NSLS-II Status and Capabilities: Examples of SAXS and XRD Characterization of RPV Steels and Nanostructured Ferritic Alloys

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DOE Cross-cutting Materials Meting

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MATERIALS UC Santa Barbara

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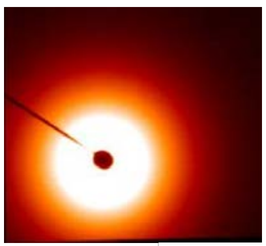
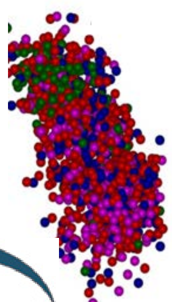
a passion for discovery

U.S. DEPARTMENT OF ENERGY | Office of Science

Presentation Outline





- New partnership between NSLS-II and NSUF
- NSLS-II capabilities including the robot
- Example – small angle X-ray scattering (SAXS) characterization of irradiated reactor pressure vessel steels
- Example – X-ray Diffraction (XRD) studies of RPV and NFA (ODS) Alloys
- This research is a very close and long-term *science* collaboration between BNL and UCSB (NEET) involving frequent scheduled conference calls, exchanges of ideas and participation by UCSB in NSLS-II beamtime and data analysis development
- Data analysis – challenges and opportunities

National Synchrotron Light Source – II - NSUF User Facility

- Formal partnership between NSUF and NSLS-II in August 2016
- Six percent beam time each year for materials for nuclear energy systems at the NSLS-II X-ray Powder Diffraction (XPD) beamline
- NSLS-II is a DOE user facility with support infrastructure, guest center, training, accommodations and established proposal process
- Unique radiation handling capabilities
- ***Scientific support for proposals, experiments, and data analysis***

Obtaining Beamtime at NSLS-II XPD

- NSUF guaranteed time: proposal process outlined in the Consolidated Innovative Nuclear Research FOA

General User Proposal:

- Three submission cycles per year: January 31, May 31, September 31
- Competitive review process based on feasibility and quality of science
- https://www.bnl.gov/ps/
- We can help with process! (lecker@bnl.gov)

My Proposals

The PASS system is the central location for submitting beamtime proposals for NSLS-II. To see a list of beamtime run cycles, deadlines, and available beamlines, see the NSLS-II call for General User proposals.

It is highly recommended that you contact the beamline on which you are planning to run your experiment to ensure:

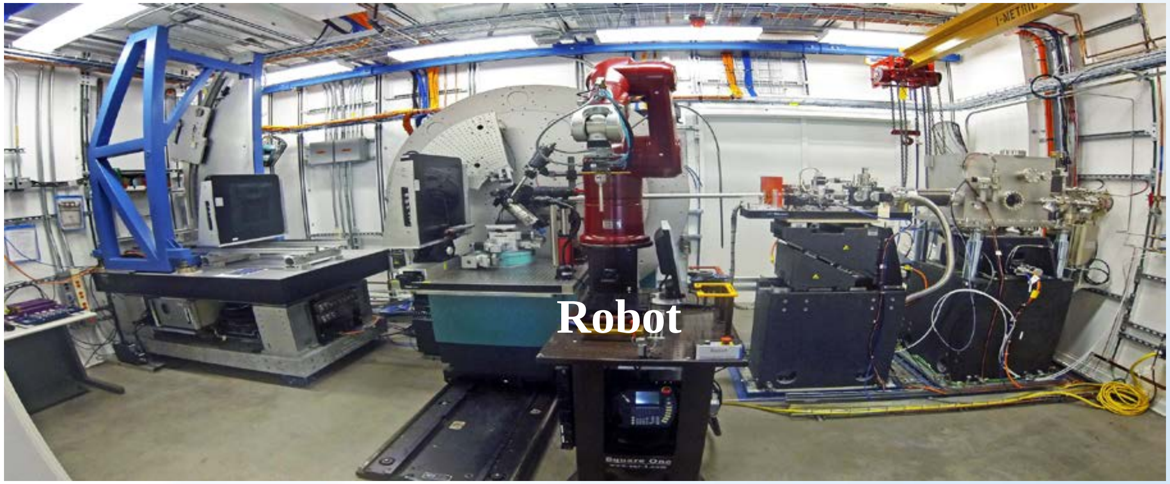
1. That your experiment is feasible
2. The beamline has the current capacity required for your experiment
3. That you are selecting the correct beamline for your experiment

NOTE: After entering your proposal information, make sure you create a time request and use the **SUBMIT** button to complete the submission process.

X-ray Powder Diffraction XPD Beamline

- Quantitative microstructural characterization of heterogeneous, real-world complex material systems
- High-throughput for high energy diffraction and scattering
- Versatile suite of techniques
- In situ experiments made possible by ultra-high x-ray flux

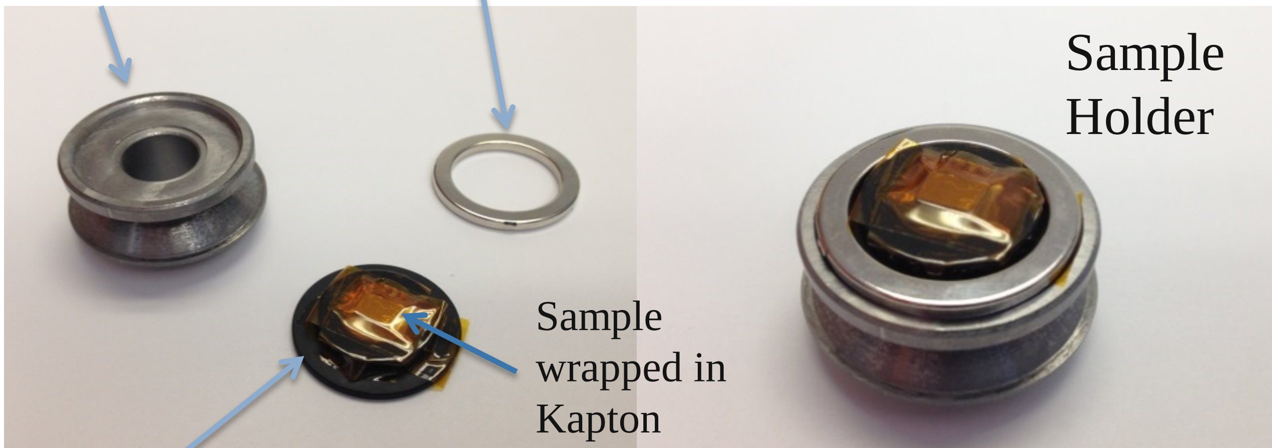
Energy Range: 30-80 keV
Flux: 10^{13} ph/s
Spot Size: mm-µm
Time Resolution: ms
Techniques: Powder diffraction, PDF, SAXS, CT, XRD-CT



XPD Robot

- High throughput at XPD: < 3 min/sample @ 25 mA (<< 500 mA max)
- Mail in access (this is the antithesis of this collaboration)
- Sample holders are bar-coded with sample ID
- Up to 100 mrem/h @ 30 cm dose rates (600mrem/h possible)

Sample Holders and Containment



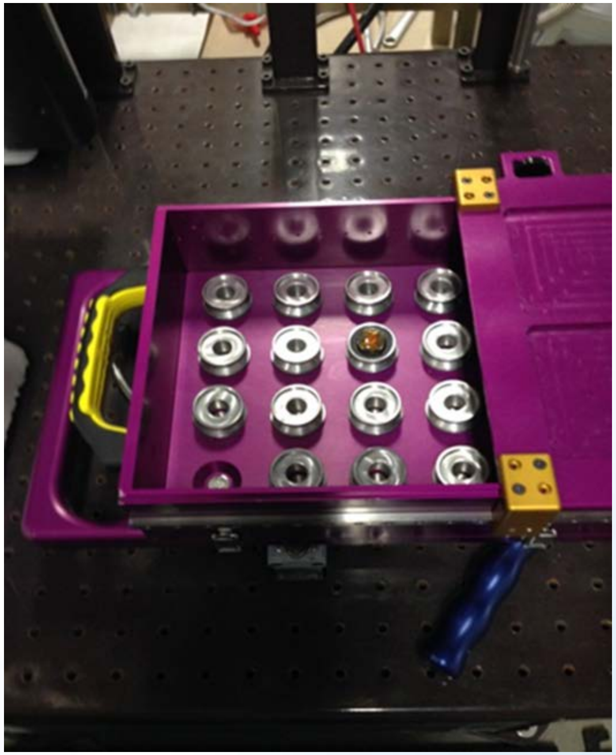
Post to remove sample from base

RPV steel sample ~2.5mm

- Sample holder provides containment
- Solid samples require single layer of containment
- Holders have a magnetic closure ring for rapid loading
- Sample holders are bar coded

Robot and Dose Rate Limits

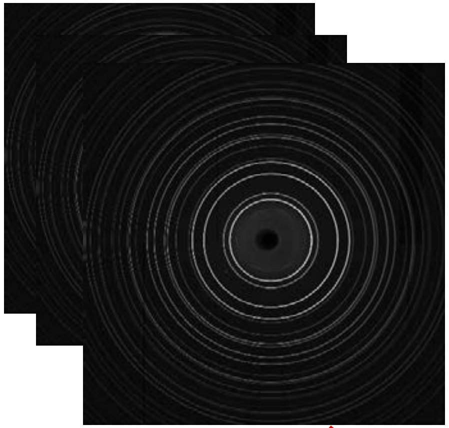
- Robot reduced dose rates enabled low fluence irradiated U-Zr alloy fuel study
- Limits for gamma at the NSLS-II are generally 5 mrem/h @ 30 cm
- Limits for gamma at XPD for steels when using the robot are 100 mrem/hr @ 30 cm
- Twenty samples loaded in the magazine - 600 mrem/h on contact including beta
- Using the robot reduces dose to hands
- Magazines hold up to *20 samples* (depending on activity)
- Users load the holders and/or magazine at their home institutions



Open magazine showing holders

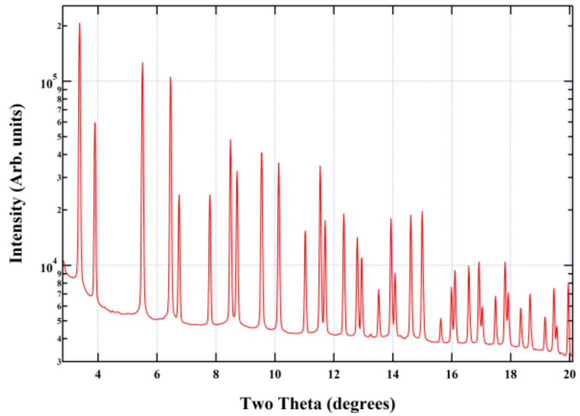
NSLS-II XPD Data Analysis

Over 12000 2D patterns for in situ experiment

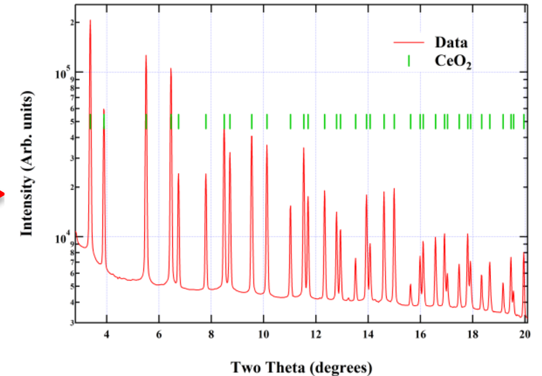


Data flow for 2D-1D and Rietveld Fitting

1D-integration
IGORPro – Wavemetrics

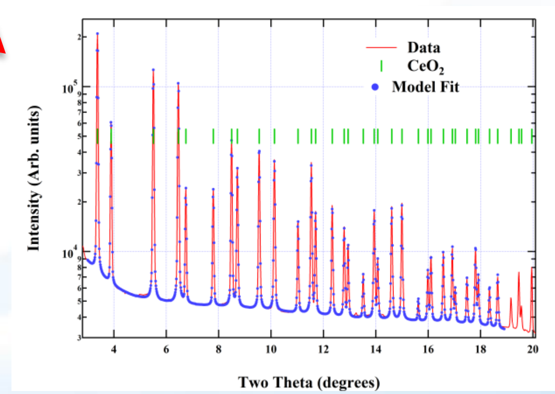


Phase ID
TOPAS and ICSD



Batch analysis

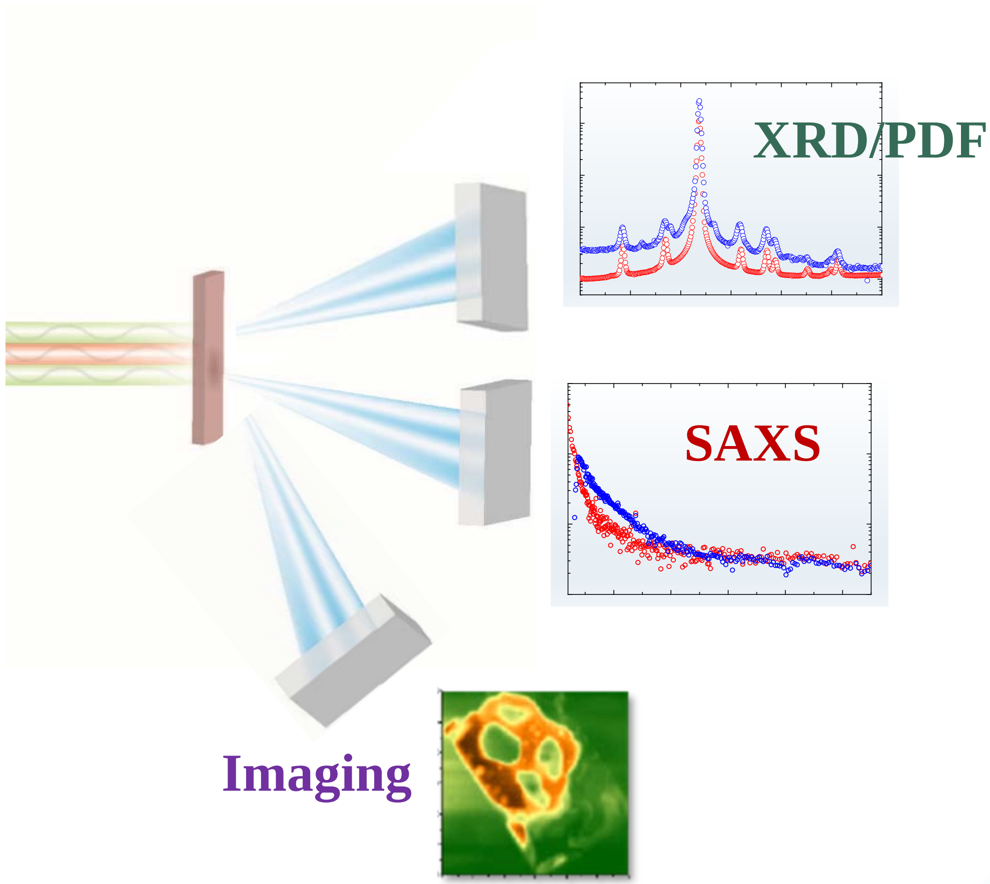
Fit/calculated pattern



Users have real time analysis and leave with data and results

Center for Data Driven Discovery at Brookhaven will specialize in large data sets

NSLS-II XPD, XRD, SAXS and PDF Analysis



Diffraction:
- Crystal structure
- Lattice parameter
- Strain (isotropic/anisotropic)
- Grain or domain Size
- Point defects and dislocations

PDF:
- Medium range order

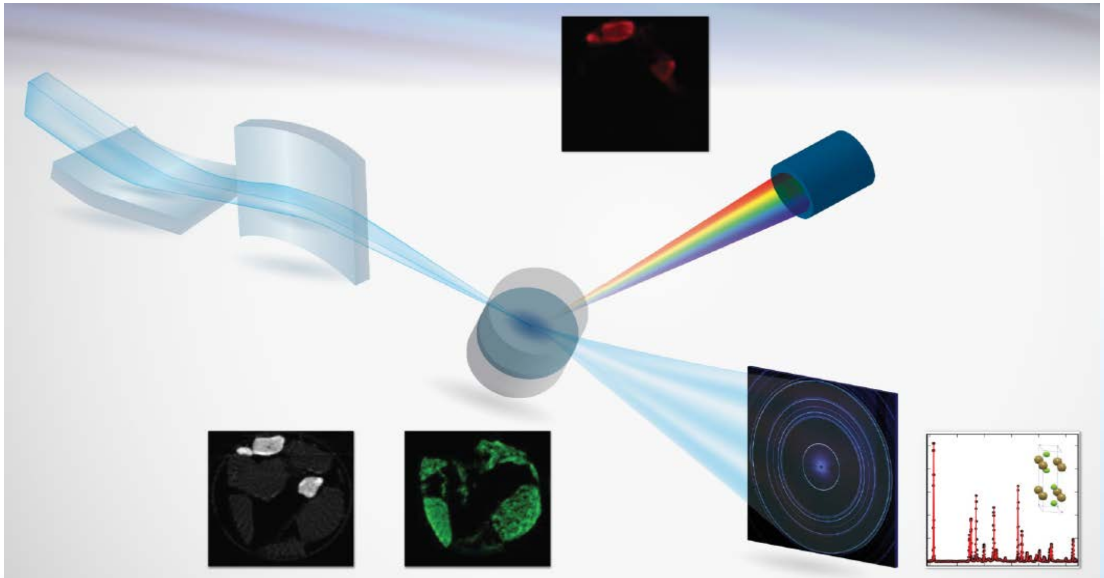
SAXS:
- Size of second phase particles
- Volume fraction
- Shape/orientation

Imaging will be available in 2017
- Morphology
- Mapping capability for information from Absorption and Diffraction

Diffraction Tomography for Nuclear Energy Materials

- Engineering scale samples for studying cracks, joining and flaws during manufacturing
- Measure the sample morphology (absorption and diffraction imaging), diffraction patterns (crystal structure, grain size and lattice strain) and fluorescence maps (chemical composition)
- Ideal for stress corrosion cracking, fatigue and eutectic formation

FY 2015 NEET Infrastructure Award

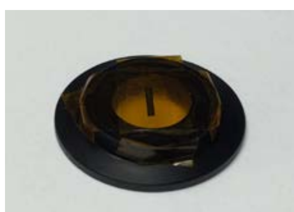


Schematic of a Diffraction Tomography Experiment

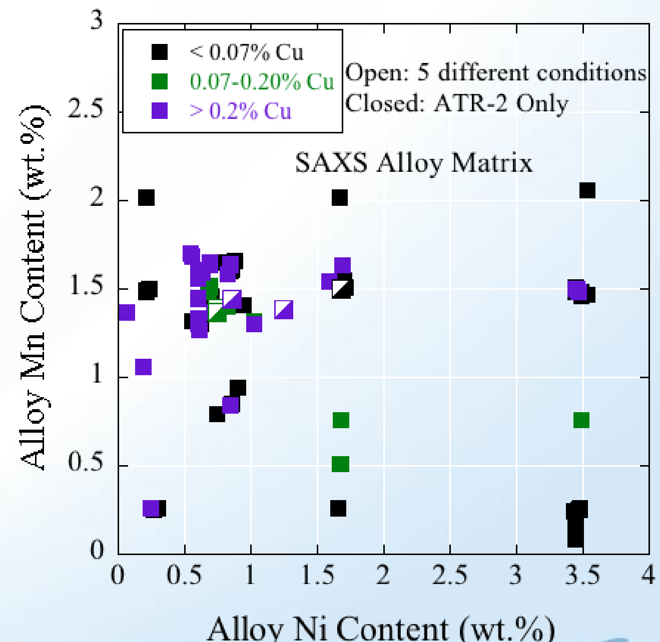
NSLS-II XPD Irradiated & Unirradiated Sample Inventory

- Ultra high speed-automated data collection provides and unparalleled opportunity for nuclear materials characterization including a new paradigm for combinatorial experiments

Samples	#	Partner institution	Technique	Mission	Achievement
RPV	> 400	UCSB	XRD + SAXS	NEET/LWS	NSLS-II First Light Experiment, BES Science highlight
RPV	20	ORNL	XRD + SAXS	NEET/LWS	
SiC	35	ORNL	XRD + PDF	NEET/AFC	
UO_2	20 (30,000 XRD patterns)	LANL	XRD	AFC	First in-situ, real time experiment at NSLS-II
UZr & UMo	80	INL	XRD + SAXS	AFC	
ODS/NFA	30	LANL UCSB	XRD + SAXS	FES	
FMS	12	LANL	XRD	AFC	
FeCrAl	18	ORNL	XRD + SAXS	NEET/LWS	
HEA	9	ORNL UT	XRD + SAXS	BES/EFRC	
Alloy 690	9		XRD		



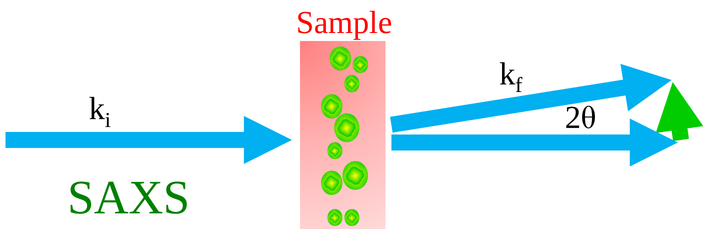
Irradiated silicon carbide sample ready for use with robot

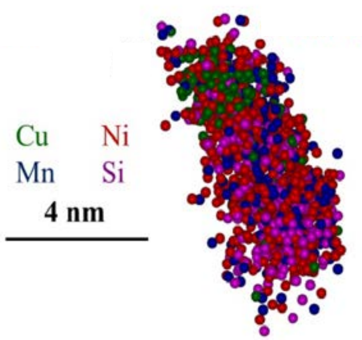


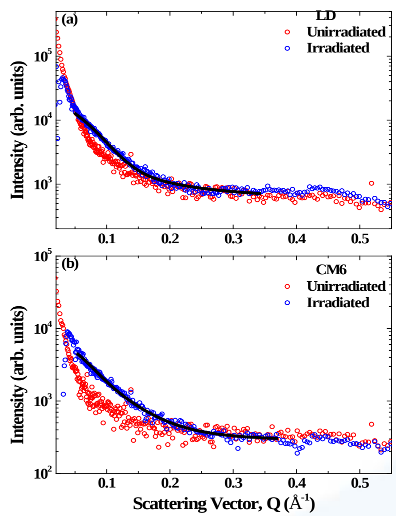
Microstructural Characterization of Irradiated RPV steels

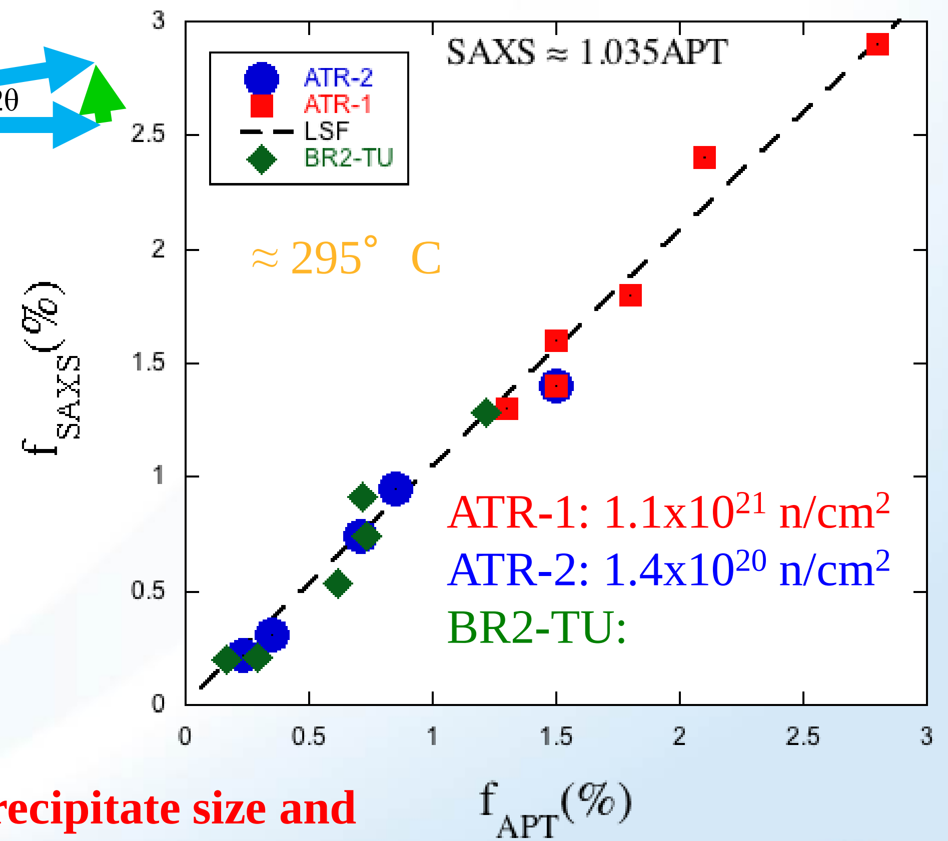
- Example of intermetallic precipitate measurements in 6 core RPV steels in UCSB irradiation experiments: ATR-1 (SAXS + XRD), ATR-2 and BR2-TU (SAXS to date)

ID	Cu	Ni	Mn	Si
LC	0.41	0.86	1.44	0.23
LD	0.38	1.25	1.38	0.23
LG	0.01	0.74	1.37	0.22
LH	0.11	0.74	1.39	0.24
LI	0.2	0.74	1.37	0.24
CM6	0.02	1.68	1.5	0.17







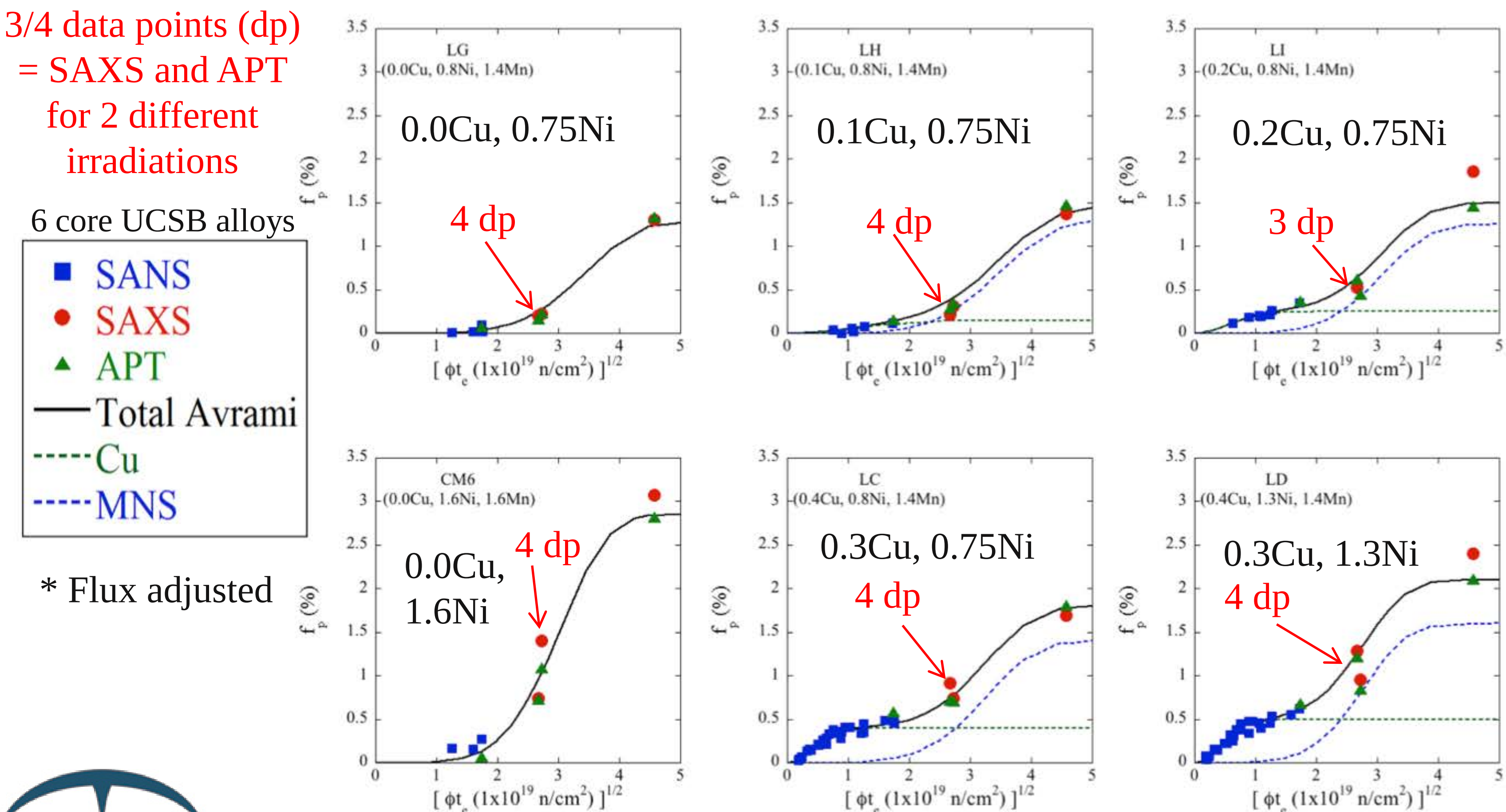


Consistent APT and SAXS precipitate size and volume fractions (f_p)

ATR-1: D. J. Sprouster et al. Scripta Mat. 113, 18 2016

Application and Impact of SAXS and XRD

- Extended life ΔT models: f_p versus effective fluence* model developed ***before*** ATR-2 SAXS (green triangles) and APT data (large red circles)



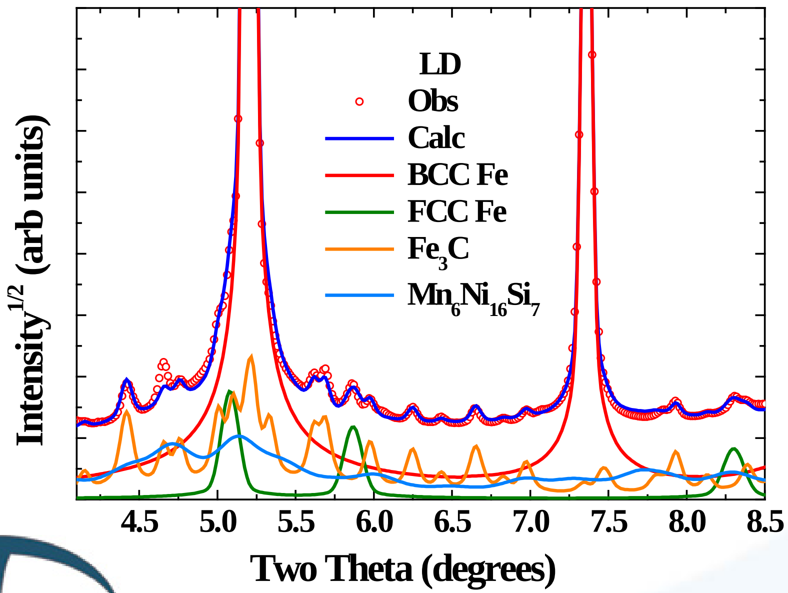
Note $\Delta T \approx C_1 \Delta\sigma_y \approx C_2 \sqrt{f_p}$

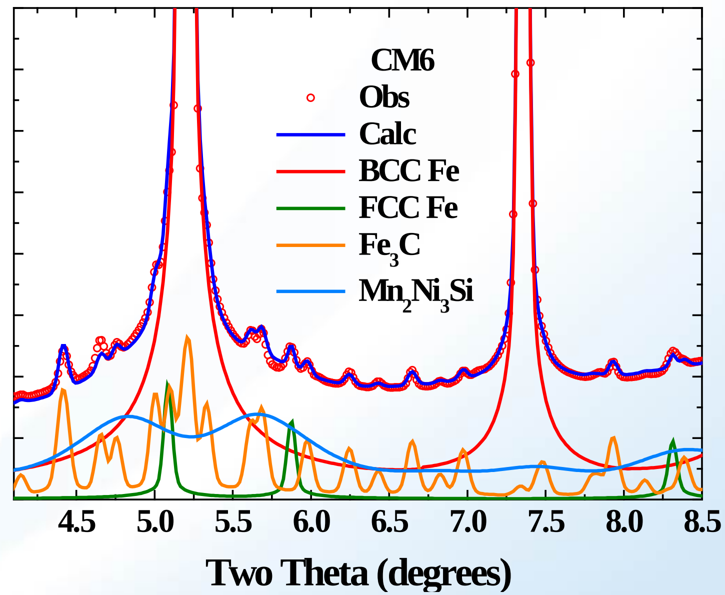
Complications - Challenges - Opportunities

- SAXS data analysis requires new procedures that are still under development and *especially* complimentary APT, XRD, SANS,TEM,…
  - Various pre-existing features - like carbides - with very large size range & different scattering characteristics – must deconvolute nano-precipitate signal
  - Irradiation may change the coarser pre-existing features
  - May be multiple populations of nano-precipitates
  - Scattering contrast factors (composition & atomic density) unknown a priori
  - Massive amount of data requires automation
- Bad news is also good news - SAXS plus XRD provide a potential *"one stop shopping"* to characterize the "entire" micro-nanostructures
- Many opportunities - X-ray microscope, glancing incidence, … - will probe ion irradiations and lab on a chip-type experiments

XRD Phase Identification - Micro to Nano

- Limited f and small d - very difficult to use XRD to characterize nanoscale features due to low signals and enormous line broadening
- Major XRD advance at NSLS-II - characterizing G- and Γ_2 phase nano precipitates - SAXS (or APT/SANS) for feature size used in a PONKCS* simulation fit to the major peak background 2θ spectrum
- Also characterized carbides and some retained austenite





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*Partially or not known crystal structure

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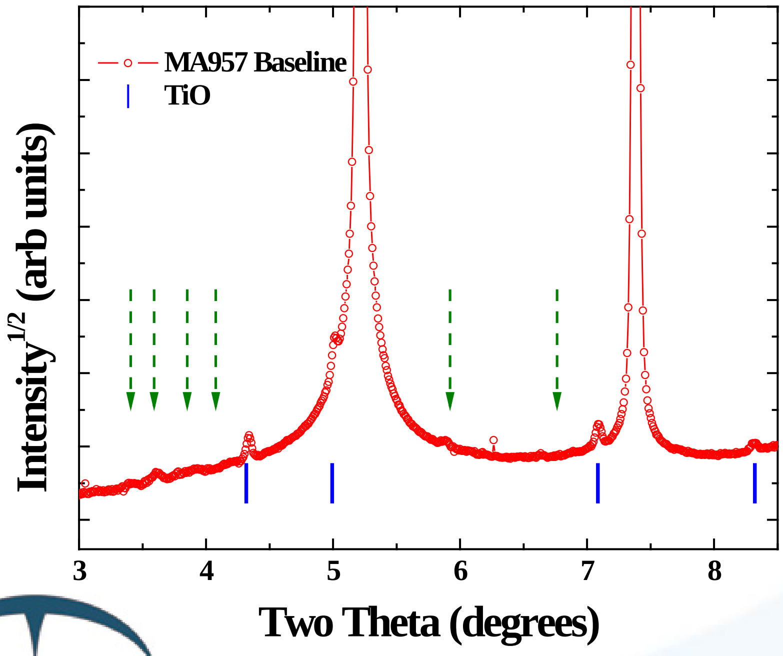
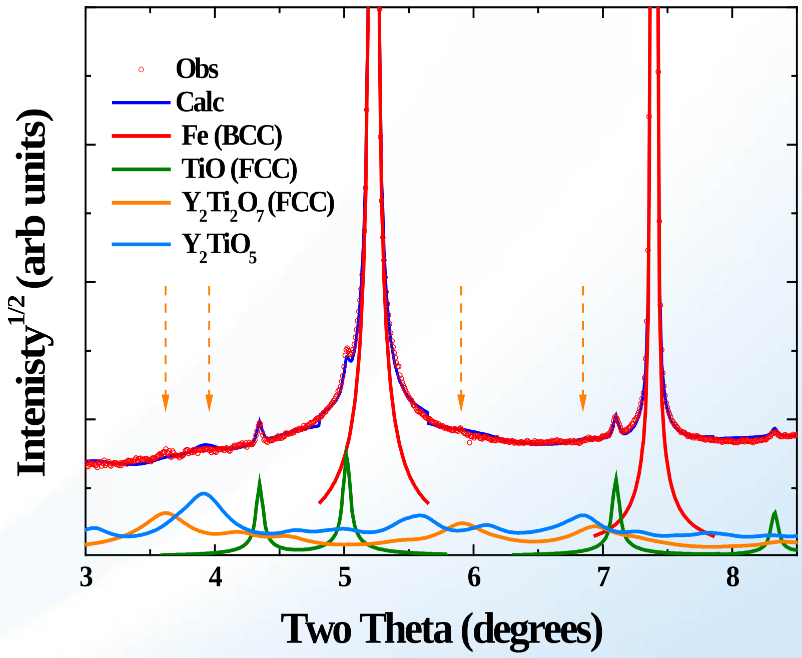
Characterization of the Nano-Oxides

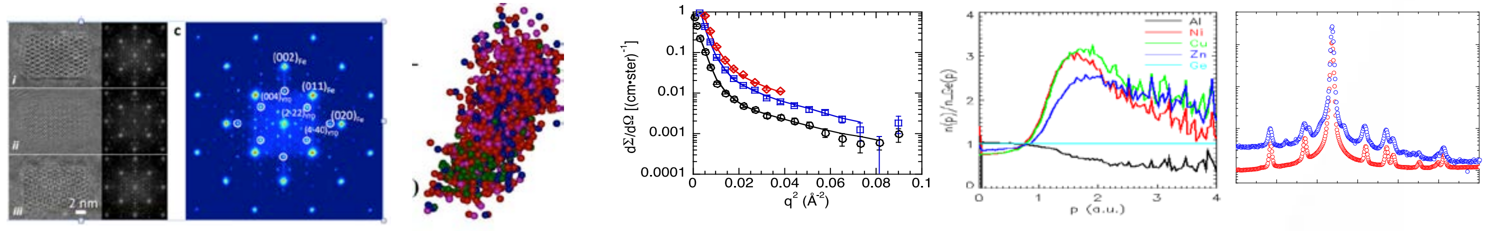
- Controversy regarding structure of nano-oxides in nanostructured ferritic alloys (aka ODS steels)
- Applied PONKCS technique to various NFA – example MA957
- Other nanoscale techniques to be explored (coherency strains?)





Multi-Tool Multi-Simulation *Computational Microscope*

TEM APT SANS(SAXS) PAS XRD

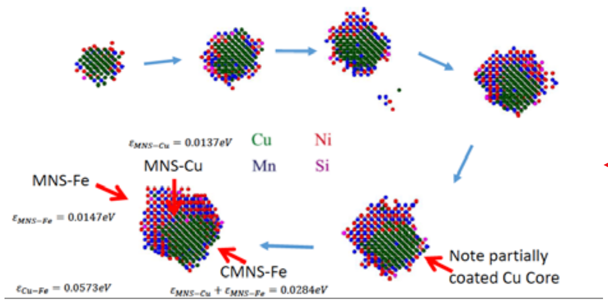


features

observables

self-consistent
'understanding'

KLMC, …

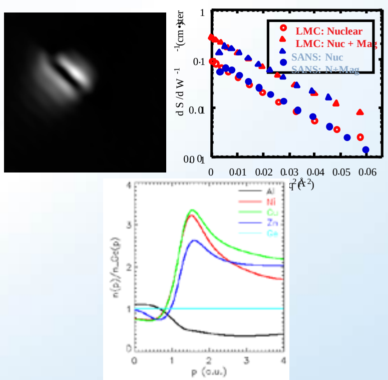


S. Shu UWisc.

predict
features

simulate
observables

TEM, SANS, XRD
Positron simulation



G. R. Odette and B. D. Wirth, "A computational microscopy study of nanostructural evolution in irradiated pressure vessel steels", JNM 251 (1997) 271

Summary and Future Opportunities

- Major XRD advance in characterizing G- and Γ_2 phase RPV steel nanoscale precipitates
- SAXS results are generally consistent with SANS and APT (much more work required)
- XRD/SAXS to help develop rigorous physical ΔT models for extended life
- XPD beam time available through NSUF - current open call
  - BNL will work with users from proposal to data analysis stages
  - Robot routinely allows use of activated materials and very high throughput
  - High resolution SAXS/XRD/PDF available
  - Tomography coming in 2017
  - Routine data analysis is automated - but complex engineering materials will require new methods and multiple techniques

Many new future capabilities and opportunities

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