Capability upgrades and future plans for nuclear materials research at NSLS-II

Mehmet Topsakal, Simerjeet K. Gill
Nuclear Science and Technologies Department
Brookhaven National Laboratory, New York.

Outline of this talk

- Introduction to NSUF facility at NSLS-II

- Capability upgrades

- Plans for future

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- Introduction to NSUF facility at NSLS-II
- Capability upgrades
- Plans for future

Who are we?

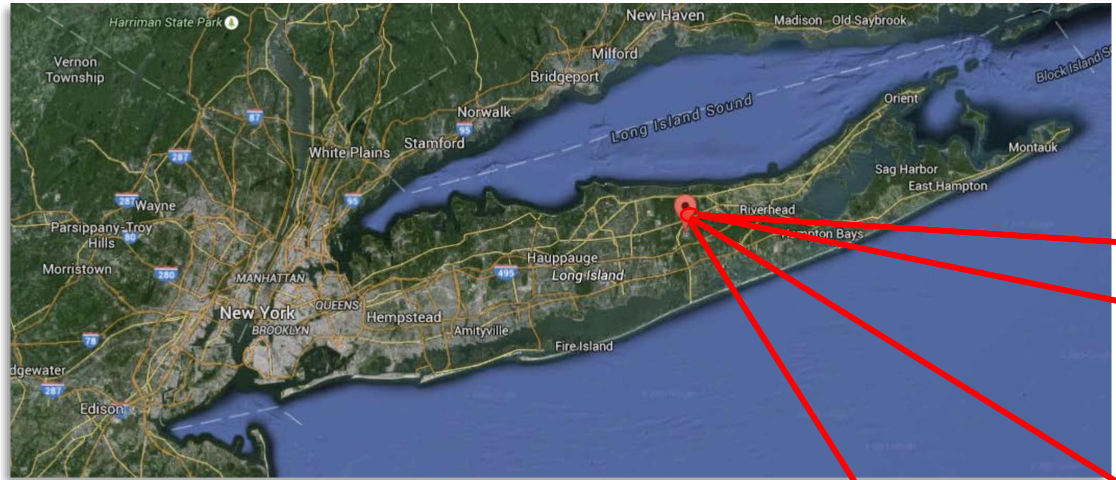
NSUF Capabilities Offer Research Opportunities

Neutron Irradiations	Ion Irradiations	Gamma Irradiations	Hot Cells & Shielded Cells	Low Activity Laboratories	Beamlines	High Performance Computing
INL Idaho National Laboratory	Wisconsin University of Wisconsin-Madison	INL Idaho National Laboratory	INL Idaho National Laboratory	INL Idaho National Laboratory	Brookhaven National Laboratory	INL Idaho National Laboratory
Oak Ridge National Laboratory	University of Michigan	Oak Ridge National Laboratory	Oak Ridge National Laboratory	CAES Center for Advanced Energy Studies	Argonne National Laboratory	
MIT Massachusetts Institute of Technology	Argonne National Laboratory	Sandia National Laboratories	PNNL	Oak Ridge National Laboratory	NC State University	
NC State University	Sandia National Laboratories		Los Alamos National Laboratory Est. 1943	Cal	Los Alamos National Laboratory Est. 1943	
Ohio State	Texas A&M University		Westinghouse	PNNL		
Sandia National Laboratories	Lawrence Livermore National Laboratory		University of Michigan	Los Alamos National Laboratory Est. 1943		
SCK•CEN Studiecentrum voor Kernenergie Centre d'Etude de l'Energie Nucléaire			SCK•CEN Studiecentrum voor Kernenergie Centre d'Etude de l'Energie Nucléaire	NC State University		
				University of Michigan		
				Purdue University		
				Wisconsin University of Wisconsin-Madison		
				University of Florida		

Visit nsuf.inl.gov for details of individual facilities

Where are we?

Brookhaven National Laboratory (BNL) is a United States **Department of Energy** national laboratory located in Upton, New York, on Long Island, and was formally established in 1947.



BNL hosts National Synchrotron Light Source-II **(NSLS-II)**

Who are we?

Since ***2017***, we are supporting nuclear science users under the umbrella of Nuclear Science User Facilities (NSUF)

NSUF Capabilities Offer Research Opportunities

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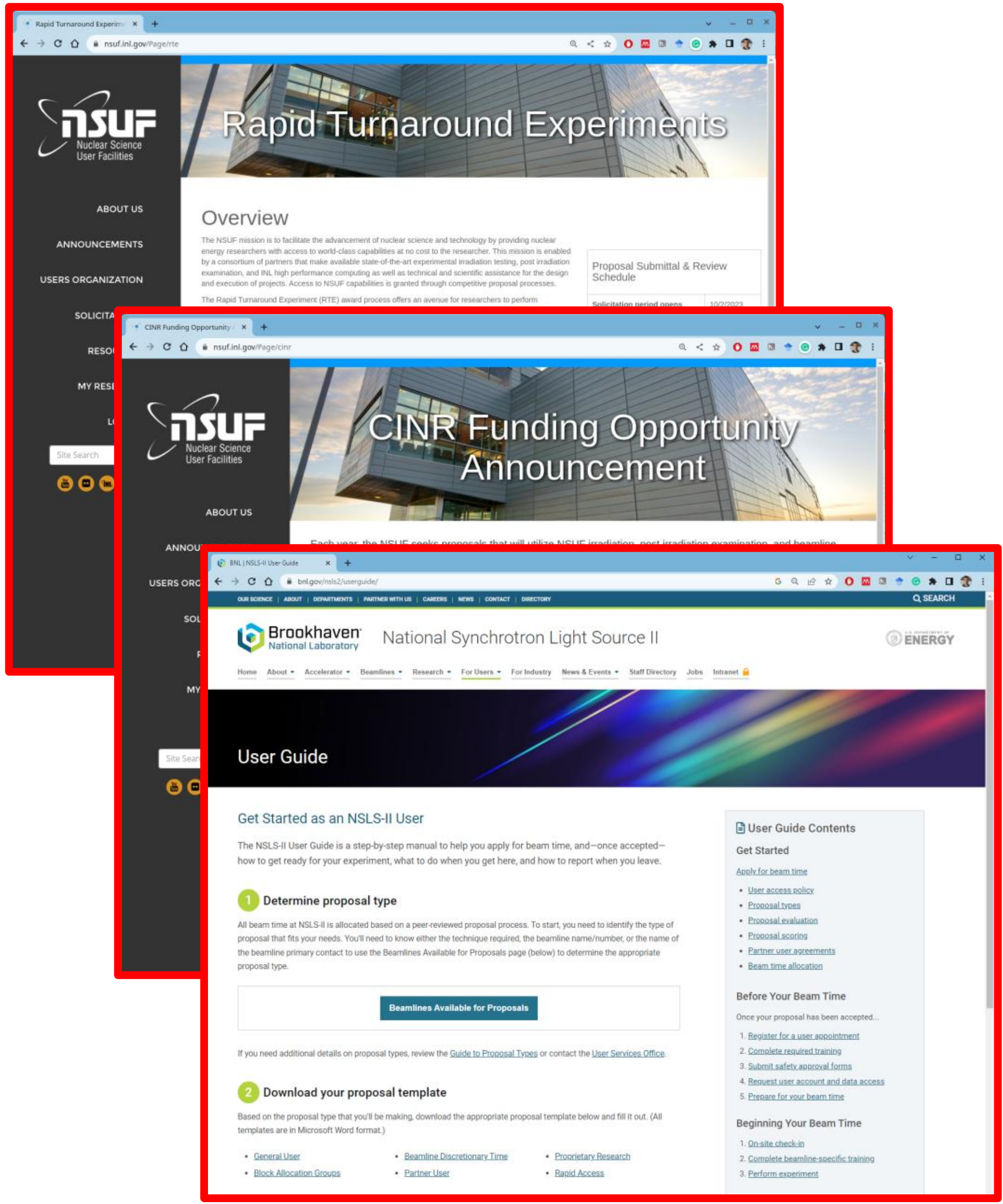
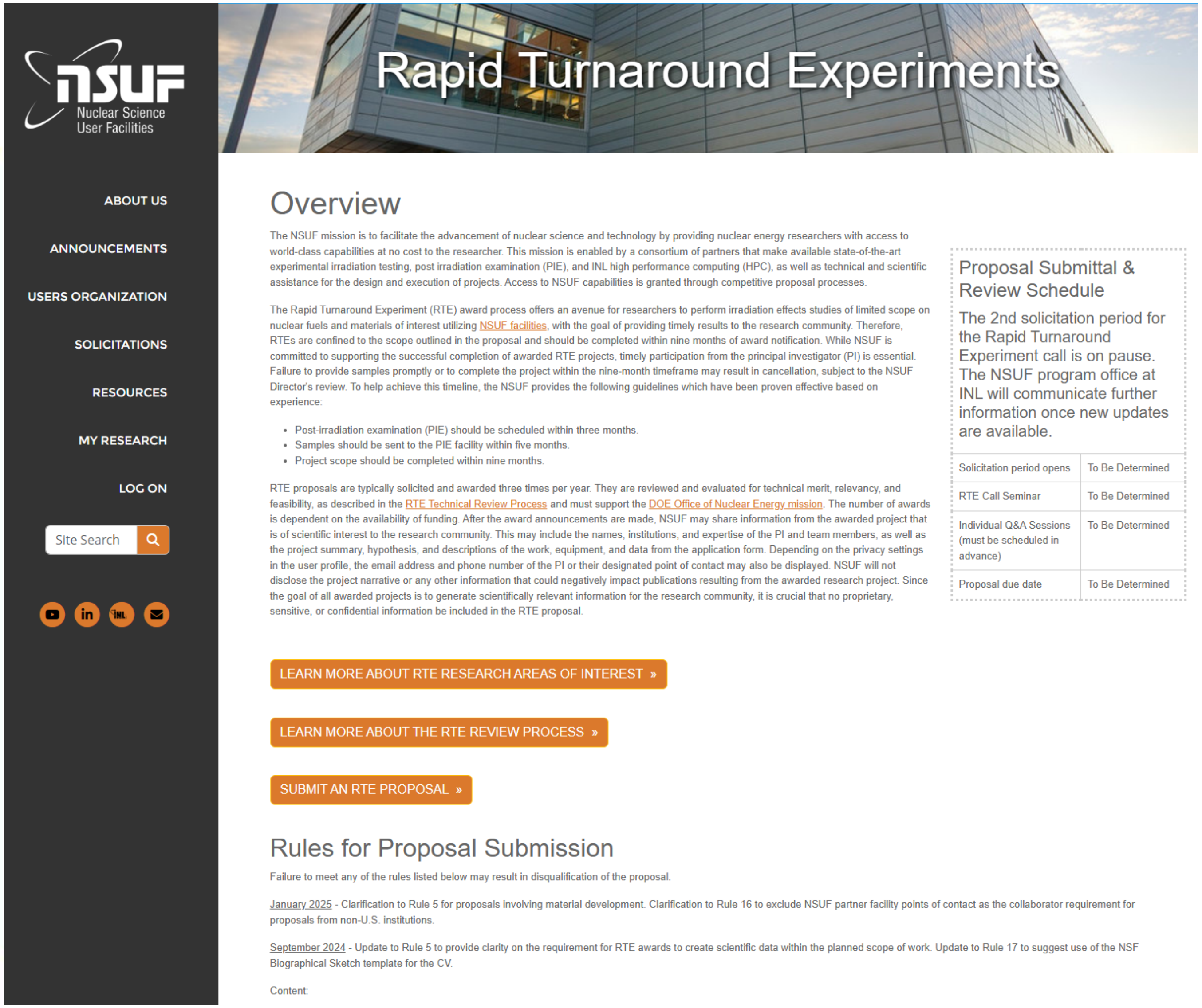


Rapid turnaround experiment (RTE) proposals are easy!


NSUF Nuclear Science User Facilities

Rapid Turnaround Experiments

ABOUT US
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USERS ORGANIZATION
SOLICITATIONS
RESOURCES
MY RESEARCH
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Overview

The NSUF mission is to facilitate the advancement of nuclear science and technology by providing nuclear energy researchers with access to world-class capabilities at no cost to the researcher. This mission is enabled by a consortium of partners that make available state-of-the-art experimental irradiation testing, post irradiation examination (PIE), and INL high performance computing (HPC), as well as technical and scientific assistance for the design and execution of projects. Access to NSUF capabilities is granted through competitive proposal processes.

The Rapid Turnaround Experiment (RTE) award process offers an avenue for researchers to perform irradiation effects studies of limited scope on nuclear fuels and materials of interest utilizing NSUF facilities, with the goal of providing timely results to the research community. Therefore, RTEs are confined to the scope outlined in the proposal and should be completed within nine months of award notification. While NSUF is committed to supporting the successful completion of awarded RTE projects, timely participation from the principal investigator (PI) is essential. Failure to provide samples promptly or to complete the project within the nine-month timeframe may result in cancellation, subject to the NSUF Director's review. To help achieve this timeline, the NSUF provides the following guidelines which have been proven effective based on experience:

- Post-irradiation examination (PIE) should be scheduled within three months.
- Samples should be sent to the PIE facility within five months.
- Project scope should be completed within nine months.

RTE proposals are typically solicited and awarded three times per year. They are reviewed and evaluated for technical merit, relevancy, and feasibility, as described in the RTE Technical Review Process and must support the DOE Office of Nuclear Energy mission. The number of awards is dependent on the availability of funding. After the award announcements are made, NSUF may share information from the awarded project that is of scientific interest to the research community. This may include the names, institutions, and expertise of the PI and team members, as well as the project summary, hypothesis, and descriptions of the work, equipment, and data from the application form. Depending on the privacy settings in the user profile, the email address and phone number of the PI or their designated point of contact may also be displayed. NSUF will not disclose the project narrative or any other information that could negatively impact publications resulting from the awarded research project. Since the goal of all awarded projects is to generate scientifically relevant information for the research community, it is crucial that no proprietary, sensitive, or confidential information be included in the RTE proposal.

Proposal Submittal & Review Schedule

The 2nd solicitation period for the Rapid Turnaround Experiment call is on pause. The NSUF program office at INL will communicate further information once new updates are available.

Solicitation period opens	To Be Determined
RTE Call Seminar	To Be Determined
Individual Q&A Sessions (must be scheduled in advance)	To Be Determined
Proposal due date	To Be Determined

LEARN MORE ABOUT RTE RESEARCH AREAS OF INTEREST »

LEARN MORE ABOUT THE RTE REVIEW PROCESS »

SUBMIT AN RTE PROPOSAL »

Rules for Proposal Submission

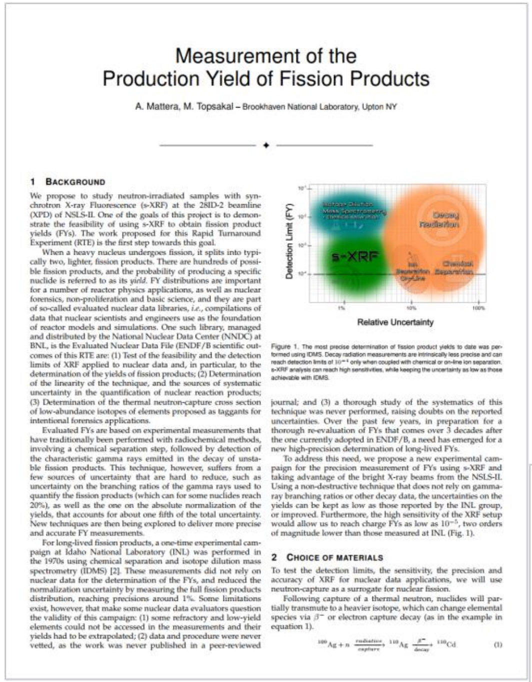
Failure to meet any of the rules listed below may result in disqualification of the proposal.

January 2025 - Clarification to Rule 5 for proposals involving material development. Clarification to Rule 16 to exclude NSUF partner facility points of contact as the collaborator requirement for proposals from non-U.S. institutions.

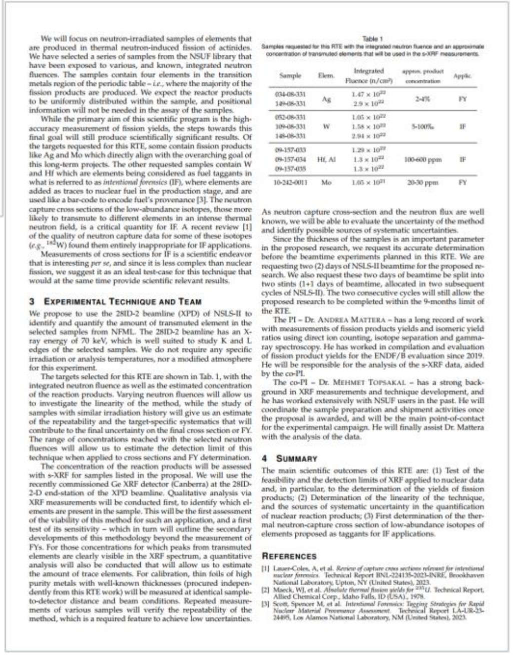
September 2024 - Update to Rule 5 to provide clarity on the requirement for RTE awards to create scientific data within the planned scope of work. Update to Rule 17 to suggest use of the NSF Biographical Sketch template for the CV.

Content:

Expected RTE proposal narratives are only 2-pages!


Measurement of the
Production Yield of Fission Products

A. Mattera, M. Topsakal – Brookhaven National Laboratory, Upton NY



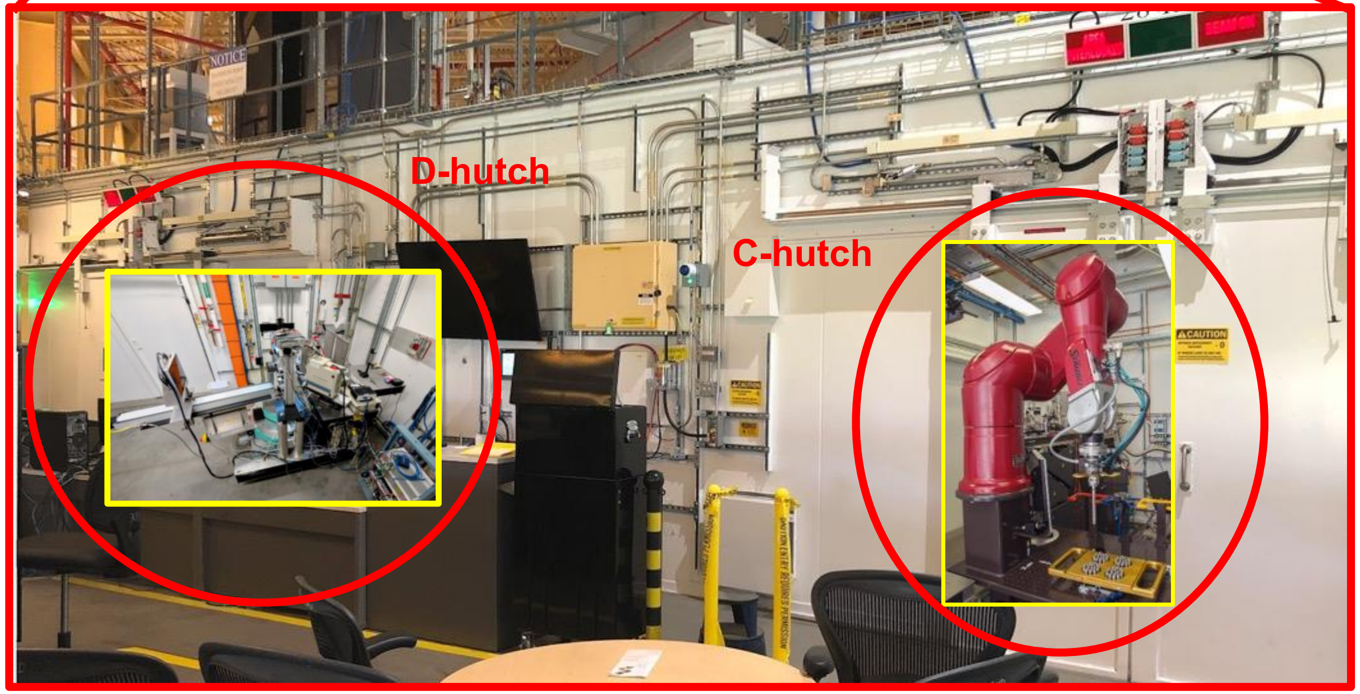
More calls are expected to be announced in 2025. Please feel free to contact us

mtopsakal@bnl.gov

gills@bnl.gov

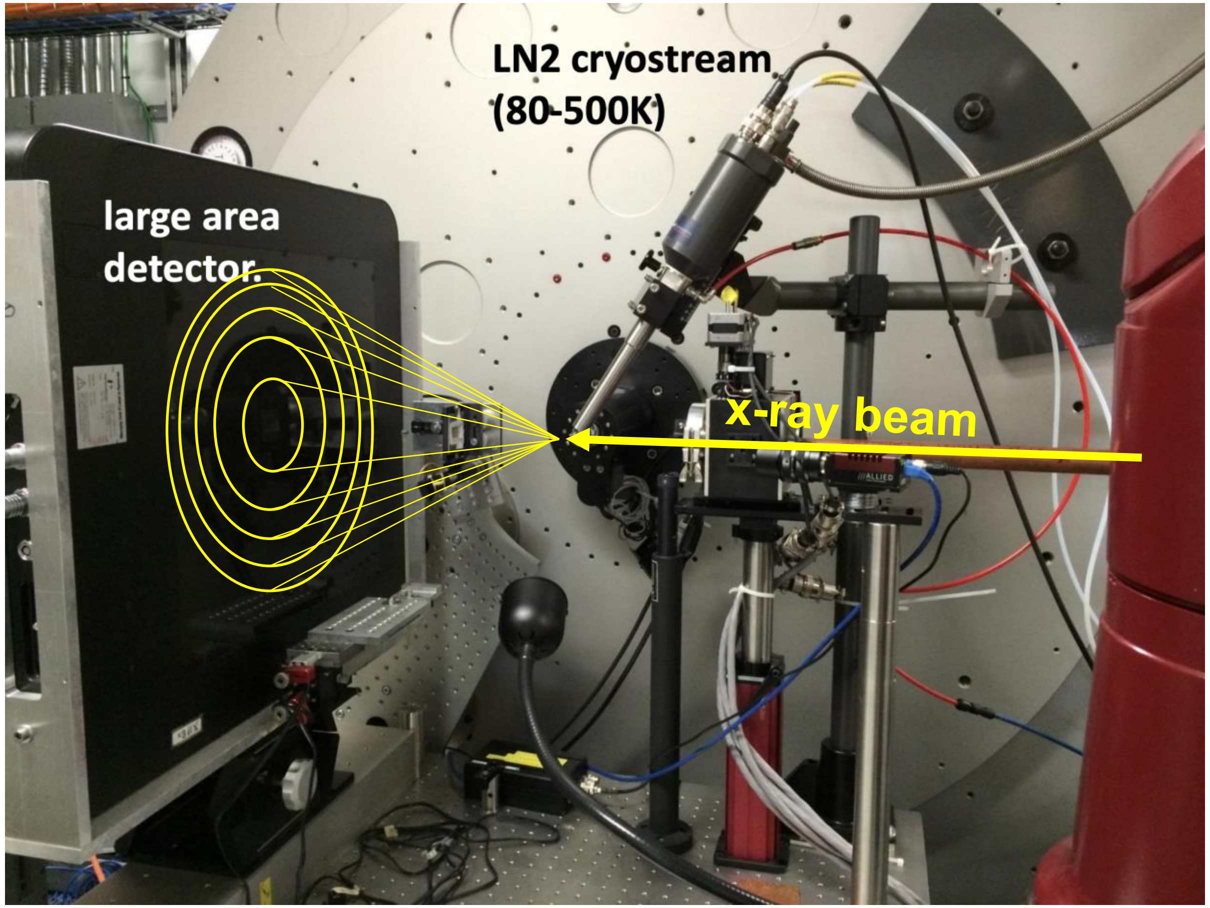
XPD beamline (28-ID) at NSLS-II

%6 of beamtime of XPD beamline is allocated to NSUF users.
(3-days of beamtime, three times in a year)

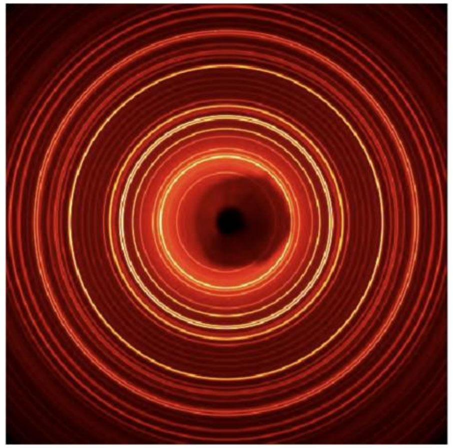


One-slide on X-ray diffraction method

A typical X-ray diffraction setup with area detector

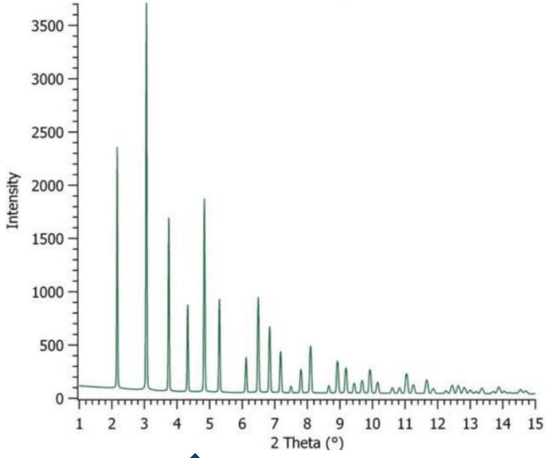


2D image



Azimuthal
integration

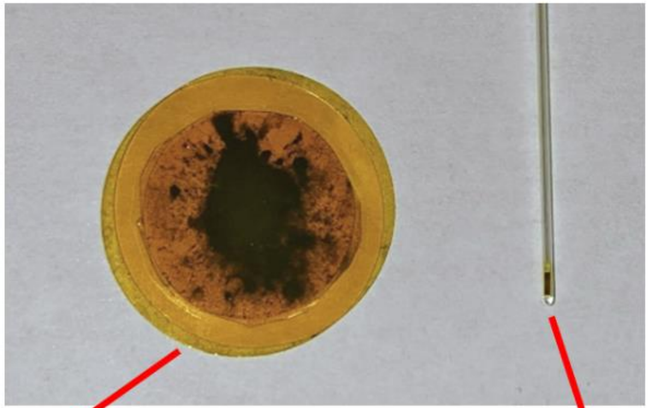
1D pattern



- Phase information
- Lattice parameters
- Phase mass fraction
- Micro strain
- Crystalline size
- Stacking faults

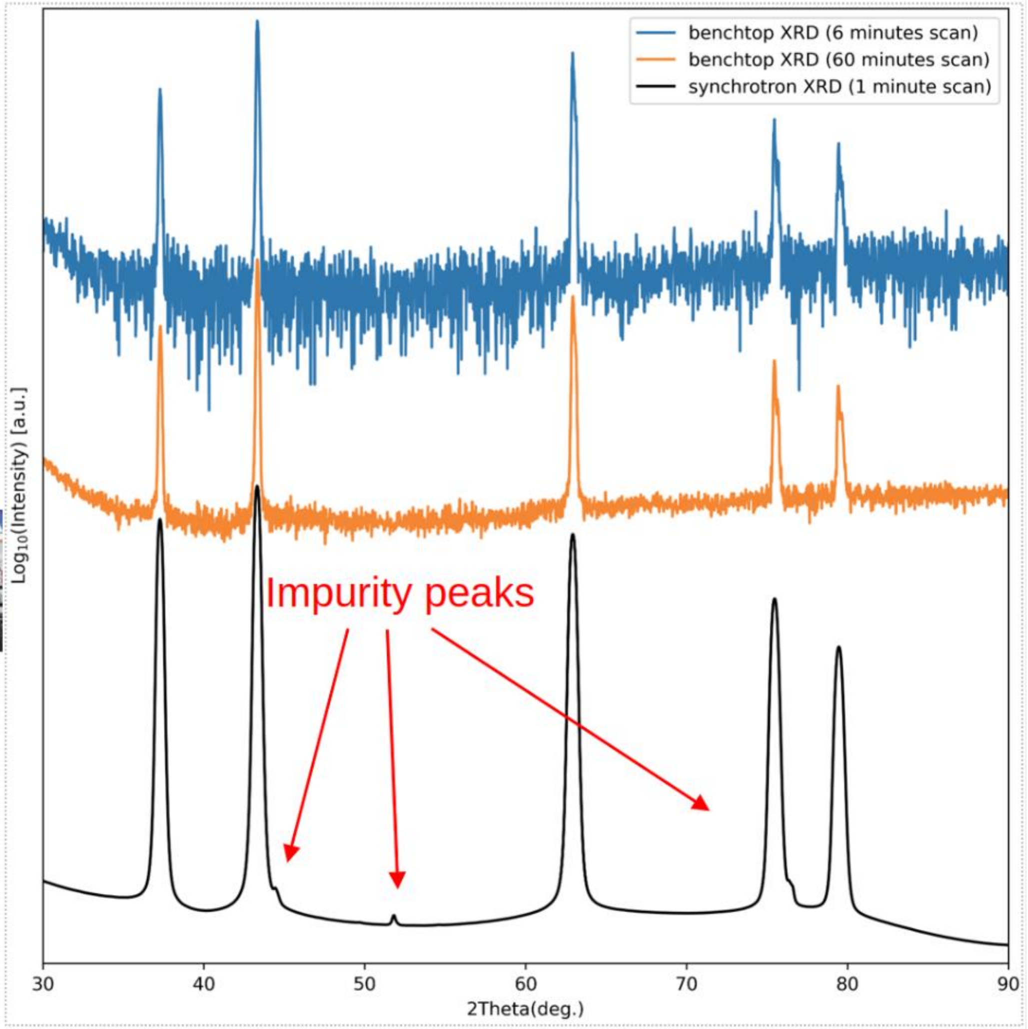
Why do we need synchrotron resources ?

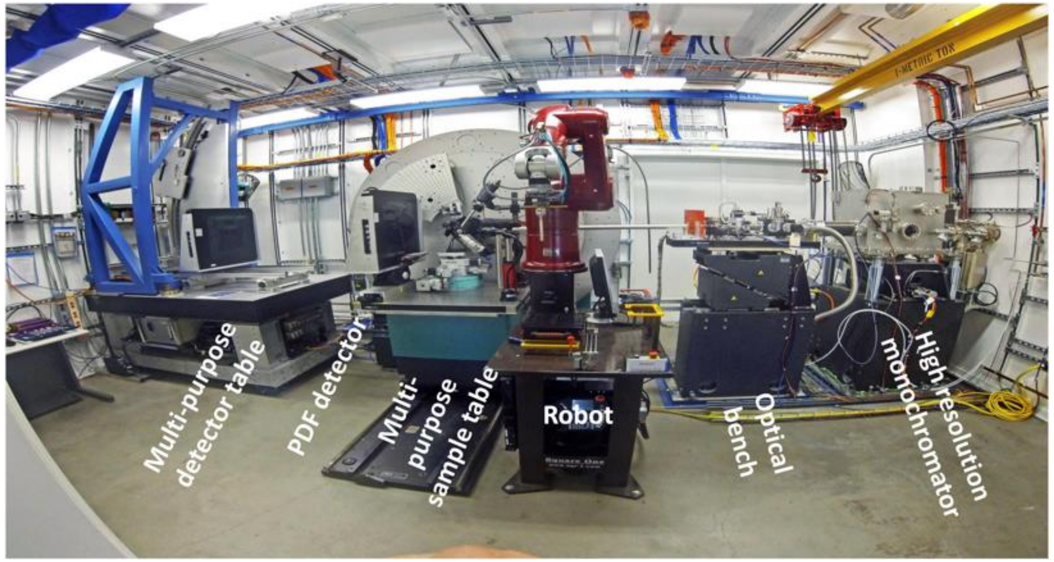
NiO powder test sample

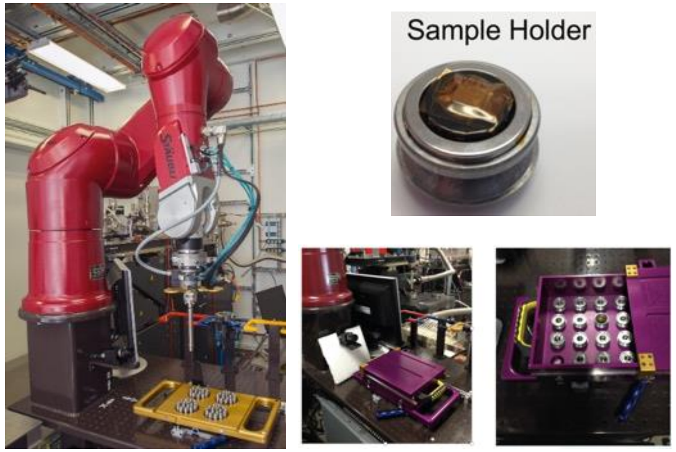


Benchtop XRD (Rigaku)

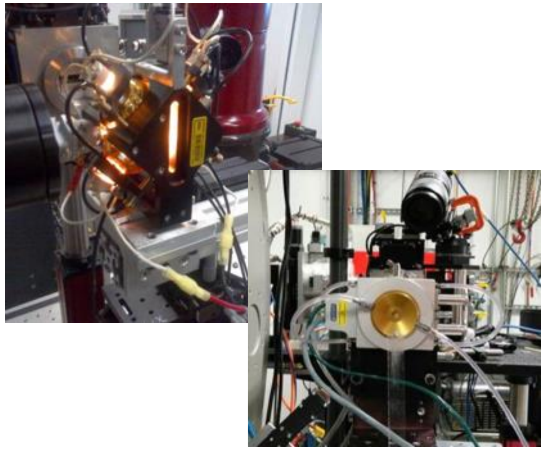
NSLS-II (XPD beamline)



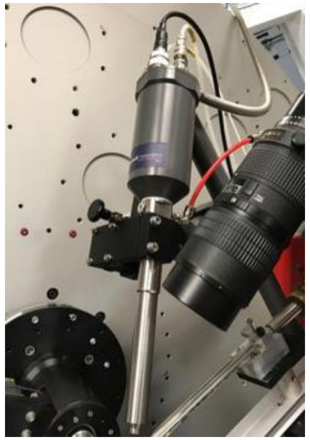




Robot for high-throughput sample changing

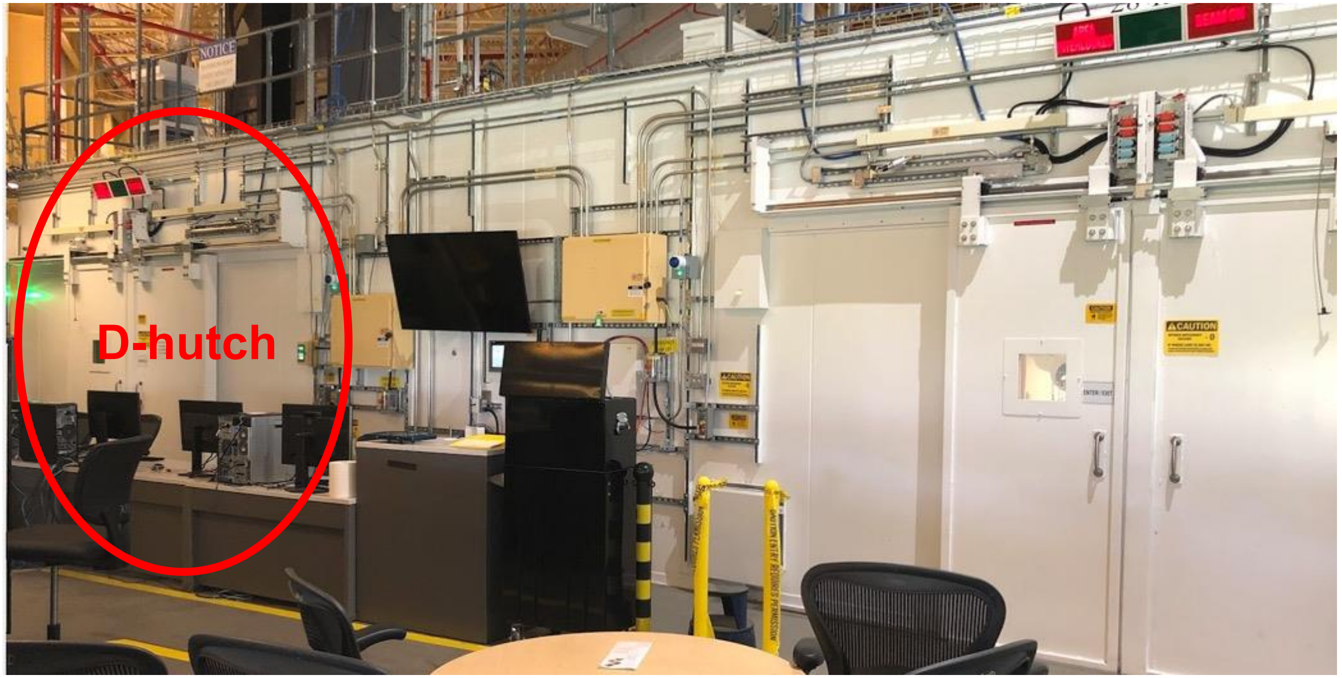


Heating up to 2000^{o}C

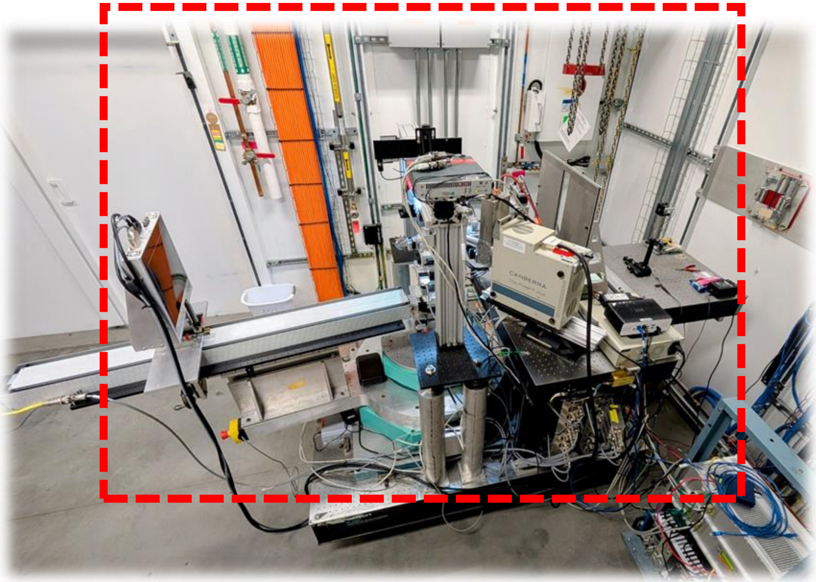


Cryostream (80K-500K)

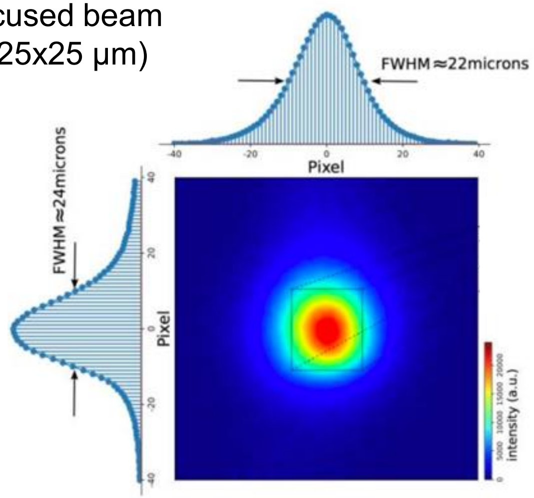
Corrosion cell



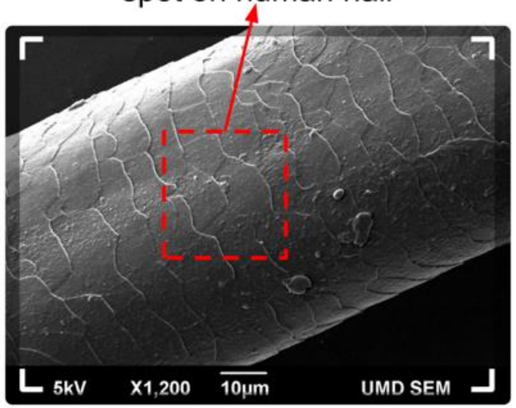
proudly supported by



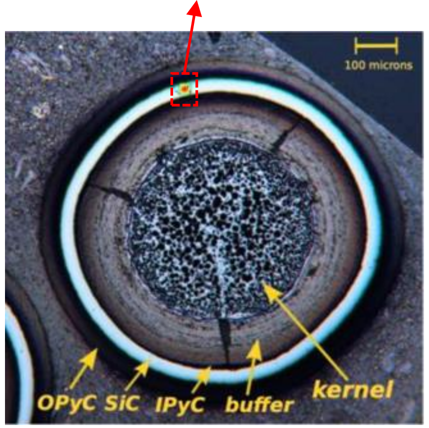
Focused beam (~25x25 μm)



25 microns x 25 microns spot on human hair



25 microns x 25 microns spot on TRISO fuel



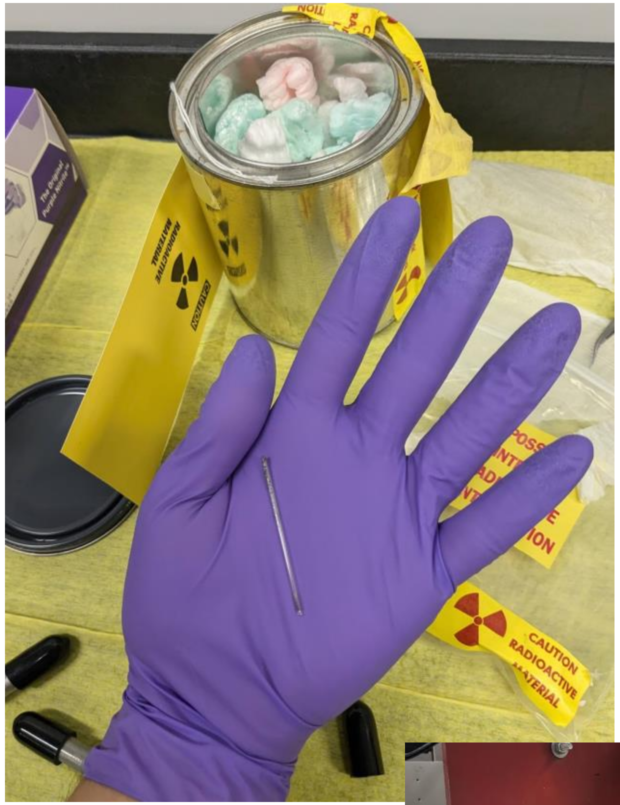
<u>Techniques enabled with small beam:</u>

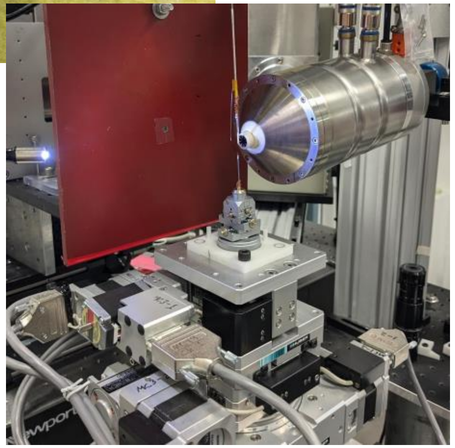
- 1D & 2D mapping (phase, lattice, strain…)
- 3D X-ray diffraction computed tomography (XRD-CT)

X-ray fluoresce spectroscopy with high-energy beam.

Multi-modal 1D, 2D, and 3D non-destructive characterization of nuclear materials.

Some pictures from a recent RTE (24-4876) experiment





UB$_4$ samples heated up to 900^{o}C

Outline of this talk

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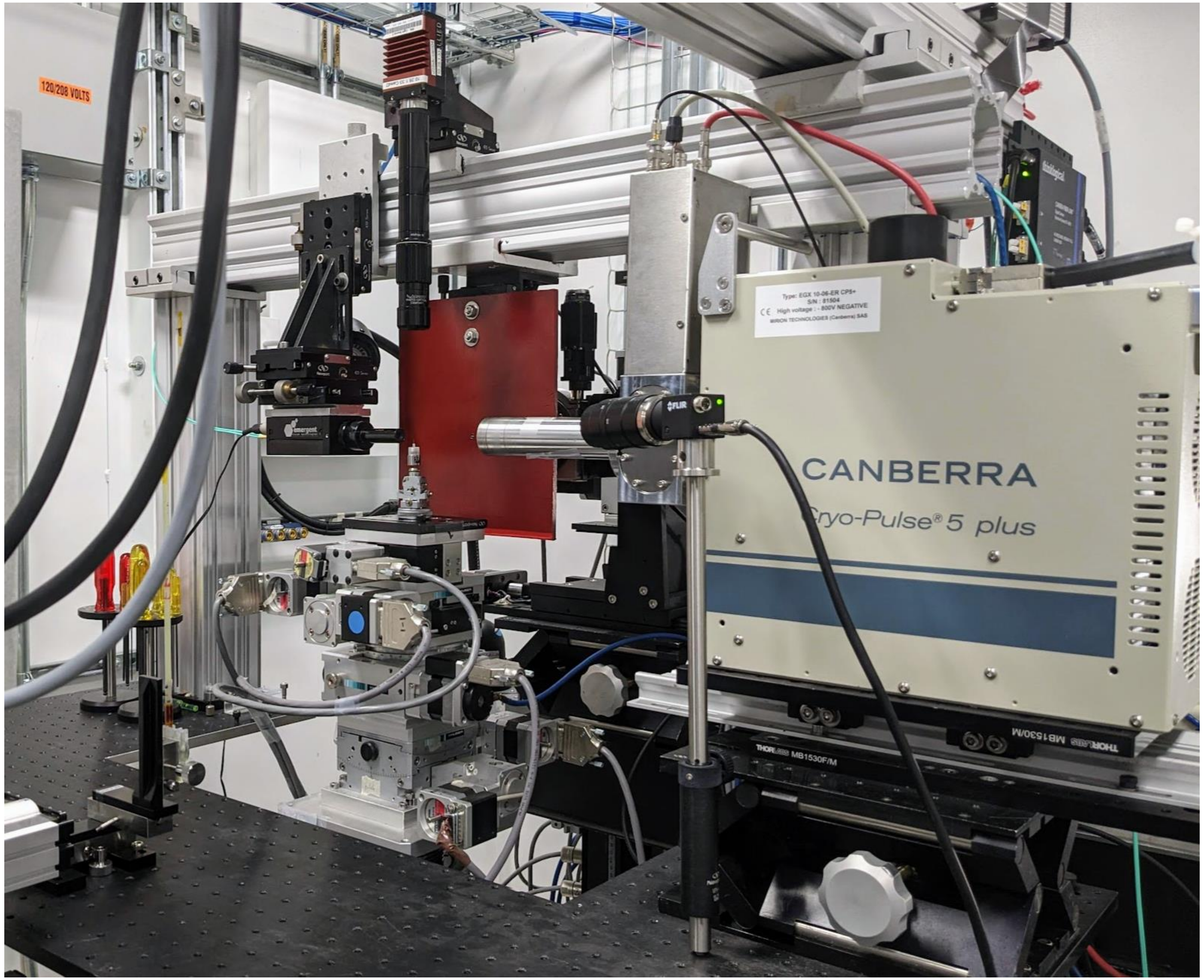
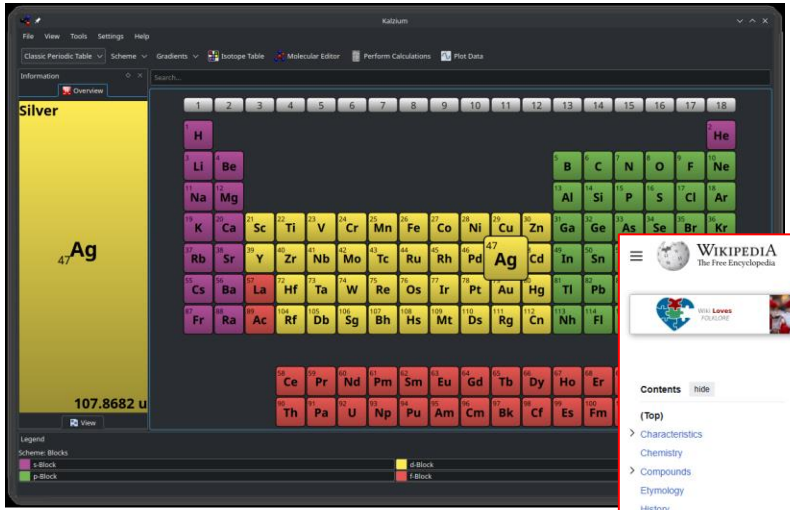
- Capability upgrades
- Plans for future

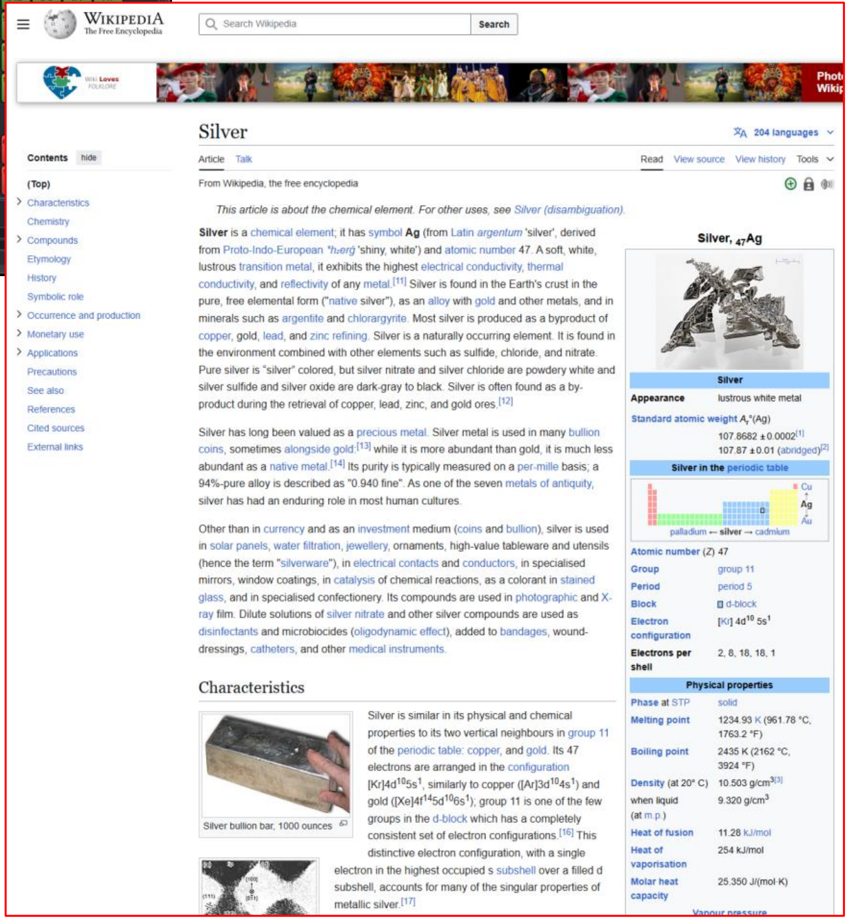
X-ray fluorescence (XRF) capability was added



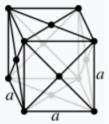
XRF capability demonstration with a science case



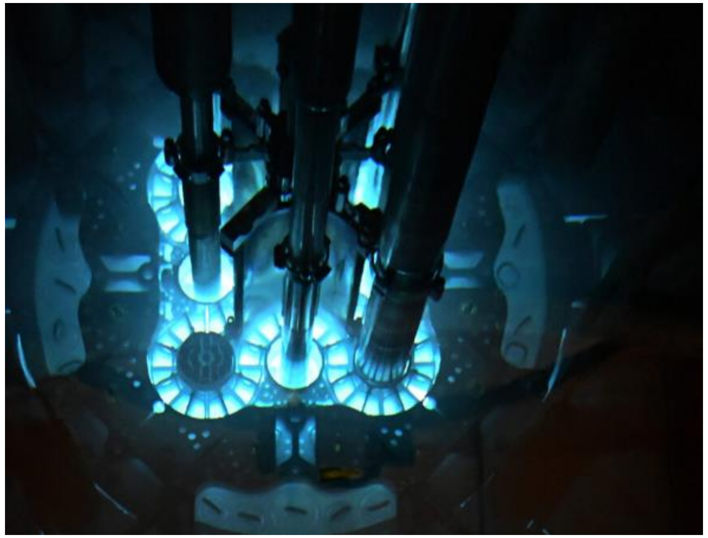
What does happen if you put silver (Ag) in ATR reactor?



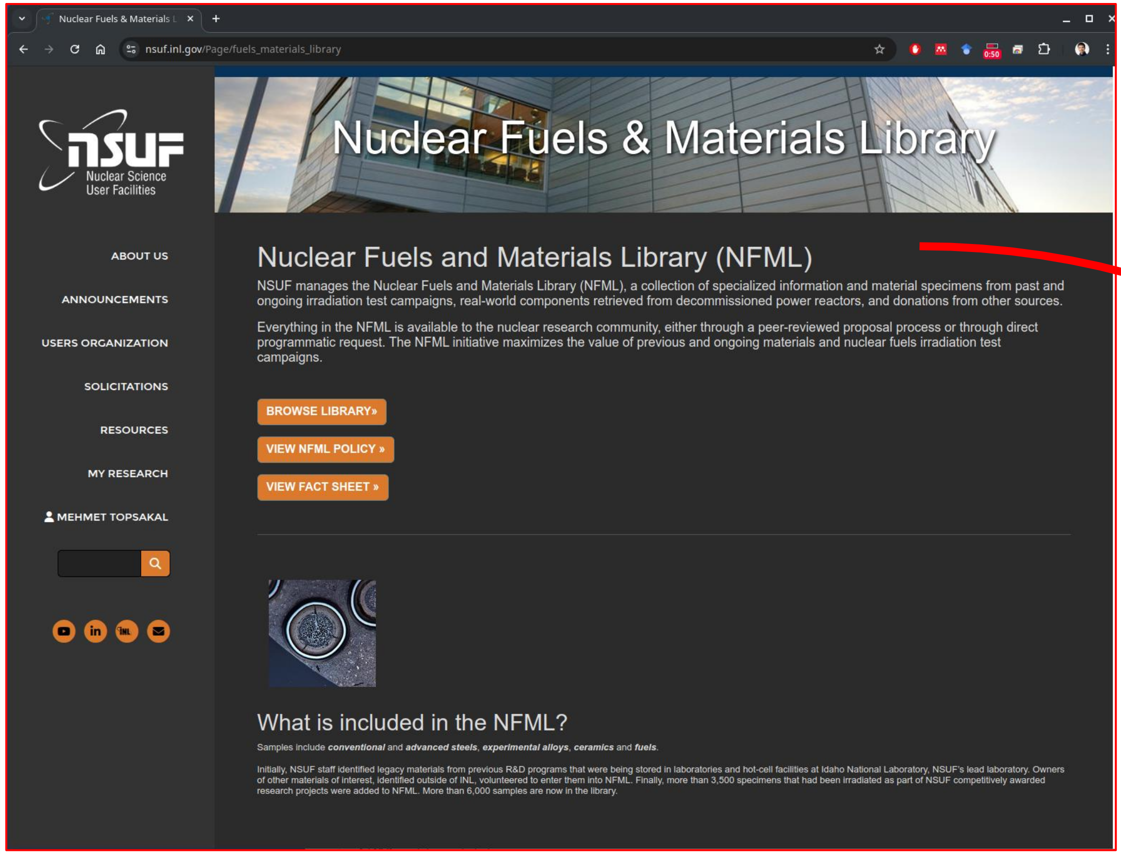
Crystal structure	face-centred cubic (fcc) (cF4)
Lattice constant	*a* = 408.60 pm (at 20 °C)[3]

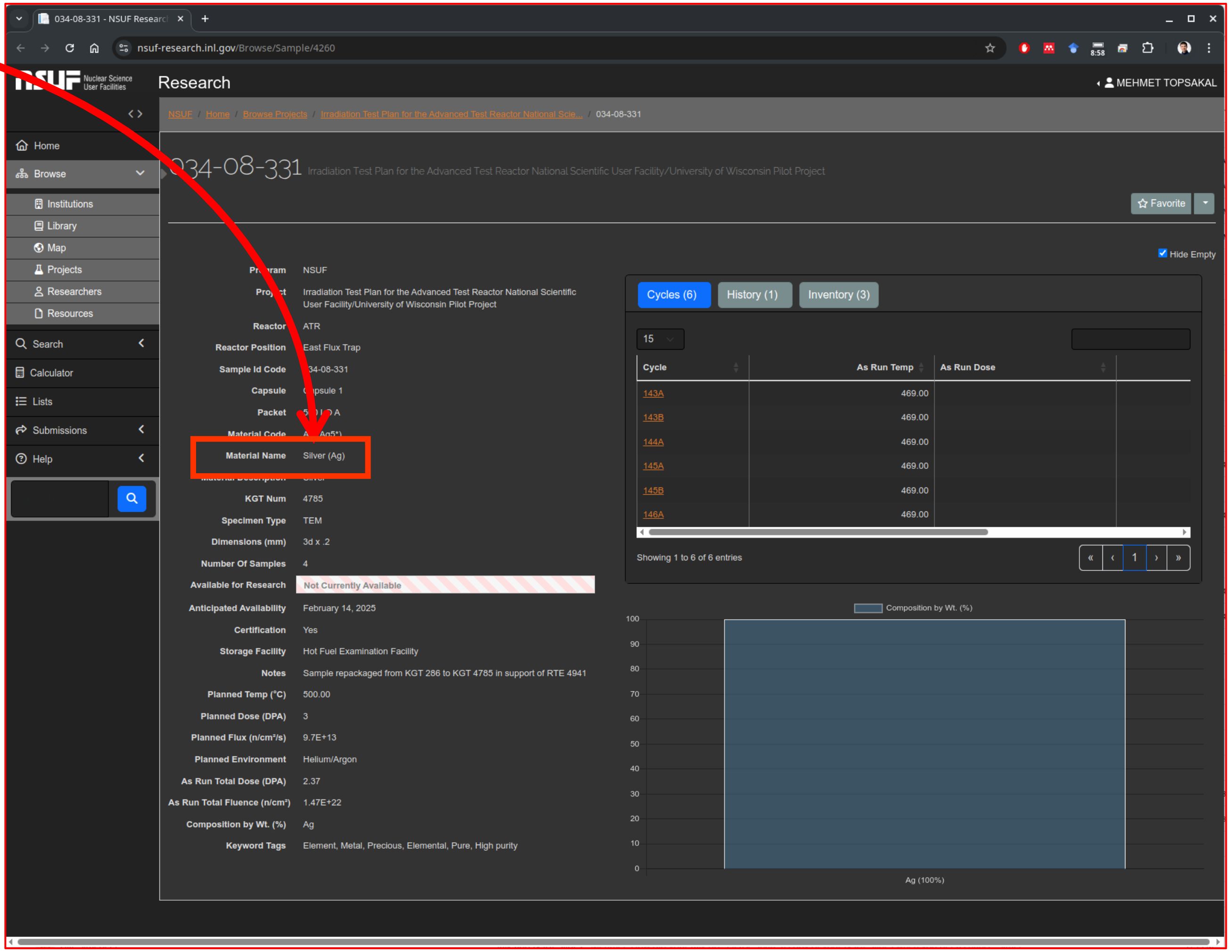


Advanced Test Reactor

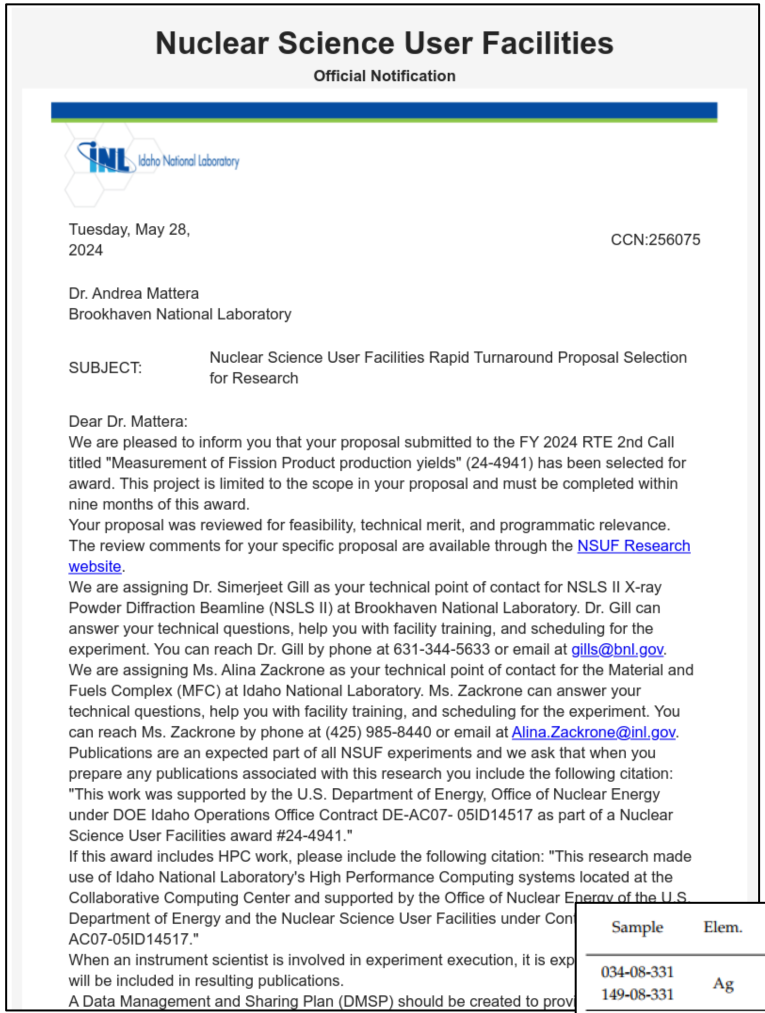


Nuclear Fuels and Materials Library (NFML) @ NSUF already has an Ag sample previously irradiated at ATR!





NSUF RTE # 24-4941 requested Ag, W, Hf, and Mo foils from NFML


Nuclear Science User Facilities
Official Notification

Idaho National Laboratory

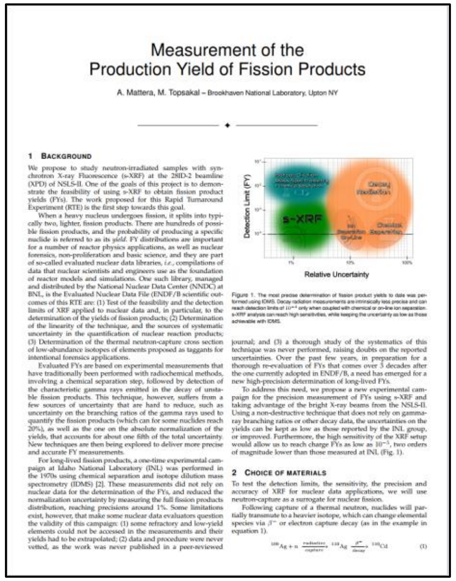
Tuesday, May 28, 2024

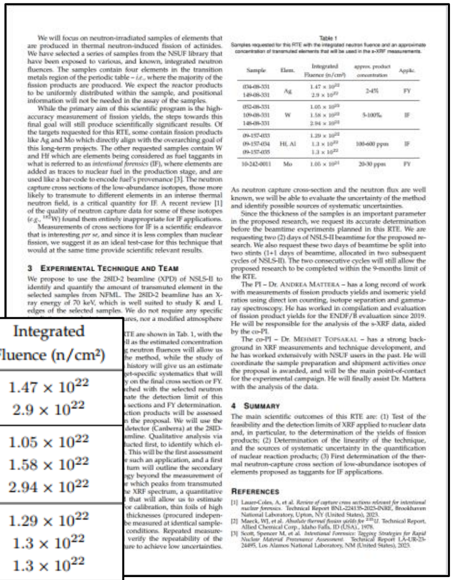
CCN:256075

Dr. Andrea Mattera
Brookhaven National Laboratory

SUBJECT: Nuclear Science User Facilities Rapid Turnaround Proposal Selection for Research

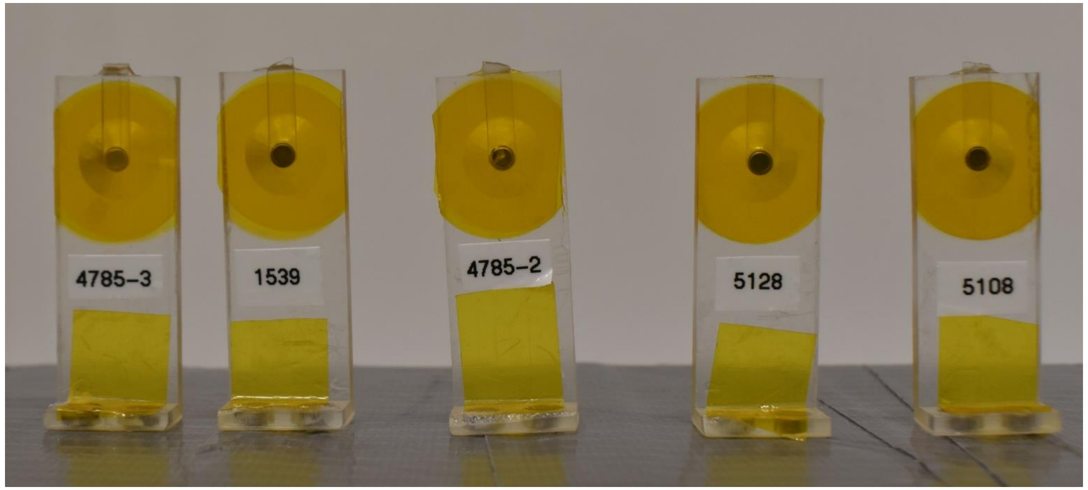
Dear Dr. Mattera:
We are pleased to inform you that your proposal submitted to the FY 2024 RTE 2nd Call titled "Measurement of Fission Product production yields" (24-4941) has been selected for award. This project is limited to the scope in your proposal and must be completed within nine months of this award.
Your proposal was reviewed for feasibility, technical merit, and programmatic relevance. The review comments for your specific proposal are available through the NSUF Research website.
We are assigning Dr. Simerjeet Gill as your technical point of contact for NSLS II X-ray Powder Diffraction Beamline (NSLS II) at Brookhaven National Laboratory. Dr. Gill can answer your technical questions, help you with facility training, and scheduling for the experiment. You can reach Dr. Gill by phone at 631-344-5633 or email at gills@bnl.gov.
We are assigning Ms. Alina Zackrone as your technical point of contact for the Material and Fuels Complex (MFC) at Idaho National Laboratory. Ms. Zackrone can answer your technical questions, help you with facility training, and scheduling for the experiment. You can reach Ms. Zackrone by phone at (425) 985-8440 or email at Alina.Zackrone@inl.gov.
Publications are an expected part of all NSUF experiments and we ask that when you prepare any publications associated with this research you include the following citation: "This work was supported by the U.S. Department of Energy, Office of Nuclear Energy under DOE Idaho Operations Office Contract DE-AC07- 05ID14517 as part of a Nuclear Science User Facilities award #24-4941."
If this award includes HPC work, please include the following citation: "This research made use of Idaho National Laboratory's High Performance Computing systems located at the Collaborative Computing Center and supported by the Office of Nuclear Energy of the U.S. Department of Energy and the Nuclear Science User Facilities under Con[...] AC07-05ID14517."
When an instrument scientist is involved in experiment execution, it is exp[...] will be included in resulting publications.
A Data Management and Sharing Plan (DMSP) should be created to prov[...]

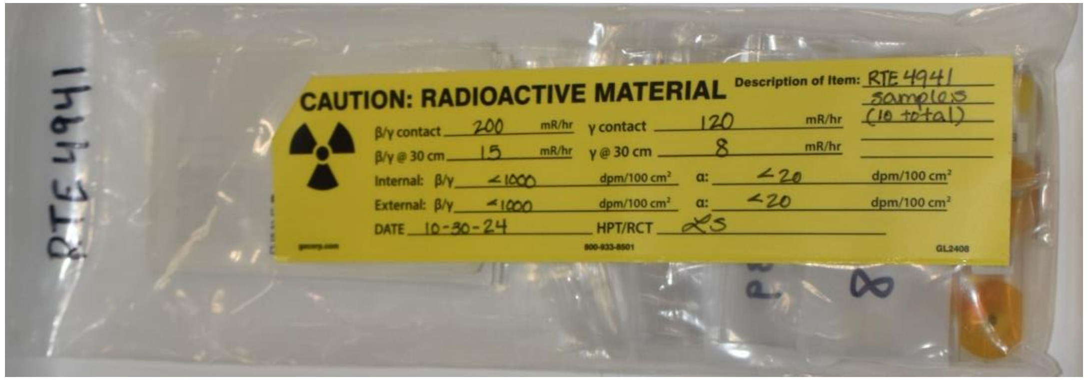




Sample	Elem.	Integrated Fluence (n/cm²)
034-08-331	Ag	1.47×10^{22}
149-08-331		2.9×10^{22}
052-08-331	W	1.05×10^{22}
109-08-331		1.58×10^{22}
148-08-331		2.94×10^{22}
09-157-033	Hf, Al	1.29×10^{22}
09-157-034		1.3×10^{22}
09-157-035		1.3×10^{22}
10-242-0011	Mo	1.05×10^{21}

As prepared samples @ INL through NSUF





Special thanks to

Alina Montrose
Experiment Manager
Nuclear Science User Facilities
Post-Irradiation Examination

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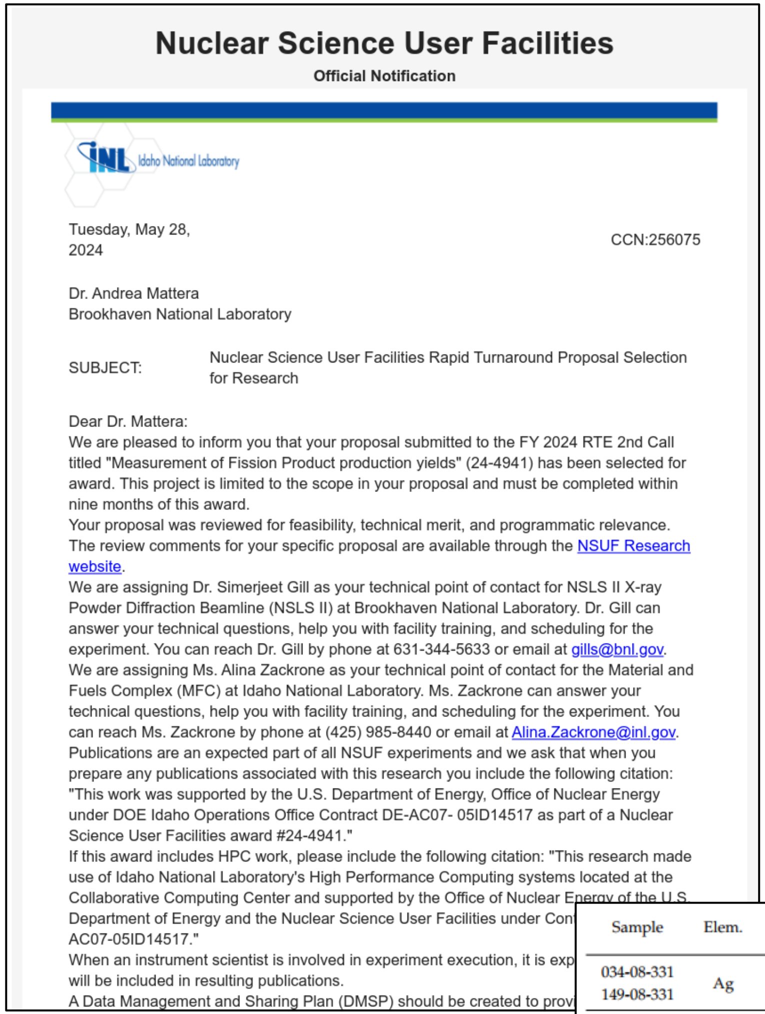

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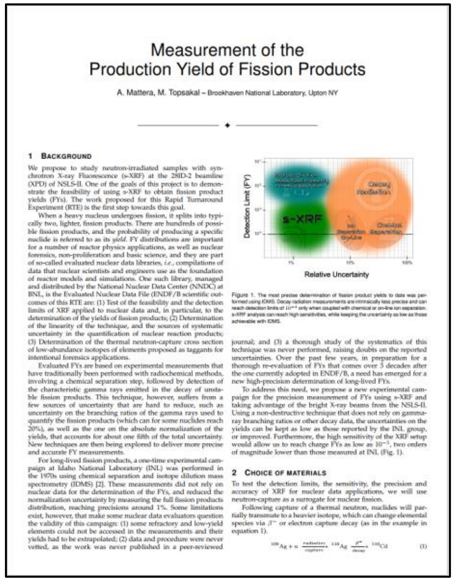
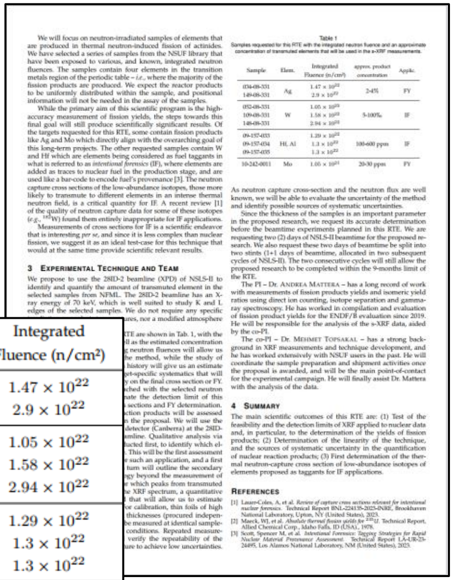


Measurement of the Production Yield of Fission Products

A. Mattera, M. Topsakal – Brookhaven National Laboratory, Upton NY

1 Background

2 Choice of Materials



3 Experimental Technique and Team

4 Summary

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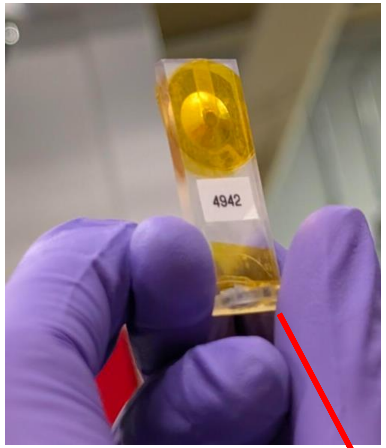
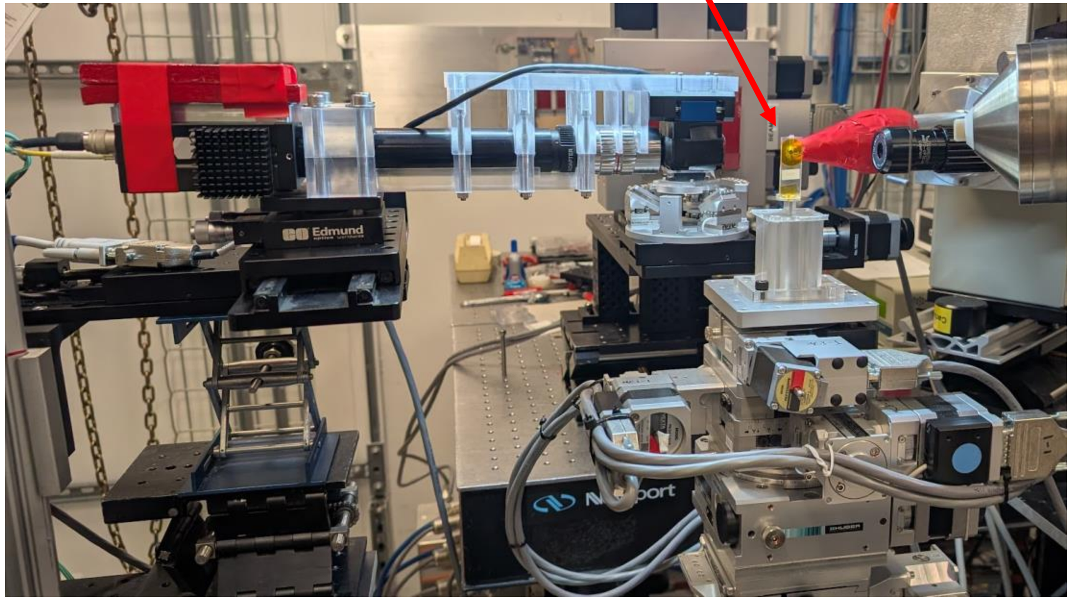
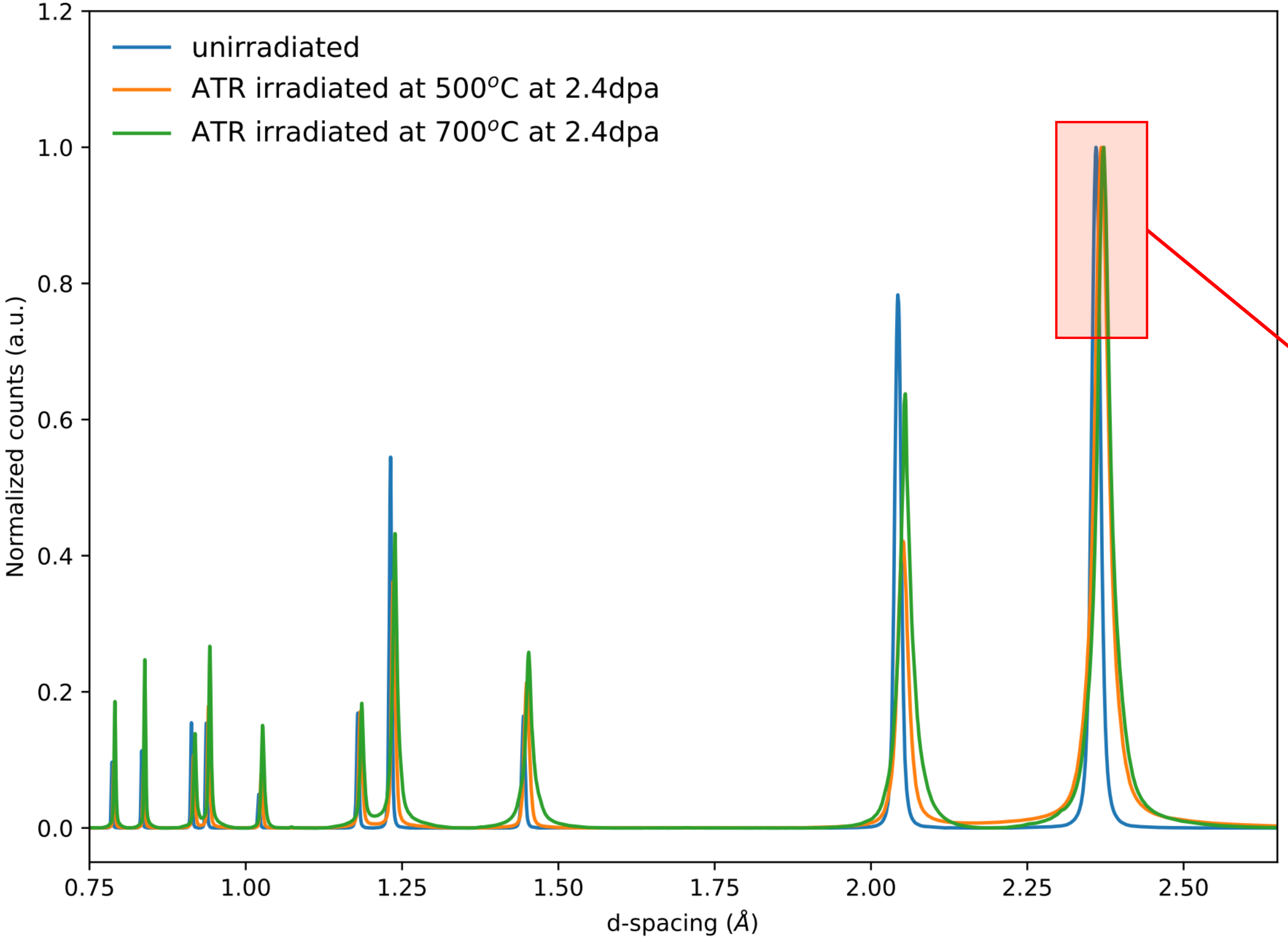
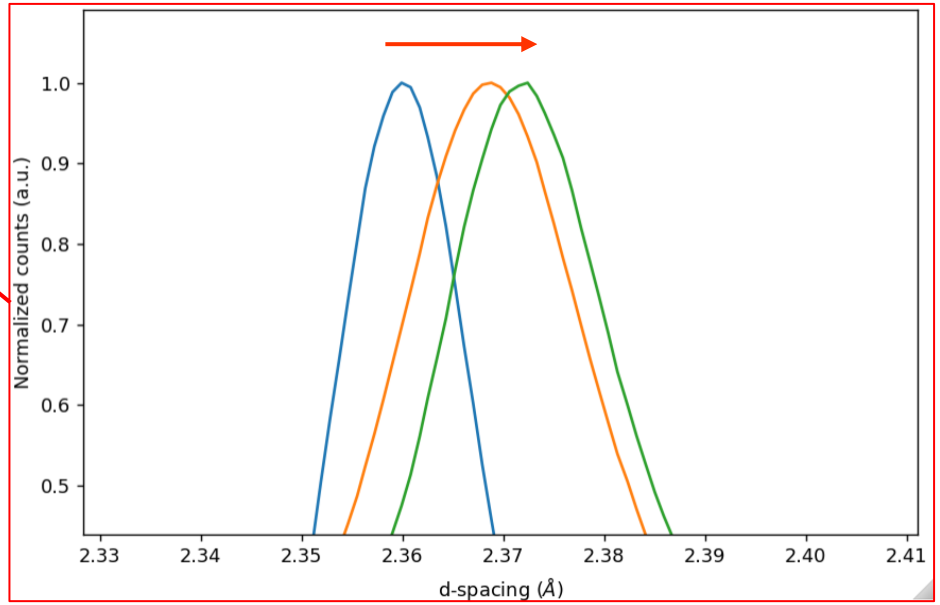




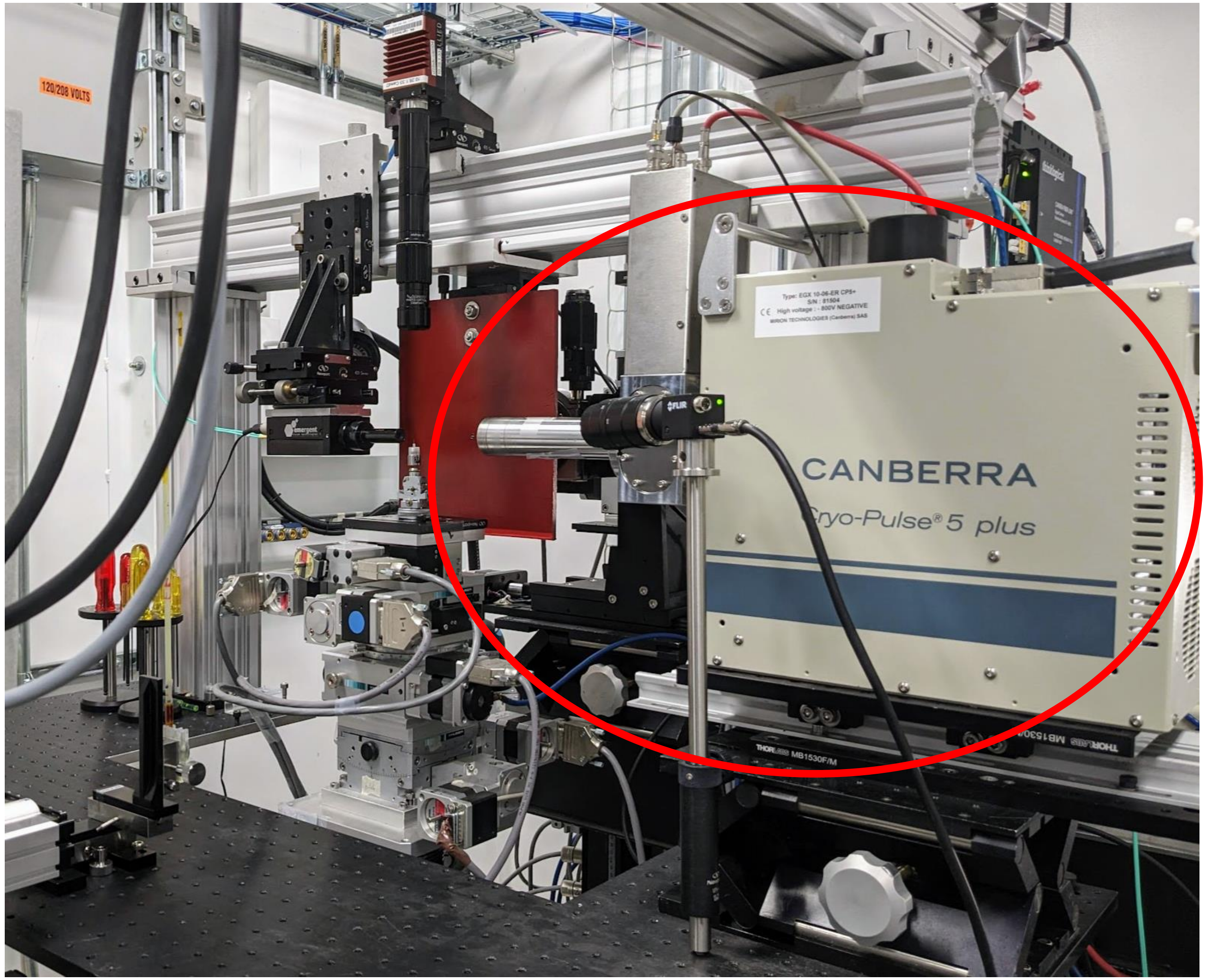


Dramatic changes with ATR irradiation!!!

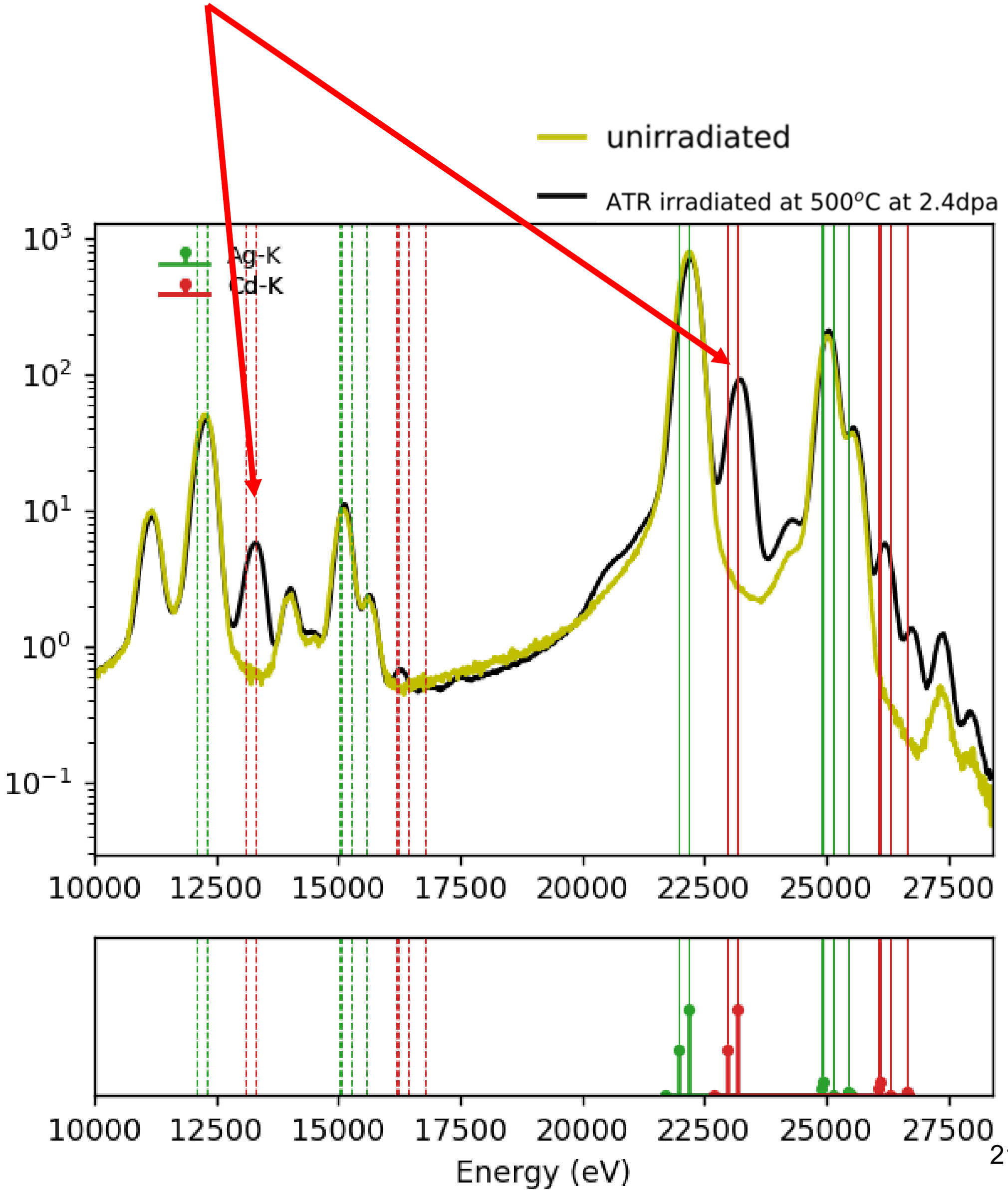


but why??

XRF capability provides additional “multi-modal information” as elemental composition

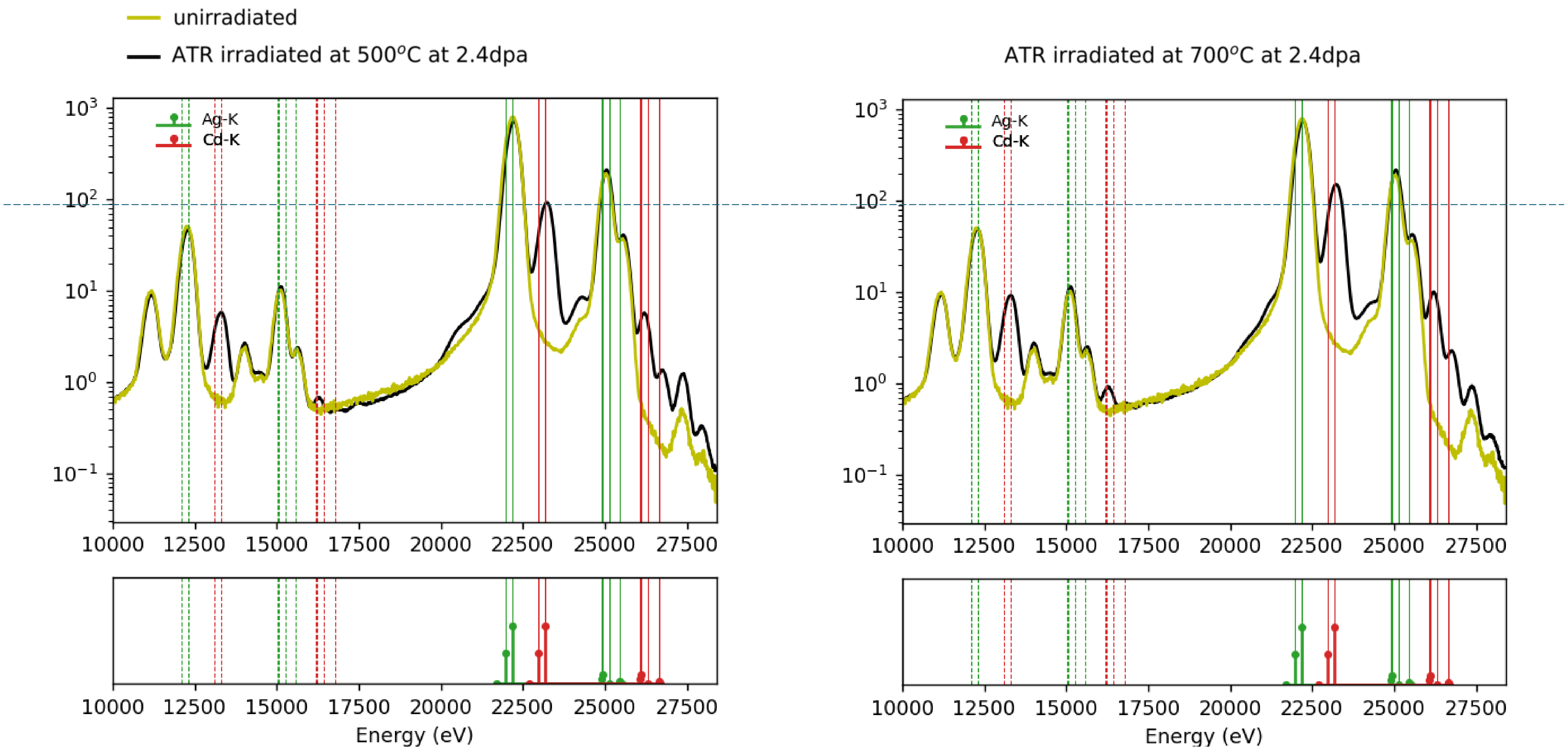


There are definitely Cd in these ATR irradiated Ag samples

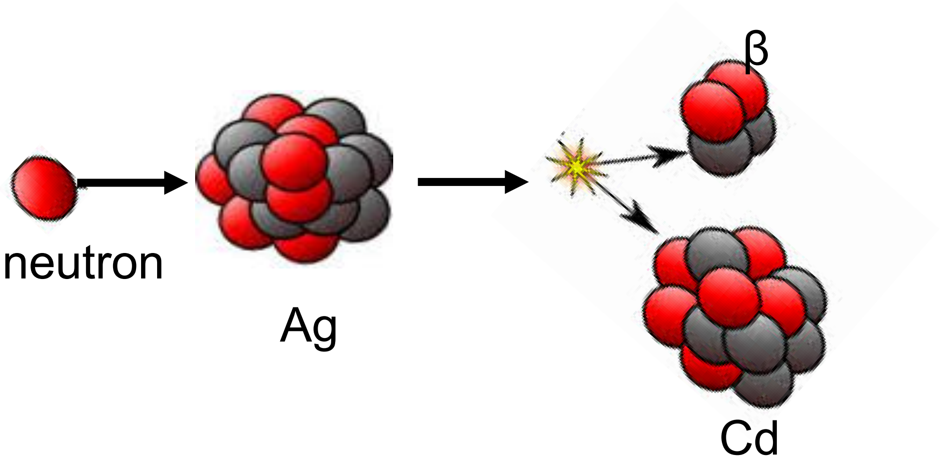


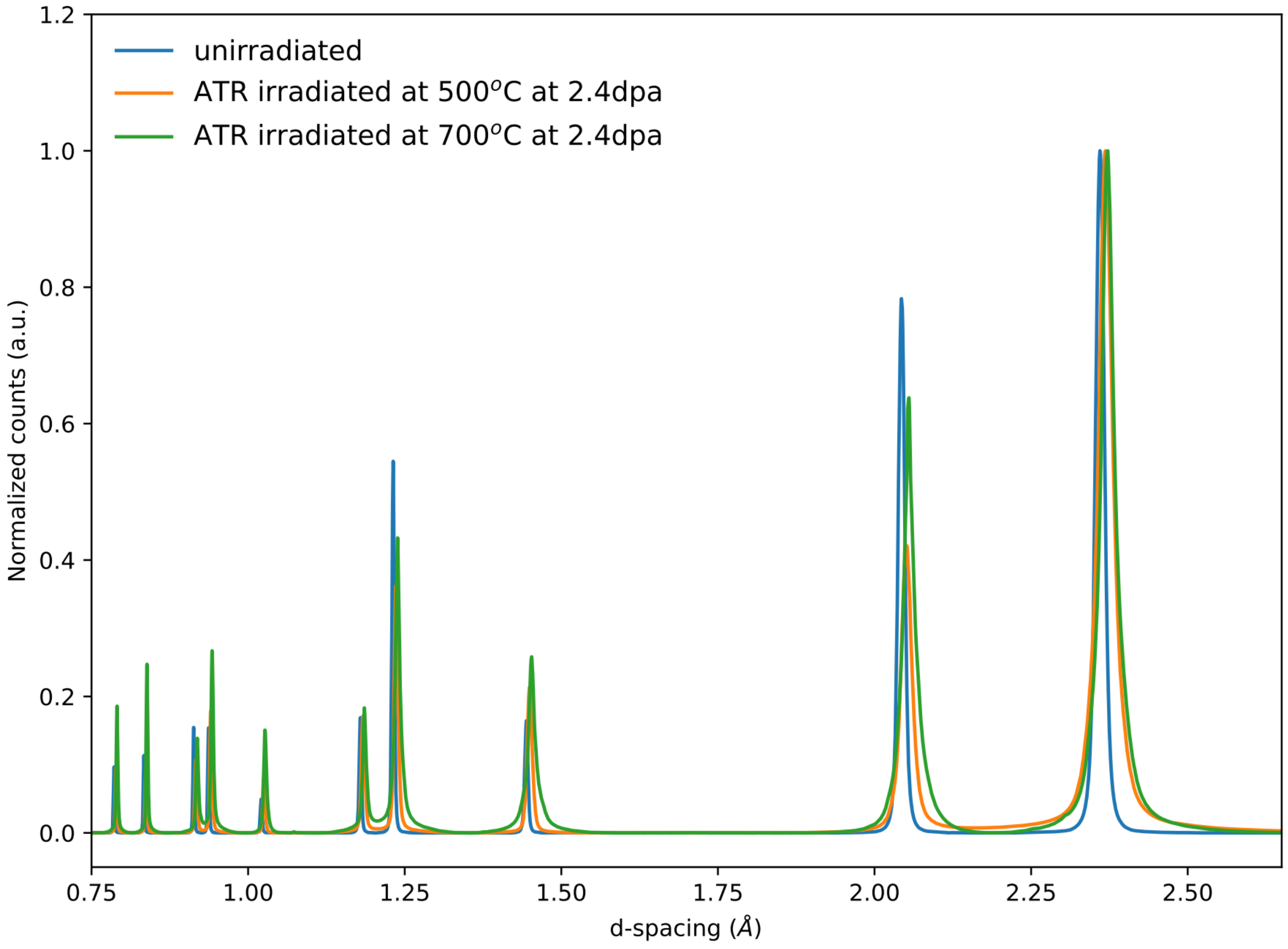
XRF capability provides additional “multi-modal information” as elemental composition

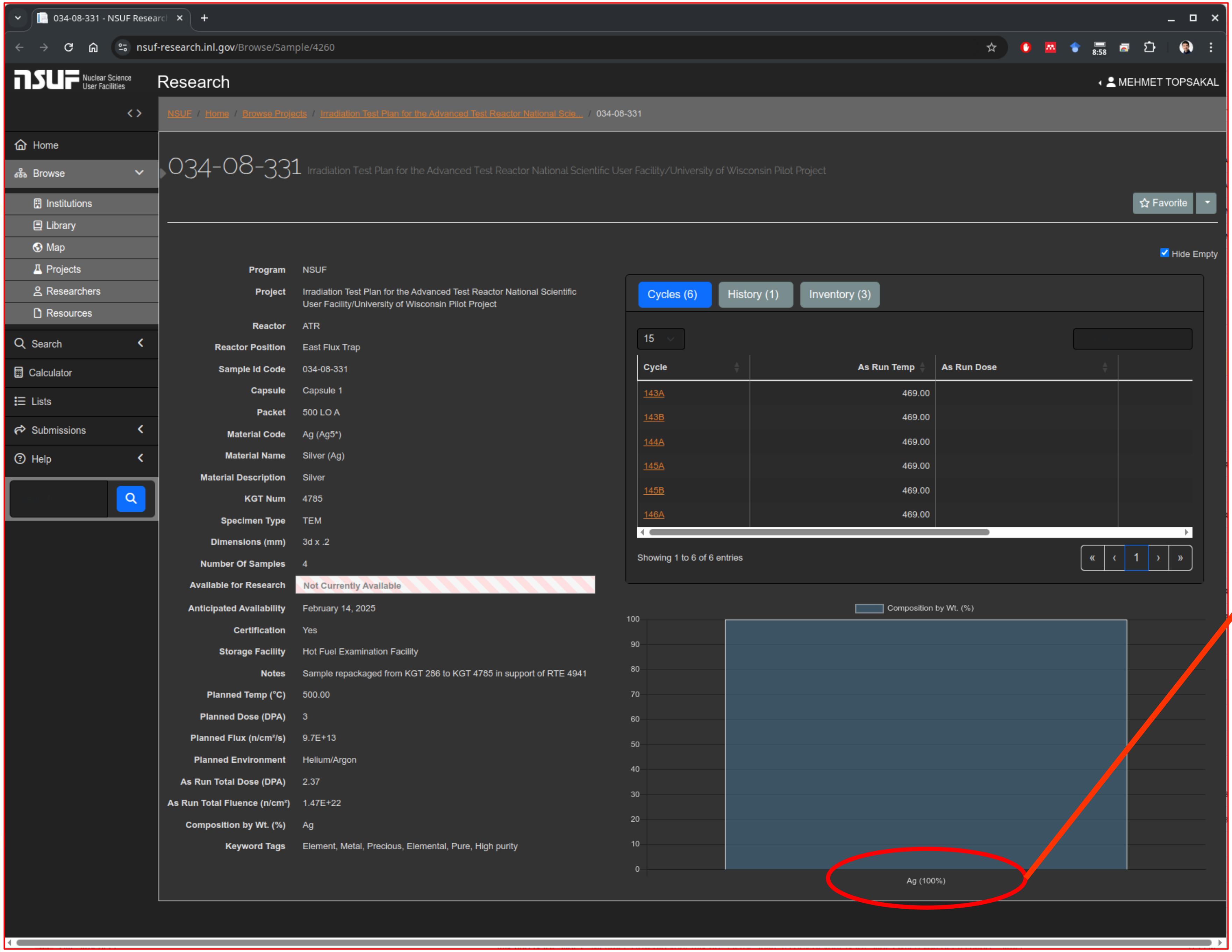
and Cd concentration increases with temperature



Transmutation of some Ag elements into Cd induces changes in lattice constant, hence d-spacing observed in XRD







This is NOT true anymore

Outline of this talk

- Introduction to NSUF facility at NSLS-II
- Capability upgrades
- Plans for future

How can we make BNL a better place for nuclear science community?

We can upgrade lab resources at BNL

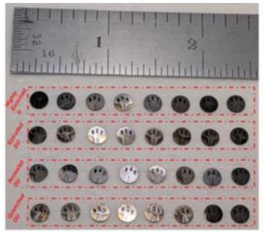
Problems:

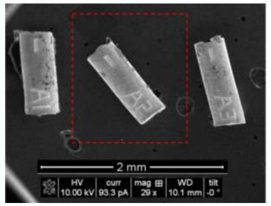
- Only short-term storage for NSUF samples.
- Far away from NSLS-II. Government vehicle is needed for sample transportation to/from NSLS-II.
- No sample preparation is allowed.

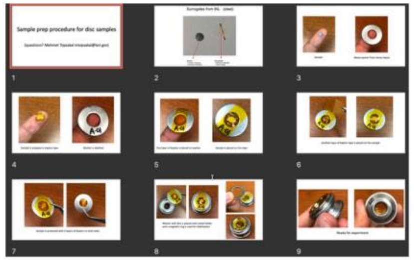
We can improve the way we support NSUF experiments at NSLS-II.

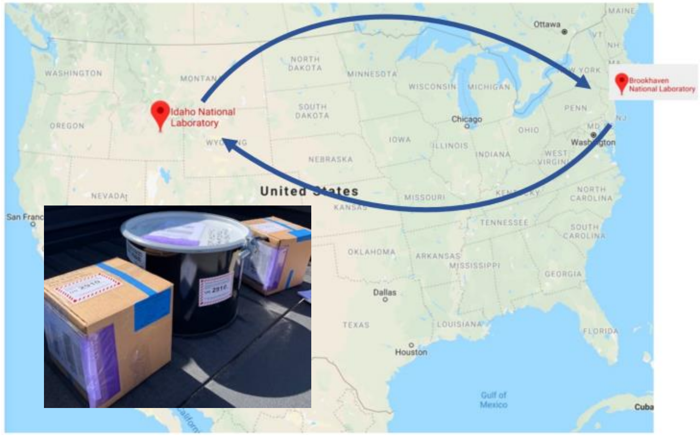
current workflow of NSUF experiments at NSLS-II can be illustrated as below

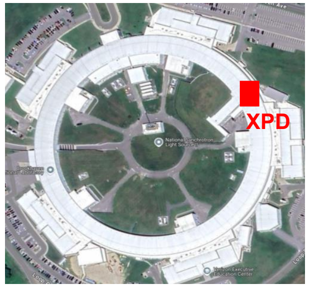
Initial samples → Sample preparation @ INL → Sample shipment from INL to BNL → Experiment at XPD beamline

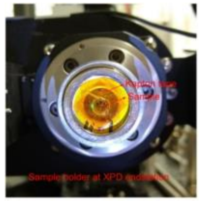


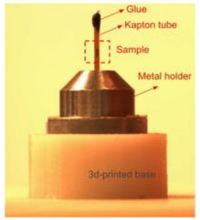


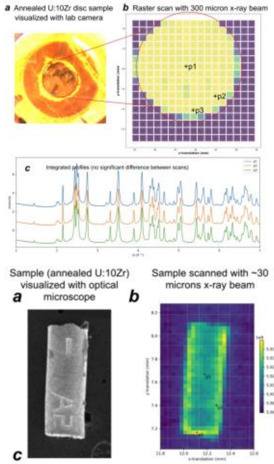












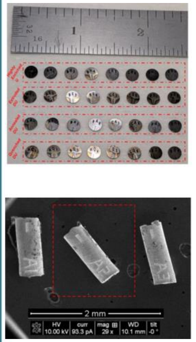
Takes usually ***a year!***

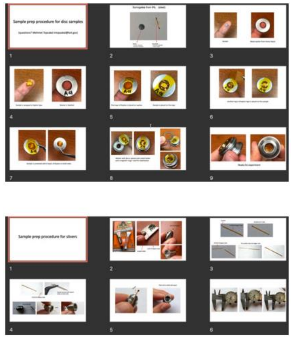
Takes usually ***2-3 days!***

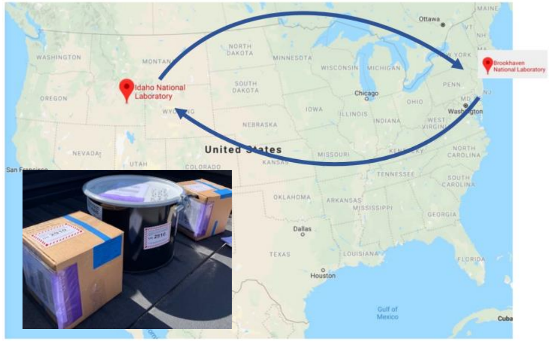
Despite all hard work spent on making samples ready for beamtime, samples go back to INL at the end of 2-3 days of beamtime without being characterized at other advanced NSLS-II beamlines.

We are considering to build a lab at NSLS-II specific for nuclear materials
Detailed characterization of samples can advance our understanding of irradiated materials

Initial samples → Sample preparation @ INL → Sample shipment from INL to BNL →





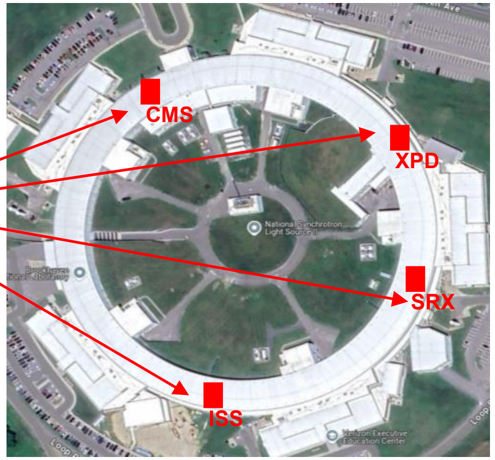


a new lab at NSLS-II

with lab-based
pre-characterization tools

→ Initial characterization and temporary storage →

Advanced characterization
at various NSLS-II beamlines

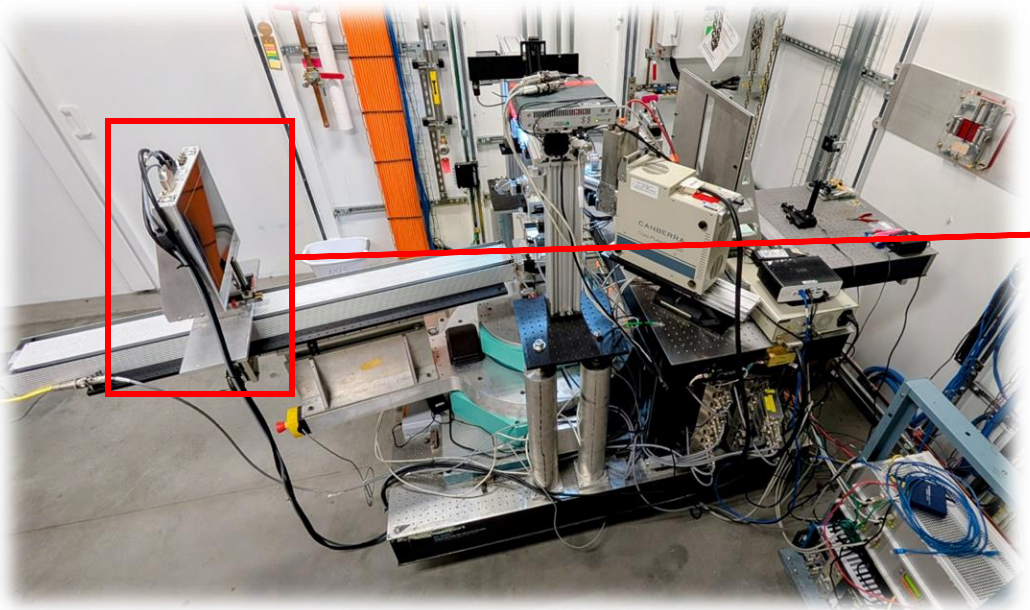


New lab at NSLS-II will enable us to do a pre-characterization and temporary storage of nuclear samples before synchrotron characterization

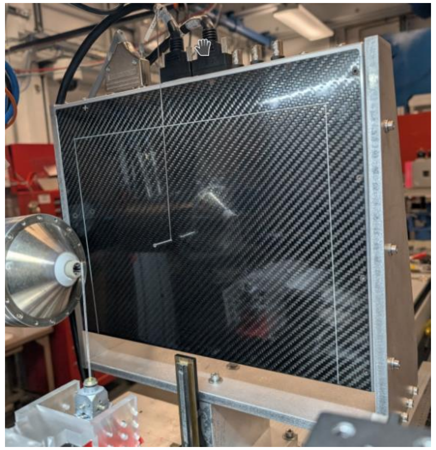
How can we make BNL a better place for nuclear science community?

>>>We can upgrade the existing resources at XPD beamline for faster data collection

Enhancing existing equipment at 28ID-2-D to enable faster and efficient data collection



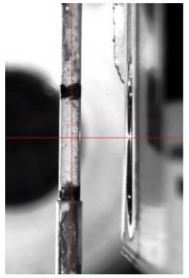
Current XRD detector

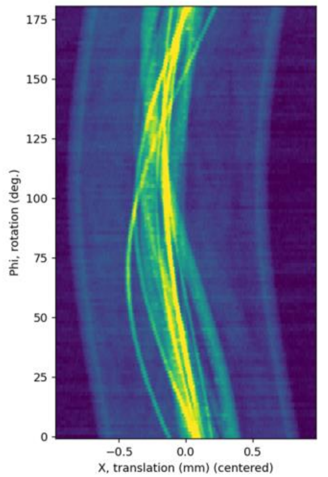


20 Hz frame rate

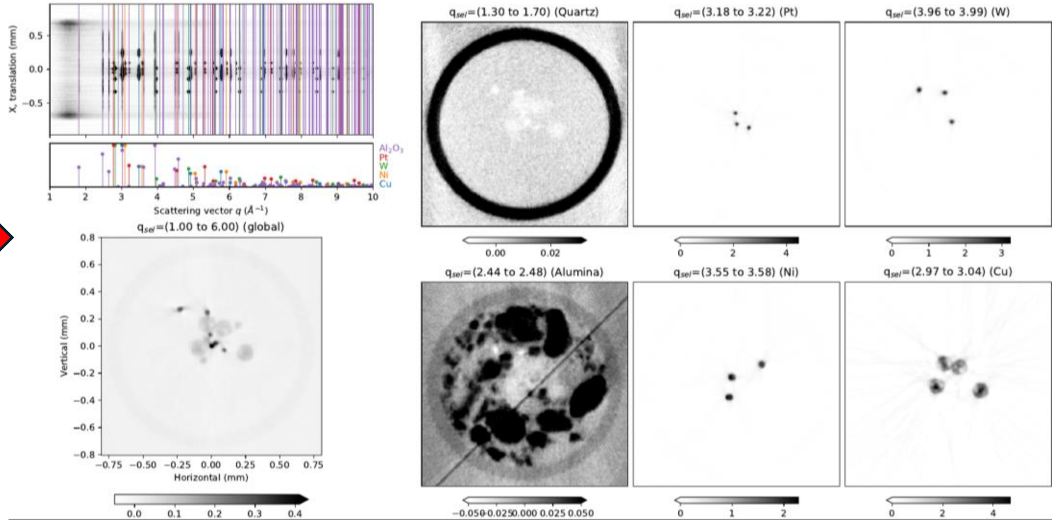
Better XRD detector

500 Hz frame rate





~480 frames were collected at each translation step (one frame every 6 microns sample travel). Coloring in above map is from total counts on detector.

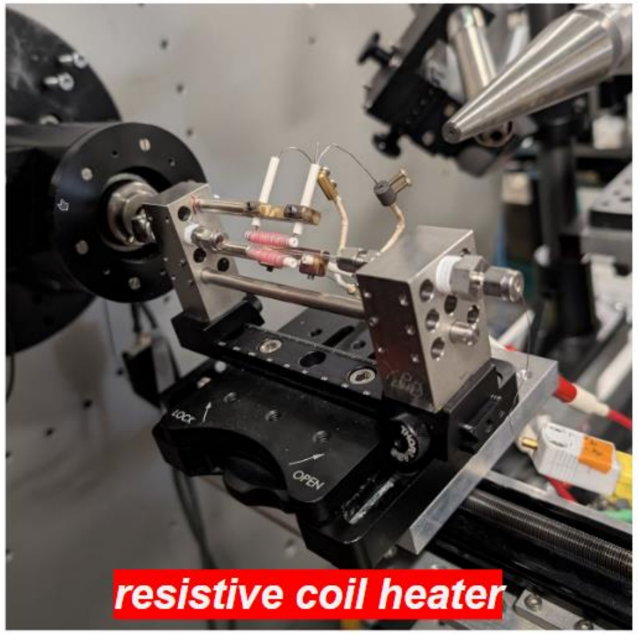


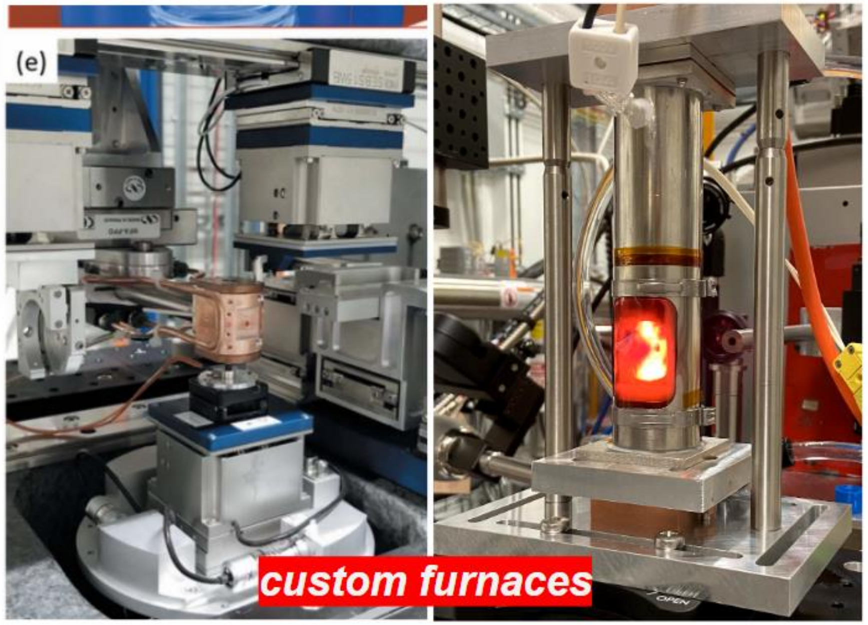
Utilized beamtime with:

- **current detector: 3 hours**
- **better detector: ~10 minutes**

In addition to upgrading the detector, we are considering to update in-situ heating capabilities for studying nuclear materials.

Conventional heating systems that are currently available at NSLS-II are shown below.



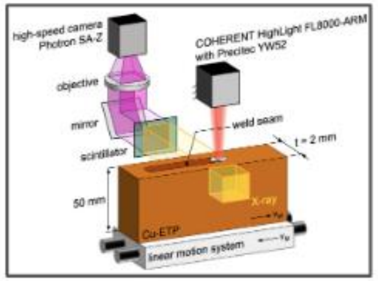


Some of drawbacks of all these systems are:

- they can't go beyond ~1000°C which is well below Tungsten melting point of 3422°C
- most of the generated heat is exposed to air and beamline components
- some are not suitable for hard-x-ray tomography (XCT, XRD-CT)

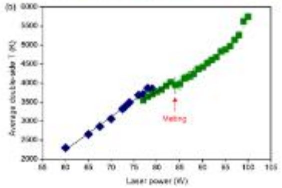
We are planning to develop a new sample-heating system that will be based on lasers for rapid and ultra-high T.

@ ESRF - The European Synchrotron Radiation Facility



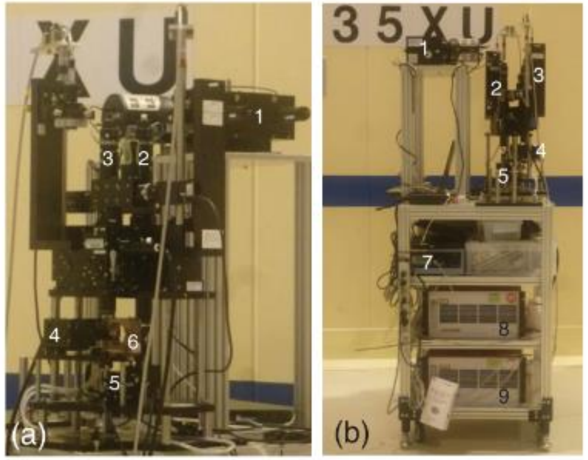
International Journal of Machine Tools & Manufacture 204 (2025) 104224

@ APS - Advanced Photon Source



Review of Scientific Instruments 86, 072201 (2015)

@ SPring-8 - Japan synchrotron



Rev. Sci. Instrum. 84, 113902 (2013)

An established setup at APS that we can get inspirations

REVIEW OF SCIENTIFIC INSTRUMENTS **86**, 072201 (2015)

New developments in laser-heated diamond anvil cell with *in situ* synchrotron x-ray diffraction at High Pressure Collaborative Access Team

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An overview of the *in situ* laser heating system at the High Pressure Collaborative Access Team, with emphasis on newly developed capabilities, is presented. Since its establishment at the beamline 16-ID-B a decade ago, laser-heated diamond anvil cell coupled with *in situ* synchrotron x-ray diffraction has been widely used for studying the structural properties of materials under simultaneous high pressure and high temperature conditions. Recent developments in both continuous-wave and modulated heating techniques have been focusing on resolving technical issues of the most challenging research areas. The new capabilities have demonstrated clear benefits and provide new opportunities in research areas including high-pressure melting, pressure-temperature-volume equations of state, chemical reaction, and time resolved studies. © *2015 AIP Publishing LLC.* [http://dx.doi.org/10.1063/1.4926895]

I. INTRODUCTION

Laser-heated diamond anvil cell (LHDAC) coupled with the *in situ* synchrotron x-ray diffraction (XRD) is a unique and powerful experimental technique for studying a broad range of material properties under extreme conditions up to megabars of pressure and several thousand degrees Kelvin of temperature. Over the last decade, this technique has evolved into a routinely used and productive experimental method at synchrotron beamlines, leading to numerous major scientific advances and a large expansion of high-pressure research in physics, chemistry, geoscience, and materials science.[1–8] One of the main applications of continuous wave laser heating (CWLH) has been the use of high temperature for overcoming kinetic barriers to phase transformation and for enabling new materials synthesis at high pressure. Thus, technical developments have emphasized long-term system stability with heating duration in a typical experiment lasting from minutes to hours.[9,10,15] Such long term temperature stability of the CWLH has made possible many studies of phase transitions,[11] materials synthesis,[6] and sample annealing for equations of state (EOS) measurement.[12] High pressure melting studies using synchrotron x-ray have been complicated by several issues including melt containment, temperature gradient, chemical reactions, and maintaining the exact alignment of the melt volume and the x-ray beam. Pressure-volume-temperature (P-V-T) EOS study is another challenging area that requires the exact alignment of heating, x-ray and temperature measurement positions at all the time during the experiment, which is not always guaranteed in the conventional systems commonly used at synchrotron beamlines.

In recent years, modulated pulse laser heating is being increasingly used for high pressure research. From the technical perspective, LHDAC in short time scale has several potential advantages. (1) It reduces the exposure of cell assembly to high temperature conditions. This helps to maintain the cell assembly's structure integrity and stability, thus improves the consistency and quality of experimental measurements and increases the potential for reaching higher pressure and temperature. (2) The short heating duration helps to suppress thermally activated chemical diffusion and reaction. (3) Heating at short time scales and improved temporal resolution of temperature measurement have been very useful for high-pressure melting studies, and studies of phase transition dynamics under high pressure. From the scientific perspective, current 3rd and 4th generation synchrotron sources provide opportunities to explore a wide range of physical and chemical phenomena occurring in increasingly short time scales down to femtosecond level. There is a need for LHDAC development to match the time scale of the light sources.

Our technical development objective in recent years has been to advance the experimental capabilities that address specific issues in the most challenging areas of high pressure research, specifically high-pressure melting and P-V-T EOS. In this paper, we summarize new techniques established at the beamline 16-ID-B in recent years, including (1) on-line heating-spot size adjustment to provide effective and uniform heating on various-sized samples in the diamond anvil cell; (2) mirror pinhole setup to allow direct viewing of the temperature sampling area relative to the heating area and x-ray beam, and online adjustment to ensure the ideal alignment for reliable experimental measurements; and (3) modulated laser heating technique synchronized with XRD, temperature measurement, and thermal imaging for high pressure melting and time-resolved studies of phase transition dynamics under high PT conditions.

II. SYSTEM OVERVIEW

The integrated system of LHDAC with *in situ* XRD is located at High Pressure Collaborative Access Team's

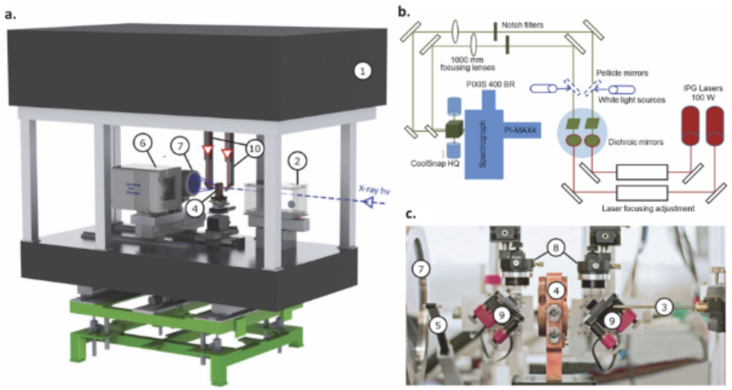
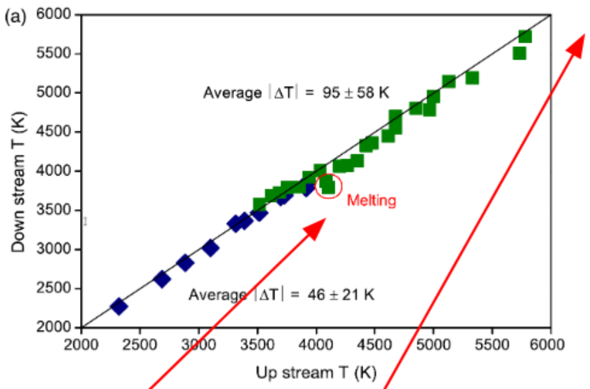


FIG. 1. The integrated system of laser-heated DAC with *in situ* XRD. (a) A computer-aided-design (CAD) drawing of the laser heating experiment table in 16-ID-B, (b) a schematic drawing of the optical system on the top experiment table (labeled as ①), and (c) an image of the setup around DAC. The numbered label ② denotes KB mirrors for XR focusing, ③ XR clean-up pinhole, ④ diamond anvil cell, ⑤ XR beam stopper, ⑥ Pilatus 1M XR detector, ⑦ MAR CCD XR detector ⑧ the apochromatic objective lenses, ⑨ coated amorphous carbon mirrors, and ⑩ movable holders for laser heating system components ⑤ and ⑨.



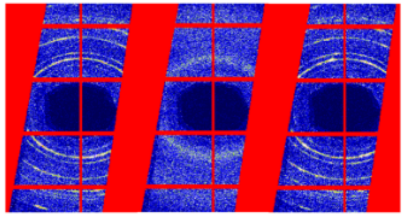
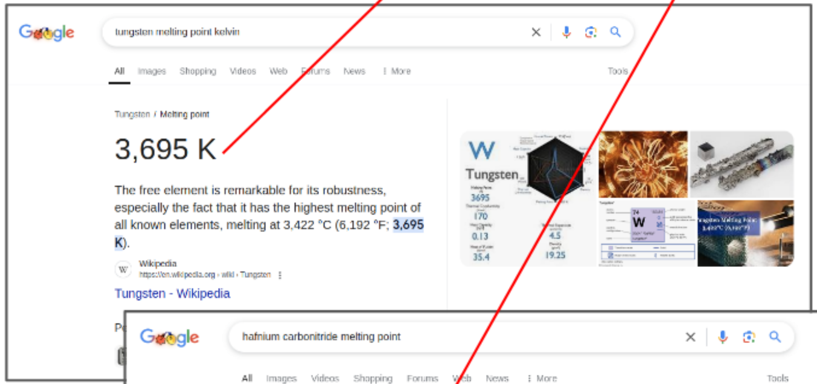
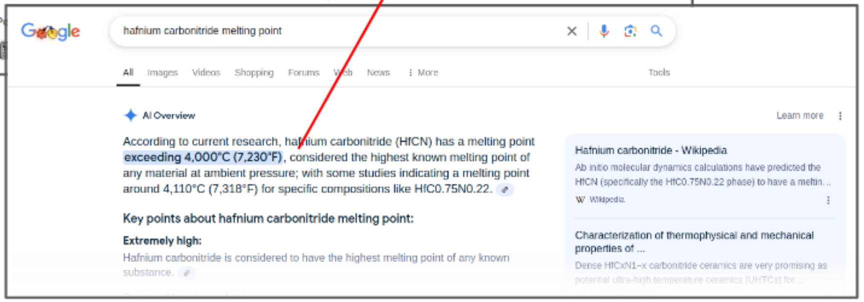
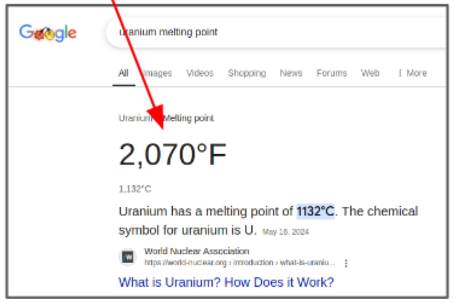


FIG. 10. Melting of uranium at high pressure captured using the pulse synchronization displayed in Figure 9(b), before (left), during (middle), and after (right) the heating pulse (in collaboration with Hyunchae Cynn).







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