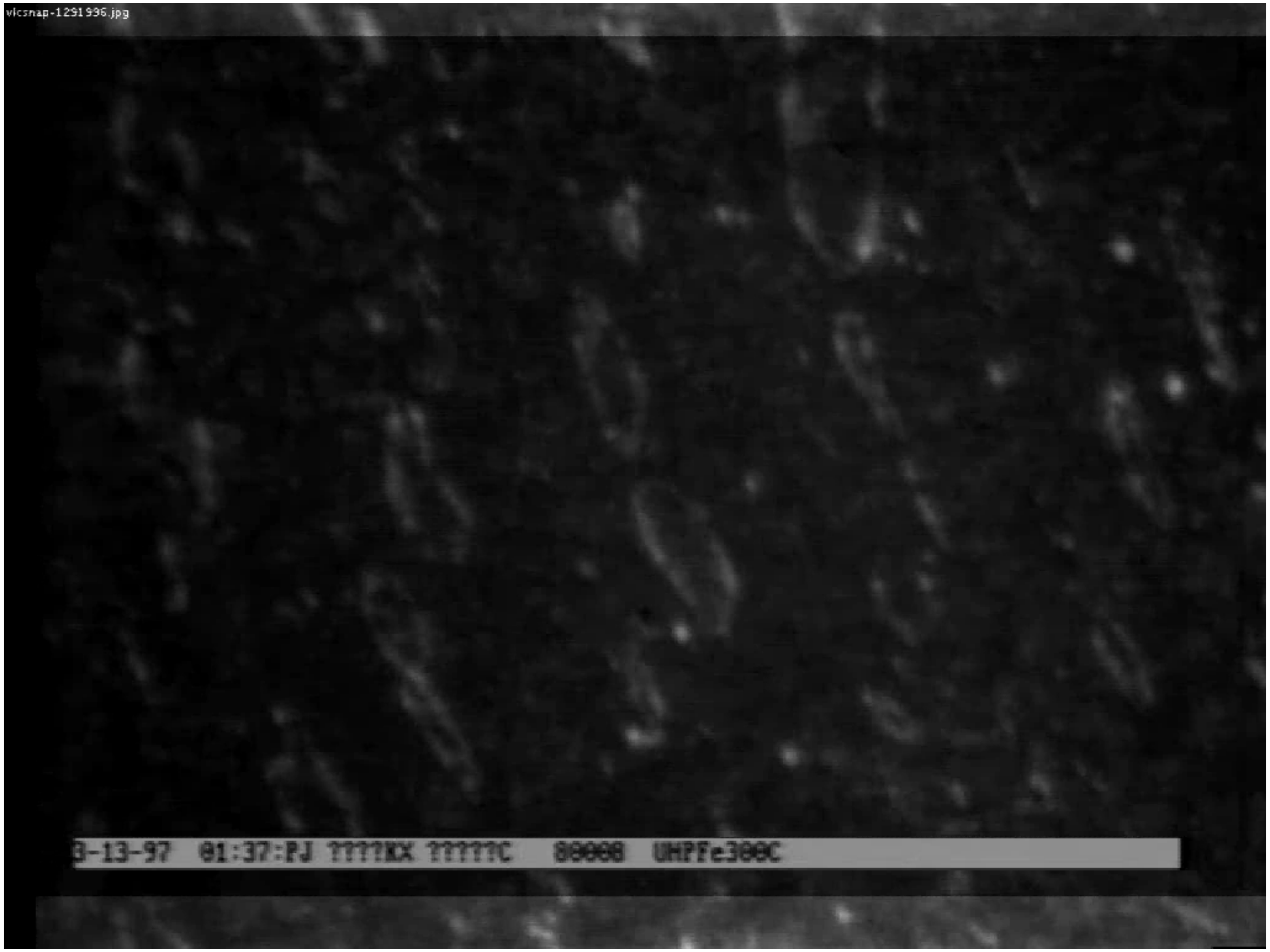
The stewardship of Argonne's core capabilities — its user facilities, fundamental science, and applied science and engineering programs — remains at the top of the Laboratory's strategic agenda. None of the great discovery- or use-inspired research performed at Argonne is possible without a substantial combination of people, facilities, and equipment having unique or world-leading components. The core capabilities are described briefly here and plans for advancing them to address the grand scientific challenges of the future are discussed.

Scientist Mark Kirk works with the IVEM-Tandem, one of the unique instruments in the Electron Microscopy Center. The instrument consists of a 300 kV transmission electron microscope interfaced to two ion accelerators, allowing *in-situ* real-time observation of ion beam modification and associated effects in materials to ~ 0.25 nanometer resolution.

22

# Study Irradiation Defect Kinetics

*Z. Yao (Queen's U), M. Hernandez-Mayoral (CIEMAT), M. L. Jenkins (U. Oxford), M. A. Kirk (ANL)*

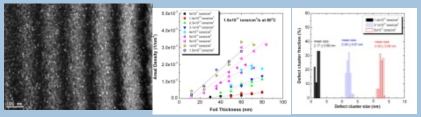


# Predict Neutron Damage using Ion Damage Data

*Meimei Li (NE/ANL), Mark Kirk (MSD/ANL), Donghua Xu, Brian Wirth (U. Tennessee)*

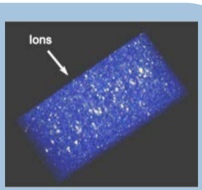
## *In situ* Ion Irradiation Experiments

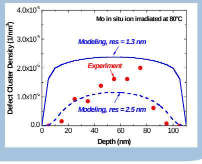
Well-controlled TEM with *in situ* ion irradiation experiments of thin films were designed to improve and validate computer models. Experimental data provide a complete set of high-quality, quantitative information, and described the defect behavior at a level of detail unavailable before.



## Experiment-Simulation Comparison

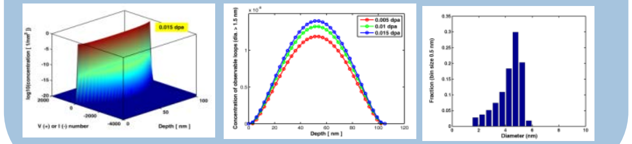
Quantitative, absolute comparisons between experiments and modeling at the same spatial and time scales have led to the establishment of accurate, reliable computer models.





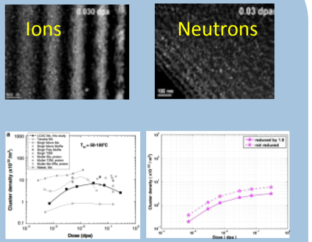
## Computer Simulations

Multiscale modeling to simulate defect evolution from atomic-scale, pico-second events to nanometer-scale, hour evolution of defect structures.



## Prediction of Neutron Damage in Reactors

The experimentally-validated model for ion irradiated thin foils is used to predict neutron damage in Mo irradiated in a reactor, and validated by neutron irradiation data



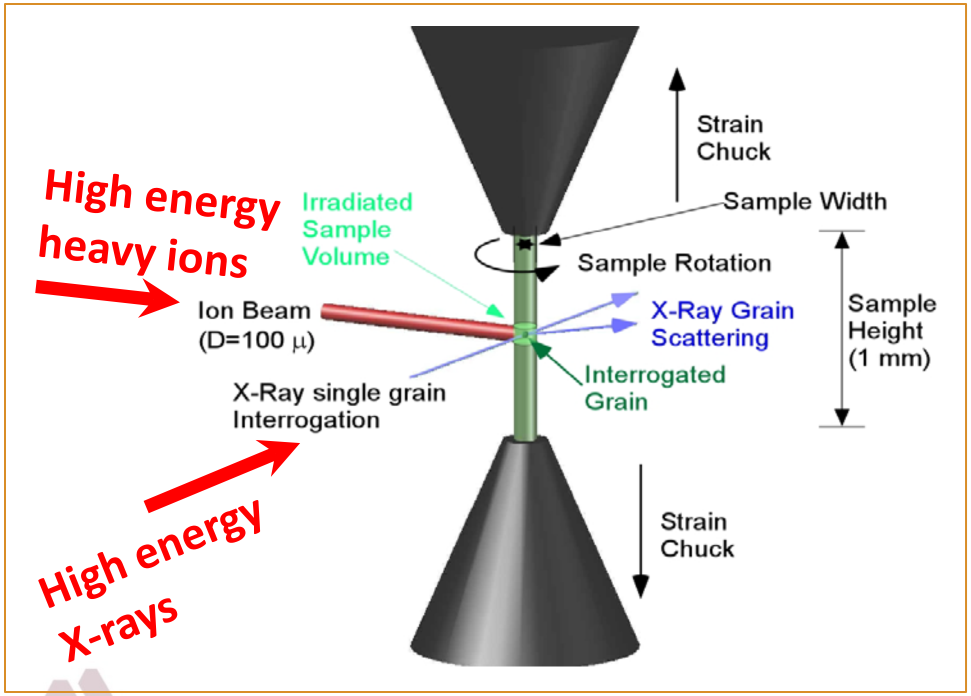
**Neutron experiment**

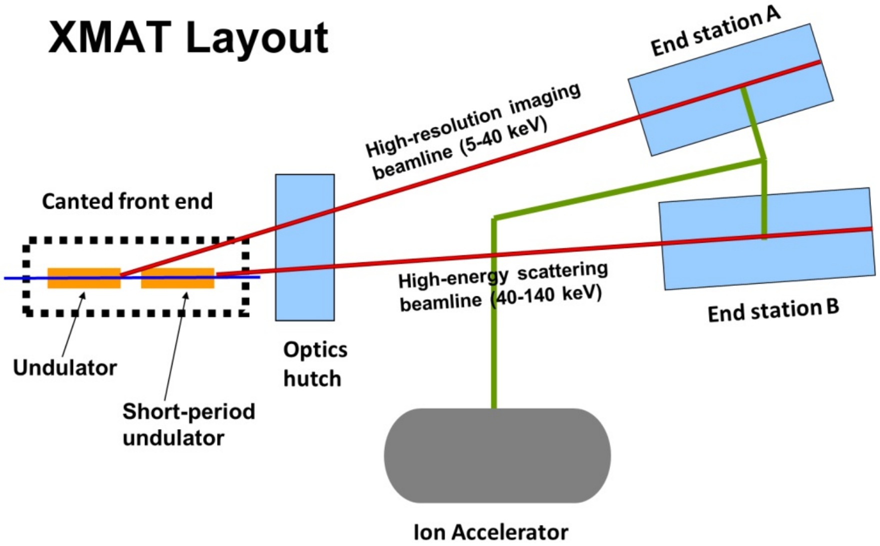
**Model**

# Proposal - eXtreme MATerials beamline (XMAT)

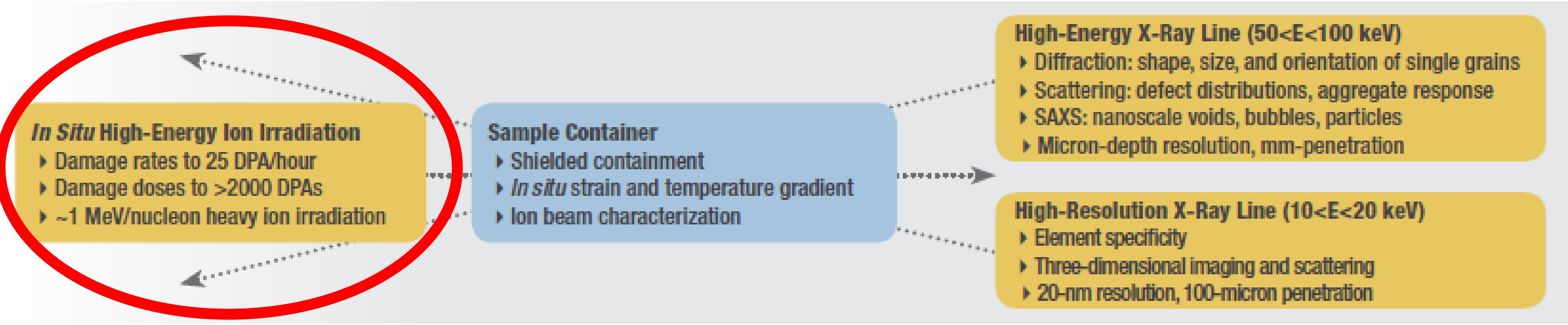
A new beamline at APS for *in situ* studies of nuclear energy materials under irradiation, temperature,  stress, and environment.

- XMAT will provide multiple x-ray probes for *in-situ* study of materials in simulated nuclear reactor environments, enabling rapid evaluation of new materials and fuels performance under extreme service conditions including for the first time nuclear fuels as well as structural materials.





# What's Unique? – High Energy, Heavy Ion Irradiation

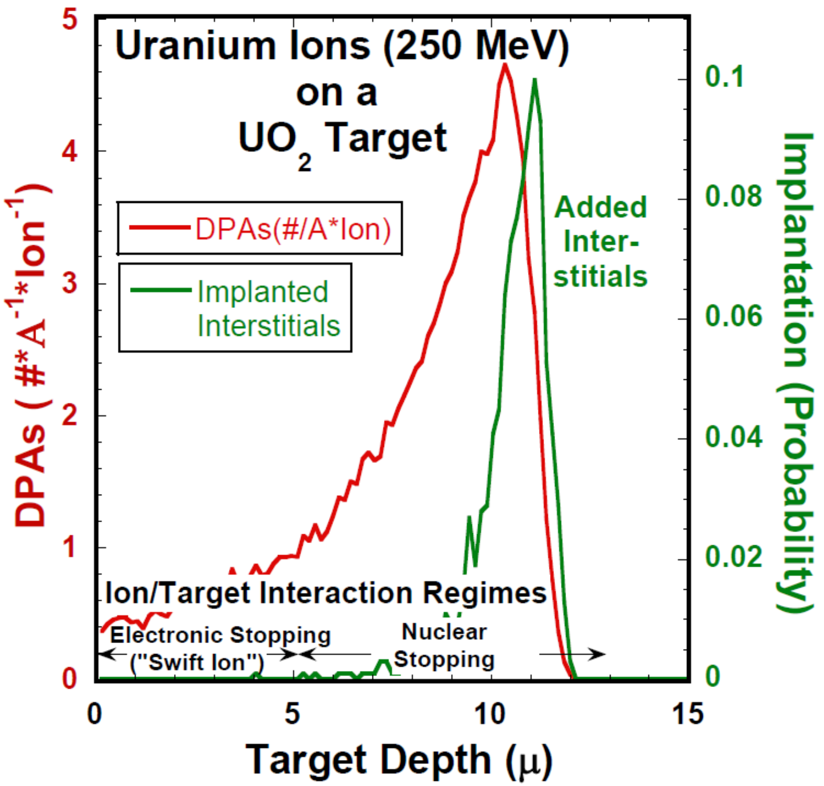


***In Situ* High-Energy Ion Irradiation**
- Damage rates to 25 DPA/hour
- Damage doses to >2000 DPAs
- ~1 MeV/nucleon heavy ion irradiation

**Sample Container**
- Shielded containment
- *In situ* strain and temperature gradient
- Ion beam characterization

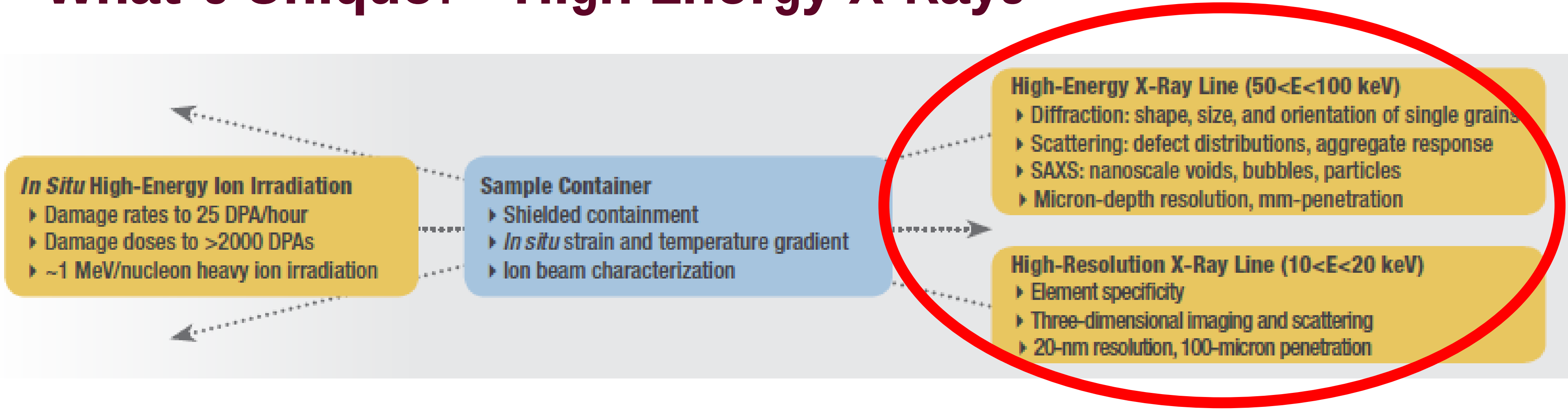
**High-Energy X-Ray Line (50<E<100 keV)**
- Diffraction: shape, size, and orientation of single grains
- Scattering: defect distributions, aggregate response
- SAXS: nanoscale voids, bubbles, particles
- Micron-depth resolution, mm-penetration

**High-Resolution X-Ray Line (10<E<20 keV)**
- Element specificity
- Three-dimensional imaging and scattering
- 20-nm resolution, 100-micron penetration



- **High irradiation doses**
- **Heavy ion irradiations create damage close to neutron irradiation**
- **Deeper penetration – "bulk" effect**
- **Fission fragment damage (80-100 MeV)**
- **Transmutation**
  - Transmutation - added Interstitials
  - Additions of H, He ion irradiation
- **Separation of irradiation effects**

# What’s Unique? – High Energy X-Rays



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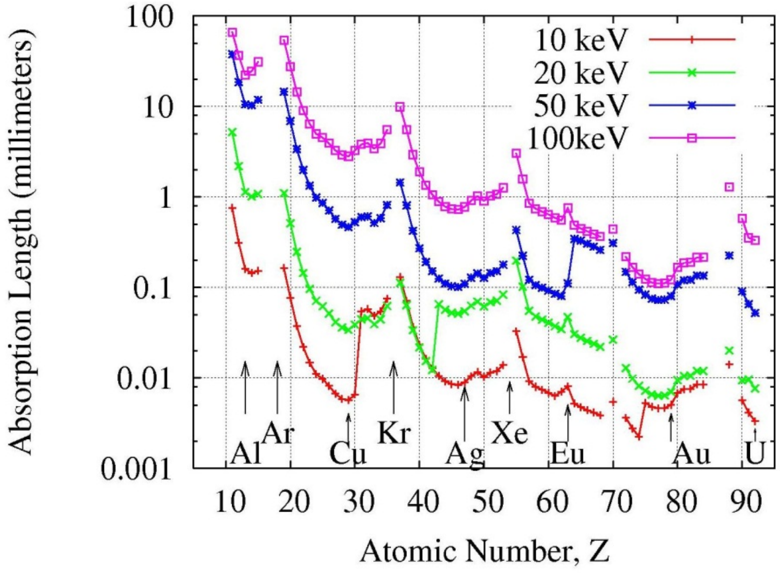
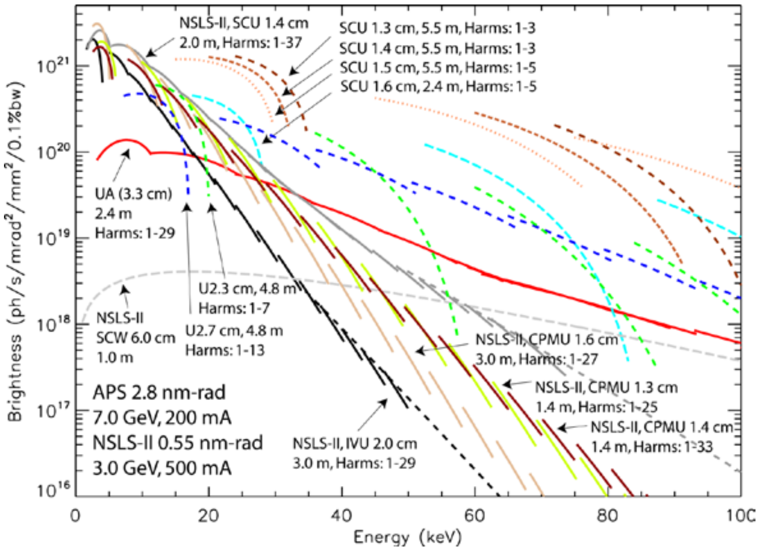


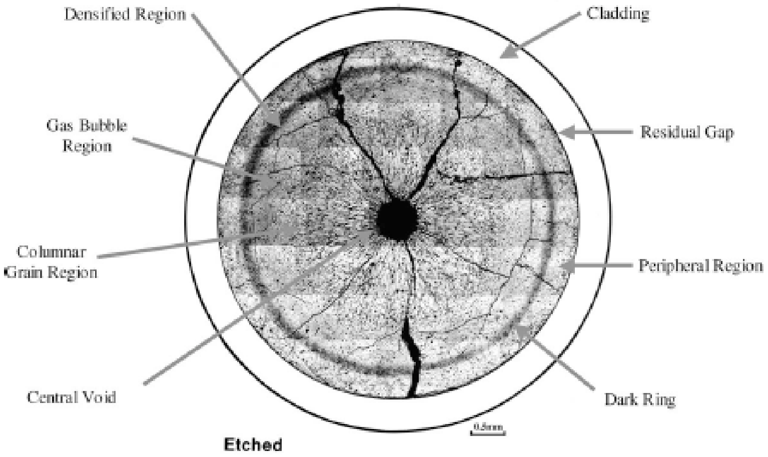


Higher x-ray energy, deeper penetration in a sample – **“bulk” effect of heavy elements (actinides)**.

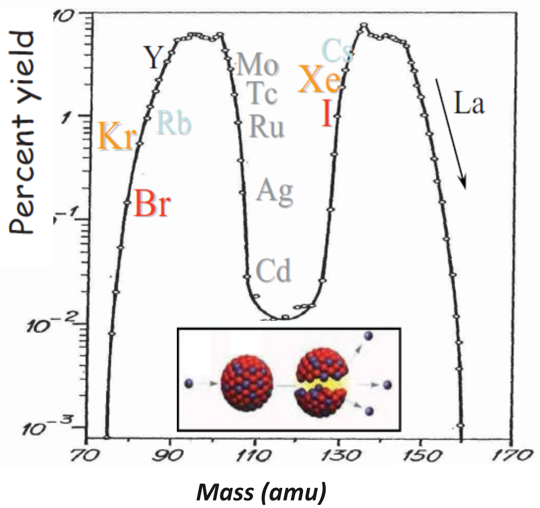
High brilliance, high flux, high resolution.

# Example - Study High Burn-up Nuclear Fuels

- **Fuel is subjected to extremely high radiation damage, ~1 dpa/day in LWRs**
  - XMAT allows high irradiation damage rate, high doses
  - High burn-up -> >2000 DPAs (cladding ~150 DPAs)
- **Fission Damage Effects**
  - XMAT delivers any fragment Ions at fission fragment energies
  - Electronic excitation effects
  - Nuclear stopping (responsible for radiation damage)
  - Added interstitials: fission products, production of transuranium elements
- **Fission bubble formations, thermal gradients in fuels, cracking, etc.**
- **XMAT allows study of each of the unique damage processes that occur in fuels.**

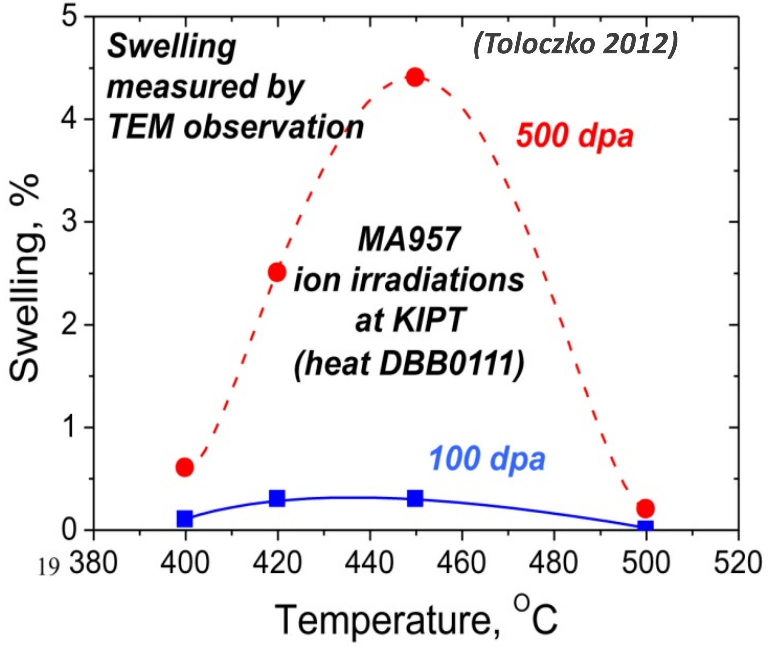


**Rim Effect in $UO_2$ Fuel**



# Example – Study High-dose Irradiation Damage

- **XMAT facility will allow specimens to be irradiated to high doses that are unachievable in a nuclear reactor in a realistic time frame**
  - Nuclear reactors: ~10-30 dpa/year
  - Ion accelerators: up to ~100 dpa/day
- **XMAT is designed to receive low-activity, low-dose neutron-irradiated specimens for high radiation damage dose experiments.**
  - Damage can be “seeded” by initial neutron exposure
  - Pre-neutron irradiation followed by ion irradiation allows defect nucleation and growth to be studied separately.

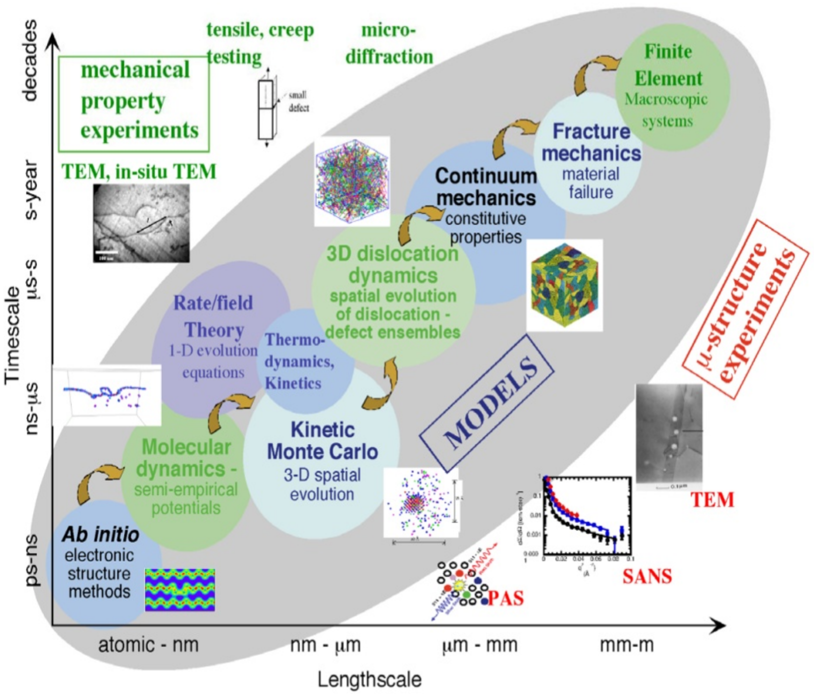


*Void swelling in ODS MA957 steel was drastically increased after irradiation to dose of 500 dpa.*

# Example – Develop Predictive Models

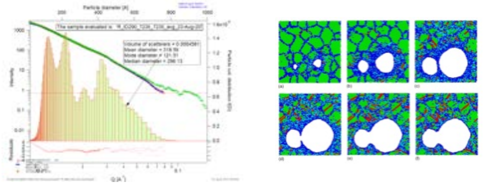
- Traditionally, problems have been approached by sequentially coupled length or time scales. XMAT allows moving toward concurrent multi-scale modeling.

**Combination of techniques envisioned for *in situ* x-ray studies of nuclear fuels and materials in irradiation environments - concurrent, scale bridging, and real-time**

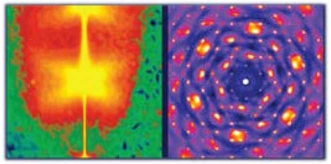


*B. Wirth et al (2004),* Barabash et al (2009), Suter et al (2012)
Dongare et al (2009), Oddershede et al (2010, 2011)

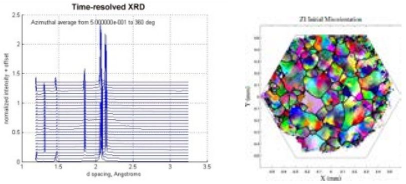
**SAXS:**
**voids, bubbles, precipitates**



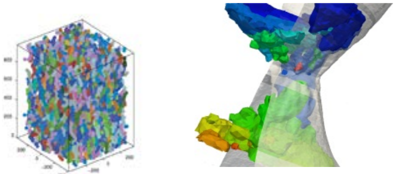
**Diffuse scattering:**
**SIA and vacancy clusters**



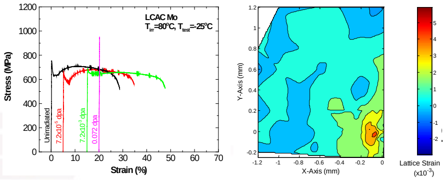
**XRD:**
**Phases, grains, dislocations, lattice strain, texture**



**Tomography:**
**Cracks, voids, grain structure**



**mechanical loading:**
**macroscale stress-strain response**



# Path forward -

- Define and refine beamline concept, including scientific questions of focus, technical aspects of an ion accelerator and x-ray techniques, cost, and schedule.
- Build a core team with a right mix of expertise and representing various groups in the community.
- Engage the nuclear materials and fuels community, and gain broad community support
- Engage all possible funding sources, particularly DOE NE.
- ………………………