

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

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@BrookhavenLab

Outline of this talk

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

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Who are we?

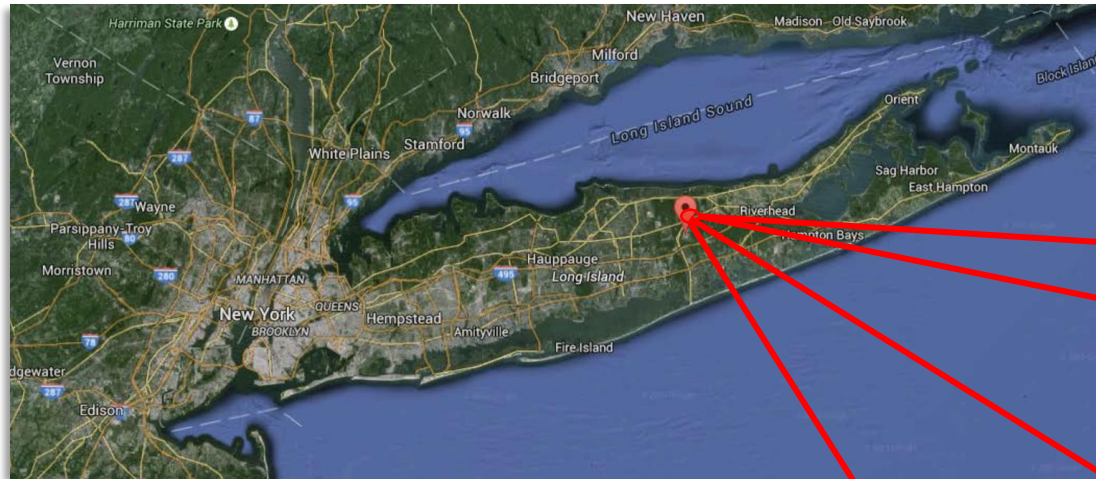
NSUF Capabilities Offer Research Opportunities

Neutron Irradiations	Ion Irradiations	Gamma Irradiations	Hot Cells & Shielded Cells	Low Activity Laboratories	Beamlines	High Performance Computing
 Idaho National Laboratory	 WISCONSIN UNIVERSITY OF WISCONSIN-MADISON	 Idaho National Laboratory	 Idaho National Laboratory	 Idaho National Laboratory	 BROOKHAVEN NATIONAL LABORATORY	 Idaho National Laboratory
 OAK RIDGE National Laboratory	 M 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	 PNL	 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	 ATM		 Westinghouse	 PNL		
 Sandia National Laboratories	 TEXAS A&M UNIVERSITY		 M UNIVERSITY OF MICHIGAN	 Los Alamos NATIONAL LABORATORY EST. 1943	 NC STATE UNIVERSITY	 M UNIVERSITY OF MICHIGAN
 SCK • CEN STUOIECENTRUM VOOR KERNEENERGIE CENTRE D'ETUDE DE L'ENERGIE NUCLEAIRE	 Lawrence Livermore National Laboratory		 SCK • CEN STUOIECENTRUM VOOR KERNEENERGIE CENTRE D'ETUDE DE L'ENERGIE NUCLEAIRE	 PURDUE UNIVERSITY		
				 WISCONSIN UNIVERSITY OF WISCONSIN-MADISON		

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.



- Located on Long Island
- Land: 5322 acres
- 319 buildings

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

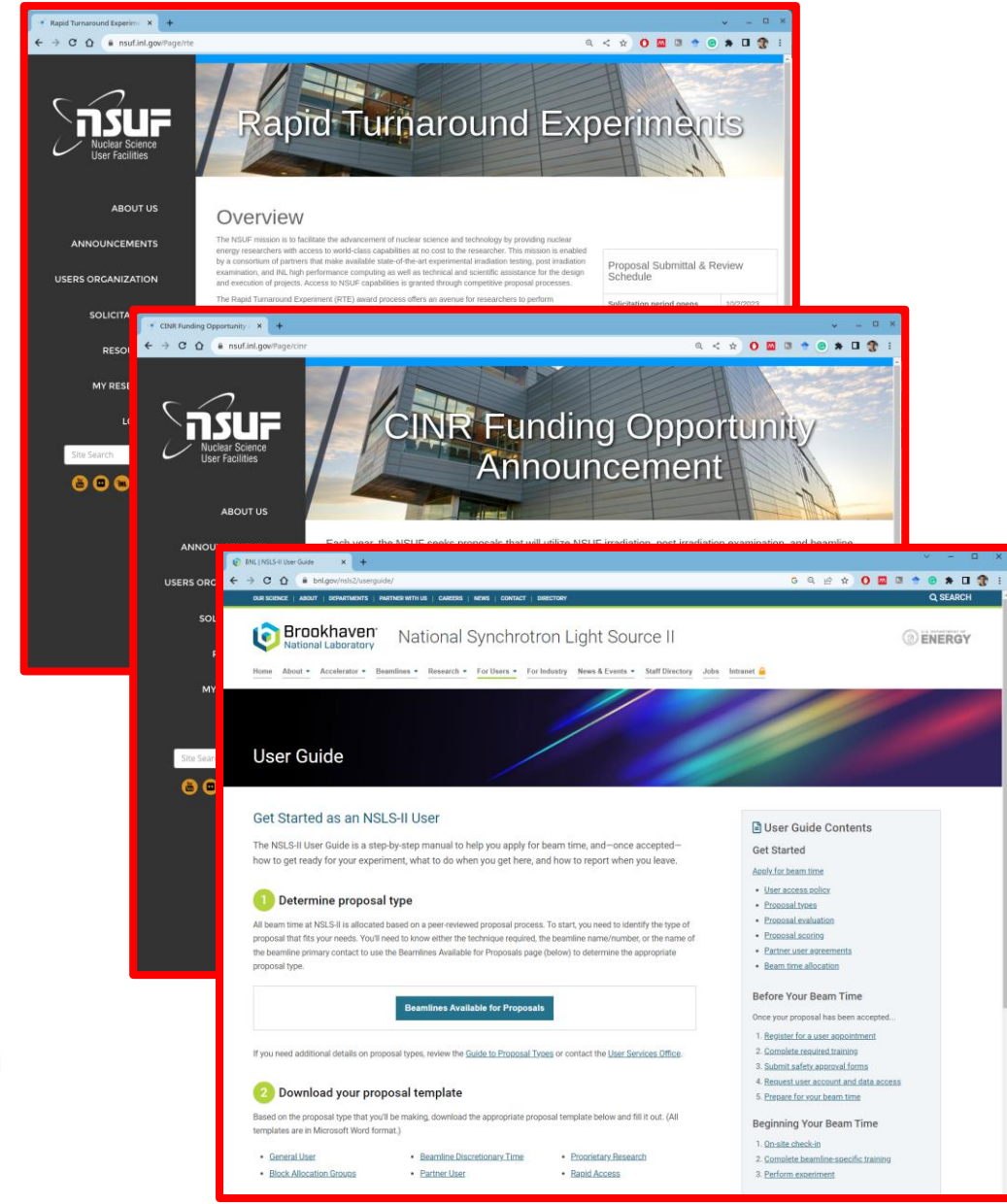
Who are we?

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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 OAK RIDGE National Laboratory	 UNIVERSITY OF MICHIGAN	 OAK RIDGE National Laboratory	 OAK RIDGE National Laboratory	 Center for Advanced Energy Studies	 Argonne NATIONAL LABORATORY	
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 OHIO STATE UNIVERSITY	 TEXAS A&M UNIVERSITY	 UNIVERSITY OF MICHIGAN	 UNIVERSITY OF MICHIGAN	 NC STATE UNIVERSITY	 UNIVERSITY OF MICHIGAN	
 Sandia National Laboratories	 Lawrence Livermore National Laboratory	 SCK-CEN STUDIECENTRUM VOOR KERNENERGIE CENTRE D'ETUDE DE L'ENERGIE NUCLEAIRE	 SCK-CEN STUDIECENTRUM VOOR KERNENERGIE CENTRE D'ETUDE DE L'ENERGIE NUCLEAIRE	 PURDUE UNIVERSITY	 UNIVERSITY OF FLORIDA	
 SCK-CEN STUDIECENTRUM VOOR KERNENERGIE CENTRE D'ETUDE DE L'ENERGIE NUCLEAIRE				 WISCONSIN UNIVERSITY OF WISCONSIN		

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Rapid turnaround experiment (RTE) proposals are easy!



Rapid Turnaround Experiments

- ABOUT US
- ANNOUNCEMENTS
- USERS ORGANIZATION
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- RESOURCES
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Site Search



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.

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:

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

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

1 BACKGROUND

We propose to study neutron-irradiated samples with synchrotron X-ray Fluorescence (sXRF) at the 2B2D2 beamline (XPD of NSLS-II). One of the goals of this project is to demonstrate the feasibility of using sXRF to obtain fission product yields (FYs). The work proposed for this Rapid Turnaround Experiment (RTE) is the first step towards this goal.

When a heavy nucleus undergoes fission, it splits into typically two, lighter fission products. There are hundreds of possible fission products, and the probability of producing a specific nuclide is referred to as its yield. FY distributions are important for a number of nuclear physics applications, as well as nuclear forensics, non-proliferation and basic science, and they are part of so-called evaluated nuclear data libraries, i.e., compilations of data that nuclear scientists and engineers use as the foundation of reactor models and simulations. One such library, managed and distributed by the National Nuclear Data Center (NNDC) at BNL, is the Evaluated Nuclear Data File (ENDF/B) scientific outcomes of this RTE are: (1) Test of the feasibility and the detection limits of XRF applied to nuclear data and, in particular, to the determination of the yields of fission products; (2) Determination of the linearity of the technique, and the sources of systematic uncertainty in the quantification of nuclear reaction products; (3) Determination of the thermal neutron-capture cross section of low-abundance isotopes of elements proposed as ligands for intentional fission applications.

Evaluated FYs are based on experimental measurements that have traditionally been performed with radiochemical methods involving a chemical separation step, followed by detection of the characteristic gamma rays emitted in the decay of unstable fission products. This technique, however, suffers from a few sources of uncertainty that are hard to reduce, such as uncertainty on the branching ratios of the gamma rays used to quantify the fission products (which can for some nuclides reach 20%), as well as the one on the absolute normalization of the yields, that accounts for about one fifth of the total uncertainty. New techniques are thus being explored to deliver more precise and accurate FY measurements.

For long-lived fission products, a one-time experimental campaign at Idaho National Laboratory (INL) was performed in the 1970s using chemical separation and isotope dilution mass spectrometry (IDMS) [2]. These measurements did not rely on nuclear data for the determination of the FYs, and reduced the normalization uncertainty by measuring the full fission product distribution, reaching precisions around 1%. Some limitations exist, however, that make some nuclear data evaluation question the validity of this campaign: (1) some refractory and low-yield elements could not be accessed in the measurements and their yields had to be extrapolated; (2) data and procedures were never vetted, as the work was never published in a peer-reviewed journal; and (3) a thorough study of the systematic of this technique was never performed, raising doubts on the reported uncertainties. Over the past few years, in preparation for a thorough re-evaluation of FYs that comes over 3 decades after the one currently adopted in ENDF/B, a need has emerged for a new high-precision determination of long-lived FYs.

To address this need, we propose a new experimental campaign for the precision measurement of FYs using sXRF and taking advantage of the bright X-ray beams from the NSLS-II. Using a non-destructive technique that does not rely on gamma-ray branching ratios or other decay data, the uncertainties on the yields can be kept as low as those reported by the INL group, or improved. Furthermore, the high sensitivity of the XRF setup would allow us to reach charge FYs as low as 10^{-5} , two orders of magnitude lower than those measured at INL (Fig. 1).

2 CHOICE OF MATERIALS

To test the detection limits, the sensitivity, the precision and accuracy of XRF for nuclear data applications, we will use neutron-capture as a surrogate for nuclear fission.

Following capture of a thermal neutron, nuclides will partially transmute to a heavier isotope, which can change elemental species via β^- or electron capture decay (as in the example in equation 1).

$$^{100}\text{Ag} + n \rightarrow \text{transmutation} \rightarrow ^{101}\text{Ag} \xrightarrow{\beta^-} ^{101}\text{Cd} \quad (1)$$

We will focus on neutron-irradiated samples of elements that are produced in thermal neutron-induced fission of actinides. We have selected a series of samples from the NNDC library that have been reported to various, and known, integrated neutron fluxes. The sample contain four elements in the transition metals range of the periodic table, i.e., where the majority of the fission products are produced. We expect the nuclear products to be uniformly distributed within the sample, and positional information will not be needed in the assay of the samples.

While the primary aim of this scientific program is the high-accuracy measurement of fission yields, the steps towards this final goal will still produce scientifically significant results. Of the targets requested for this RTE, some contain fission products like Ag and Mo which directly align with the overarching goal of this long-term project. The other requested samples contain W and Hf which are elements being considered as fuel ligands in what is referred to as an intentional fission (IF), where elements are added as tracers to nuclear fuel in the production stage, and are used like a bar-code to encode fuel provenance [3]. The neutron capture cross sections of the low-abundance isotopes, those more likely to transmute to different elements in an intense thermal neutron field, is a critical quantity for IF. A recent review [1] of the quality of neutron capture data for some of these isotopes (e.g., ^{187}W) found them entirely inappropriate for IF applications. Measurements of cross sections for IF is a scientific endeavor that is interesting per se, and since it is less complex than nuclear fission, we suggest it as an ideal test-bed for this technique that would at the same time provide scientific relevant results.

3 EXPERIMENTAL TECHNIQUE AND TEAM

We propose to use the 2B2D2 beamline (XPD) of NSLS-II to identify and quantify the amount of transmuted element in the selected samples from NNDC. The 2B2D2 beamline has an X-ray energy of 20 keV, which is well suited to study K and L edges of the selected samples. We do not require any specific irradiation or analysis temperatures, nor a modified atmosphere for the experiment.

The targets selected for this RTE are shown in Tab. 1, with the integrated neutron fluence as well as the estimated concentration of the reaction products. Varying neutron fluences will allow us to investigate the linearity of the method, while the study of samples with similar irradiation history will give us an estimate of the repeatability and the target-specific systematic that will contribute to the final uncertainty on the final cross-section or FY. The range of concentrations reached with the selected neutron fluences will allow us to estimate the detection limit of this technique when applied to cross sections and FY determination.

The concentration of the reaction products will be assessed with sXRF for samples listed in the proposal. We will use the recently commissioned Ge XRF detector (Canberra) at the 2B2D2-2-D evaluation of the XPD beamline. Qualitative analysis via XRF measurements will be conducted first, to identify which elements are present in the sample. This will be the first assessment of the viability of this method for such an application, and a first test of its sensitivity - which in turn will define the secondary developments of this methodology beyond the measurement of FYs. For those concentrations for which peaks from transmuted elements are clearly visible in the XRF spectrum, a quantitative analysis will also be conducted that will allow us to estimate the amount of trace elements. For calibration, thin foils of high-purity metals with well-known thicknesses (precisely independent from this RTE work) will be measured at identical sample-to-detector distance and beam conditions. Repeated measurements of various samples will verify the repeatability of the method, which is a required feature to achieve low uncertainties.

Figure 1. The most precise determination of fission product yields to date was performed using IDMS. Decay radiation measurements are intrinsically less precise and can reach detection levels of 10^{-4} only when coupled with chemical or online ion separation. sXRF analysis can reach high sensitivities, while keeping the uncertainty as low as those achieved with IDMS.

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Table 1
Samples requested for this RTE with the integrated neutron fluence and an approximate concentration of transmuted elements that will be used in the sXRF analysis.

Sample	Elem.	Integrated Fluence (n/cm ²)	approx. product concentration	applic.
104048-331	Ag	1.47×10^{22}	2-6%	FY
104048-331	Ag	2.3×10^{22}		
024048-331		1.01×10^{22}		
104048-331	W	1.58×10^{22}	5-10%	IF
104048-331		9.04×10^{21}		
06-157-053		1.29×10^{22}		
06-157-054	He, Al	1.3×10^{22}	100-600 ppm	IF
06-157-055		1.3×10^{22}		
10-242-0011	Mo	1.01×10^{22}	20-30 ppm	FY

As neutron capture cross-section and the neutron flux are well known, we will be able to evaluate the uncertainty of the method and identify possible sources of systematic uncertainties.

Since the thickness of the samples is an important parameter in the proposed research, we request its accurate determination before the beamtime experiments planned in this RTE. We are requesting two (2) days of NSLS-II beamtime for the proposed research. We also request these two days of beamtime be split into two shifts (1+1 days of beamtime, allocated in two subsequent cycles of NSLS-II). The two consecutive cycles will still allow the proposed research to be completed within the 9-months limit of the RTE.

The PI – Dr. ANDREA MATTERA – has a long record of work with measurements of fission products yields and isotopic yield ratios using direct ion counting, isotope separation and gamma-ray spectroscopy. He has worked in compilation and evaluation of fission product yields for the ENDF/B evaluation since 2019. He will be responsible for the analysis of the sXRF data, aided by the co-PI.

The co-PI – Dr. MIHAIL TOPSAKAL – has a strong background in XRF measurements and technique development, and he has worked extensively with NSUF users in the past. He will coordinate the sample preparation and shipment activities once the proposal is awarded, and will be the main point-of-contact for the experimental campaign. He will finally assist Dr. Mattera with the analysis of the data.

4 SUMMARY

The main scientific outcomes of this RTE are: (1) Test of the feasibility and the detection limits of XRF applied to nuclear data and, in particular, to the determination of the yields of fission products; (2) Determination of the linearity of the technique, and the sources of systematic uncertainty in the quantification of nuclear reaction products; (3) First determination of the thermal neutron-capture cross section of low-abundance isotopes of elements proposed as ligands for IF applications.

REFERENCES

[1] Lauer-Giles, A. et al. Review of capture cross section releases for intentional nuclear fission. Technical Report BNL-25110-2023-001, Brookhaven National Laboratory, Upton, NY (United States), 2023.

[2] March, W.L. et al. Idaho National Reactor Project for U.S. Technical Report, Allied Chemical Corp., Idaho Falls, ID (USA), 1979.

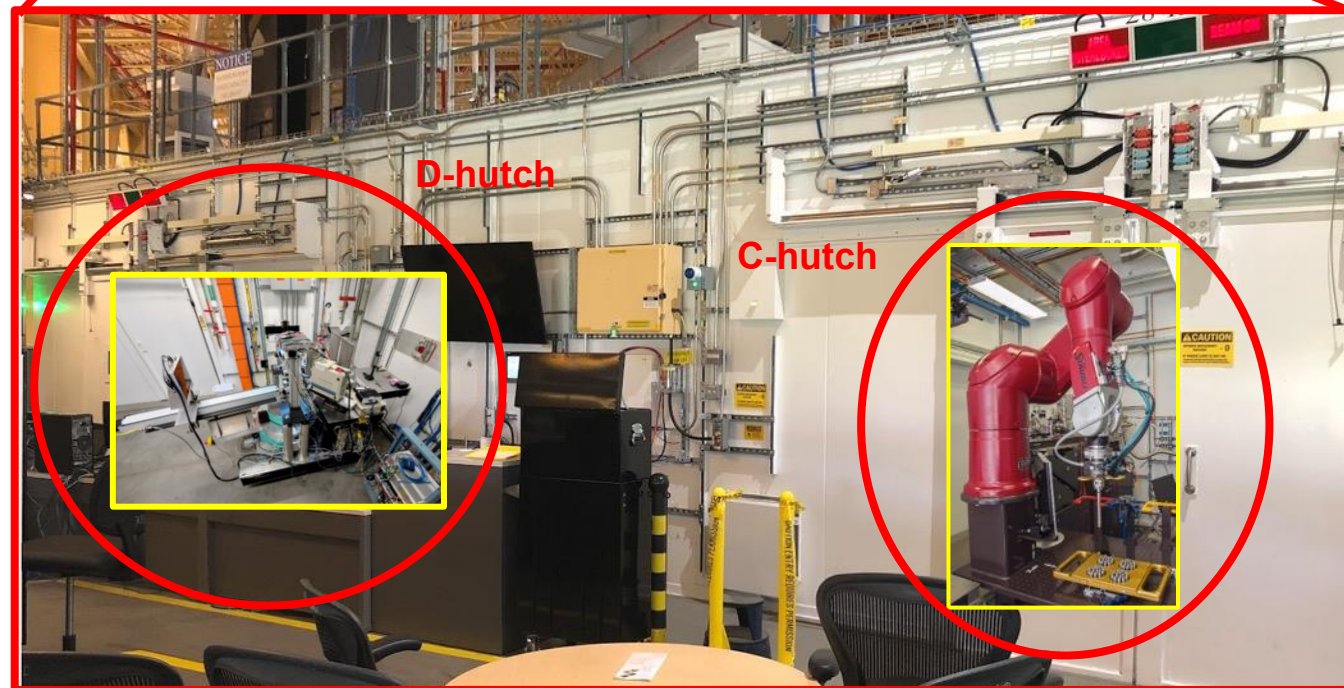
[3] Smith, Spencer M. et al. Intentional Fission: Design Strategies for Rapid Nuclear Material Provenance Assessment. Technical Report LBNL-25-25460, Lawrence National Laboratory, Livermore National Lab., 2023.

More calls are expected to be announced in 2025. Please feel free to contact us at mtopsakal@bnl.gov or gills@bnl.gov



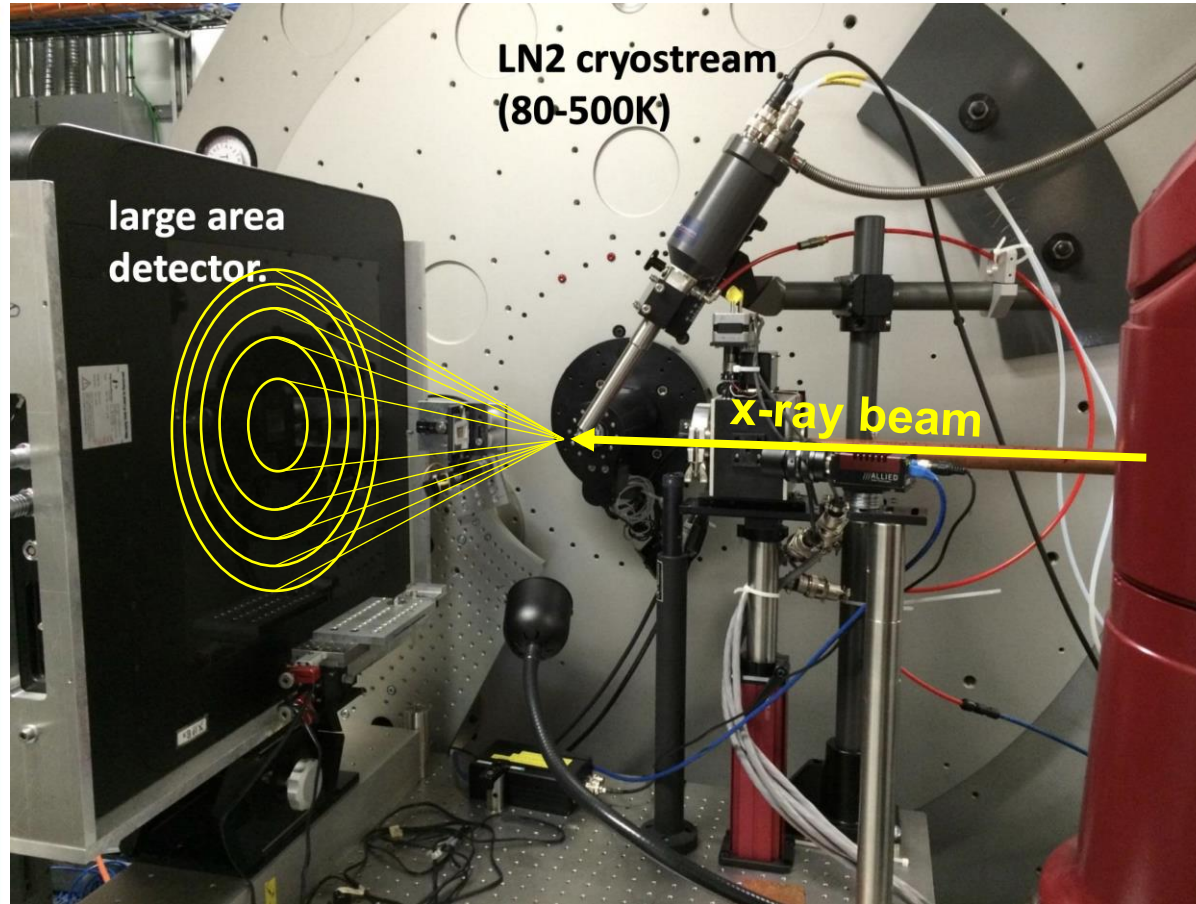
XPD beamline (28-ID) at NSLS-II

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

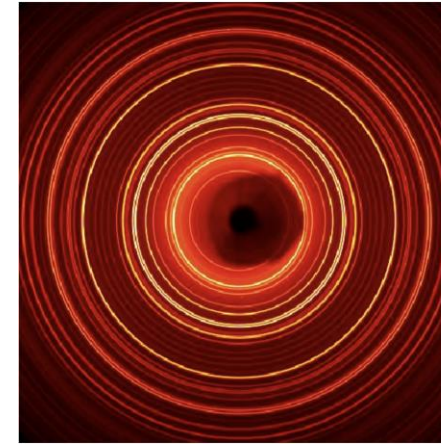


One-slide on X-ray diffraction method

A typical X-ray diffraction setup with area detector

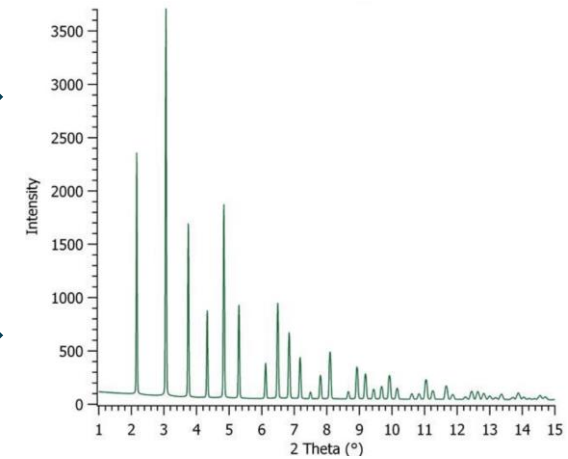


2D image



Azimuthal
integration

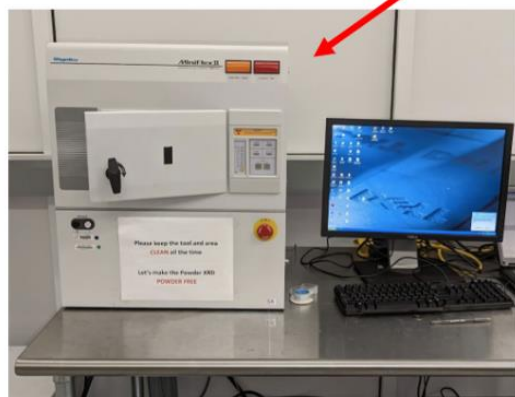
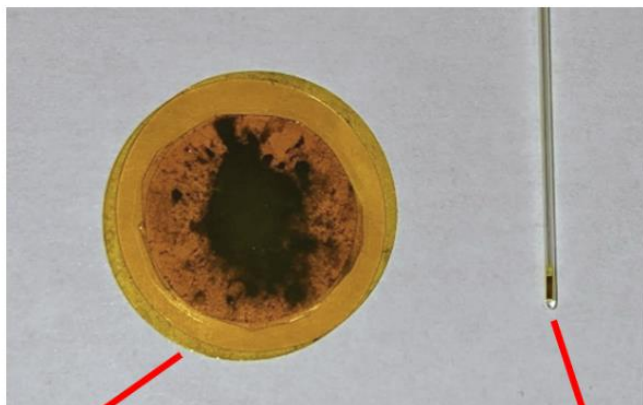
1D pattern



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

Why do we need synchrotron resources ?

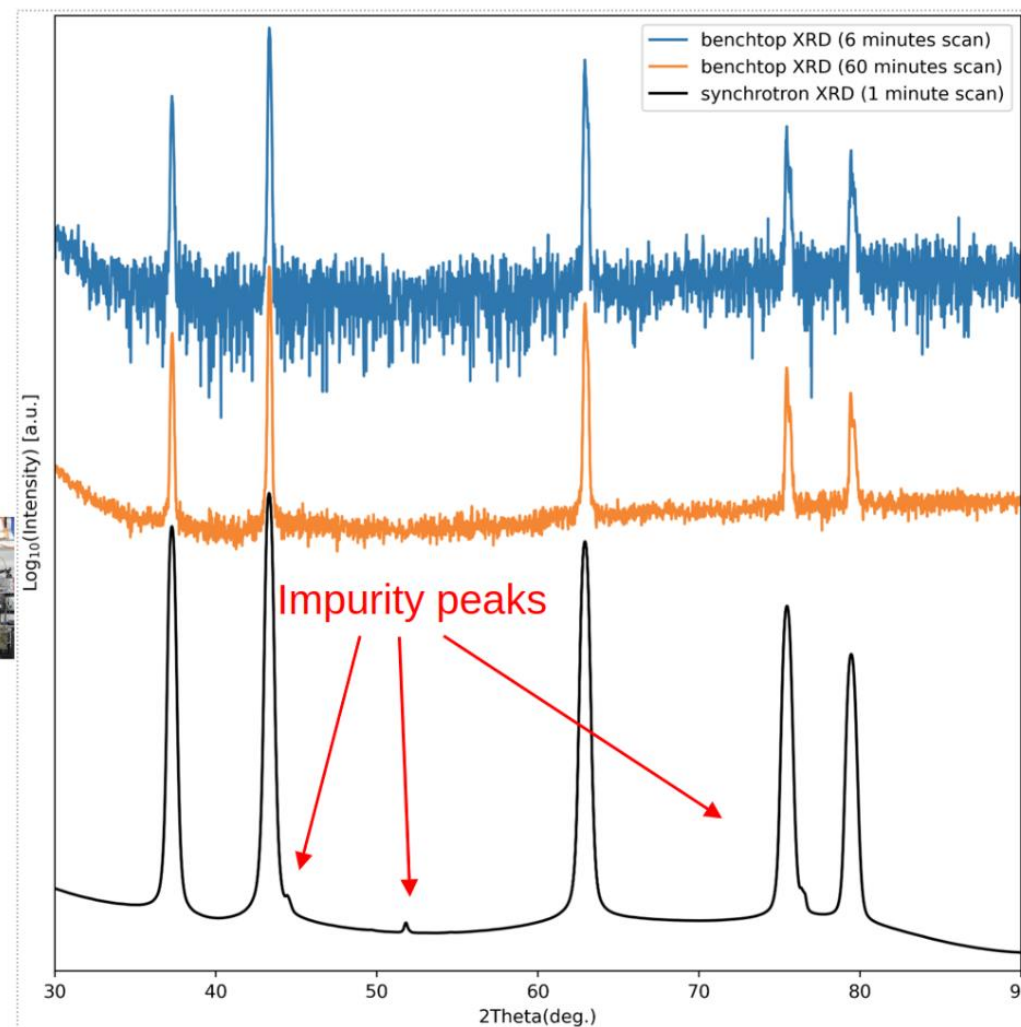
NiO powder test sample

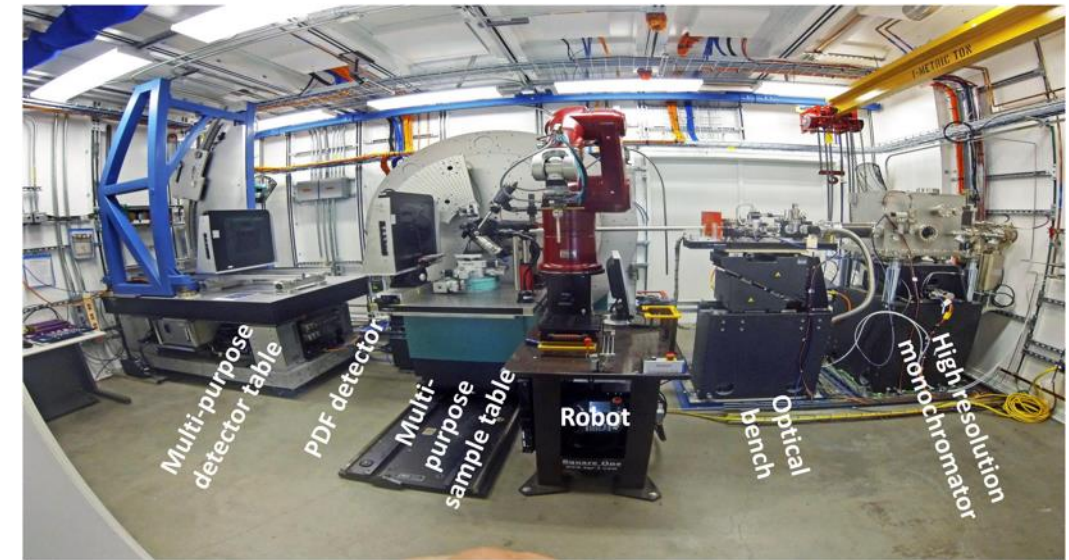


Benchtop XRD (Rigaku)

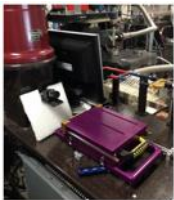


NSLS-II (XPD beamline)

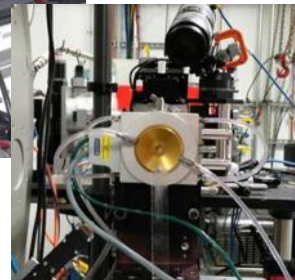
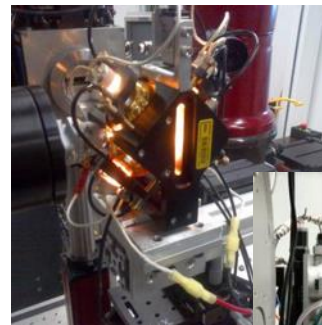




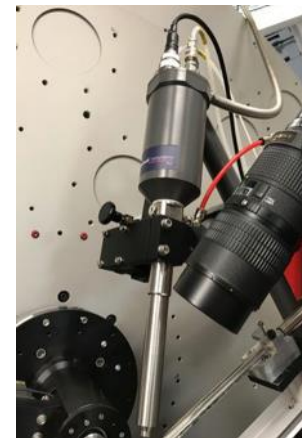
Sample Holder



Robot for high-throughput sample changing



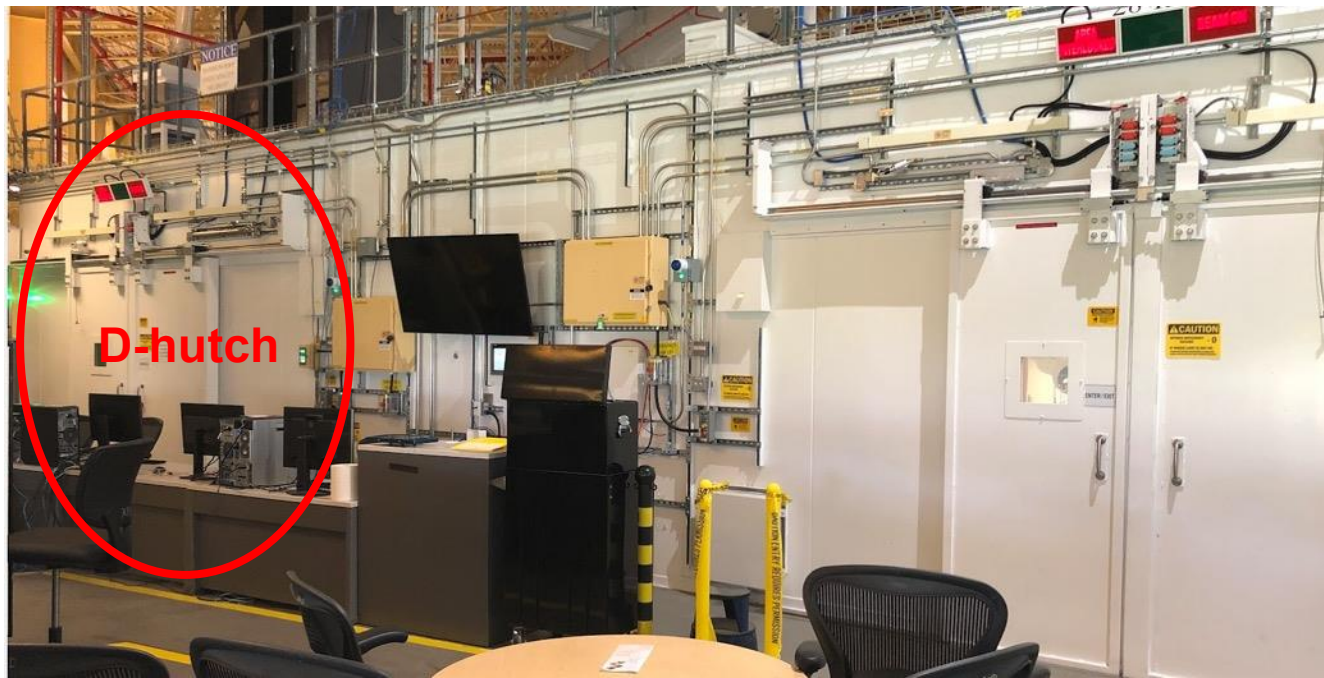
Heating up to 2000°C



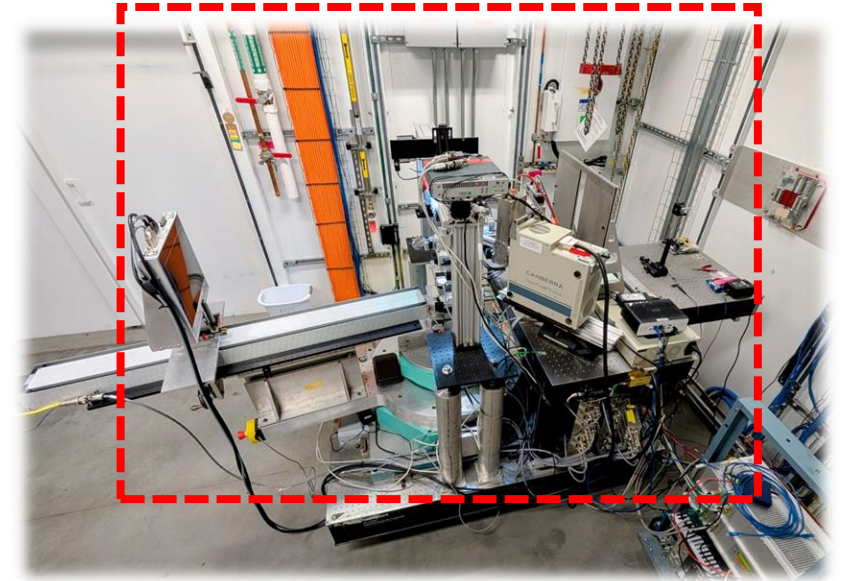
Cryostream (80K-500K)



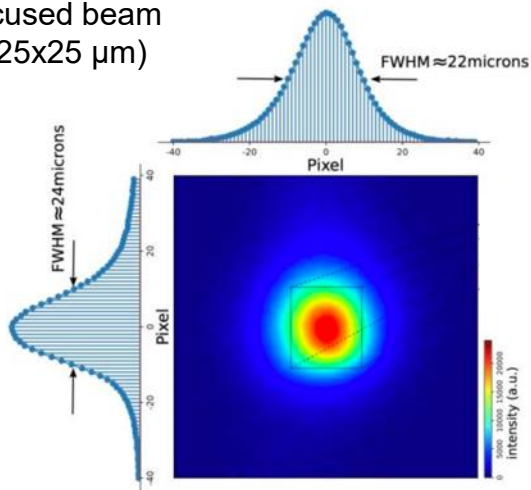
Corrosion cell



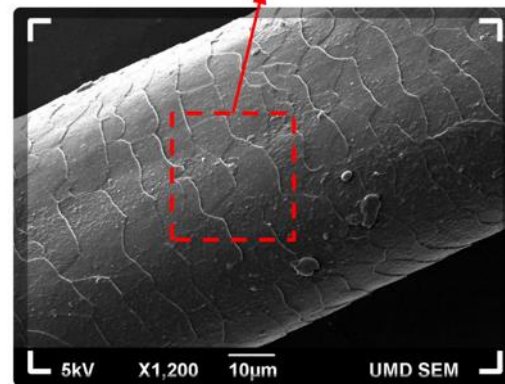
proudly
supported by



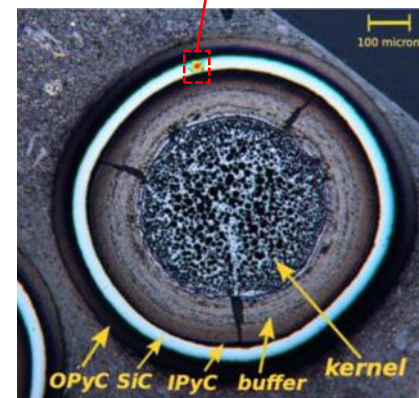
Focused beam
(~25x25 μm)



25 microns x 25 microns
spot on human hair



25 microns x 25 microns
spot on TRISO fuel



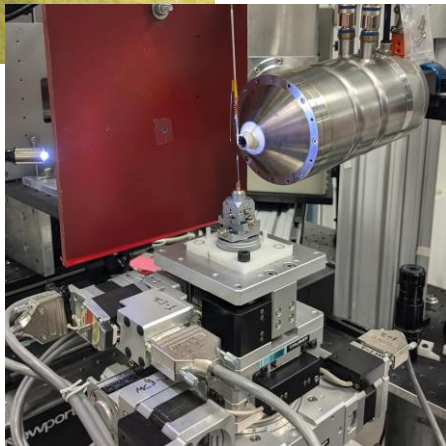
Techniques enabled with small beam:

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

X-ray fluorescence 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



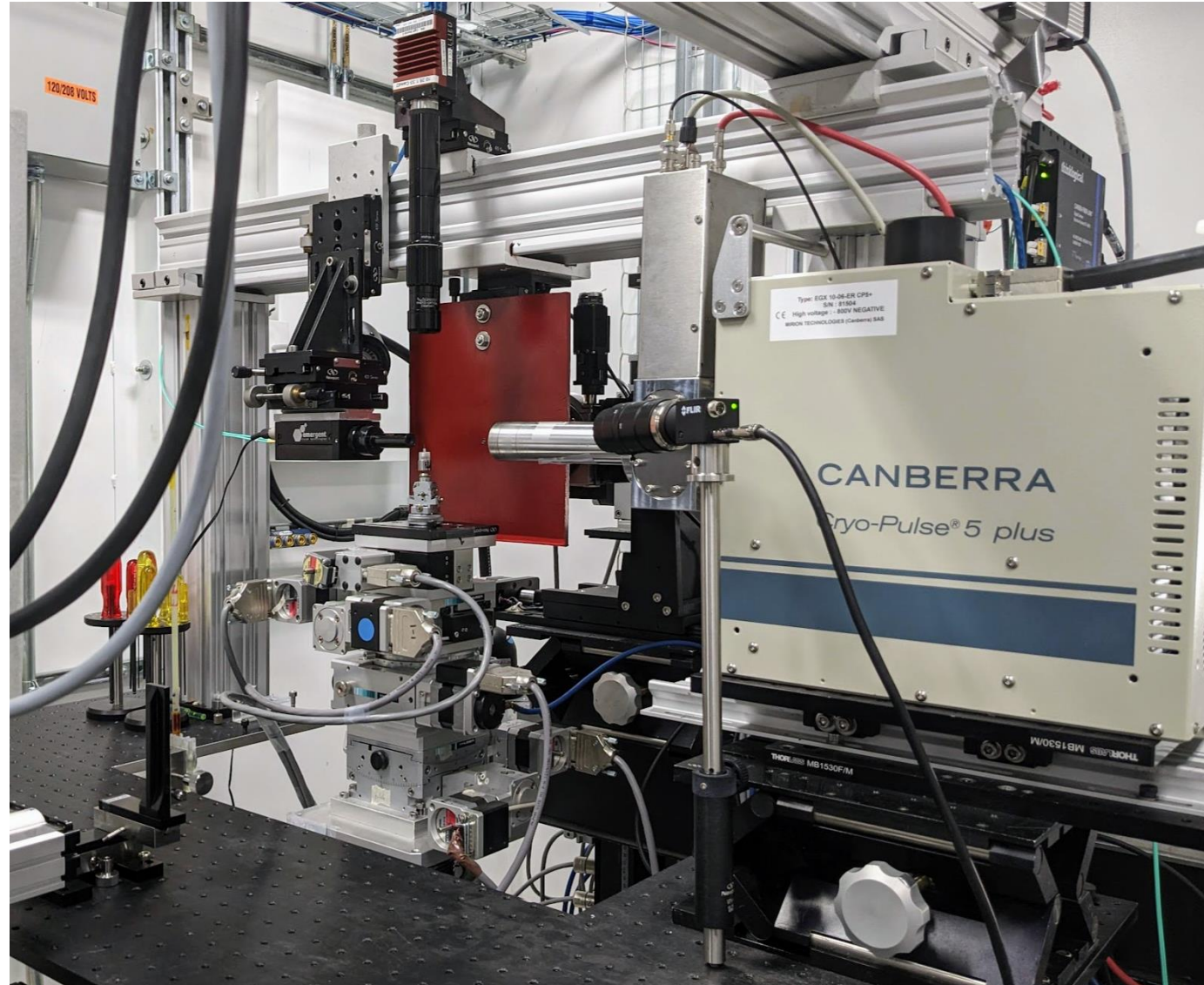
UB₄ samples heated up to 900°C



Outline of this talk

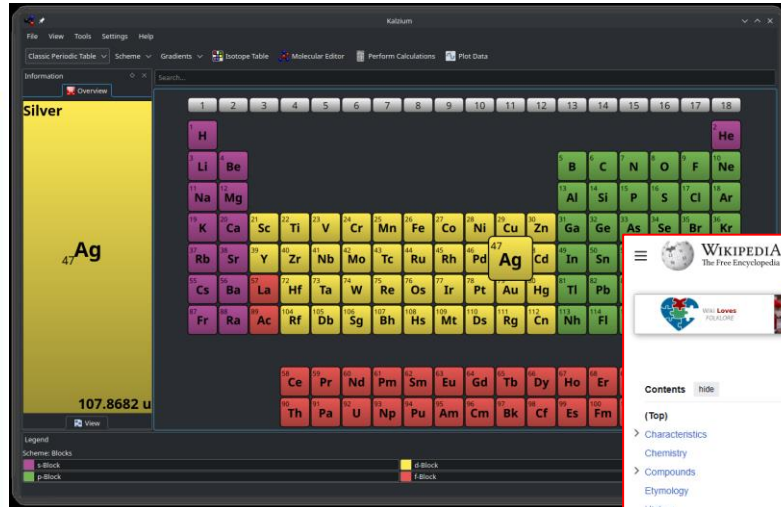
- Introduction to NSUF facility at NSLS-II
- **Capability upgrades**
- Plans for future

X-ray fluorescence (XRF) capability was added



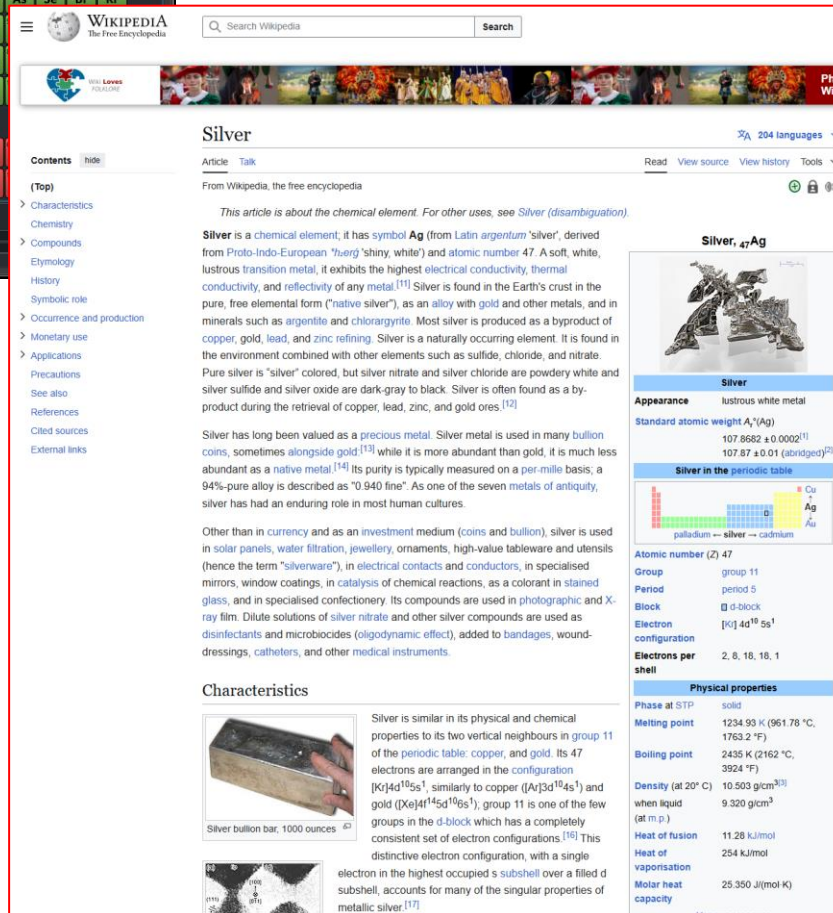
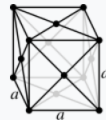
XRF capability demonstration with a science case

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



Crystal structure face-centred cubic (fcc)
(cF4)

Lattice constant $a = 408.60 \text{ pm}$
(at 20 °C)^[3]



Silver

Article Talk

From Wikipedia, the free encyclopedia

This article is about the chemical element. For other uses, see [Silver \(disambiguation\)](#).

Silver is a chemical element, it has symbol **Ag** (from Latin *argentum* 'silver', derived from Proto-Indo-European **h₂erǵ* 'shiny, white') and atomic number 47. A soft, white, lustrous transition metal, it exhibits the highest electrical conductivity, thermal conductivity, and reflectivity of any metal.^[1] Silver is found in the Earth's crust in the pure, free elemental form ("native silver"), as an alloy with gold and other metals, and in minerals such as *argenteite* and *chlorargente*. Most silver is produced as a byproduct of copper, gold, lead, and zinc refining. Silver is a naturally occurring element. It is found in the environment combined with other elements such as sulfide, chloride, and nitrate. Pure silver is "silver" colored, but silver nitrate and silver chloride are powdery white and silver sulfide and silver oxide are dark-gray to black. Silver is often found as a by-product during the retrieval of copper, lead, zinc, and gold ores.^[2]

Silver has long been valued as a precious metal. Silver metal is used in many bullion coins, sometimes alongside gold^[13] while it is more abundant than gold, it is much less abundant as a native metal.^[14] Its purity is typically measured on a per-mille basis; a 94%-pure alloy is described as "0.940 fine". As one of the seven metals of antiquity, silver has had an enduring role in most human cultures.

Other than in currency and as an investment medium (coins and bullion), silver is used in solar panels, water filtration, jewellery, ornaments, high-value tableware and utensils (hence the term "silverware"), in electrical contacts and conductors, in specialised mirrors, window coatings, in catalysis of chemical reactions, as a colorant in stained glass, and in specialised confectionery. Its compounds are used in photographic and X-ray film. Dilute solutions of silver nitrate and other silver compounds are used as disinfectants and microbicides (oligodynamic effect), added to bandages, wound-dressings, catheters, and other medical instruments.

Characteristics

Silver is similar in its physical and chemical properties to its two vertical neighbours in group 11 of the periodic table: copper, and gold. Its 47 electrons are arranged in the configuration $[\text{Kr}]4d^{10}5s^1$, similarly to copper ($[\text{Ar}]3d^{10}4s^1$) and gold ($[\text{Xe}]4f^{14}5d^{10}6s^1$); group 11 is one of the few groups in the d-block which has a completely consistent set of electron configurations.^[16] This distinctive electron configuration, with a single electron in the highest occupied s subshell over a filled d subshell, accounts for many of the singular properties of metallic silver.^[17]

Silver, 47Ag

Appearance lustrous white metal

Standard atomic weight $A_r^\circ(\text{Ag})$
107.8682 ± 0.0002^[1]
107.87 ± 0.01 (abridged)^[2]

Silver in the periodic table

Atomic number (Z) 47

Group group 11

Period period 5

Block d-block

Electron configuration $[\text{Kr}] 4d^{10} 5s^1$

Electrons per shell 2, 8, 18, 18, 1

Physical properties

Phase at STP solid

Melting point 1234.93 K (961.78 °C, 1763.2 °F)

Boiling point 2435 K (2162 °C, 3924 °F)

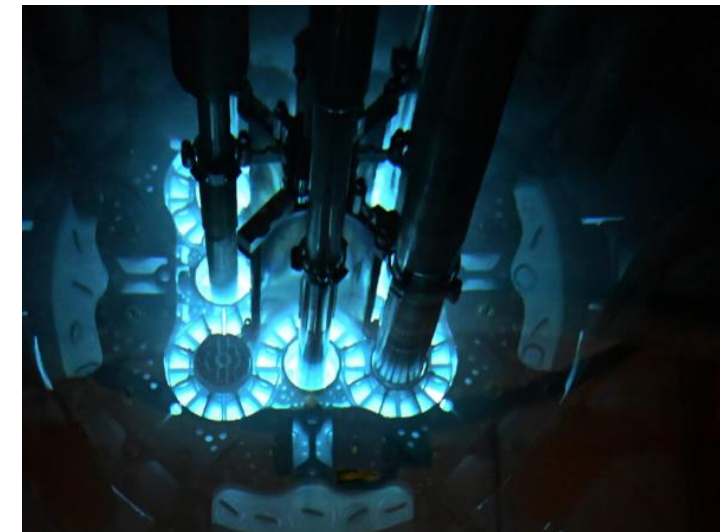
Density (at 20 °C) 10.503 g/cm³^[3]
when liquid (at m.p.) 9.320 g/cm³

Heat of fusion 11.28 kJ/mol

Heat of vaporisation 254 kJ/mol

Molar heat capacity 25.350 J/(mol K)

Advanced Test Reactor



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

The screenshot shows the homepage of the Nuclear Fuels & Materials Library (NFML) at NSUF. The header features the NSUF logo and the title "Nuclear Fuels & Materials Library". The main content area includes a description of the library, a search bar, and a sidebar with navigation links. A red arrow points from the "Material Name" field in the sample details to the "BROWSE LIBRARY" button.

Nuclear Fuels & Materials Library (NFML)

NSUF manages the Nuclear Fuels and Materials Library (NFML), a collection of specialized information and material specimens from past and ongoing irradiation test campaigns, real-world components retrieved from decommissioned power reactors, and donations from other sources.

Everything in the NFML is available to the nuclear research community, either through a peer-reviewed proposal process or through direct programmatic request. The NFML initiative maximizes the value of previous and ongoing materials and nuclear fuels irradiation test campaigns.

[BROWSE LIBRARY](#)

[VIEW NFML POLICY](#)

[VIEW FACT SHEET](#)

What is included in the NFML?

Samples include *conventional and advanced steels, experimental alloys, ceramics and fuels*.

Initially, NSUF staff identified legacy materials from previous R&D programs that were being stored in laboratories and hot-cell facilities at Idaho National Laboratory, NSUF's lead laboratory. Owners of other materials of interest, identified outside of INL, volunteered to enter them into NFML. Finally, more than 3,500 specimens that had been irradiated as part of NSUF competitively awarded research projects were added to NFML. More than 6,000 samples are now in the library.

The screenshot shows the details page for sample 034-08-331. The page includes a sidebar with navigation links, a main content area with sample details, and a table of cycles. A red arrow points from the "Material Name" field in the sample details to the "BROWSE LIBRARY" button in the NFML homepage screenshot.

Research

034-08-331 Irradiation Test Plan for the Advanced Test Reactor National Scientific User Facility/University of Wisconsin Pilot Project

[Home](#) [Browse](#) [Institutions](#) [Library](#) [Map](#) [Projects](#) [Researchers](#) [Resources](#)

Sample Details:

- Program: NSUF
- Project: Irradiation Test Plan for the Advanced Test Reactor National Scientific User Facility/University of Wisconsin Pilot Project
- Reactor: ATR
- Reactor Position: East Flux Trap
- Sample Id Code: 34-08-331
- Capsule: Capsule 1
- Packet: 5.11 A
- Material Code: A-3000
- Material Name: Silver (Ag)
- Material Description: Silver
- KGT Num: 4785
- Specimen Type: TEM
- Dimensions (mm): 3d x 2
- Number Of Samples: 4
- Available for Research: Not Currently Available
- Anticipated Availability: February 14, 2025
- Certification: Yes
- Storage Facility: Hot Fuel Examination Facility
- Notes: Sample repackaged from KGT 286 to KGT 4785 in support of RTE 4941
- Planned Temp (°C): 500.00
- Planned Dose (DPA): 3
- Planned Flux (n/cm²/s): 9.7E+13
- Planned Environment: Helium/Argon
- As Run Total Dose (DPA): 2.37
- As Run Total Fluence (n/cm²): 1.47E+22
- Composition by Wt. (%): Ag
- Keyword Tags: Element, Metal, Precious, Elemental, Pure, High purity

Cycles (6) History (1) Inventory (3)

Cycle	As Run Temp	As Run Dose
143A	469.00	
143B	469.00	
144A	469.00	
145A	469.00	
145B	469.00	
146A	469.00	

Showing 1 to 6 of 6 entries

Composition by Wt. (%)

Ag (100%)

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

Nuclear Science User Facilities

Official Notification



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 Contract DE-AC07-05ID14517."

When an instrument scientist is involved in experiment execution, it is expected to be included in resulting publications.

A Data Management and Sharing Plan (DMSP) should be created to provide

Measurement of the Production Yield of Fission Products

A. Mattera, M. Toppikal - Brookhaven National Laboratory, Upton NY

1 BACKGROUND

We propose to study neutron-irradiated samples with synchrotron X-ray fluorescence (XRF) at the 2802.5 keV line (XRF) of NSLS-II. One of the goals of this project is to demonstrate the feasibility of using XRF to obtain fission product yields (FPY). The work proposed for this Rapid Turnaround Experiment (RTE) is the first step towards this goal.

When a heavy nucleus undergoes fission, it splits into typically two, lighter fission products. There are hundreds of possible fission products, and the probability of producing a specific fission product is referred to as its yield. Fission product yields are important for a number of nuclear physics applications, as well as nuclear forensics, non-proliferation and basic science, and they are part of so-called evaluated nuclear data libraries, i.e., compilations of data that nuclear scientists and engineers use as the foundation of nuclear models and simulations. One such library, managed and distributed by the National Nuclear Data Center (NNDC) at INEL, is the Evaluated Nuclear Data File (ENDF/B) scientific notation of this RTE are: (1) Test of the feasibility and the detection limits of XRF applied to nuclear data and, in particular, to the determination of the yields of fission products; (2) Determination of the linearity of the technique and the sources of systematic uncertainty in the quantification of nuclear reaction products; (3) Determination of the thermal neutron-capture cross section of low-abundance isotopes of elements proposed as targets for international fission applications.

Evaluated FPYs are based on experimental measurements that have traditionally been performed with radiochemical methods, involving a chemical separation step, followed by detection of the characteristic gamma rays emitted in the decay of unstable fission products. This technique, however, suffers from a few sources of uncertainty that are hard to reduce, such as uncertainty on the branching ratios of the gamma rays used to quantify the fission products (which can for some nuclides reach 20%), as well as the use as the absolute normalization of the yields, that accounts for about one fifth of the total uncertainty. New techniques are then being explored to deliver more precise and accurate FPY measurements.

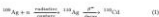
For long-lived fission products, a one-time experimental campaign at Idaho National Laboratory (INL) was performed in the 1970s using chemical separation and isotope dilution mass spectrometry (IDMS) [2]. These measurements did not rely on nuclear data for the determination of the FPYs, and reduced the normalization uncertainty by measuring the full fission products distribution, making precisions around 1%. Some limitations exist, however, that make some nuclear data evaluation question the validity of the campaign: (1) some refractory and low-yield elements could not be accounted in the measurements and their yields had to be extrapolated; (2) data and procedures were never vetted, as the work was never published in a peer-reviewed journal; and (3) a thorough study of the systematics of this technique was never performed, raising doubts on the reported uncertainties. Over the past few years, in preparation for a thorough re-evaluation of FPYs that comes over a decade after the one currently adopted in ENDF/B, a need has emerged for a new high-precision determination of long-lived FPYs.

To address this need, we propose a new experimental campaign for the precise measurement of FPYs using XRF and taking advantage of the bright X-ray beams from the NSLS-II. Using a non-destructive technique that does not rely on gamma-ray branching ratios or other decay data, the uncertainty on the yields can be kept as low as those reported by the INL group, or improved. Furthermore, the high sensitivity of the XRF setup would allow us to reach change FPYs as low as 10^{-7} , two orders of magnitude lower than those measured at INL (Fig. 1).

2 CHOICE OF MATERIALS

To test the detection limits, the sensitivity, the precision and accuracy of XRF for nuclear data applications, we will use neutron-capture as a surrogate for nuclear fission.

Following capture of a thermal neutron, nuclides will partially transform to heavier isotopes, which can change elemental species via β^- or electron capture decay (or in the example in equation 1).



We will focus on neutron-irradiated samples of elements that are produced in thermal neutron-induced fission of actinides. We have selected a series of samples from the NSUF library that have been exposed to various, and known, integrated neutron fluences. The samples contain four elements in the transition metal region of the periodic table - i.e., where the majority of the fission products are produced. We expect the nuclear products to be uniformly distributed within the sample, and positional information will not be needed in the case of the samples.

While the primary aim of this scientific program is the high-accuracy measurement of fission yields, the steps towards this goal will still produce scientifically significant results. Of the targets requested for this RTE, some contain fission products like Ag and Mo which directly align with the overarching goal of this long-term project. The other requested samples contain W and Hf which are elements being considered as fuel additives in what is referred to as advanced reactors (AR), where elements are added as tracers to nuclear fuel in the production stage, and are used like a bar-code to track fuel provenance [3]. The neutron capture cross sections of the low-abundance isotopes, those more likely to transmute to evaluate the uncertainty of the method and identify possible sources of systematic uncertainties.

If the quality of neutron capture data for some of these isotopes (e.g., ^{181}W) found them entirely inappropriate for IF applications. Measurements of cross sections data for a scientific endeavor that is interesting per se and since it is less complex than nuclear fission, we suggest it as an ideal use-case for this technique that would at the same time provide scientific relevant results.

3 EXPERIMENTAL TECHNIQUE AND TEAM

We propose to use the 2802.5 keV line (XRF) of NSLS-II to identify and quantify the amount of transmutated element in the selected samples from NSUF. The 2802.5 keV line has a X-ray energy of 70 keV, which is well suited to study K and L shell transitions. The use of this energy does not require any specific edge of the selected samples, nor a modified atmosphere.

The RTE is shown in Tab. 1, with the list of the elements to be studied and the amount of transmutated element in the selected samples from NSUF. The 2802.5 keV line has a X-ray energy of 70 keV, which is well suited to study K and L shell transitions. The use of this energy does not require any specific edge of the selected samples, nor a modified atmosphere.

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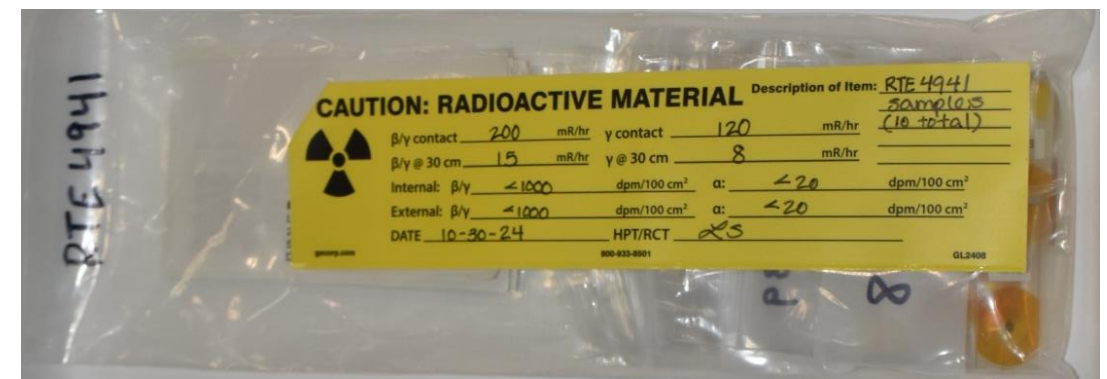
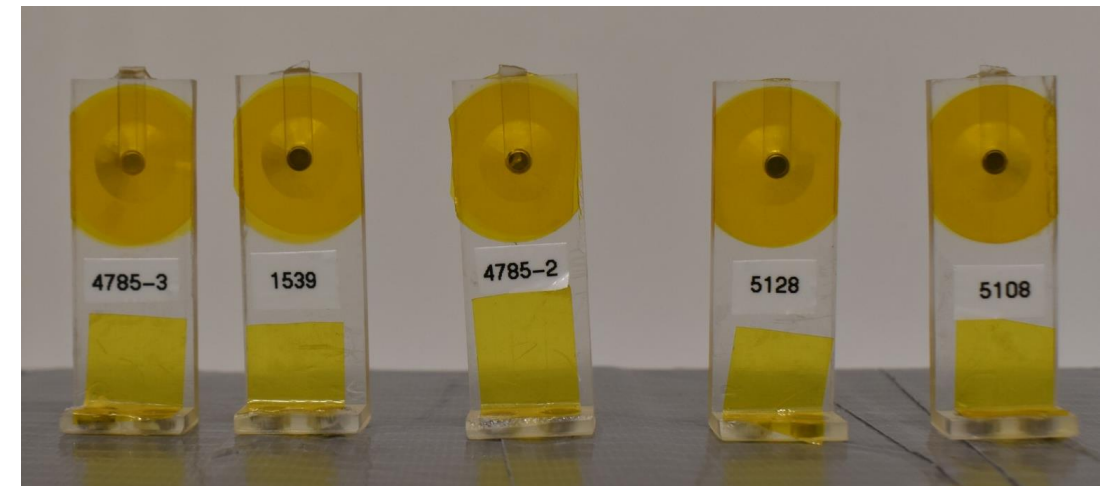
REFERENCES

[1] Lopez-Casas, A. et al. Review of neutron-capture cross-sections relevant for international nuclear forensics. Technical Report INF-2023-020202020202, Brookhaven National Laboratory, Upton, NY, 2023.

[2] National Nuclear Data Center, Evaluated Nuclear Data File (ENDF/B), 2023.

[3] From Simerjeet M. et al. International Nuclear Energy Agency project for legal nuclear material provenance assessment. Technical Report IAEA-2023-2023, 2023.

As prepared samples @ INL through NSUF



Alina Montrose

Experiment Manager
Nuclear Science User Facilities
Post-Irradiation Examination

Special thanks to

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

As measured samples @ NSLS-II

Nuclear Science User Facilities

Official Notification



Tuesday, May 28,
2024

CCN:256075

Dr. Andrea Matterna
Brookhaven National Laboratory

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

Dear Dr. Matterna:

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](#).

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A Data Management and Sharing Plan (DMSP) should be created to provide

Sample	Elem.	Integrated Fluence (n/cm ²)
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	Hf, Al	2.94 × 10 ²²
09-157-033		1.29 × 10 ²²
09-157-034	Mo	1.3 × 10 ²²
09-157-035		1.3 × 10 ²²
10-242-0011		1.05 × 10 ²¹

Measurement of the Production Yield of Fission Products

A. Matterna, M. Toppikal - Brookhaven National Laboratory, Upton NY

1 BACKGROUND

We propose to study neutron-irradiated samples with synchrotron X-ray fluorescence (XRF) at the 2802.5 beamline (XRF) of NSLS-II. One of the goals of this project is to demonstrate the feasibility of using XRF to obtain fission product yields (FPY). The work proposed for this Rapid Turnaround Experiment (RTE) is the first step towards this goal.

When a heavy nucleus undergoes fission, it splits into typically two, lighter fission products. There are hundreds of possible fission products, and the probability of producing a specific fission product is referred to as its yield. FPY distributions are important for a number of nuclear physics applications, as well as nuclear forensics, non-proliferation and basic science, and they are part of so-called evaluated nuclear data libraries, i.e., compilations of data that nuclear scientists and engineers use as the foundation of reactor models and simulations. One such library, managed and distributed by the National Nuclear Data Center (NNDC) at INEL, is the Evaluated Nuclear Data File (ENDF/B) scientific outcome of this RTE are: (1) Test of the feasibility and the detection limits of XRF applied to nuclear data, and, in particular, to the determination of the yields of fission products; (2) Determination of the linearity of the technique, and the sources of systematic uncertainty in the quantification of nuclear reaction products; (3) Determination of the thermal neutron-capture cross section of low-abundance isotopes of elements proposed as targets for international fission applications.

Evaluated FPYs are based on experimental measurements that have traditionally been performed with radiochemical methods, involving a chemical separation step, followed by detection of the characteristic gamma rays emitted in the decay of unstable fission products. This technique, however, suffers from a few sources of uncertainty that are hard to reduce, such as uncertainty on the branching ratios of the gamma rays used to quantify the fission products (which can for some nuclides reach 20%), as well as the use as the absolute normalization of the yields, that accounts for about one fifth of the total uncertainty. New techniques are then being explored to deliver more precise and accurate FPY measurements.

For long-lived fission products, a one-time experimental campaign at Idaho National Laboratory (INL) was performed in the 1970s using chemical separation and isotope dilution mass spectrometry (IDMS) [2]. These measurements did not rely on nuclear data for the determination of the FPYs, and reduced the normalization uncertainty by measuring the full fission products distribution, reaching precisions around 1%. Some limitations exist, however, that make nuclear data evaluation questions the validity of the campaign: (1) some refractory and low-yield elements could not be accounted in the measurements and their yields had to be extrapolated; (2) data and procedures were never vetted, as the work was never published in a peer-reviewed journal; and (3) a thorough study of the systematics of this technique was never performed, causing doubts on the reported uncertainties. Over the past few years, in preparation for a thorough re-evaluation of FPYs that comes over a decade after the one currently adopted in ENDF/B, a need has emerged for a new high-precision determination of long-lived FPYs.

To address this need, we propose a new experimental campaign for the precise measurement of FPYs using XRF and taking advantage of the bright X-ray beams from the NSLS-II. Using a non-destructive technique that does not rely on gamma-ray branching ratios or other decay data, the uncertainties on the yields can be kept as low as those reported by the INL group, or improved. Furthermore, the high sensitivity of the XRF setup would allow us to reach change FPYs as low as 10⁻³, two orders of magnitude lower than those measured at INL (Fig. 1).

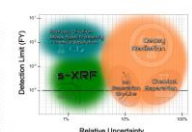


Figure 1: The most precise determination of fission product yields to date was performed at INEL. These relative measurements are presented here as a scatter plot and are used to determine the detection limit of XRF applied to nuclear data and, in particular, to the determination of the yields of fission products. (2) Determination of the linearity of the technique, and the sources of systematic uncertainty in the quantification of nuclear reaction products; (3) Determination of the thermal neutron-capture cross section of low-abundance isotopes of elements proposed as targets for international fission applications.

2 CHOICE OF MATERIALS

To test the detection limits, the sensitivity, the precision and accuracy of XRF for nuclear data applications, we will use neutron-capture as a surrogate for nuclear fission.

Following capture of a thermal neutron, nuclides will partially transmute to heavier isotopes, which can change detection species via β^- or electron capture decay (as in the example in equation 1).

$$^{109}\text{Ag} + n \rightarrow ^{110}\text{Ag} \rightarrow ^{110}\text{Cd} + \beta^- + \bar{\nu}_e \quad (1)$$

We will focus on neutron-irradiated samples of elements that are produced in thermal neutron-induced fission of actinides. We have selected a set of samples from the NSUF library that have been exposed to various, and known, integrated neutron fluences. The samples contain four elements in the transition metal region of the periodic table (i.e., where the majority of the fission products are produced). We expect the nuclear products to be uniformly distributed within the sample, and positional information will not be needed in the case of the samples.

While the primary aim of this scientific program is the high-accuracy measurement of fission yields, the steps towards this final goal will still produce scientifically significant results. Of the targets requested for this RTE, some contain fission products like Ag and Mo which directly align with the overarching goal of this long-term project. The other requested samples contain W and Hf which are elements being considered as fuel ingredients in what is referred to as advanced reactors [3], where elements are added as tracers to nuclear fuel in the production stage, and are used like a bar-code to monitor fuel's provenance [3]. The neutron capture cross sections of the low-abundance isotopes, those more likely to transmute to different elements in an intense thermal neutron field, is a critical quantity for IF. A recent review [4] of the quality of nuclear data for use in these isotopes (e.g., ^{109}Ag) found them entirely unsuitable for IF applications.

Measurements of cross sections for IF as a scientific endeavor that is interesting per se and since it is less complex than nuclear fission, we suggest it as an ideal test-case for this technique that would at the same time provide scientific relevant results.

3 EXPERIMENTAL TECHNIQUE AND TEAM

We propose to use the 2802.5 beamline (XRF) of NSLS-II to identify and quantify the amount of transmutated element in the selected samples from NNDC. The 2802.5 beamline has an X-ray energy of 70 keV, which is well suited to study K and L edges of the selected samples. We do not require any specific design of the selected samples, nor a modified atmosphere.

As shown in Tab. 1, with the use of the estimated concentration of fission products, we will use the detector (Canberra) of the 2802.5 beamline. Qualitative analysis via XRF will be the first measurement, to identify which elements are present in the sample, and a first test will outline the secondary steps beyond the measurement of which peaks from transmutated XRF spectrum, a quantitative that will allow us to estimate the fission product yields. The high-resolution (precursor) detectors measured at identical sample conditions. Reported measurements of the reproducibility of the sum to achieve low uncertainties.

Sample	Elem.	Integrated Fluence (n/cm ²)	FPY
034-08-331	Ag	1.47 × 10 ²²	2.4%
149-08-331	Ag	2.9 × 10 ²²	2.4%
052-08-331	W	1.05 × 10 ²²	5.00%
109-08-331	W	1.58 × 10 ²²	5.00%
148-08-331	Hf, Al	2.94 × 10 ²²	100-000 ppm
09-157-033	Hf, Al	1.29 × 10 ²²	100-000 ppm
09-157-034	Mo	1.3 × 10 ²²	100-000 ppm
09-157-035	Mo	1.3 × 10 ²²	100-000 ppm

As neutron capture cross-section and the neutron flux are well known, we will be able to evaluate the uncertainty of the method and identify possible sources of systematic uncertainties. Since the thickness of the samples is an important parameter in the proposed methods, we request in accurate determination of the sample thickness. We are requesting two (2) days of NSLS-II beamtime for the proposed work. We also request these two days for the sample preparation and the analysis of the data. The two consecutive cycles will still allow the proposed research to be completed within the 9-months limit of the RTE.

The Dr. ANDREA MATTENA - has a long record of work with measurements of fission products yields and isotopic yield ratios using direct ion counting, isotope separation and gamma-ray spectroscopy. He has worked in compilation and evaluation of fission product yields for the ENDF/B evaluation since 2010. He will be responsible for the analysis of the XRF data, aided by the staff.

The Dr. M. Toppikal - has a strong background in XRF measurements and technique development, and he has worked extensively with NSLS-II in the past. He will coordinate the sample preparation and alignment activities over the proposed 2 months, and will be the main point-of-contact for the experimental campaign. He will finally assist Dr. Matterna with the analysis of the data.

4 SUMMARY

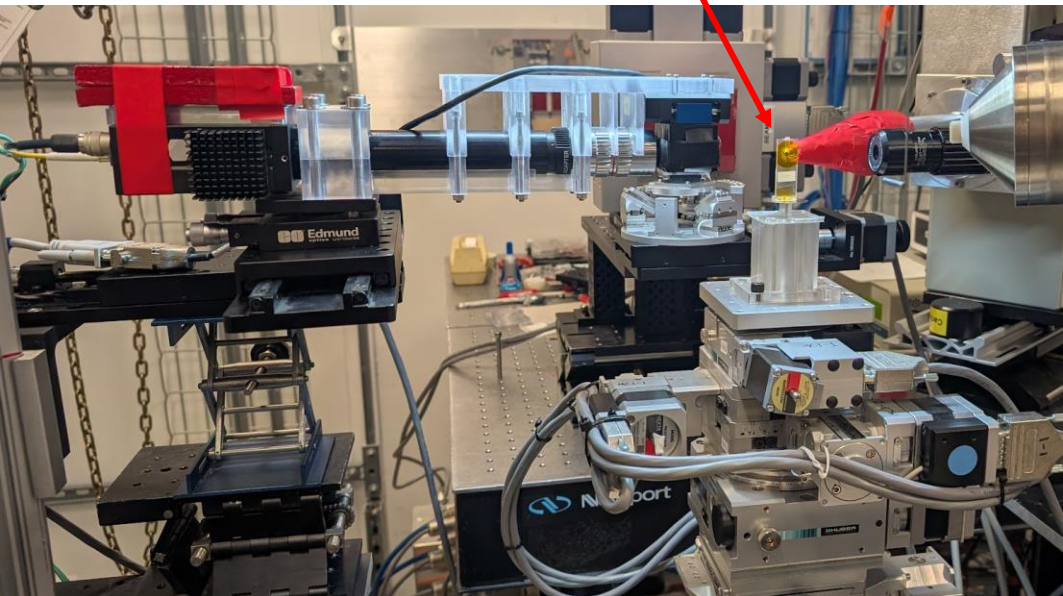
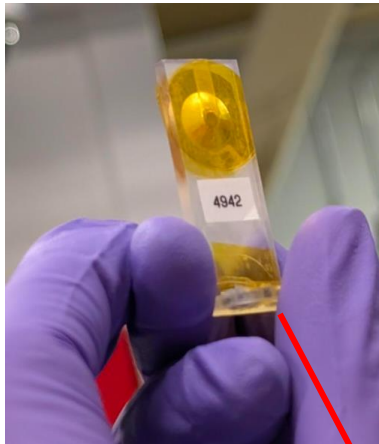
The main scientific outcomes of this RTE are: (1) Test of the feasibility and the detection limits of XRF applied to nuclear data and, in particular, to the determination of the yields of fission products; (2) Determination of the linearity of the technique, and the sources of systematic uncertainty in the quantification of nuclear reaction products; (3) Test of the feasibility of the thermal neutron-capture cross section of low-abundance isotopes of elements proposed as targets for international fission applications.

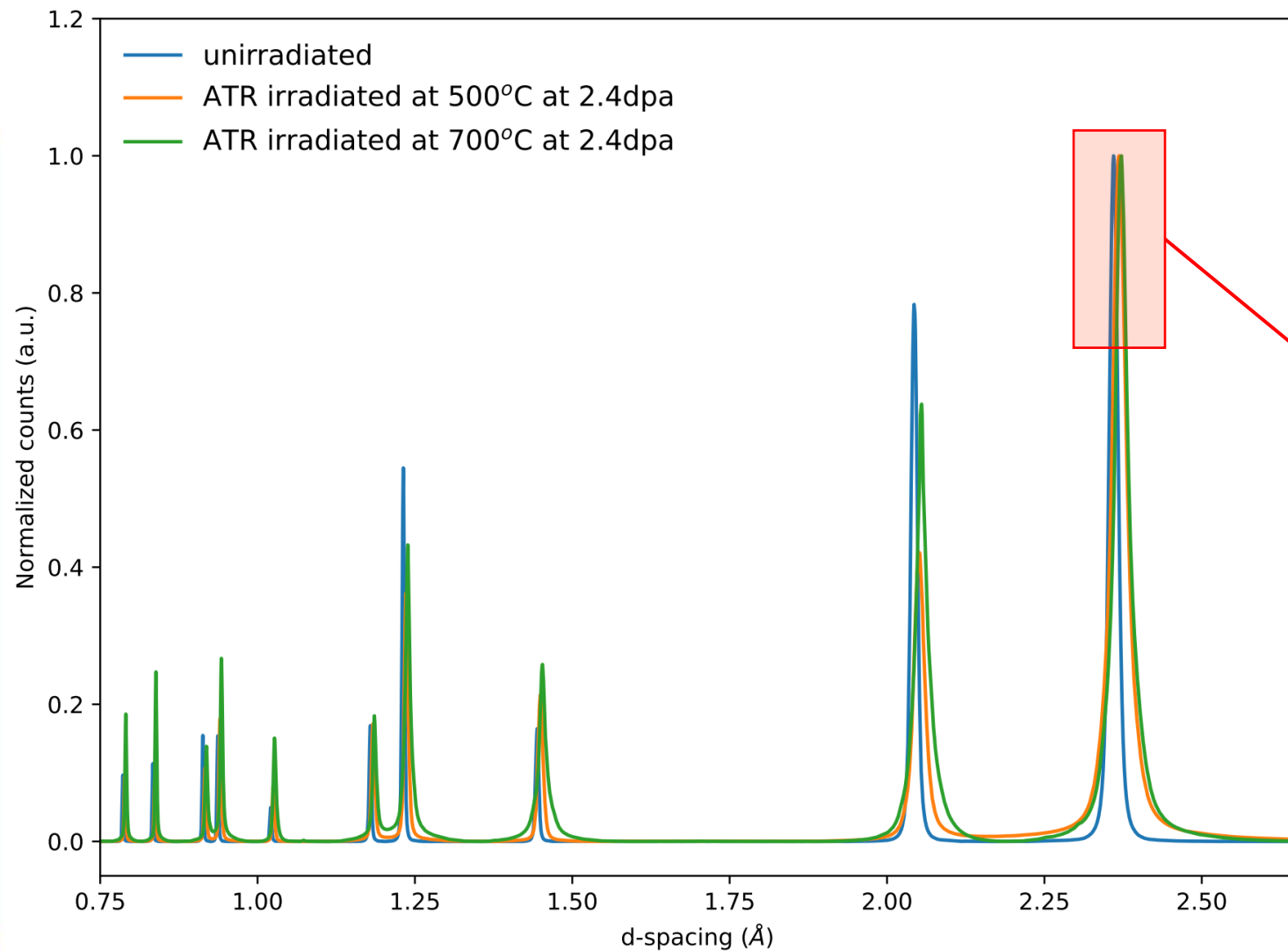
REFERENCES

[1] Langer, A. et al. *Review of nuclear data for international nuclear fission*. Technical Report NNDC-2015-02-001, Brookhaven National Laboratory, Upton, NY, United States, 2015.

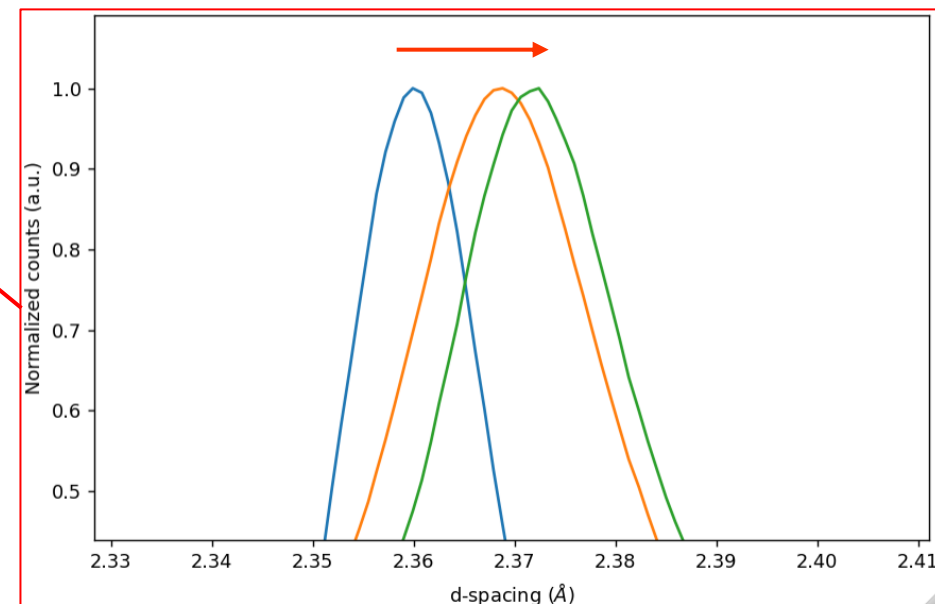
[2] Langer, A. et al. *Review of nuclear data for international nuclear fission*. Technical Report NNDC-2015-02-001, Brookhaven National Laboratory, Upton, NY, United States, 2015.

[3] Langer, A. et al. *Review of nuclear data for international nuclear fission*. Technical Report NNDC-2015-02-001, Brookhaven National Laboratory, Upton, NY, United States, 2015.



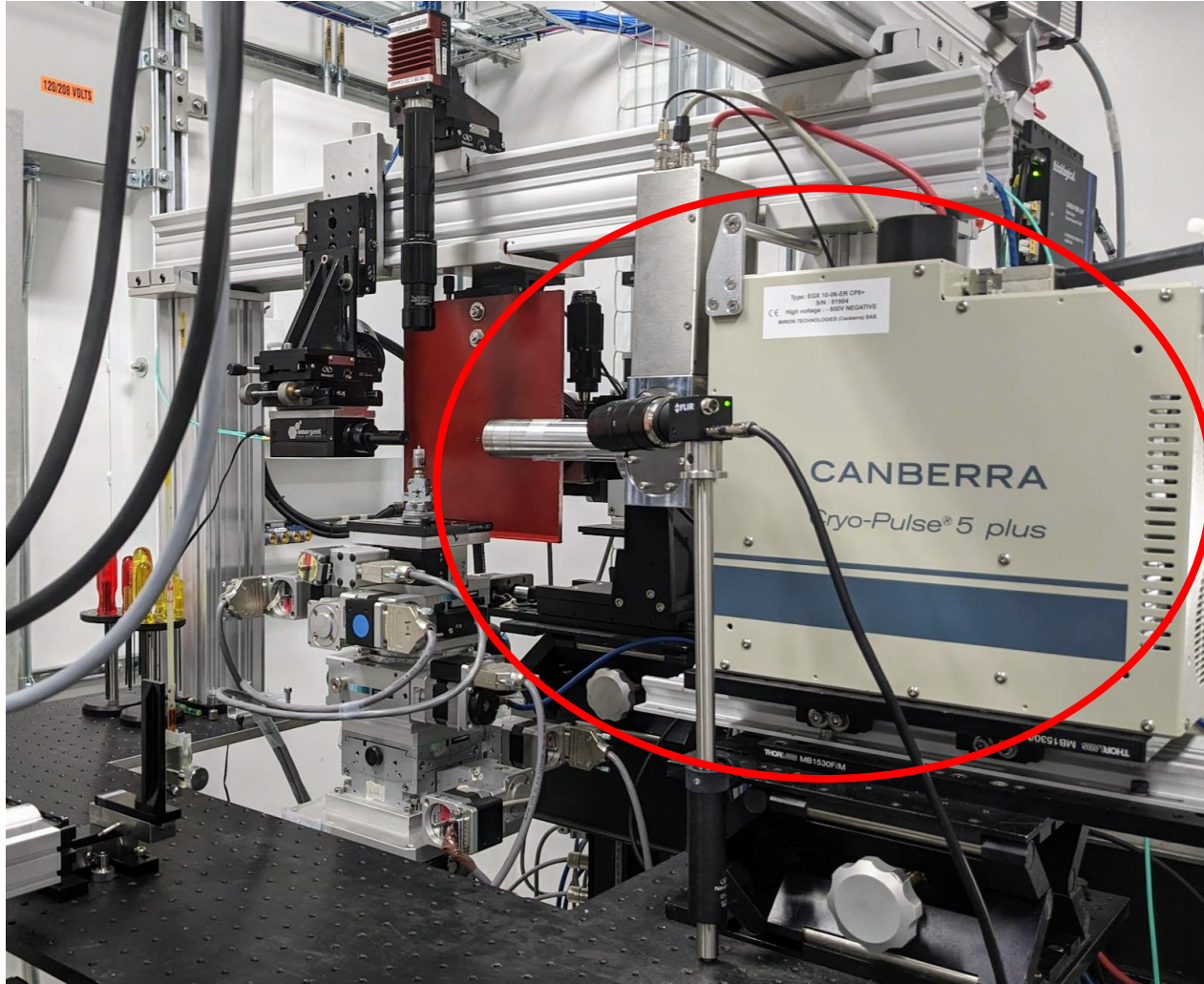


Dramatic changes with ATR irradiation!!!

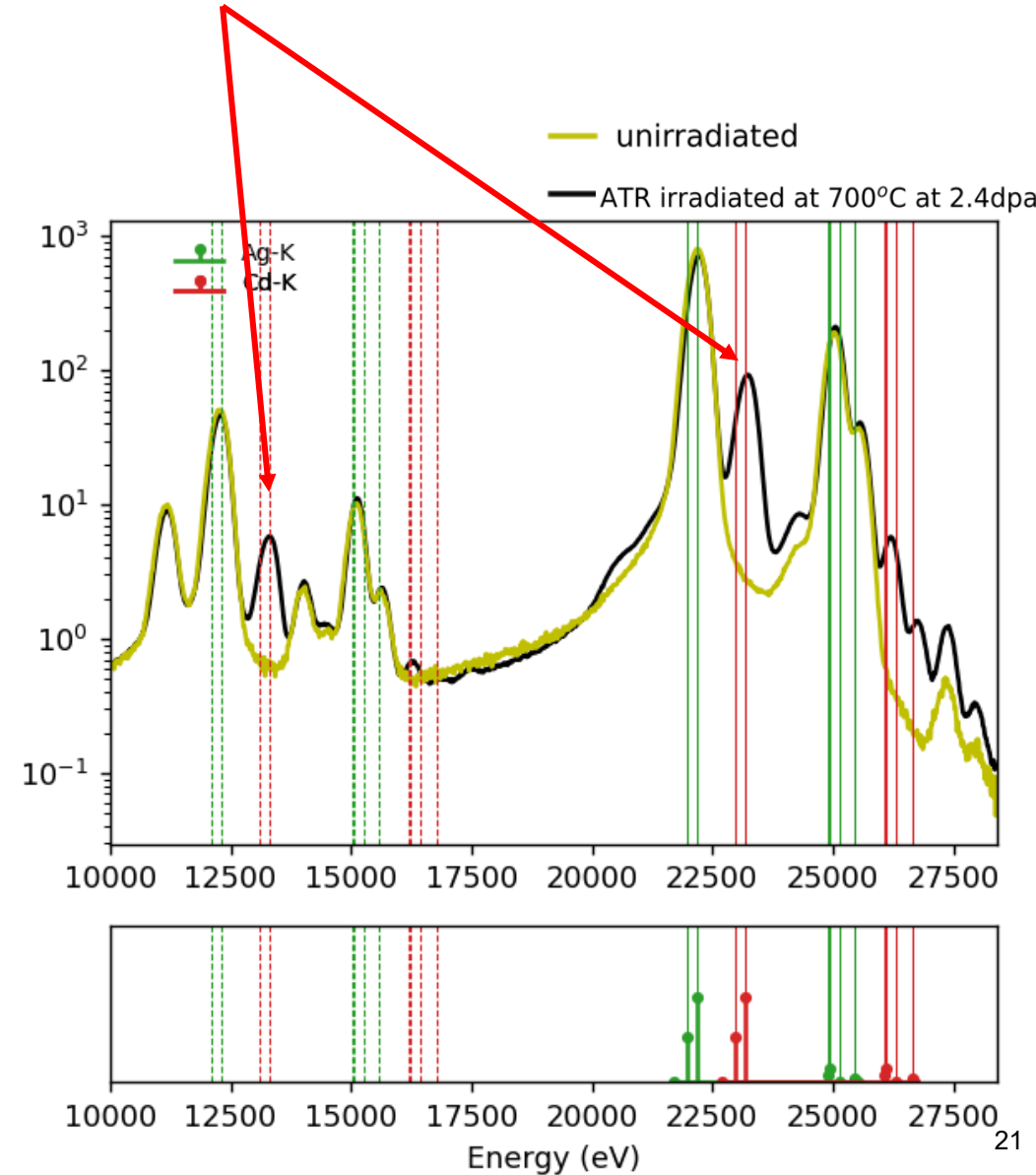


but why??

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



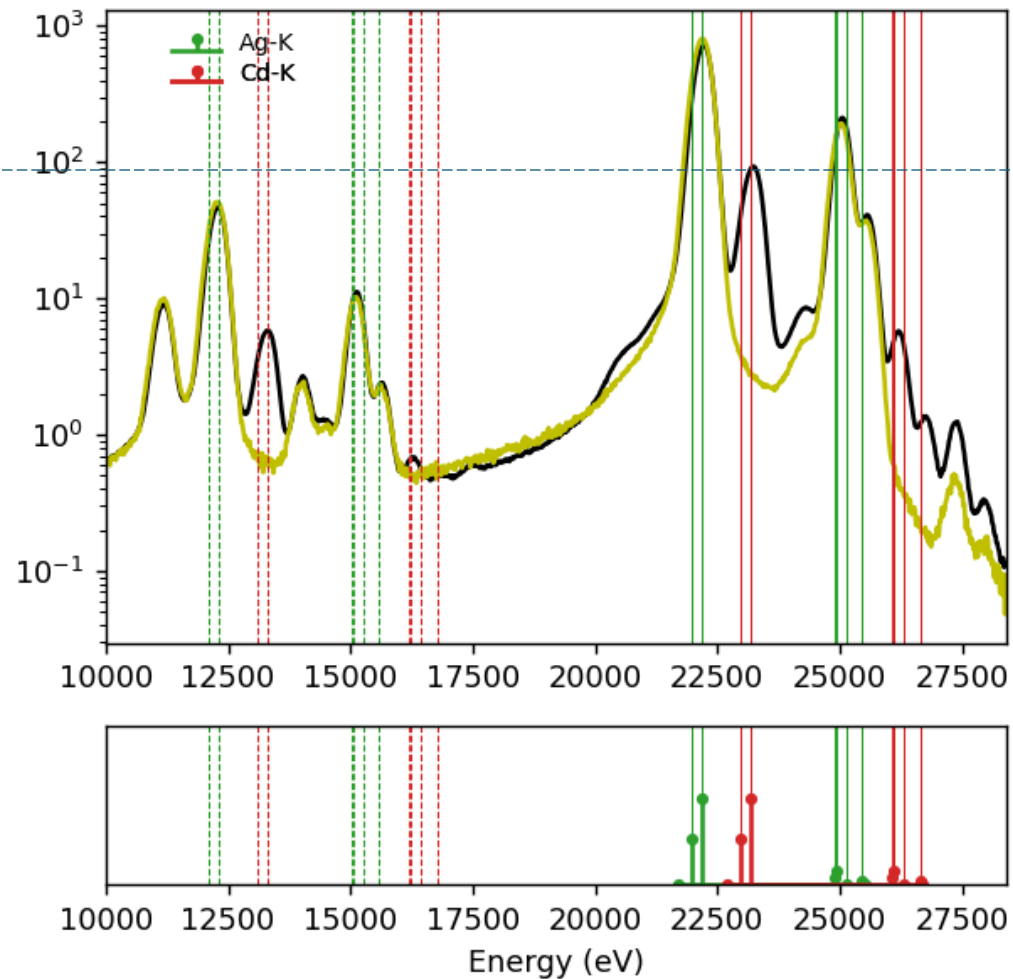
There are definitely Cd in these ATR irradiated Ag samples



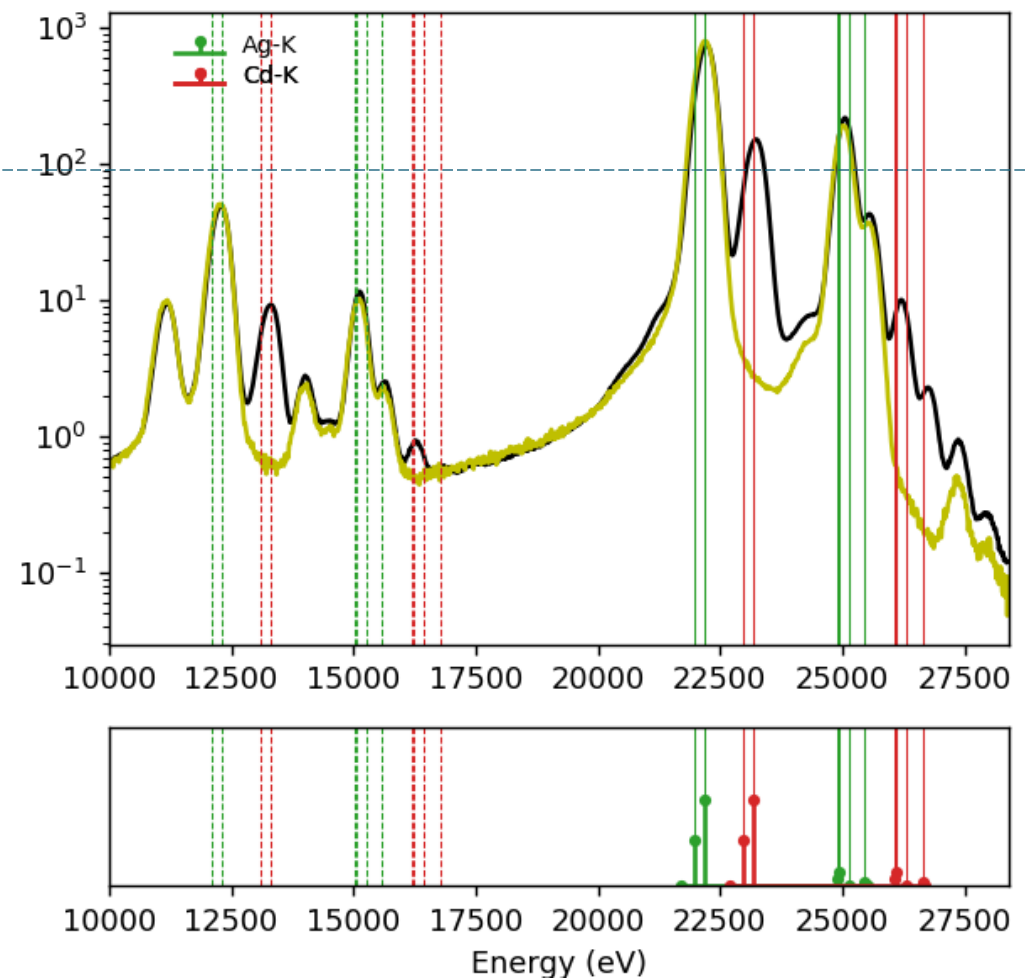
XRF capability provides additional “multi-modal information” as elemental composition

and Cd concentration increases with temperature

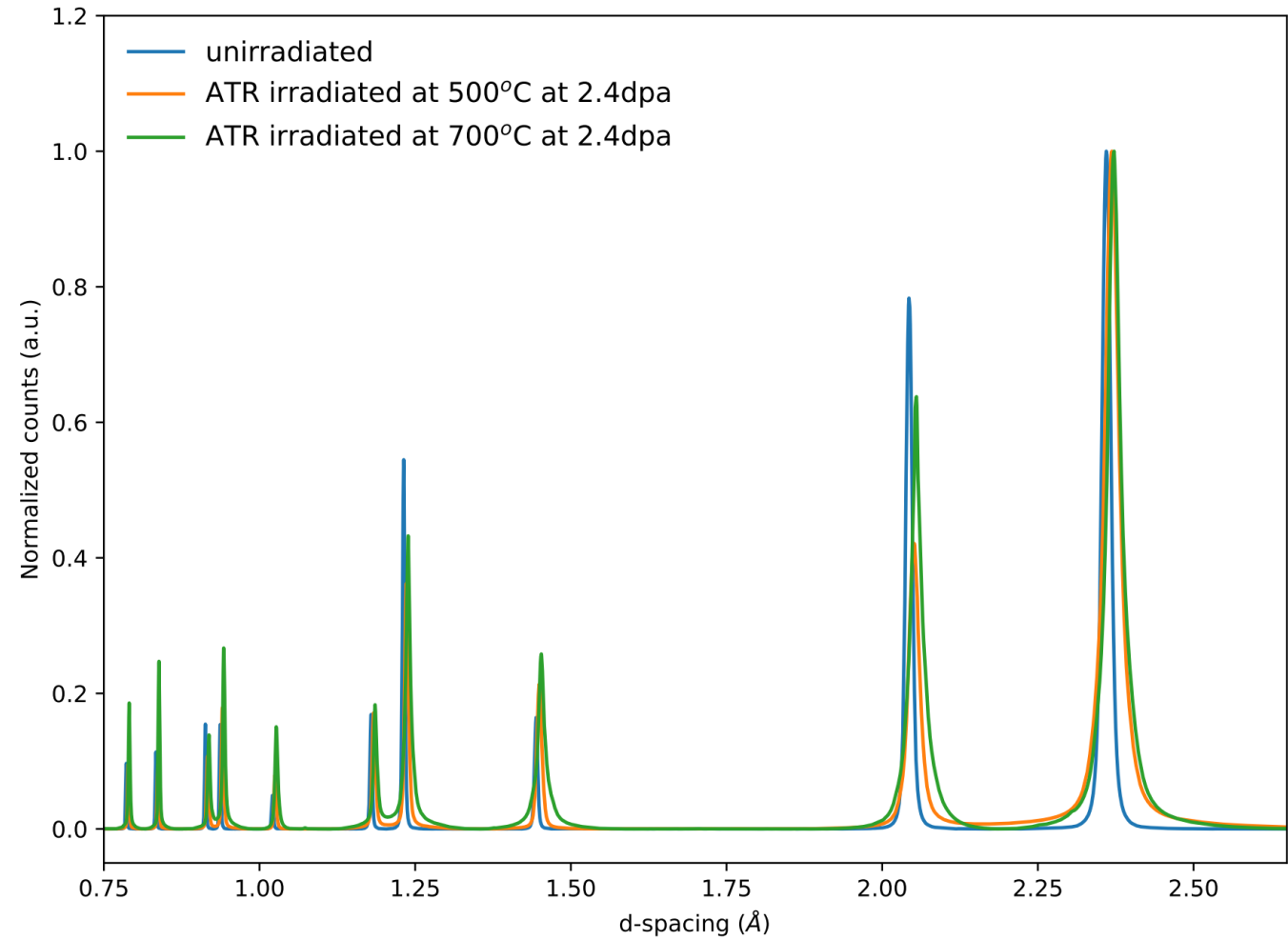
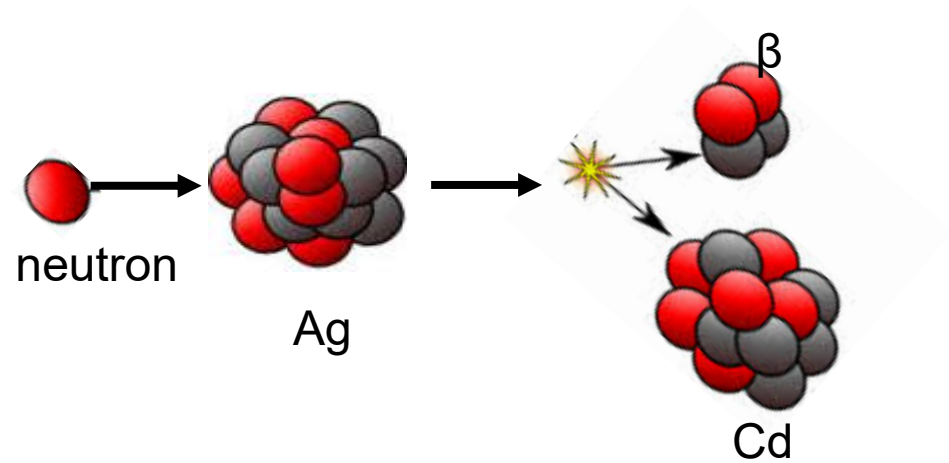
— unirradiated
— ATR irradiated at 500°C at 2.4dpa



ATR irradiated at 700°C at 2.4dpa



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



034-08-331 - NSUF Research

nsuf-research.inl.gov/Browse/Sample/4260

NSUF Nuclear Science User Facilities

Research

MEHMET TOPSAKAL

034-08-331 Irradiation Test Plan for the Advanced Test Reactor National Scientific User Facility/University of Wisconsin Pilot Project

Favorite

Hide Empty

Program: NSUF

Project: Irradiation Test Plan for the Advanced Test Reactor National Scientific User Facility/University of Wisconsin Pilot Project

Reactor: ATR

Reactor Position: East Flux Trap

Sample Id Code: 034-08-331

Capsule: Capsule 1

Packet: 500 LO A

Material Code: Ag (Ag⁵)

Material Name: Silver (Ag)

Material Description: Silver

KGT Num: 4785

Specimen Type: TEM

Dimensions (mm): 3d x .2

Number Of Samples: 4

Available for Research: Not Currently Available

Anticipated Availability: February 14, 2025

Certification: Yes

Storage Facility: Hot Fuel Examination Facility

Notes: Sample repackaged from KGT 286 to KGT 4785 in support of RTE 4941

Planned Temp (°C): 500.00

Planned Dose (DPA): 3

Planned Flux (n/cm²/s): 9.7E+13

Planned Environment: Helium/Argon

As Run Total Dose (DPA): 2.37

As Run Total Fluence (n/cm²): 1.47E+22

Composition by Wt. (%): Ag

Keyword Tags: Element, Metal, Precious, Elemental, Pure, High purity

Cycles (6) History (1) Inventory (3)

15

Cycle	As Run Temp	As Run Dose
143A	469.00	
143B	469.00	
144A	469.00	
145A	469.00	
145B	469.00	
146A	469.00	

Showing 1 to 6 of 6 entries

Composition by Wt. (%)

Ag (100%)

This is NOT true anymore

Outline of this talk

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

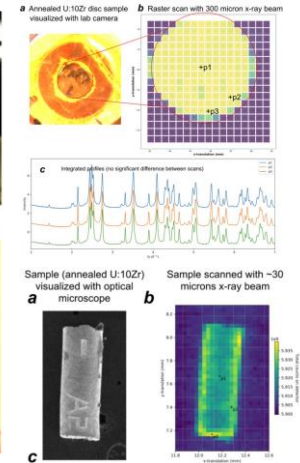
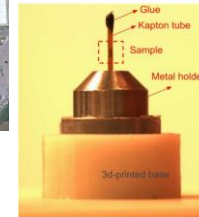
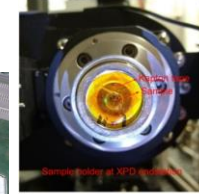
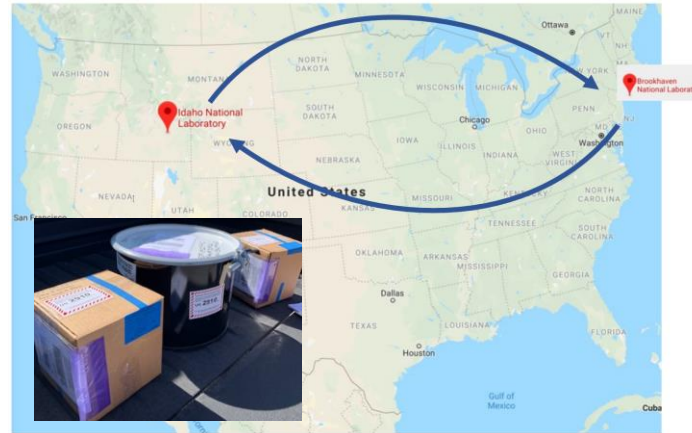
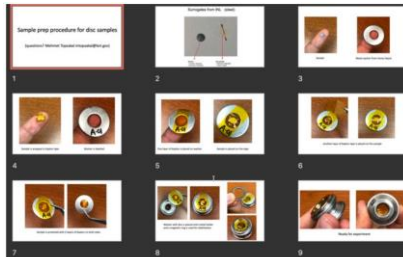
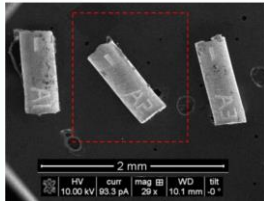
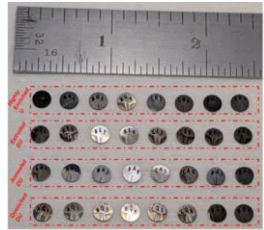
current workflow

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.

desired workflow

a new lab at NSLS-II

Advanced characterization
at various NSLS-II beamlines

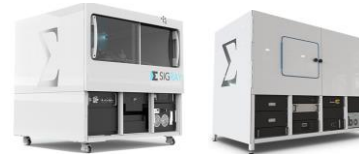
Initial samples

Sample preparation @ INL

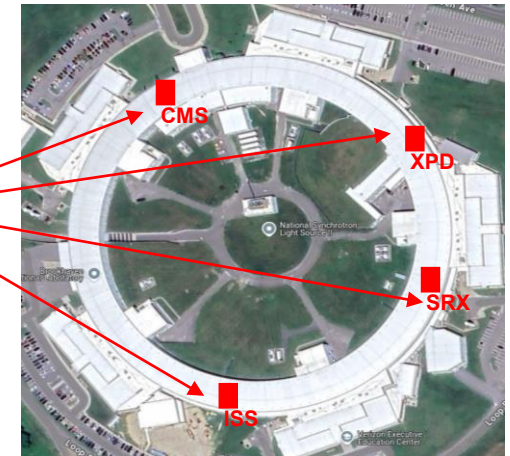
Sample shipment from INL to BNL



with lab-based
pre-characterization tools

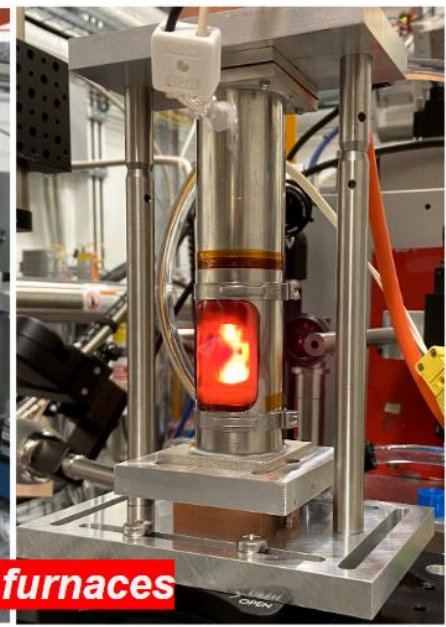
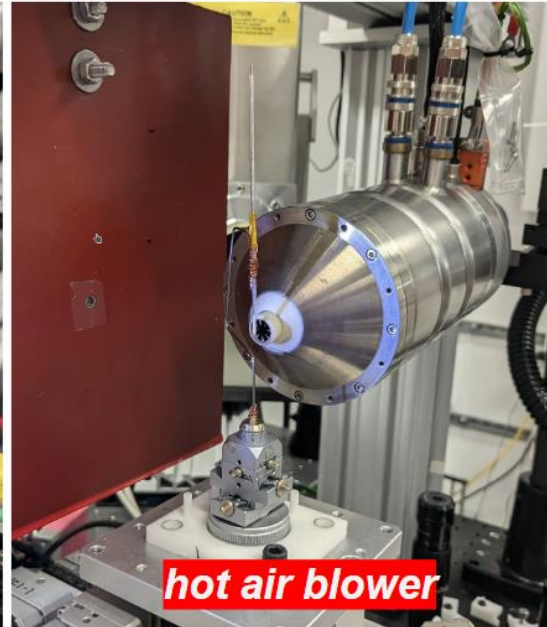
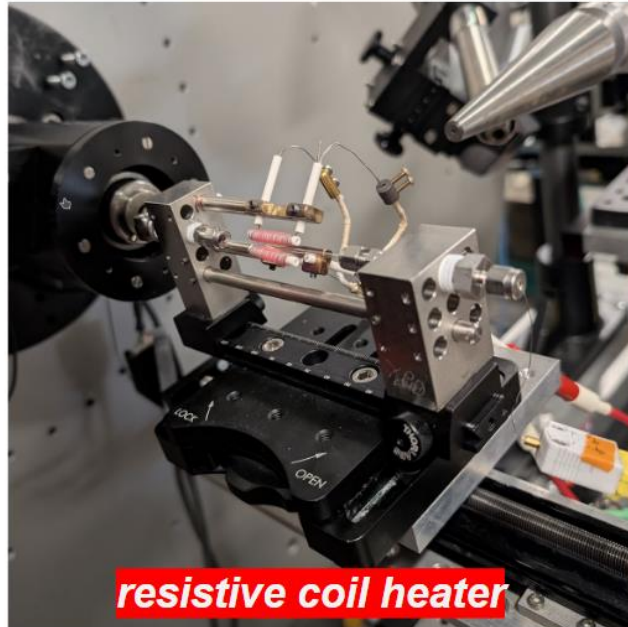


Initial
characterization
and
temporary storage



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

Conventional heating systems that are currently available at NSLS-II

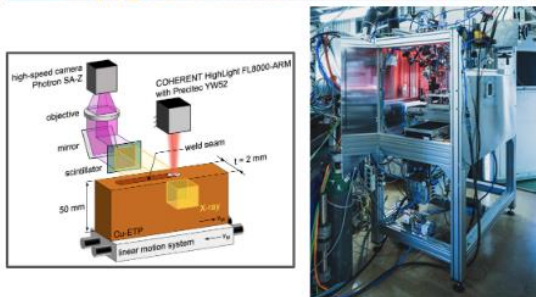


Some of drawbacks of all these systems are:

- they can't go beyond $\sim 1000^{\circ}\text{C}$ 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)

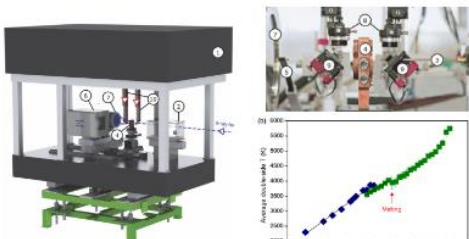
Ultra-high temperature laser heating systems that are NOT available at NSLS-II, but available at other synchrotrons

@ ESRF - The European Synchrotron Radiation Facility



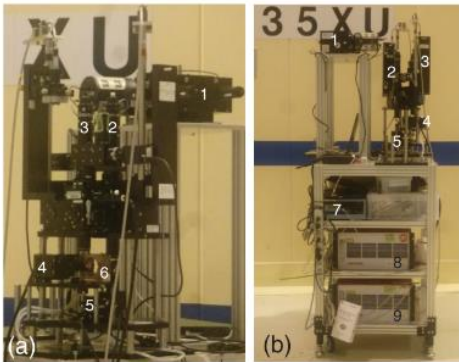
International Journal of Machine Tools & Manufacture 204 (2025) 104224

@ APS - Advanced Photon Source



Review of Scientific Instruments 86, 072201 (2015)

@ SPring-8 - Japan synchrotron



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

@ NSLS-II



**Currently
Not Available**

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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¹HPCAT, Geophysical Laboratory, Carnegie Institution of Washington, Argonne, Illinois 60565, USA
²Geophysical Laboratory, Carnegie Institution of Washington, Washington, DC 20015, USA

(Received 6 April 2015; accepted 28 June 2015; published online 17 July 2015)

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 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

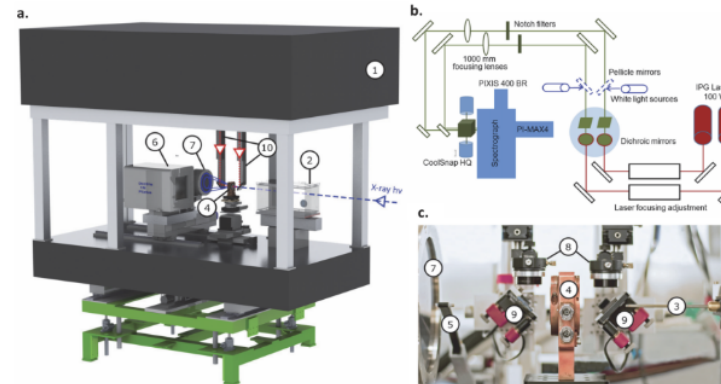


FIG. 1. The integrated system of laser-heated DAC with *in situ* XRD. (a) A computer-aided-design (CAD) drawing of the laser heating experiment table in 16-ID-B, (b) a schematic drawing of the optical system on the top experiment table (labeled as ①), and (c) an image of the setup around the diamond anvil cell. 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 IM 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 ⑥.

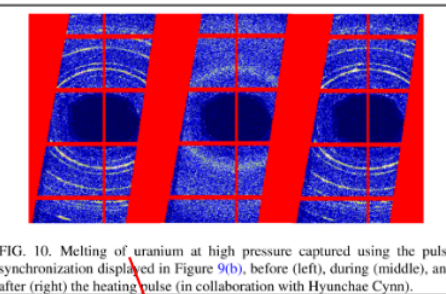
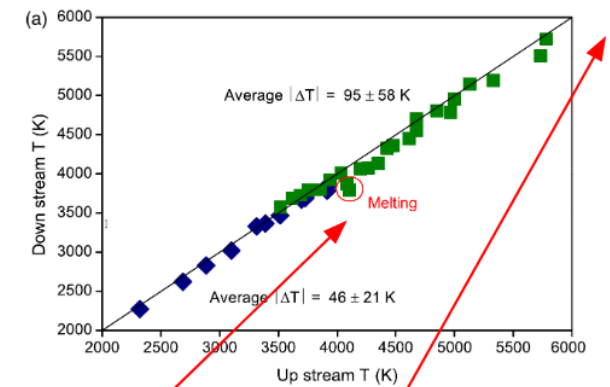


FIG. 10. Melting of uranium at high pressure captured using the pulse synchronization displayed in Figure 9(b), before (left), during (middle), and after (right) the heating pulse (in collaboration with Hyunhae Cynn).

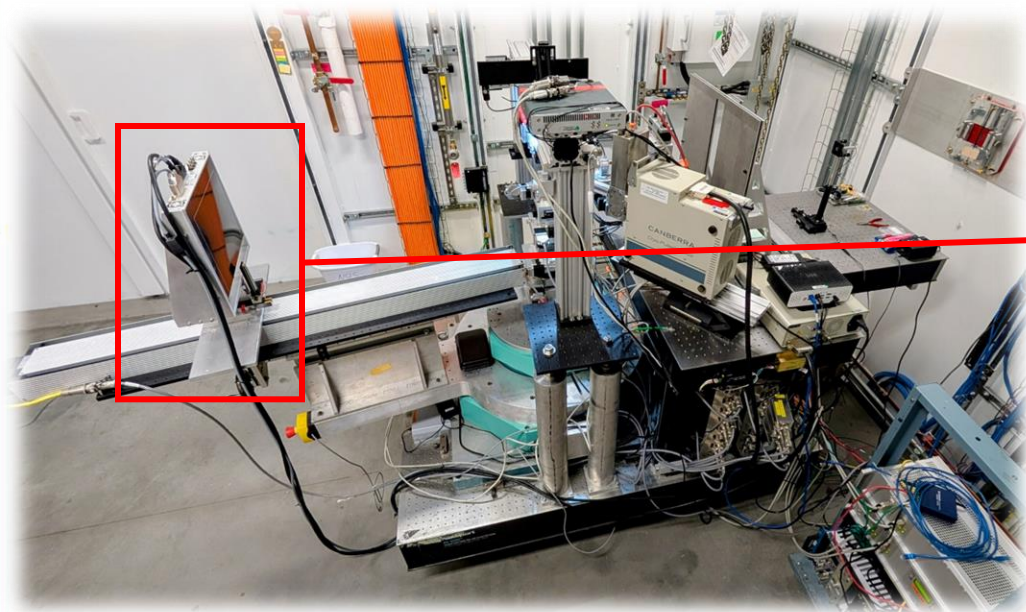


Google search for "Uranium melting point" showing a result for 2,070°F (1,132°C) with a snippet: "Uranium has a melting point of 1132°C. The chemical symbol for uranium is U, May 15, 2024".

Google search for "tungsten melting point kelvin" showing a result for 3,695 K with a snippet: "The free element is remarkable for its robustness, especially the fact that it has the highest melting point of all known elements, melting at 3,422 °C (6,192 °F; 3,695 K)".

Google search for "hafnium carbonitride melting point" showing a result for 4,000°C (7,230°F) with a snippet: "According to current research, hafnium carbonitride (HfCN) has a melting point exceeding 4,000°C (7,230°F), considered the highest known melting point of any material at ambient pressure; with some studies indicating a melting point around 4,110°C (7,318°F) for specific compositions like HfC0.75N0.22".

Enhancing existing equipment at 28ID-2-D



Current XRD detector

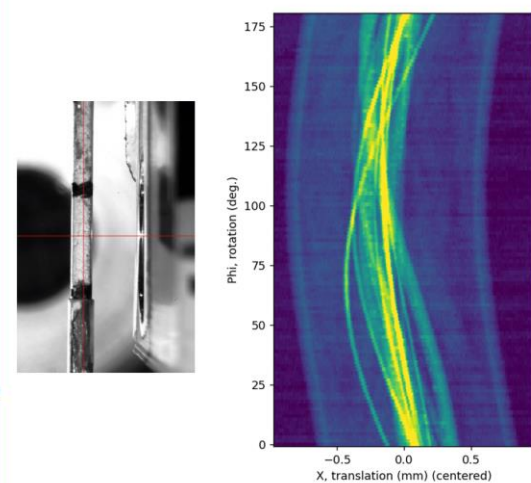


20 Hz frame rate

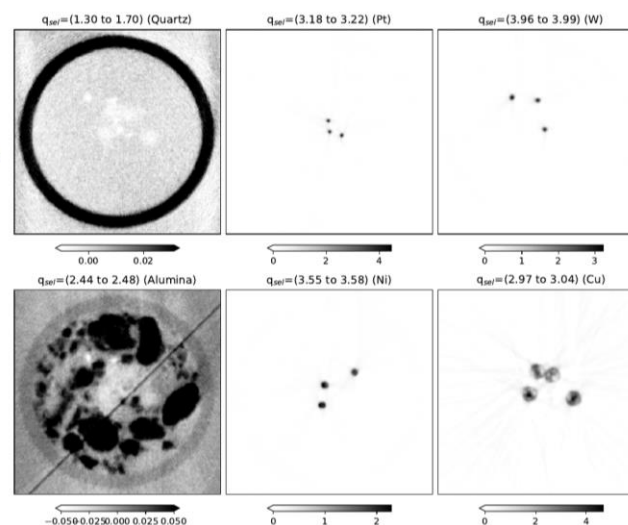
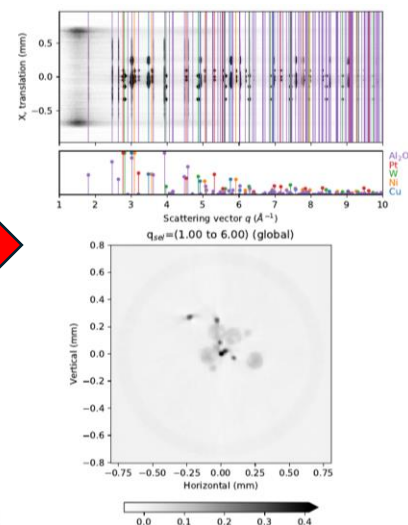
Better XRD detector



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

Acknowledgements

- Sanjit Ghose
- Eric Dooryhee
- Michael Drakopoulos
- Jiaming Bai
- John Trunk
- Lynne Ecker
- David Sprouster
- Kim Wehunt
- Steven Woodburn
- ...





Thanks