



# BNL Capabilities to Support Advanced Material Studies

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June 17, 2021



# Outline of Presentation

- Overview of the Isotope Program
- Present capabilities at BNL in irradiation and post-irradiation characterizing accelerator and nuclear materials on materials in utilizing its unique experimental facilities
- Discuss some of the previous work
- Outline a path forward in both irradiation damage capabilities, an exciting array of micro-characterization techniques at the NSLS-II synchrotron beamlines
- Present future capabilities at the Material in Radiations Environment MRE for Post Irradiation examination of radioactive materials



# Isotope Program Missions

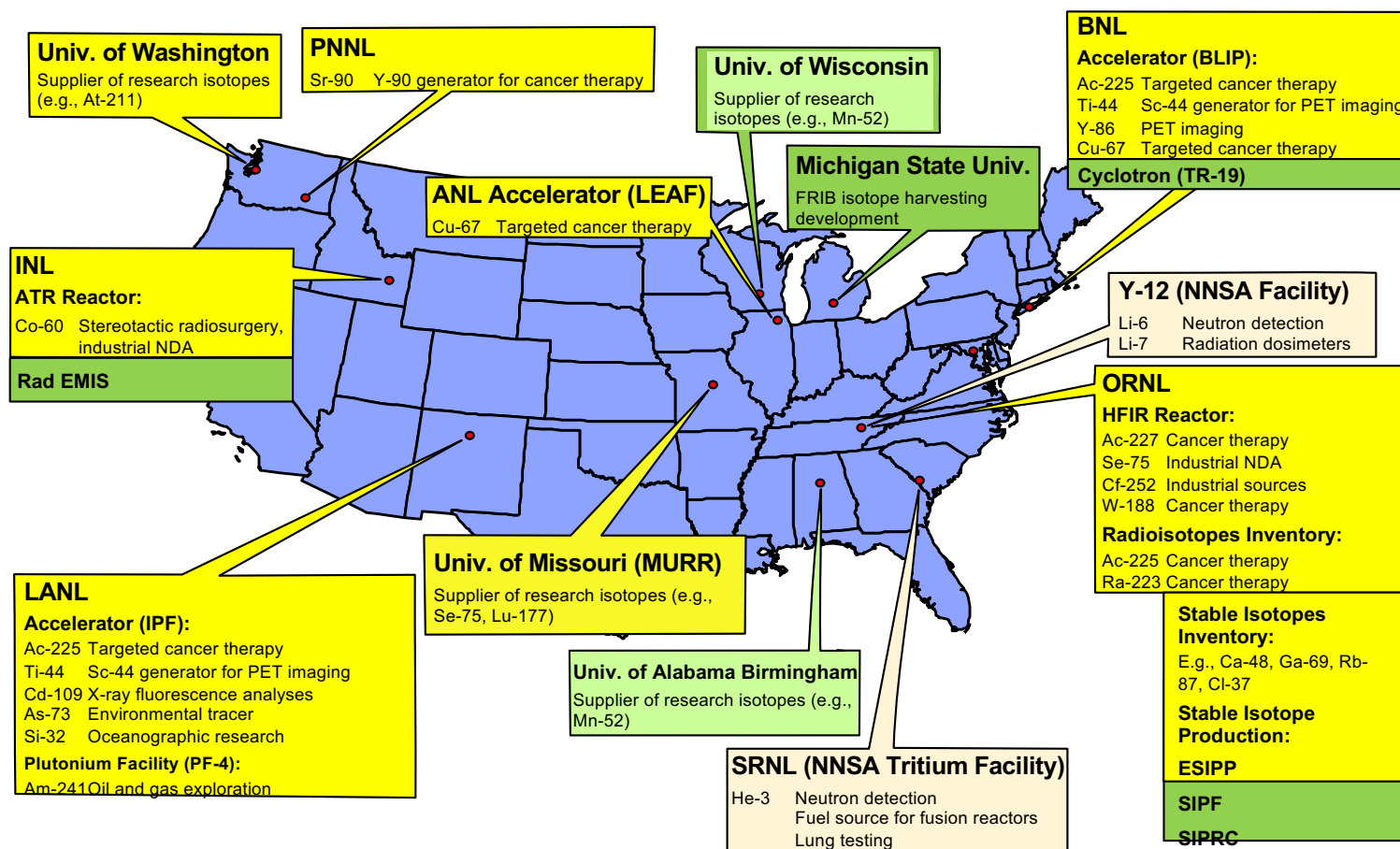
- Produce and/or distribute radioactive isotopes that are in short supply, including valuable by-products, surplus materials and related isotope services, mitigate reliance on foreign supply
- Maintain the infrastructure required to produce and supply isotope products and related services
- Conduct R&D on new and improved isotope production and processing techniques which can make available new isotopes for research and applications

## Attributes:

- Core R&D where there are programmatically stewarded activities
- Competitive R&D
- SBIR/STTR, Early Career Award Program
- Nuclear and Radiochemistry Summer School, Workforce Development

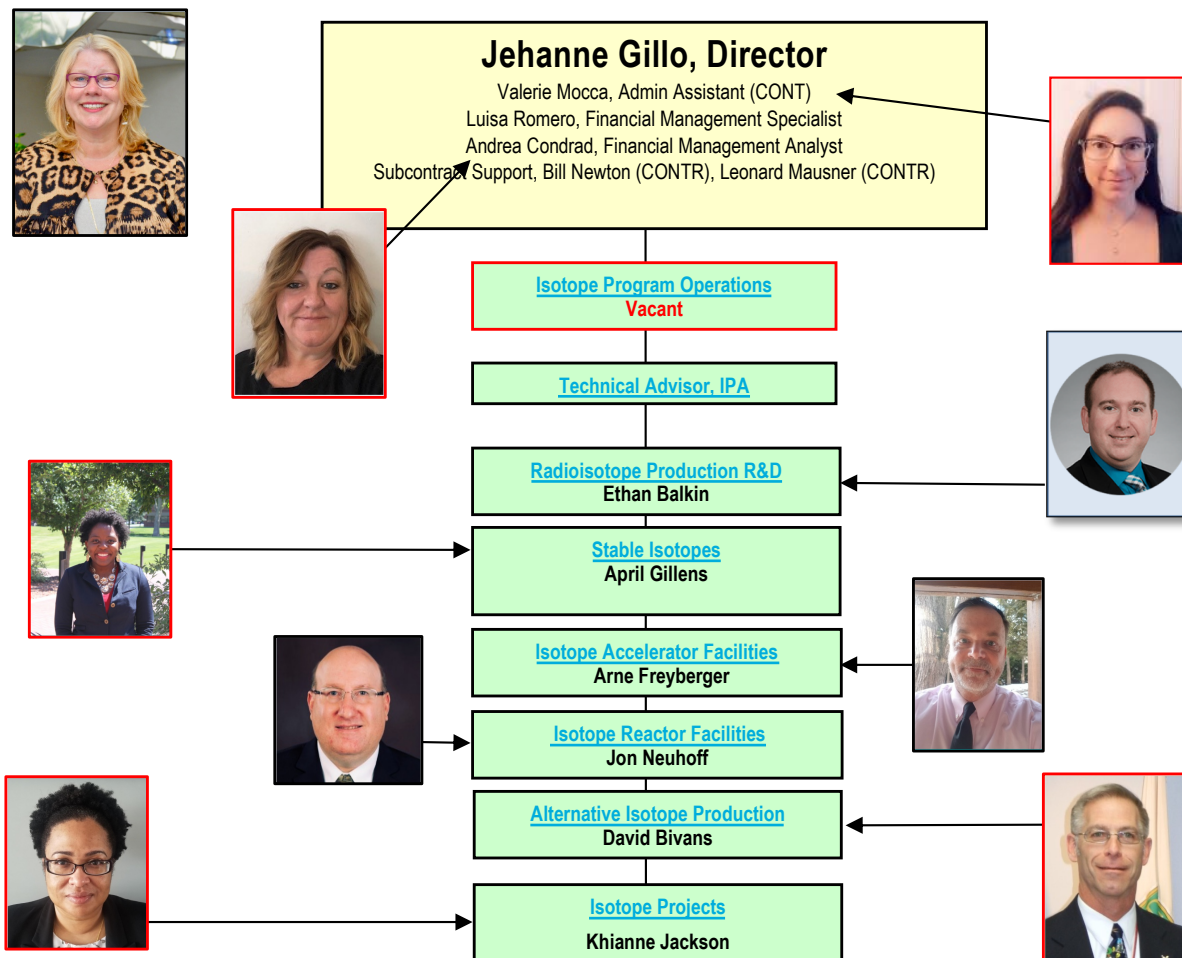


# DOE Isotope Program Production Sites

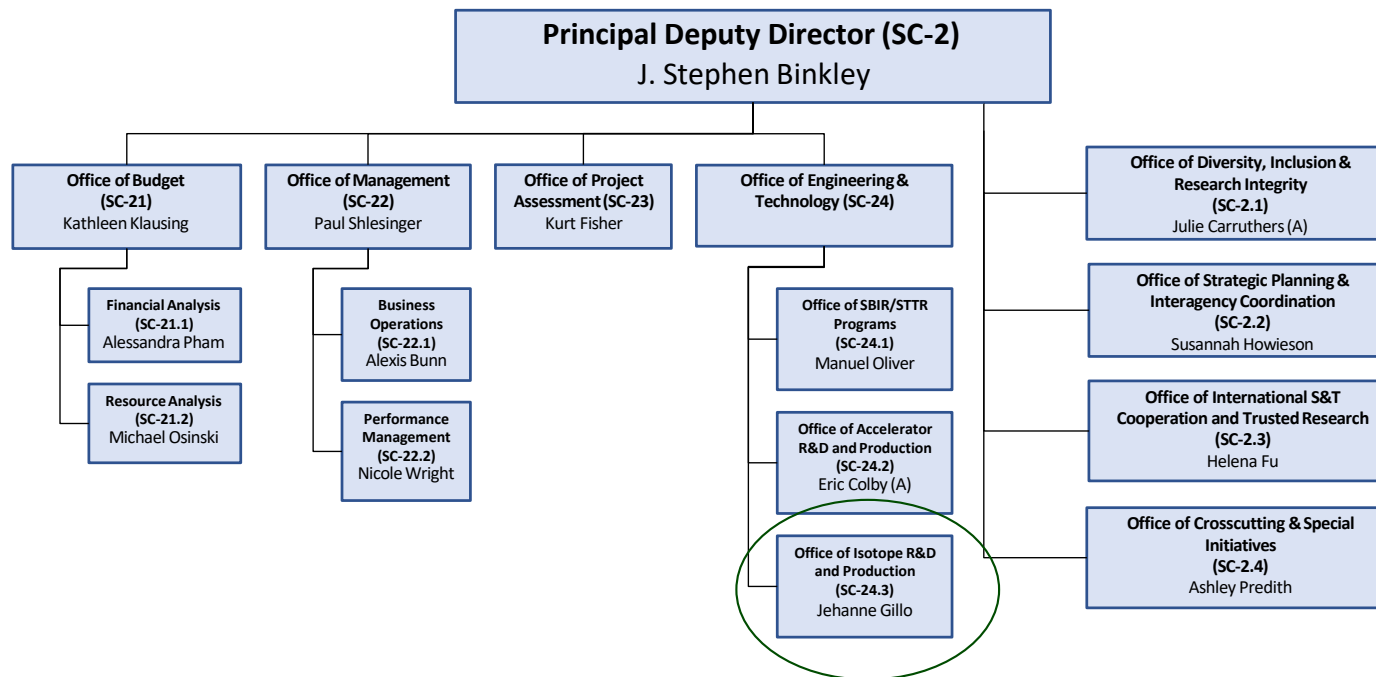




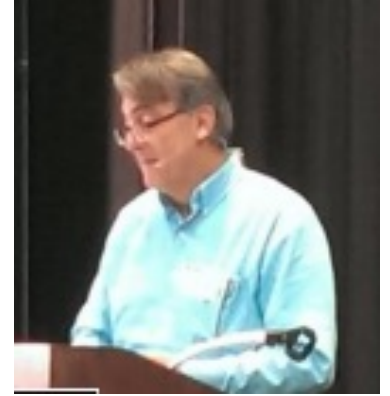
# Office of Isotope R&D and Production SC-24.3



# Office of Principal Deputy Director (SC-2)



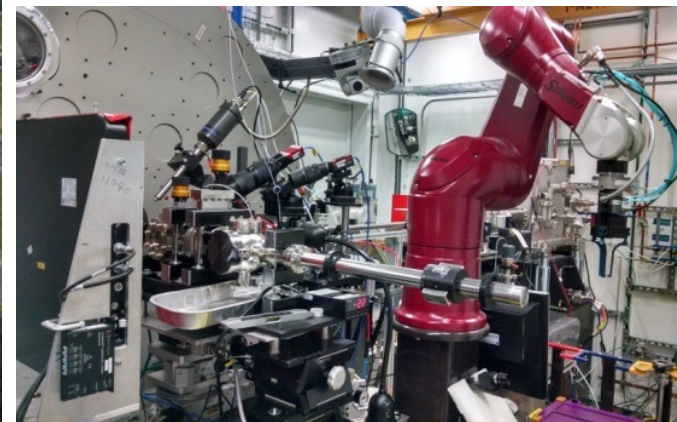
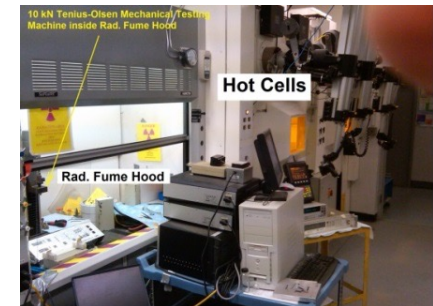
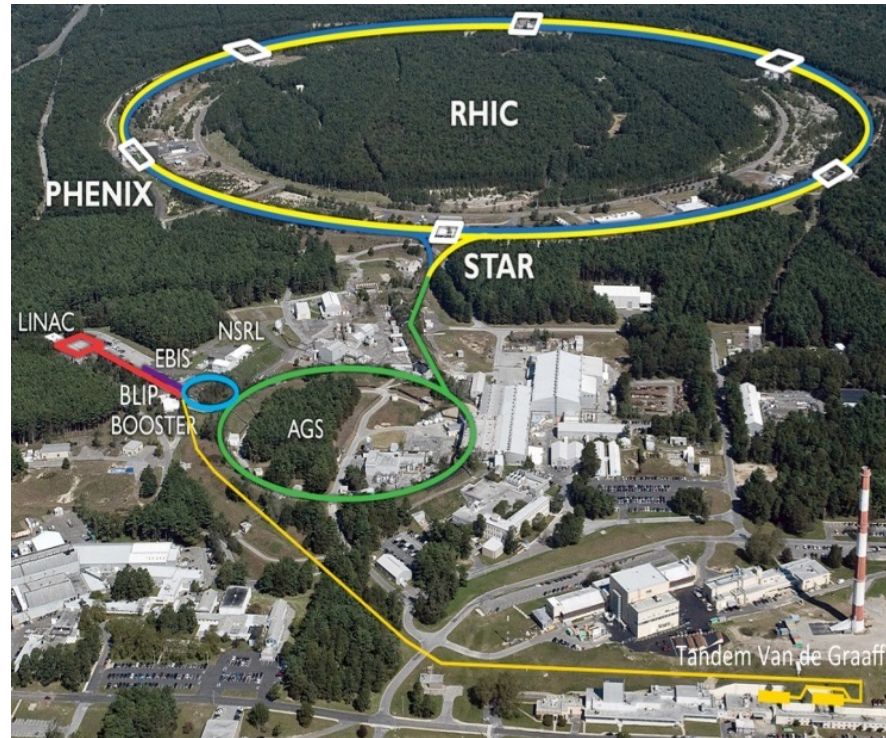
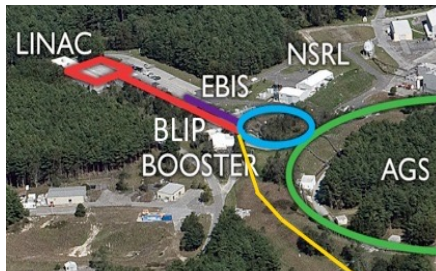
# Material Evaluations



- Need for materials for high power targetry and accelerator components
- Targetry for physics experiments and as IP needs are increasing need for targets that can survive multi-MW irradiations with particle beams.
- As has been demonstrated components that have been used for other applications are not always suitable and evaluations are necessary.
- Necessary to evaluate the long-term accumulated radiations damage as well as the short bursts that result in thermo-mechanical shock.



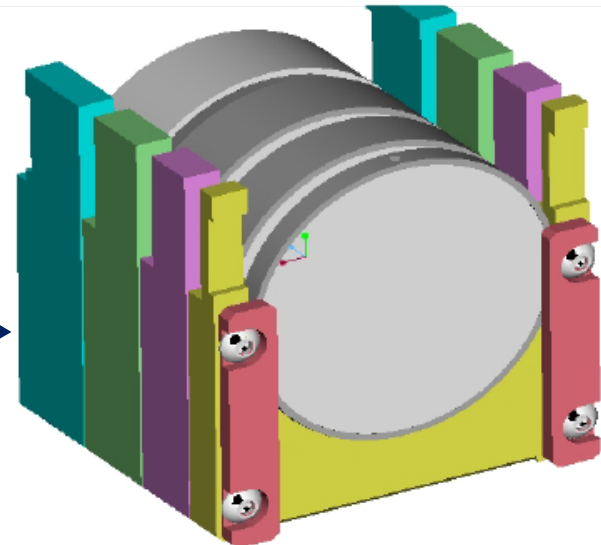
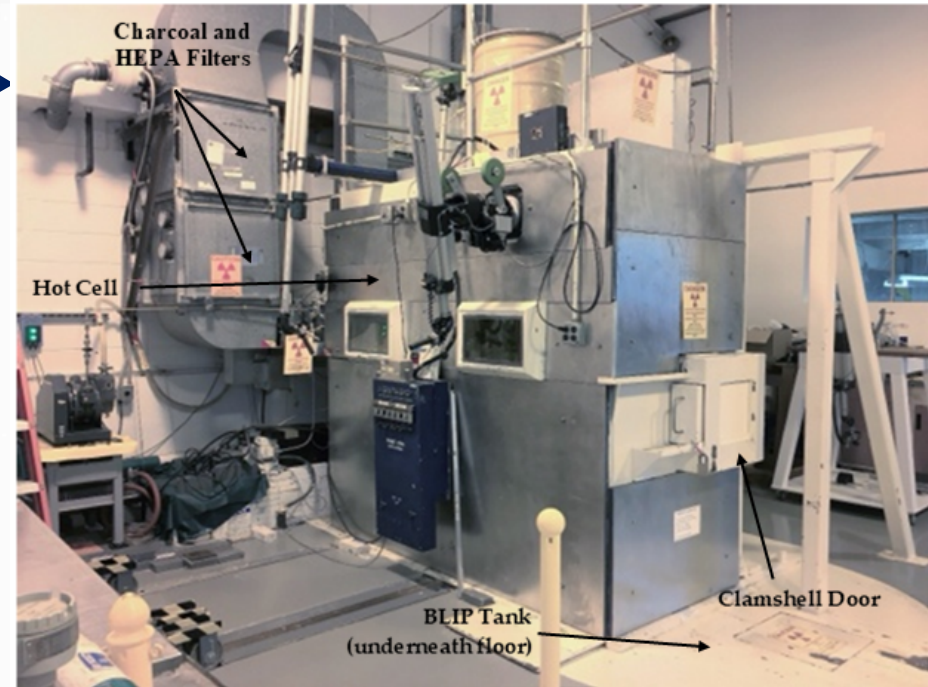
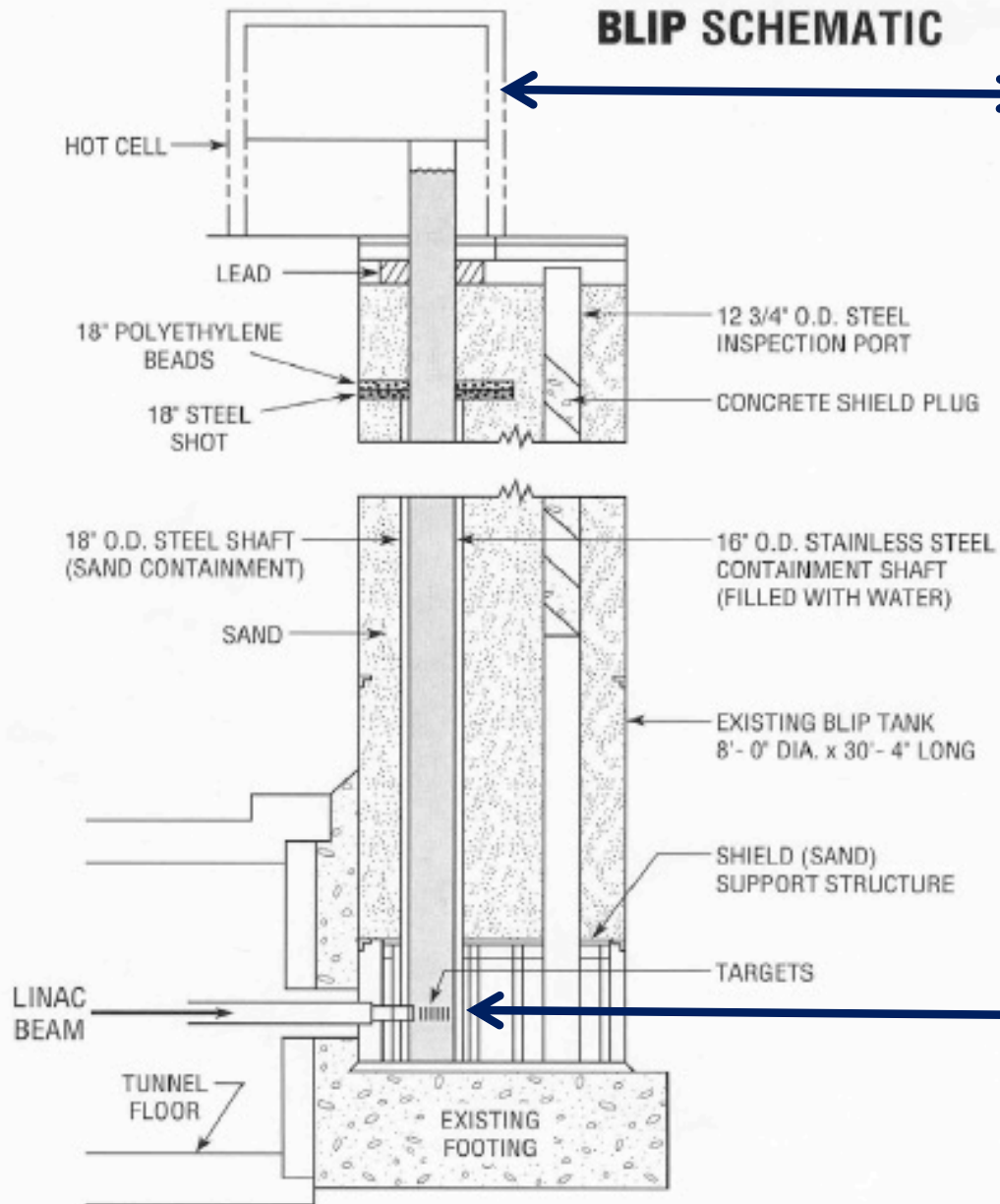
# Irradiation and Post-Irradiation





# Brookhaven Linear Isotope Producer

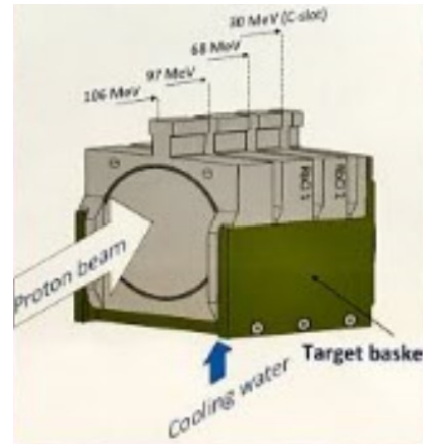
**BLIP SCHEMATIC**



# BLIP Targets



Target



- Targets are irradiated by the proton beam
  - Production targets for Sr
  - R & D targets
  - LINAC beam intensity: 50-165  $\mu$ A
  - Beam raster lowers power density on target to minimize risk of target failure enabling higher current and higher production yields
  - BLIP equipment allows as many as two target boxes per irradiation
- Targets undergo review process by Radiation Safety Sub-Committee including:
  - Radionuclides and activity expected after irradiation
  - Heating profile and heat transfer calculations
- Documentation maintained in “canning record” kept by RSC
- Metal foils used for R & D also reviewed against similar criteria as production targets
- Target arrays are configuration managed and checked before irradiations against target loading table

Target Submission Number: TS-TGT-16-01  
Appendix 2

Target Loading Table				
Title: Irradiation of thicker RbCl targets with rastered beam				
Target Array Number: TA-TGT-16-01				
Prepared by: D. Melchior				
Proposed Run Time (e.g. days, hrs.): 2 weeks, 05/18/2016-06/01/2016				
Desired Completion Date: 06/01/2016				
Requested Beam Energy (MeV): 117 incident		Requested Beam Current		
Item	Type and stamped ID (if available)	Layer #/s from "Target Array and Energy Propagation Calculations"	Thickness inch	mm
1	Water gap	6	0.200	5.080
2	SS slab degrader	7	0.058	1.473
3	Water gap	8	0.200	5.080
4	RbCl 1	9-11	0.720	18.29
5	Water gap	12	0.200	5.080
6	RbCl 2	13-15	0.065	1.651
7	Water gap	16	0.200	5.080
8	Cu stop	17	0.305	7.747
9	Water gap	18	0.200	5.080
10	Al or Cu stop	19	0.220	5.588

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2. Target Material and Properties	
Target Name: To be determined	Target & Canning No. Assign unique no. (year-000)
Target Material Properties	
Purity or Grade	≥99.8 %, Analytical grade
Chemical Formula	RbCl
Physical Characteristics at 70 °F or 21 °C	solid
Physical Form	Disc
Elements (%)	See 5. Target
Melting Point	782 °C (1440 °F)
Boiling Point	1384 °C (2503 °F)
Thermal Conductivity	2.28 W.m <sup>-1</sup> .K <sup>-1</sup>
Density	2.80 g/cm <sup>3</sup>
Specific Heat	52.4 J/kg.K
Does the Target material react with any of the following?	
Aluminum	
H <sub>2</sub> O	
Inconel 600	
Canning	
Chemical Formula	Inconel 600
Casing Wall Thickness (inches/mm)	0.012 / 0.30
Casing Dimensions (inches/mm)	Casing Diameter 1.354
Melting Point	1354 °C (2469 °F)
Thermal Conductivity	14.9 W.m <sup>-1</sup> .K <sup>-1</sup>
Density	8.44 g/cm <sup>3</sup>
Specific Heat	444 J/kg.K

6. Activation Analysis of Target Material and Casing					
Main isotopes produced in 3 week (140 $\mu$ A) irradiation of RbCl targets, their nuclear data, and activities					
Isotope	Half-life	E <sub>γ</sub> , keV (I, %)	End of Bombardment	Activity, Ci	7 days post EOB
Sr-82 (Rb-82)	25.34 d	778.52 (15.0)	16.80	13.84	
Sr-83	1.35 d	381.53 (14.0)	69.13	1.90	
Sr-85	64.85 d	762.45 (26.7)	5.04	4.68	
Rb-79	0.0159 d	688.1 (23.1)	28.59	decayed	
Rb-81	0.1905 d	190.46 (24.8)	68.93	mil	
Rb-82 (metastable)	0.270 d	554.35 (62.4)	60.71	mil	
Rb-83	86.2 d	776.52 (64.39)	10.99	11.40	
Rb-84	32.82 d	639.11 (37.98)	44.38	38.27	
Rb-86	18.642 d	520.4 (45)	18.80	14.50	
Kr-79	1.46 d	529.59 (29.3)	21.49	0.79	
Br-77	2.38 d	552.55 (16.0)	8.03	1.00	

Contribution to the dose rate from Inconel window activation is negligible  
Most prominent gamma rays are shown  
\*Calculated from cross-section data reported by E. Z. Burdette et al, Applied Radiation and Isotopes, vol.64, p.915 (2006), 10.1016/j.apradiso.2006.03.002

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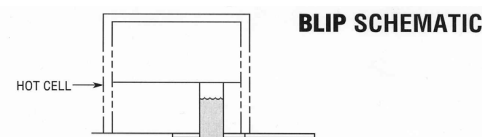
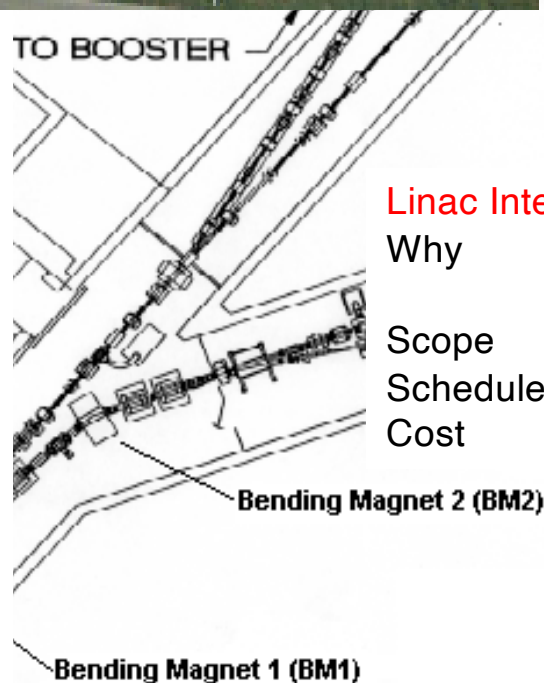


# 200 MeV H- beams LINAC/BLIP



- First beam: fall 1972
- 85-100% of all Linac pulses go to BLIP
- Target irradiation with 66 – 200 MeV, 165  $\mu\text{A}$
- Radioisotope production for diagnostic (e.g.  $^{82}\text{Sr}$ ,  $^{68}\text{Ge}$ ) and therapeutic ( $^{225}\text{Ac}$ ) use

Contact: D. Raparia



## Linac Intensity Upgrade Phase II (shovel-ready)

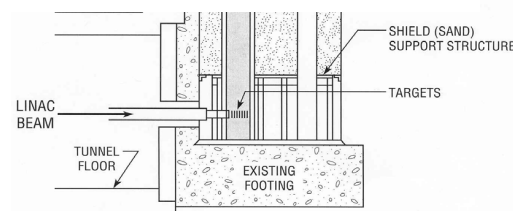
Why : 2x increase in beam current and significant increase in isotope production capacity

Scope : Linac pulse length doubling to 900 ms for  $I_{\text{avg}}$  250 mA

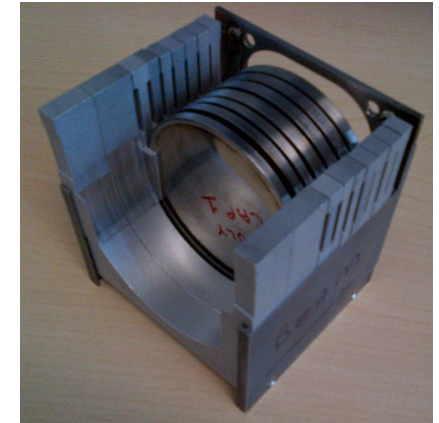
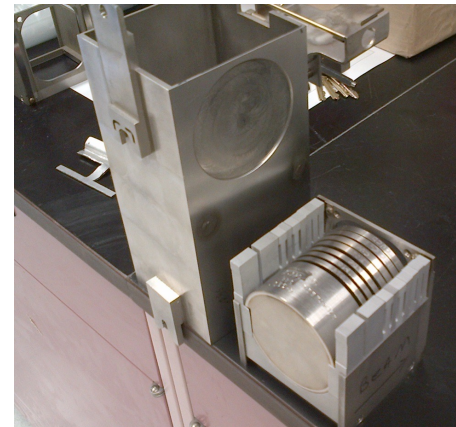
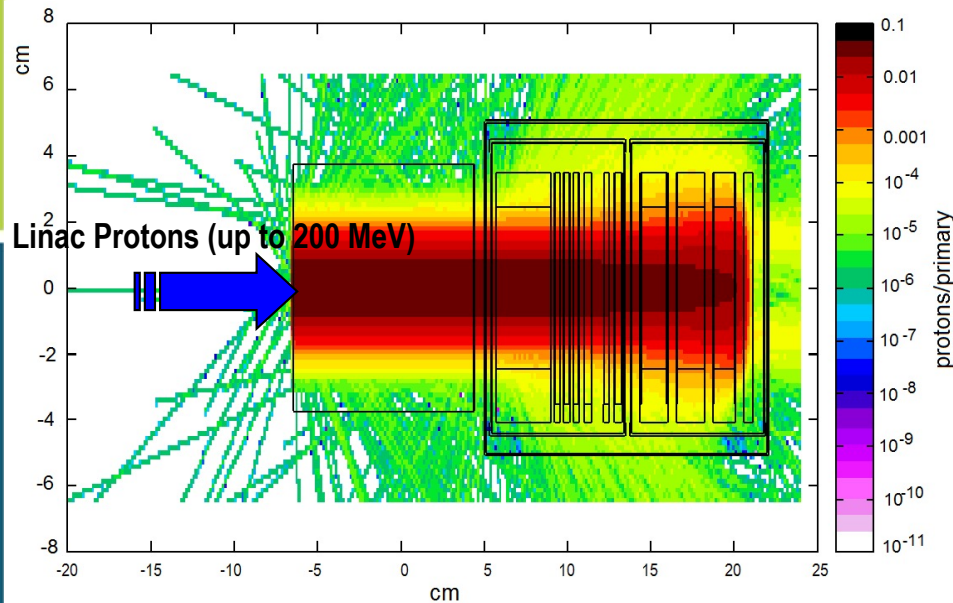
Schedule: ~3-4 years (with cost minimization)

Cost : ~\$17.4M (depending on scope and schedule)

EL  
3T  
LD PLUG  
SS STEEL  
(SHAFT  
ATER)  
ANK  
4' LONG

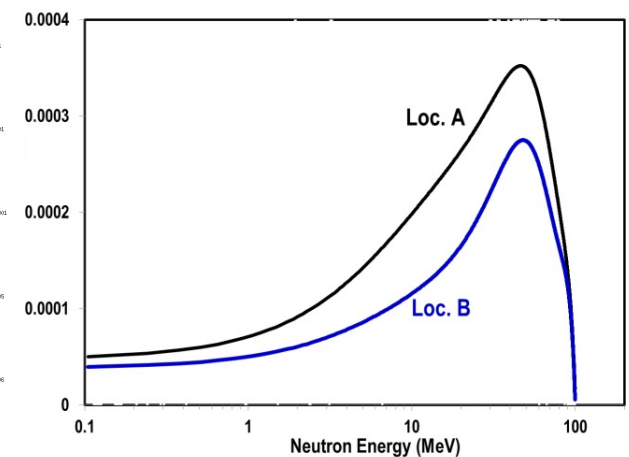
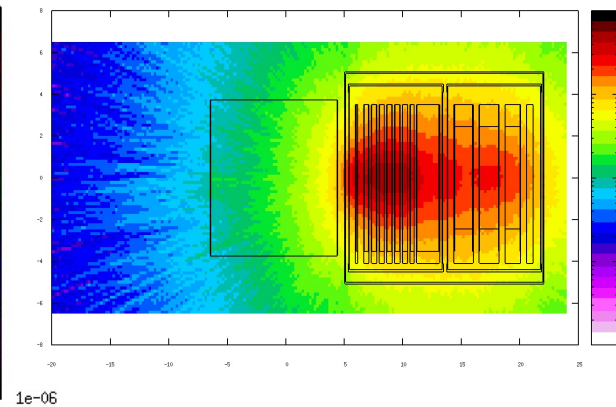
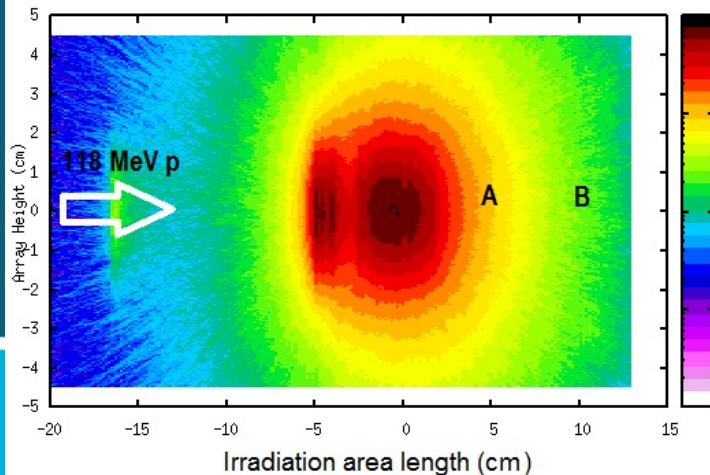


# BNL Irradiation Facilities and Capabilities



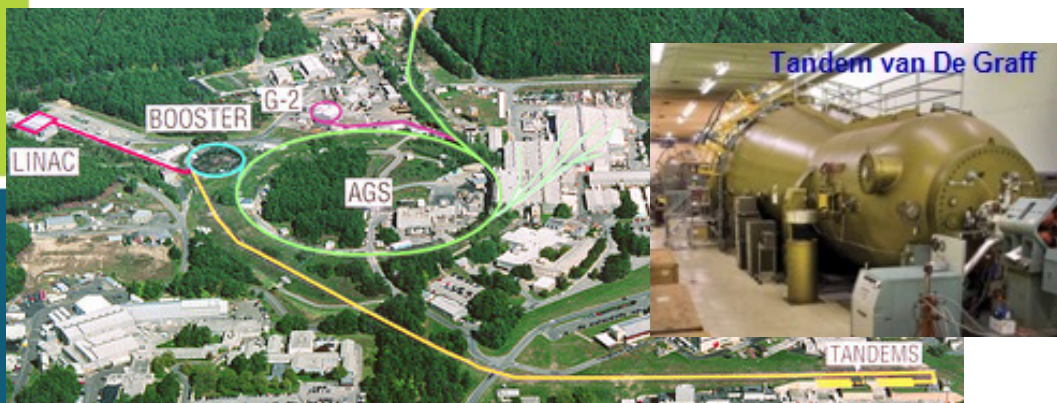
## Fast Neutron Spectra and Nuclear Materials at BNL/BLIP

Spallation Neutron Irradiation Set-up at BLIP

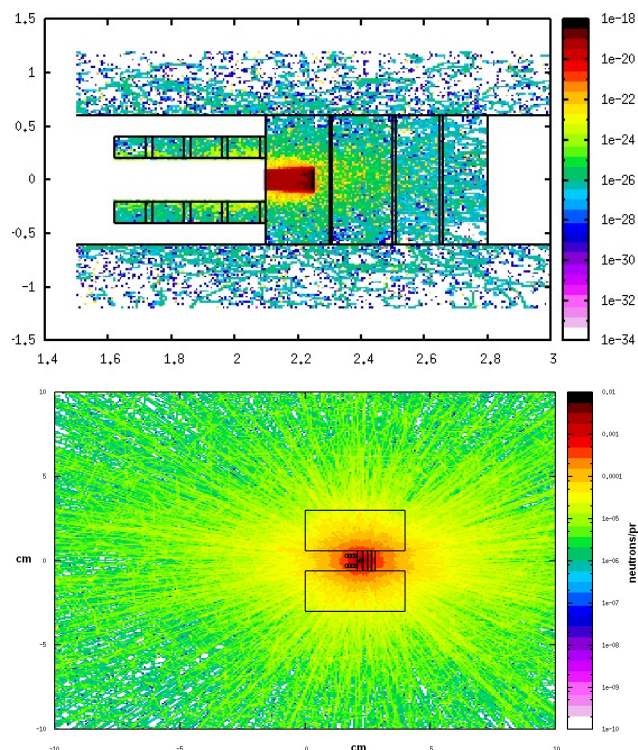
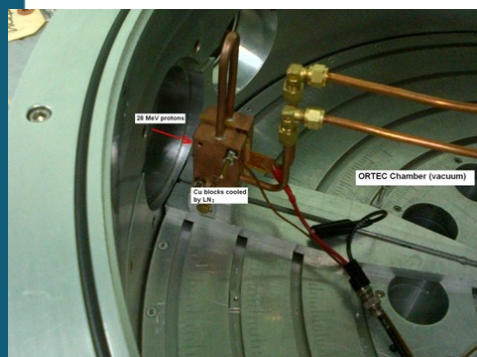




# Ion irradiation at BNL Tandem



28 MeV irradi. at sub-zero temp.



		Mass	Max Energy		Surface LET	Range	Surface LET	Range
Z	Symbol	AMU	MeV	MeV AMU	MeV mg/cm <sup>2</sup>	Microns	MeV mg/cm <sup>2</sup>	Microns
1	<sup>1</sup> H	1.0079	28.75	28.52	<a href="#">0.0153</a>	4550	<a href="#">0.0118</a>	2610
3	<sup>7</sup> Li	7.0160	57.2	8.15	<a href="#">0.369</a>	390	<a href="#">0.273</a>	240
5	<sup>11</sup> B	11.0093	85.5	7.77	<a href="#">1.08</a>	206.13	<a href="#">0.754</a>	132.55
6	<sup>12</sup> C	12.0000	99.6	8.30	<a href="#">1.46</a>	180.43	<a href="#">1.03</a>	115.82
8	<sup>16</sup> O	15.9994	128	8.00	<a href="#">2.61</a>	137.78	<a href="#">1.83</a>	88.9
9	<sup>19</sup> F	18.9954	142	7.48	<a href="#">3.51</a>	118.88	<a href="#">2.45</a>	77.12
12	<sup>24</sup> Mg	23.9927	161	6.71	<a href="#">6.01</a>	84.16	<a href="#">4.17</a>	55.13
14	<sup>28</sup> Si	28.0855	187	6.66	<a href="#">7.81</a>	77.16	<a href="#">5.42</a>	50.66
17	<sup>35</sup> Cl	34.9688	212	6.06	<a href="#">11.5</a>	64.41	<a href="#">7.93</a>	42.71
20	<sup>40</sup> Ca	39.9753	221	5.53	<a href="#">15.8</a>	51.89	<a href="#">10.9</a>	34.7
22	<sup>48</sup> Ti	47.9479	232	4.84	<a href="#">19.6</a>	47.8	<a href="#">13.4</a>	32.36
24	<sup>52</sup> Cr	51.9405	245	4.72	<a href="#">22.3</a>	45.86	<a href="#">15.3</a>	31.06
26	<sup>56</sup> Fe	55.9349	259	4.63	<a href="#">25.1</a>	44.24	<a href="#">17.2</a>	30.09
28	<sup>58</sup> Ni	57.9353	270	4.66	<a href="#">27.9</a>	44.56	<a href="#">19.1</a>	30.47
29	<sup>63</sup> Cu	62.9296	277	4.40	<a href="#">30.1</a>	42.06	<a href="#">20.6</a>	28.79
32	<sup>72</sup> Ge	71.9221	273	3.80	<a href="#">35.9</a>	37.94	<a href="#">24.4</a>	26.25
35	<sup>81</sup> Br	80.9163	287	3.55	<a href="#">41.3</a>	37.50	<a href="#">28.0</a>	26.11
41	<sup>93</sup> Nb	92.9060	300	3.23	<a href="#">47.5</a>	36.32	<a href="#">32.1</a>	25.4
47	<sup>107</sup> Ag	106.9051	313	2.93	<a href="#">59.2</a>	32.48	<a href="#">39.9</a>	22.89
53	<sup>127</sup> I	126.9045	322	2.54	<a href="#">66.9</a>	32.54	<a href="#">45.0</a>	23.17
79	<sup>197</sup> Au	196.9665	337	1.71	<a href="#">84.6</a>	29.21	<a href="#">56.2</a>	21.18



# BNL Post-Irradiation Facilities

## Isotope Extraction and Processing

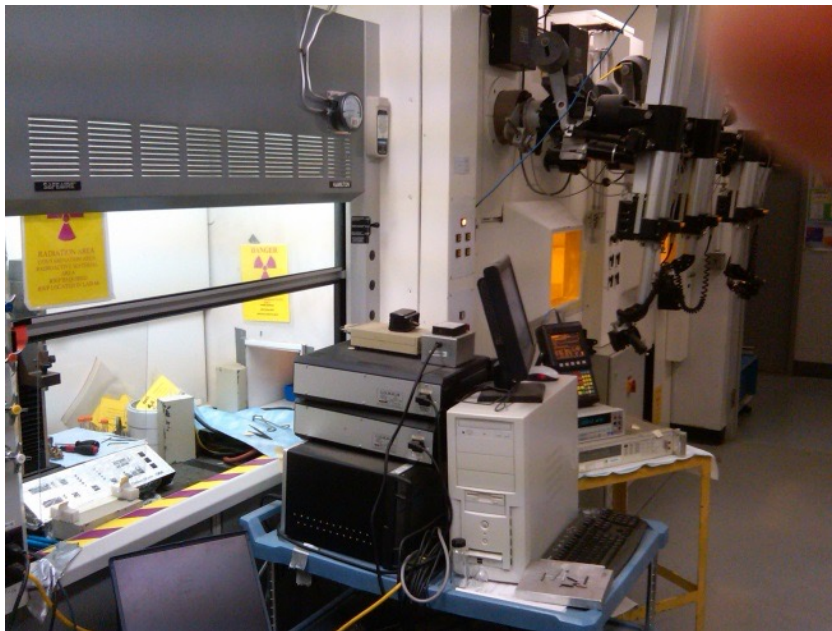
Experimental Facility occupies 2 hot cells and a HEPA-filtered fume hood

PLUS

Photon spectra and isotopic analysis

Activity measurements

Weight loss or gain

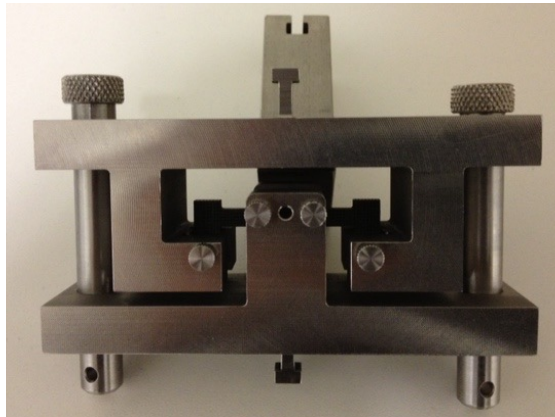
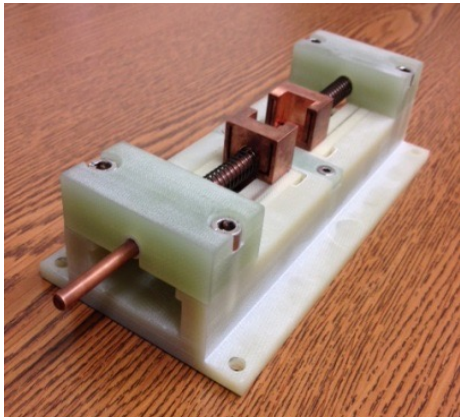
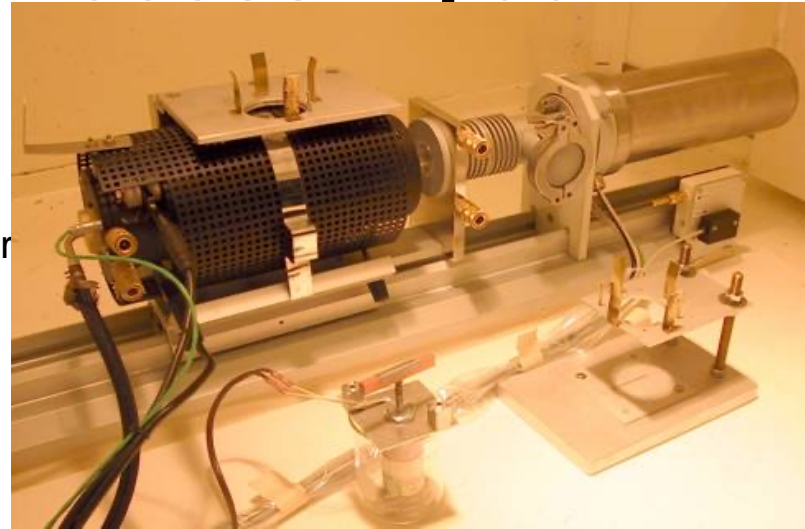


# BNL Post-Irradiation Facilities

## Isotope Extraction and Processing at BNL

### Macroscopic PIE analyses performed are:

Stress-strain (tension, 3-point and 4-point bending)  
Thermal Expansion and annealing (sensitive dilatometer Linseis)  
Thermal Conductivity (electrical resistivity)  
Magnetic Whole probe  
Ultrasonic measurements

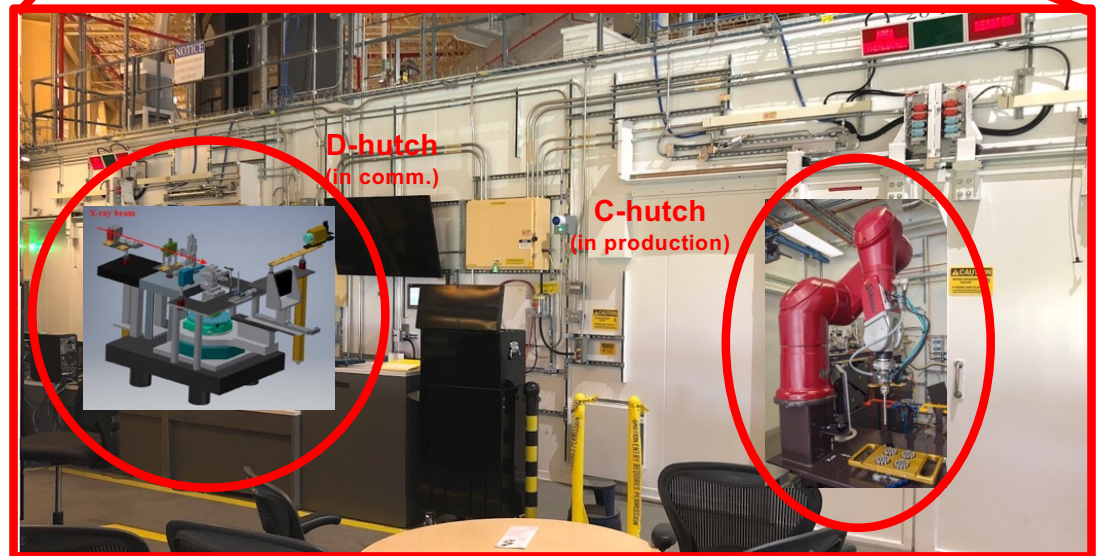




# **Post-Irradiation Characterization at BNL Synchrotrons**



# PIE: X-ray diffraction of irradiated materials

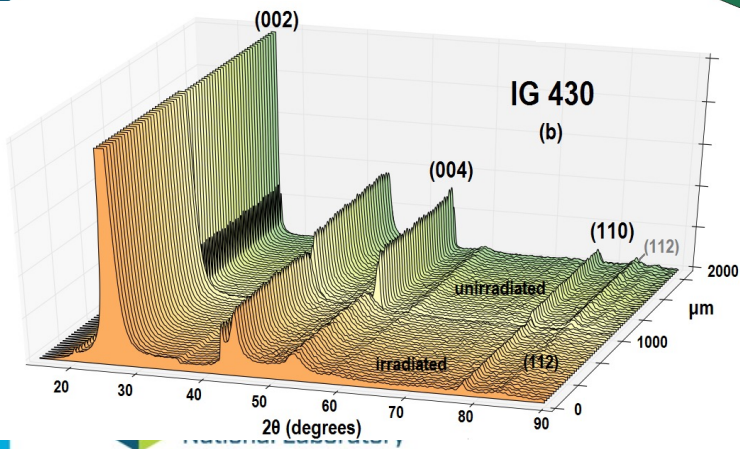
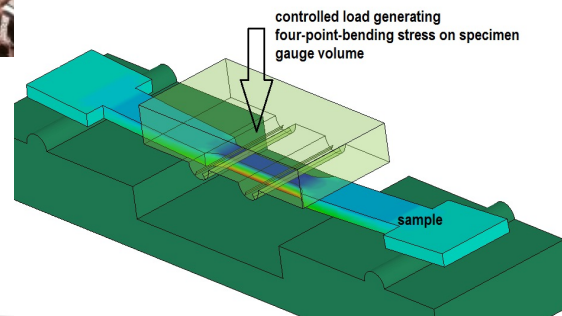
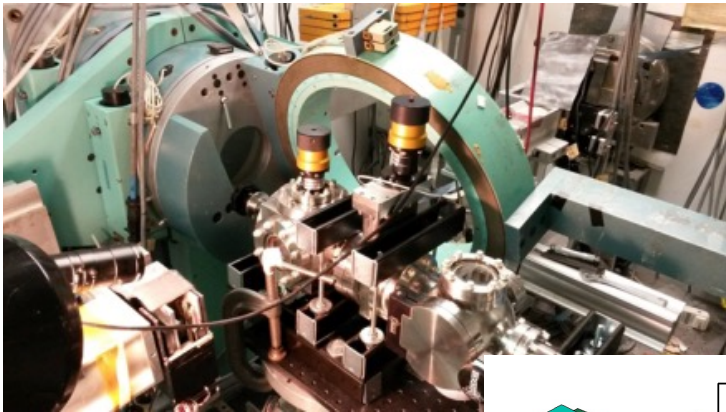


High-resolution X-ray diffraction  
beamline (XPD) at NSLS-II



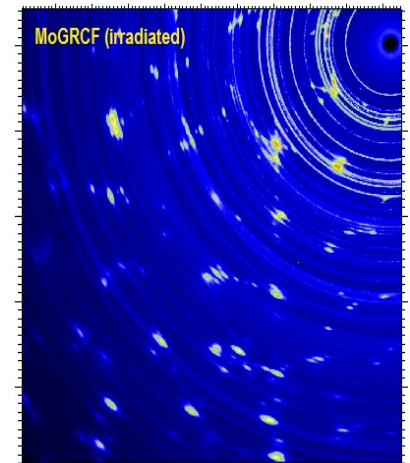
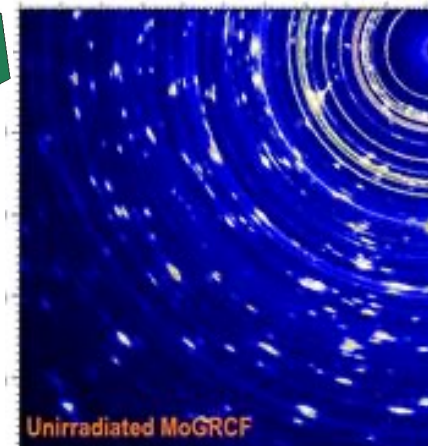
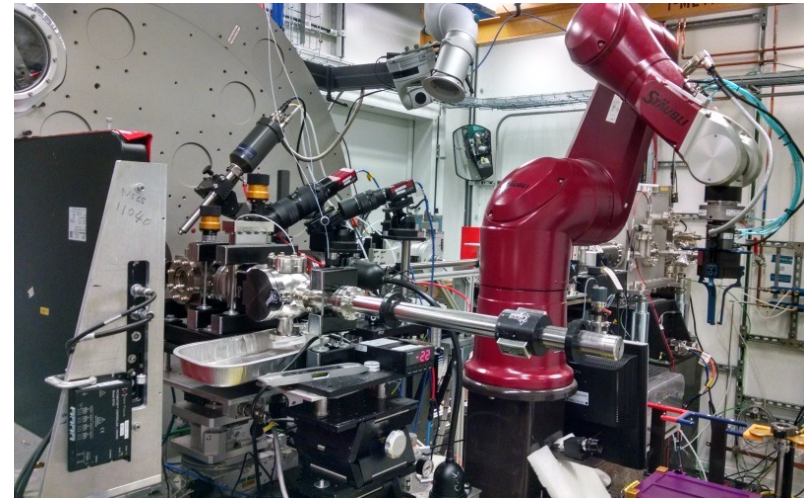
## NSLS (end of life)

- X17B1 Beamline (200 keV, polychromatic beam, EDXRD)
- X17A (70 keV, monochromatic X-rays, XRD)



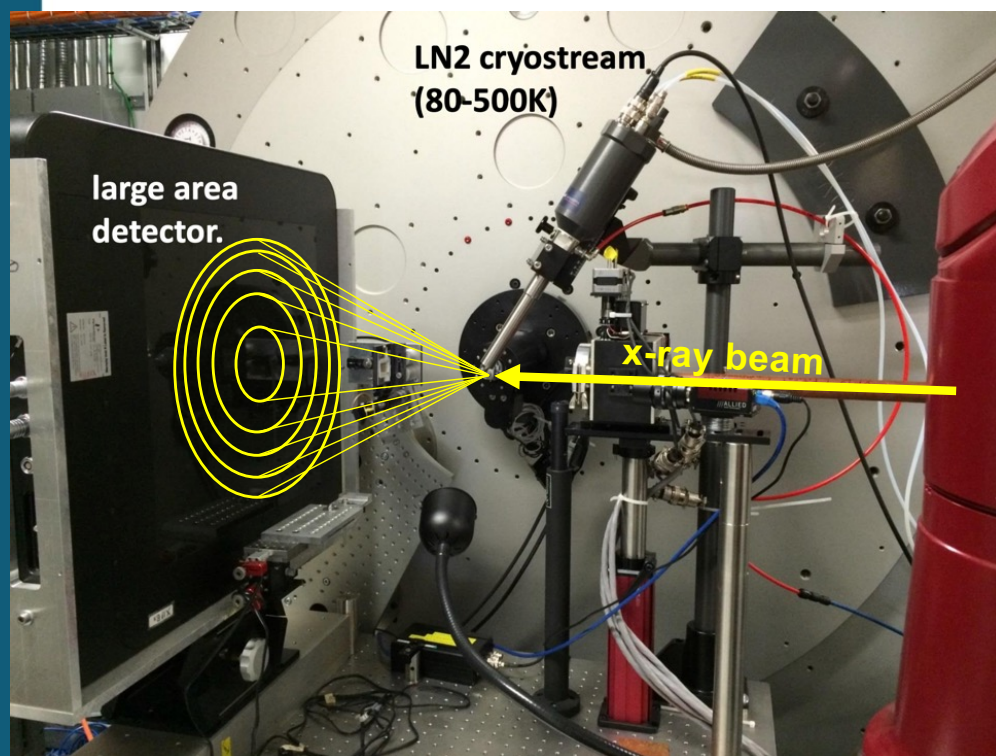
## NSLS II (XPD) Beamline

- XRD
- PDF
- X-Ray Tomography\*

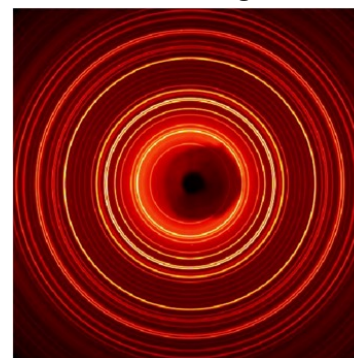


# One-slide on X-ray diffraction method

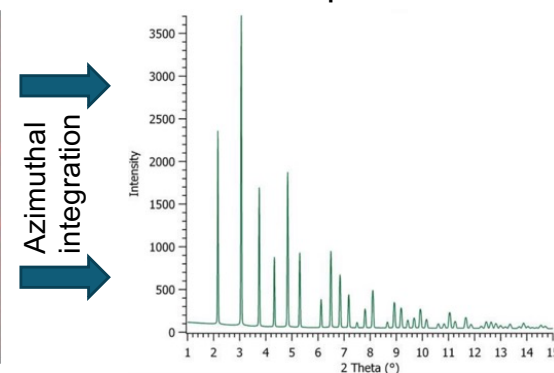
A typical X-ray diffraction setup with area detector



2D image

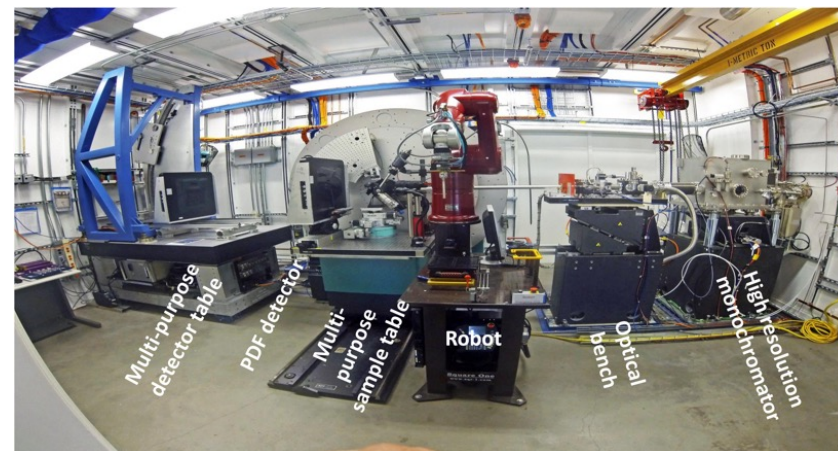


1D pattern

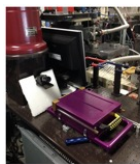


- Phase information
- Lattice parameters
- Phase mass fraction
- Micro strain
- Crystalline size
- Stacking faults

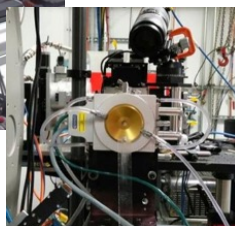
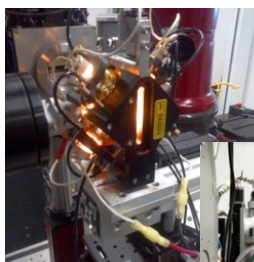




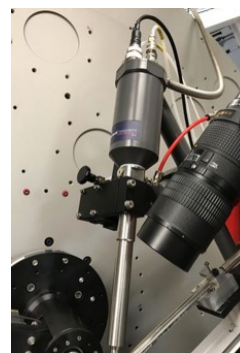
Sample Holder



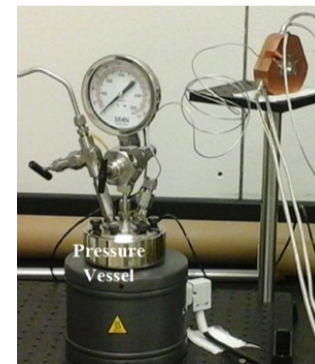
Robot for high-throughput sample changing



Heating up to 1500C



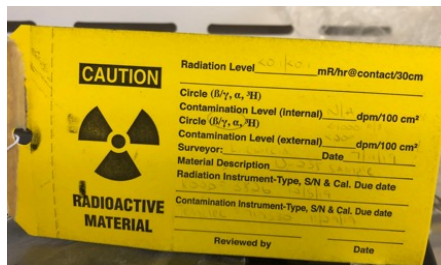
Cryostream (80K-500K)



Corrosion cell



# We are experienced with radioactive materials at NSLS-II



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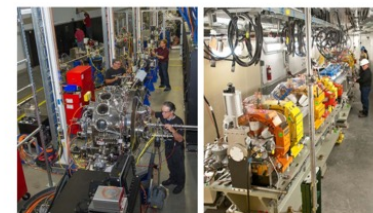
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## NSLS-II PROCEDURE: PROPERTY PROTECTION AREA (PPA) ACCESS CONTROL AND SECURITY

September 25, 2019

Version 1

L. Stiegler



**Think Safety. Act Safely.**

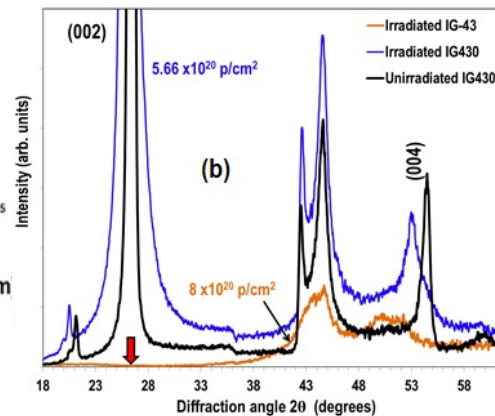
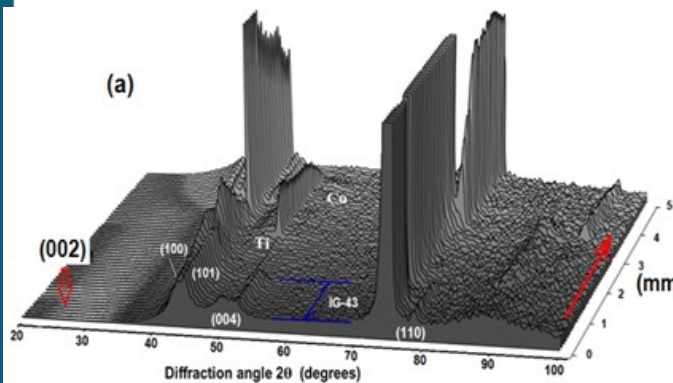
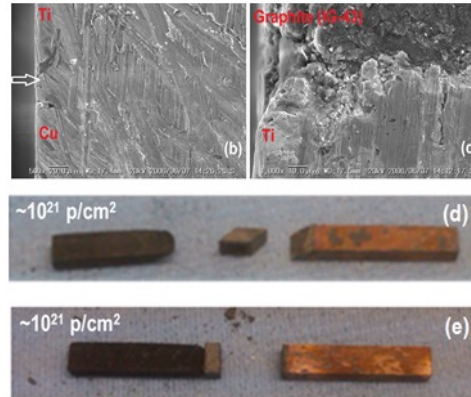
# Overview of nuclear material studies at BNL

Radiation damage effects – from Graphite to Tungsten

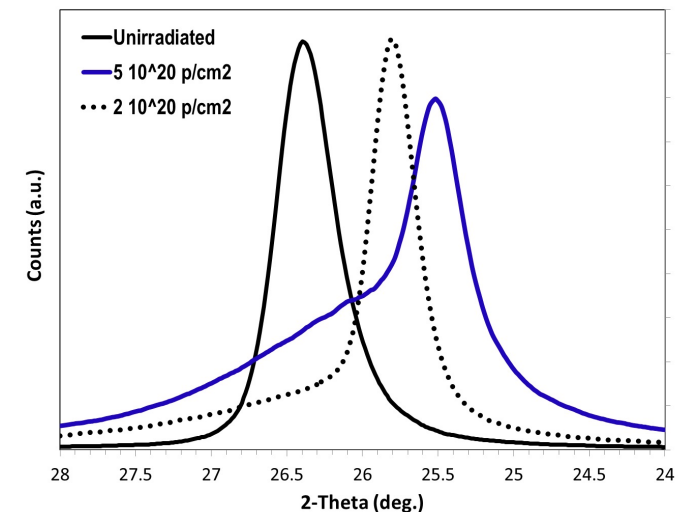
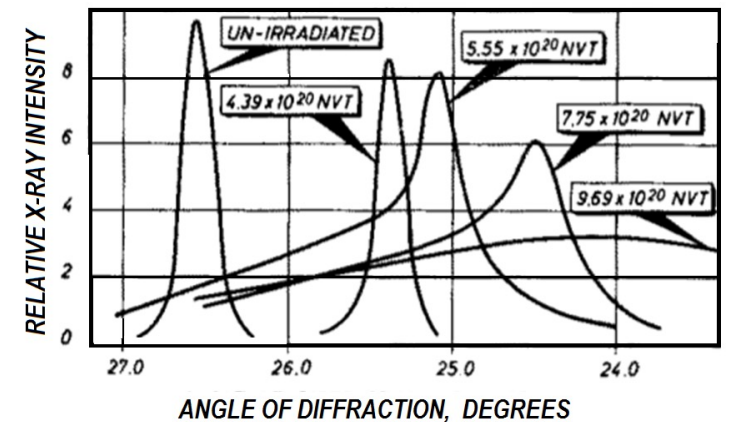
Linking macrostructure to radiation-induced lattice defects

# Research on Graphite: Irradiation → Post-irrad

J-PARC Muon target – BNL Studies



Correlating damage in GRAPHITE between neutrons and protons at BNL



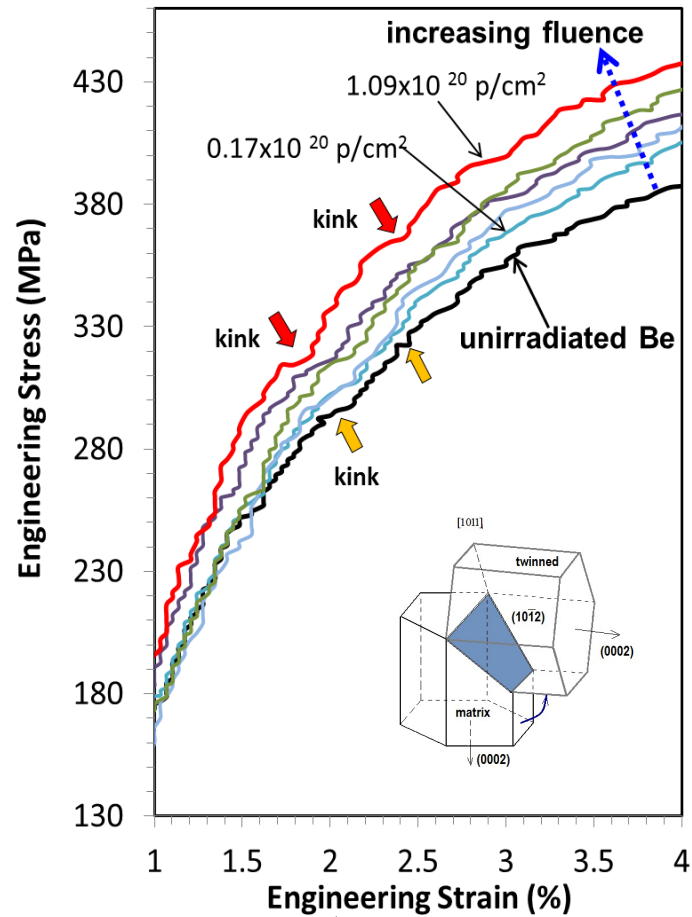
N. Simos, et al., "Solid Target Studies for Muon Colliders and Neutrino Beams," Nuclear Physics B, 155, pp. 288-290, 2006

N. Simos, H. Ludewig, et al., "Multi-MW accelerator target material properties under proton irradiation at Brookhaven National Laboratory linear isotope producer," *Physical Review Accelerators and Beams* (2018) 21 (5)

N. Simos, et al., "Proton Irradiated Graphite Grades for a Long Baseline Neutrino Facility Experiment," *Physical Review Accel. and Beams* 20, 071002 2017



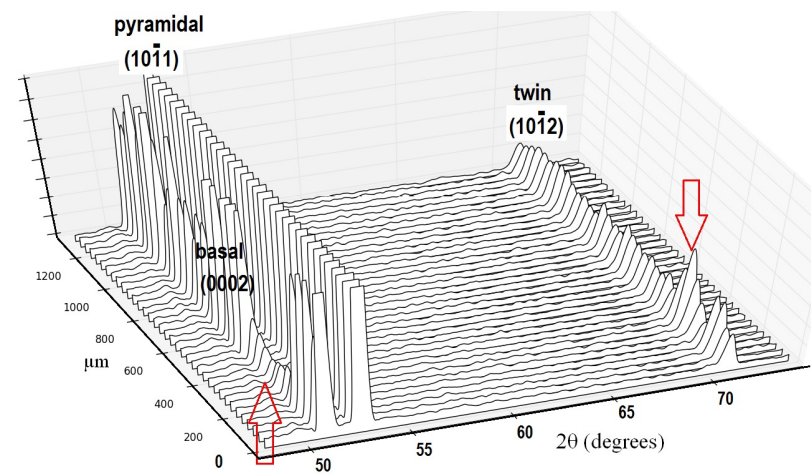
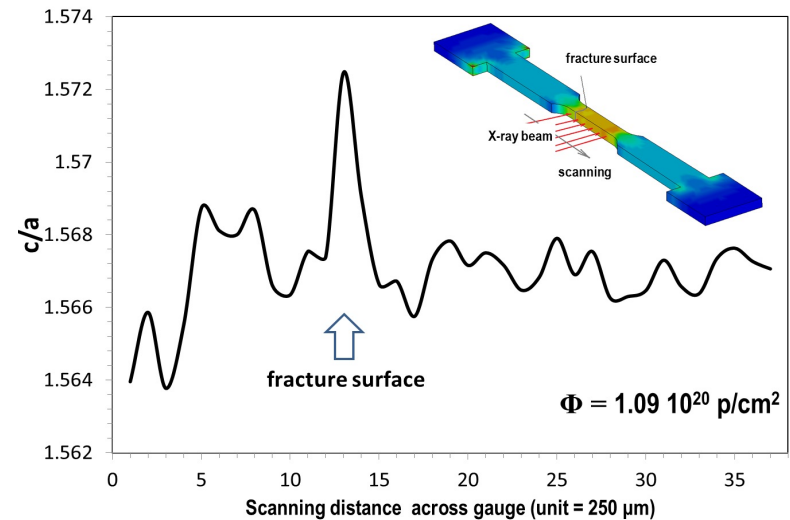
# HCP Beryllium (S 200F) at BNL



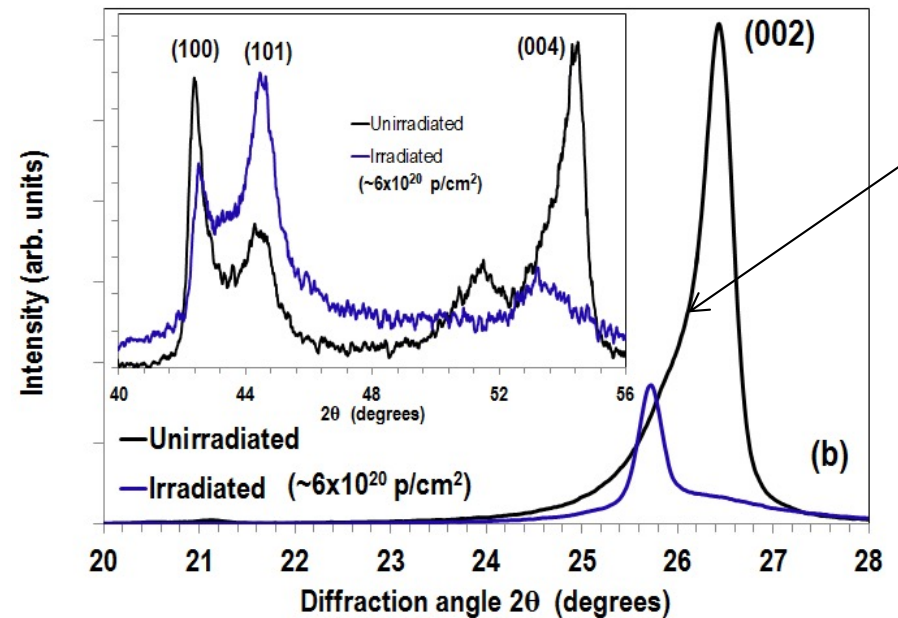
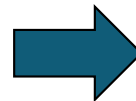
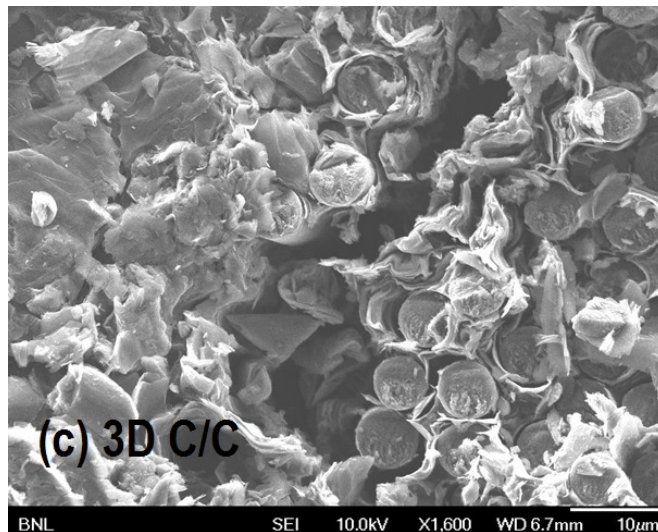
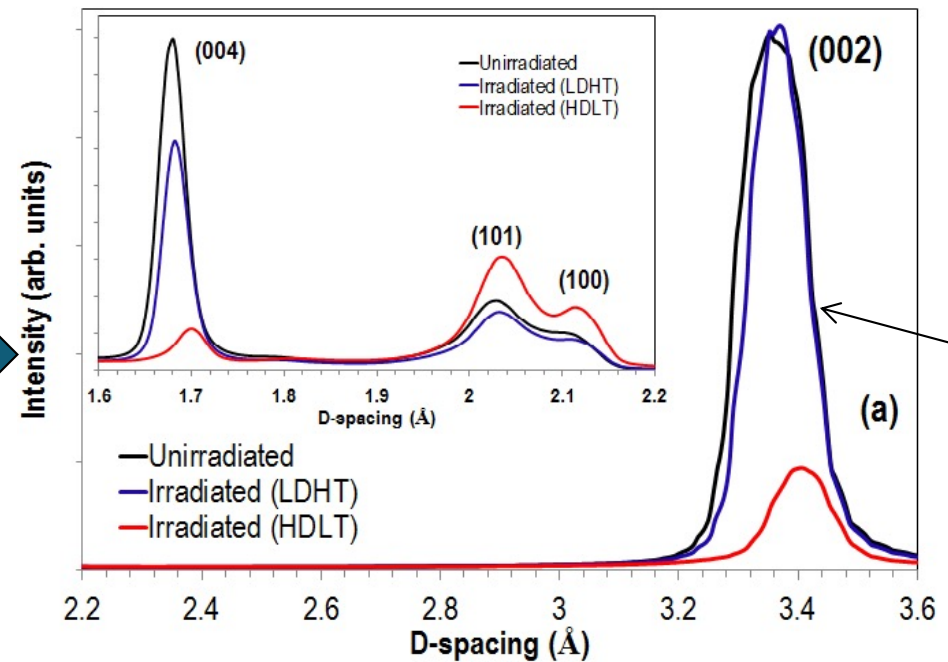
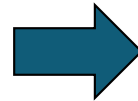
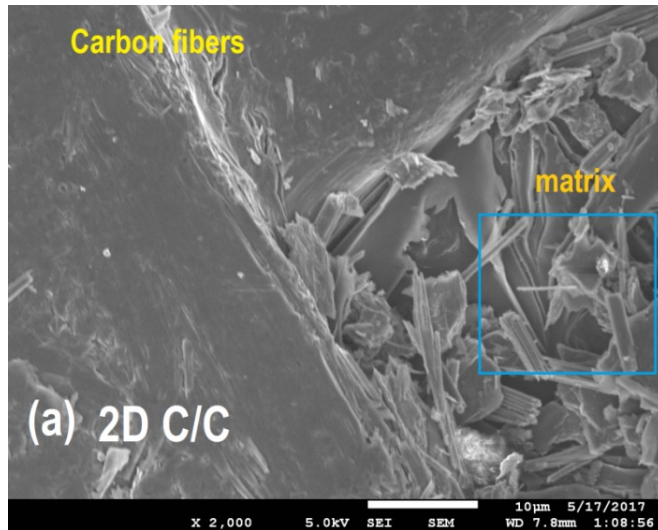
Macroscopic closer view



## Microscopic analysis



# CFC Materials



Graphitization issue/comparison

N. Simos, et al., "Radiation damage and thermal shock response of carbon-fiber-reinforced materials to intense high-energy proton beams" Phys. Rev. Accel. Beams 19, 2016





## Super Alloys

- The ( $\alpha$  +  $\beta$ ) Ti-6Al-4V alloy
- The  $\beta$ -titanium alloy **Gum metal** (Ti-21Nb-0.7Ta-2.Zr-1.2O)
- Super-Invar
- Inconel 625 and 718

## Refractory Metals

- Tantalum
- Tungsten
- Molybdenum

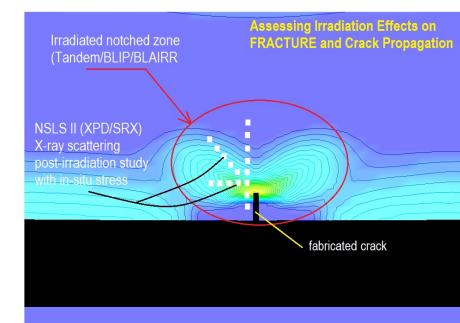
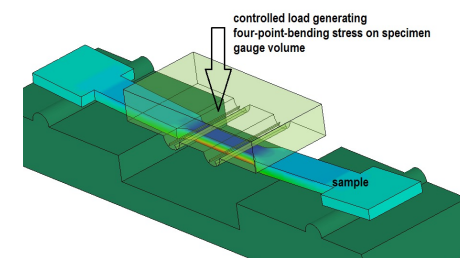
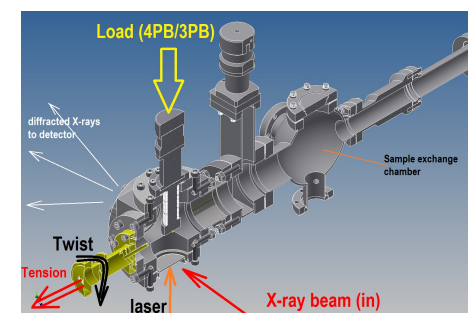
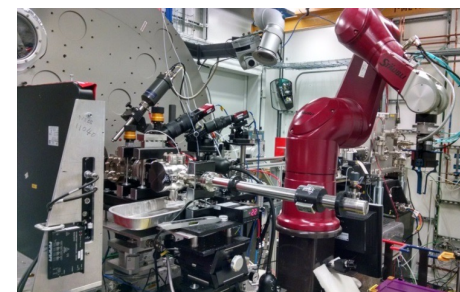
## LHC Collimation Materials

- Glidcop
- Mo-carbide-Graphite
- Copper-Diamond

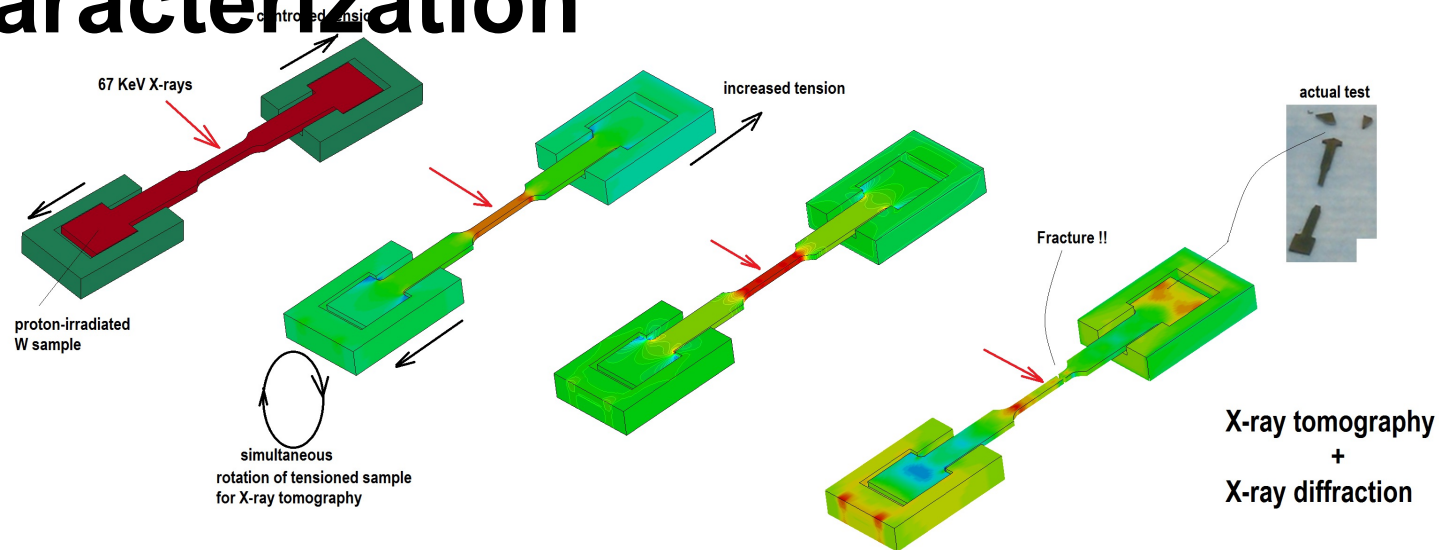
**Nuclear steels and nano-structured materials (coatings), including SiC, SiC/SiC**

# Enhancing EXTREME conditions during X-ray Characterization

- Under way with upcoming experiments at NSLS-II beamlines
- X-ray characterization under extreme, multi-dimensional stress states
- High energy X-rays and creep of irradiated materials
- High energy X-rays and fracture toughness/crack propagation
- High energy X-rays and high temperatures, aggressive environments



# BNL Microstructural Analysis Capabilities - X-ray Characterization

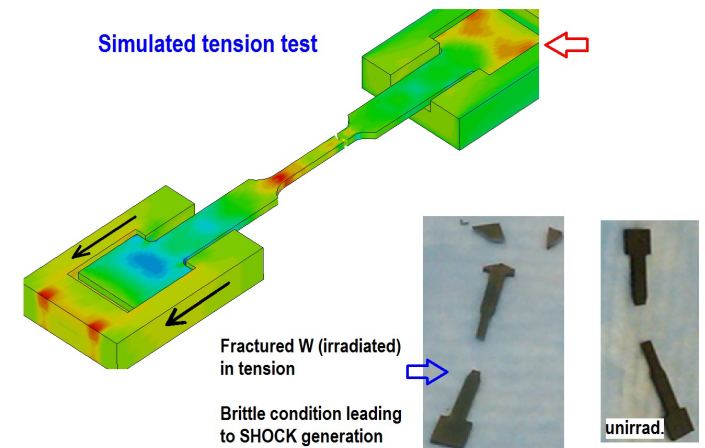


X-ray Diffraction, X-ray tomography, small angle scattering with **in-situ** multidirectional loading.

Including 3-point, 4-point bending

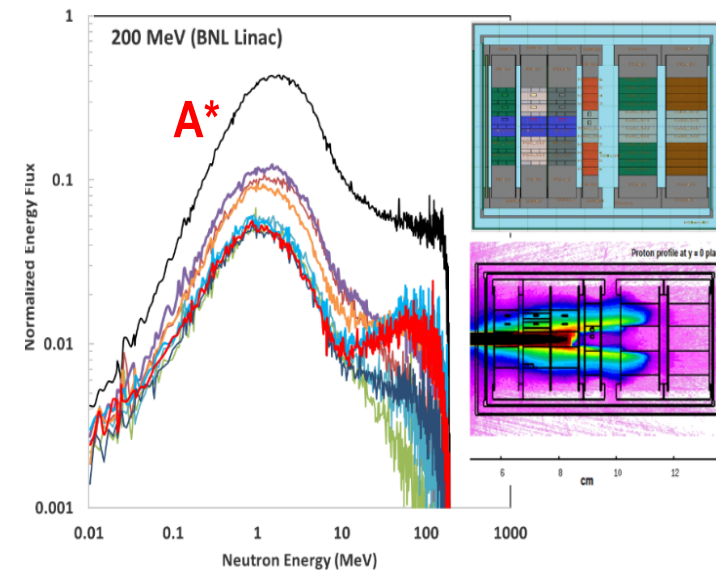
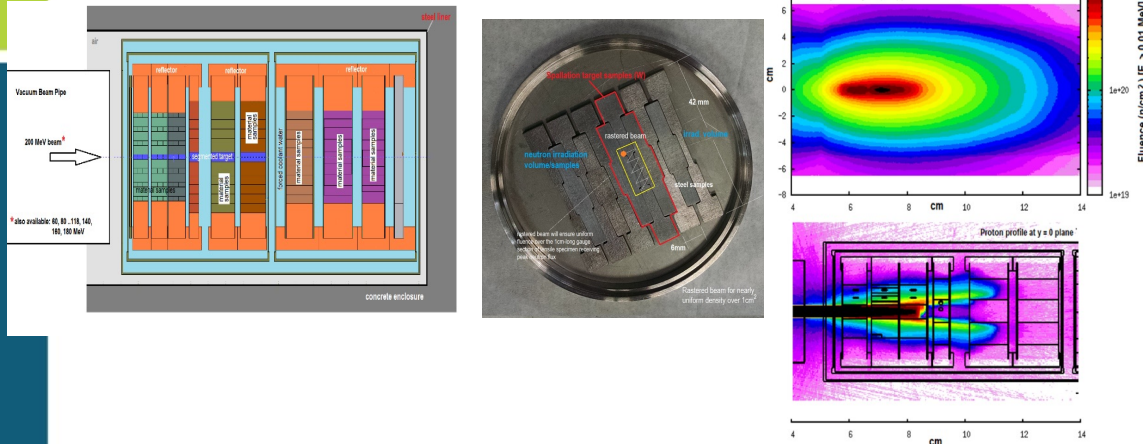
Tension (to fracture), compression and twisting in addition to the 4-point and 3-point bending

ENABLES observation of the microstructural evolution of the IRRADIATED bulk samples as the material is taken to its limits and eventual fracture.



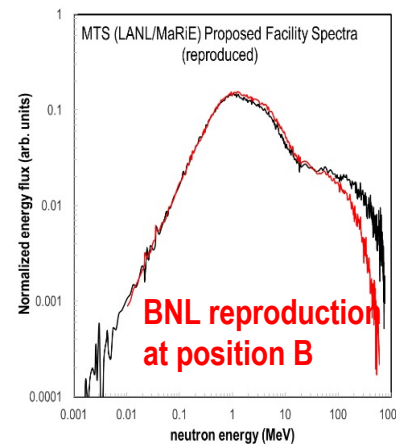
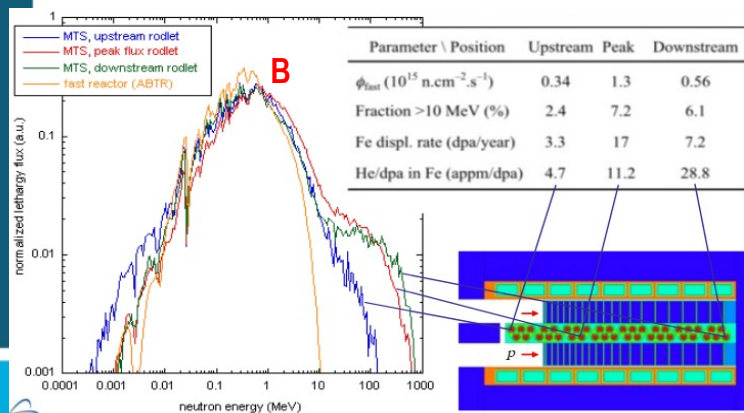


# Forward – Spallation Process optimization for fission/fusion reactor materials



Neutron energy spectra generated by the 200 MeV protons of the BNL beam at various irradiation locations within the irradiated volume

Reproduction of the MaRIE (LANL) produced neutron spectra using 800 MeV LANCE protons prior to completing the BNL option study.

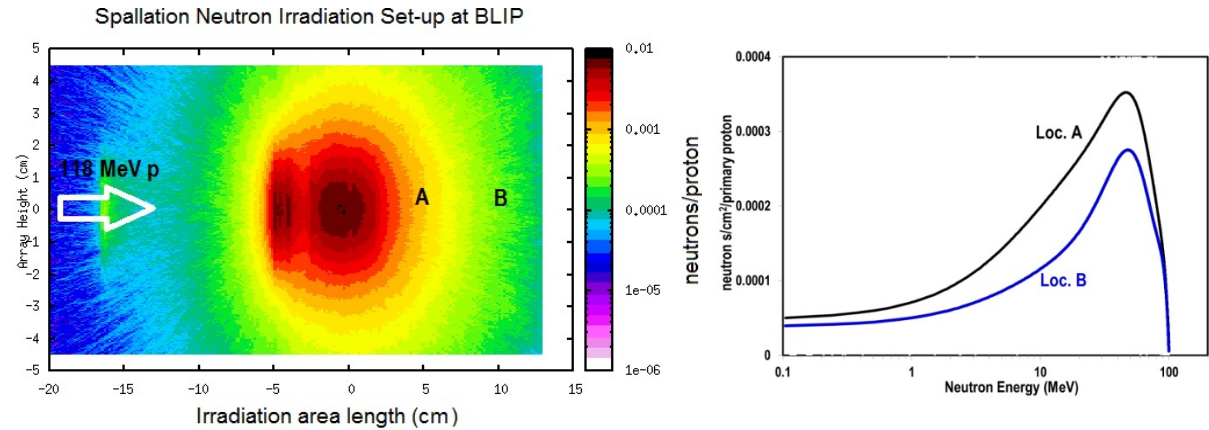


BNL, due to the potential of arranging the irradiated (fast neutron) material volume very close to the spallation target (W) CAN in the closest position match both the spectra and flux (trace A\*)

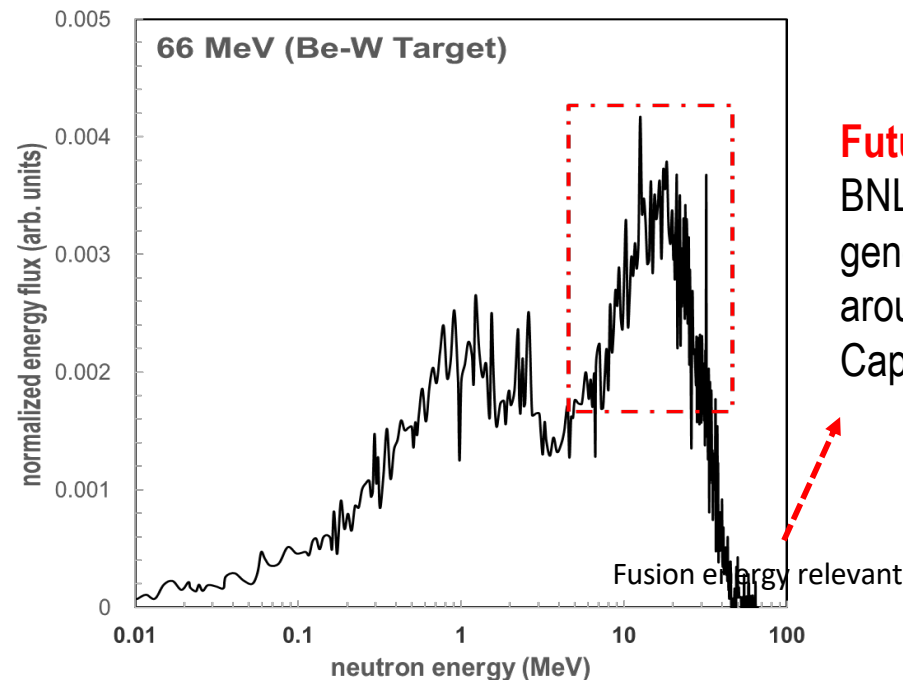
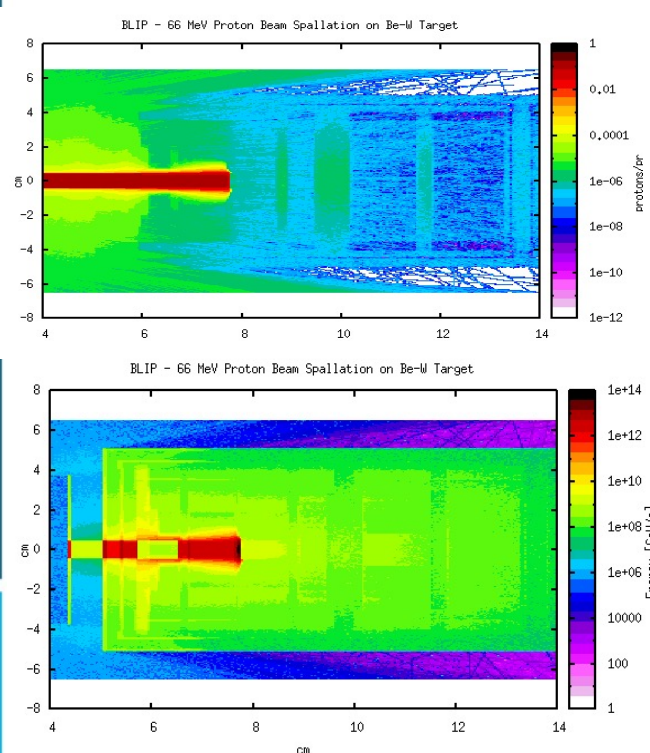
Spallation target can be studied as accelerator material under proton (200 MeV) flux.

# Forward: Fast Neutron Spectra for Fusion Reactor Materials

**Current capability** in irradiating with fast neutrons (constrained by isotope production with beam at 118 MeV, thus fast spectra at 0-degree downstream peaking at ~30 MeV)



**Future (explored):** Proton beam at 66 MeV with W-Be spallation target



**Future** capability of the BNL Linac/BLIP to generate spectra peaking around 14 MeV!!  
Capability stemmi

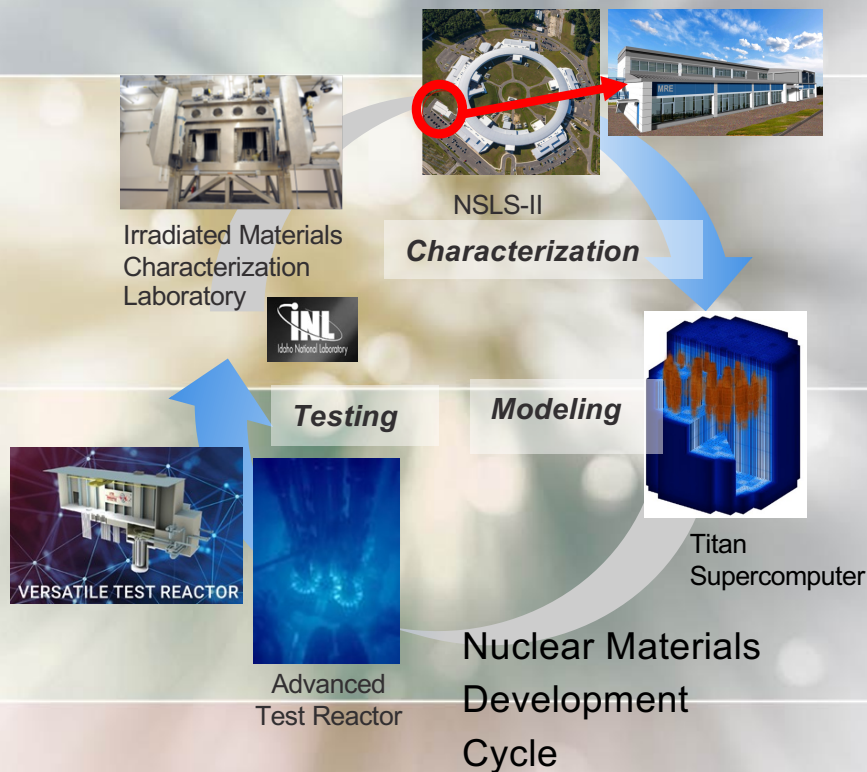
# Section Break

Materials in Radiation Damage



# Materials in Radiation Environment

A unique opportunity identified by INL and BNL to enhance DOE mission delivery



*Providing a critical tool for  
Post Irradiation  
Examination of materials  
and actinide chemistry in  
support of the DOE  
mission to address  
America's nuclear energy  
and security challenges*

November 19, 2020

**INL** Idaho National Laboratory

**BROOKHAVEN**  
NATIONAL LABORATORY

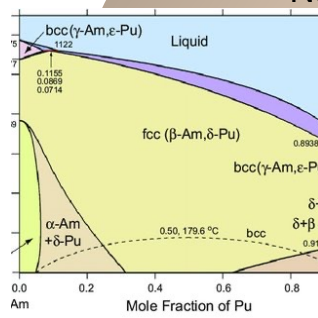
# MRE

## Addressing Questions Across the Mission of the Entire DOE

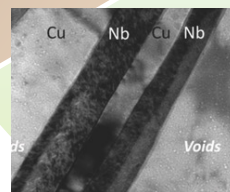
- High x-ray energy
- High photon flux
- Separate building
- Separate access and ESH envelope from NSLS-II
- Operated as a radiological facility

**DOE-NNSA**

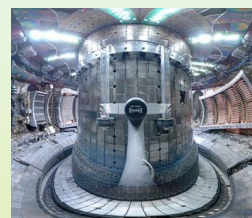
Nuclear Forensics



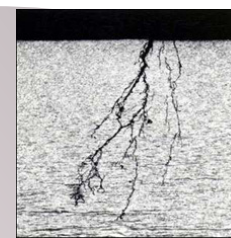
Phase diagrams for transuranics



Radiation Resistance



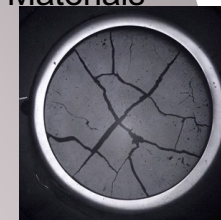
Fusion Energy Science



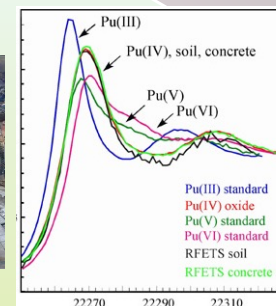
Corrosion Resistance



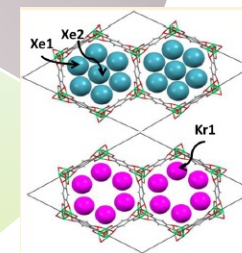
Improved Structural Materials



Studies on Spent Fuel



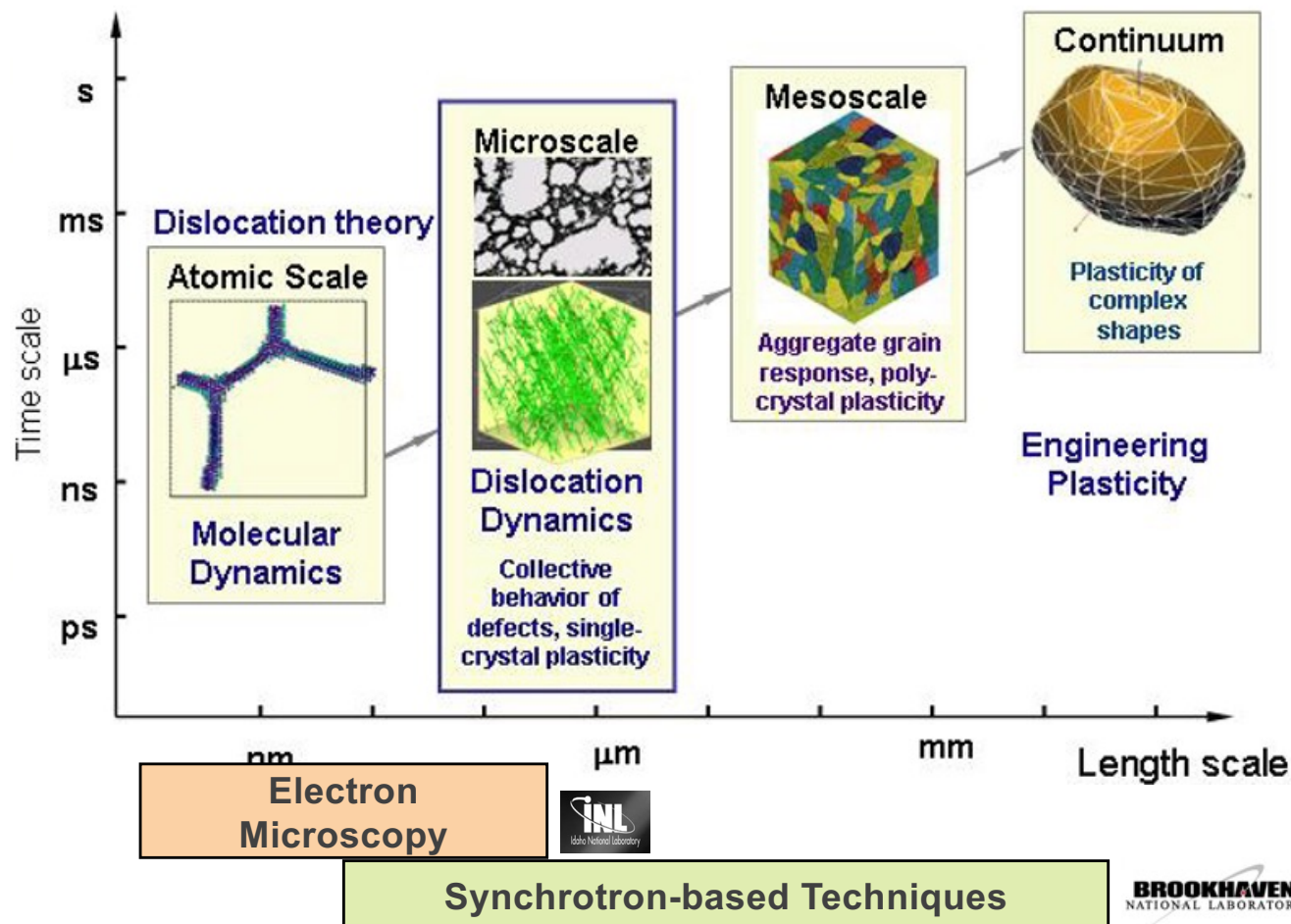
Actinide Chemistry



Separations

**DOE-BES and DOE-FES**

# Complementary tools for PIE

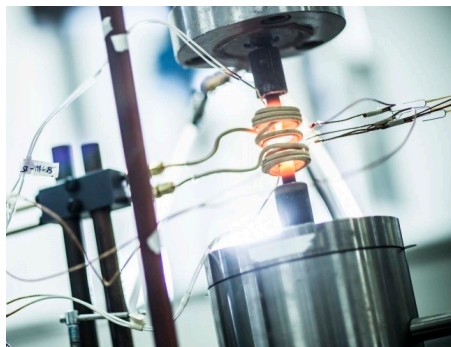


- NE Materials Research Challenges involve a range of processes with length scales spanning orders of magnitude
- Electron microscopy and synchrotron techniques provide a complementary tool set to address these challenges

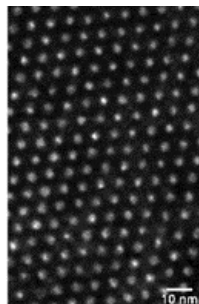


# MRE Expands Scope of SC Research on Radioactive Materials

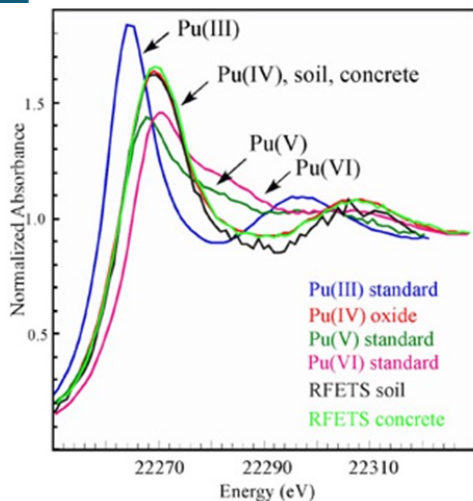
In situ experiments with actinides



Nanopatterning due to radiation damage

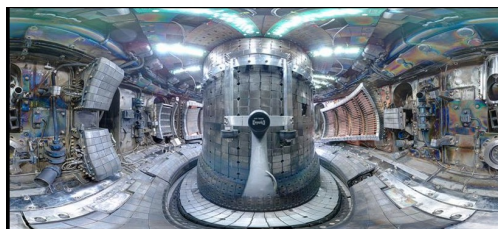


Spectroscopy of actinides



DOE-SC research interests where cutting-edge characterization of radioactive materials is critical to success

- Formation of self-organized microstructures
- Role of grain boundaries in irradiation response
- Design of radiation-resistant alloys
- Crack initiation
- Actinide chemistry / *f*-electron challenge
- Actinide-ligand bonding
- Interaction of actinides at interfaces (liquid-liquid and liquid-solid)
- Advanced materials for waste forms
- Materials degradation in complex environments across broad timescales
- Aqueous chemistry far from equilibrium due to ionizing radiation



# Looking Forward at BNL

- Utilizing the potential of NSLS II and establish/complete the spectrum of extreme conditions at the beamlines (high temperature, high and complex stress states, fatigue, oxidation/corrosion kinetics)
  - In-situ multi-dimensional stress capabilities with X-rays adding to the 3- and 4-point bending to include, Tension to failure, Compression, Torsion, High temperature, Oxidizing and other environments within the chamber

X-ray Tomography, Small Angle Scattering

and with upcoming HEX beamline: EDXRD and X-ray imaging once HEX beamline is commissioned

- Bring into the suite of characterization capabilities electron microscopy for irradiated materials.
  - The introduction of scanning electron microscopy (SEM) in the TPL laboratory is currently under study in coordination with the BNL Center of Functional Nanomaterials (CFN), a capability that will enhance the characterization of materials at TPL.
- LINAC/BLIP upgrade: reached 205  $\mu\text{A}$  current → UPGRADE goal 300  $\mu\text{A}$
- Materials in Radiation Environment

**Novel materials**, alloys and composites for next generation reactors and/or accelerators require assessment under extremes. Key to such assessment is the ability for post-irradiation characterization of materials of interest

BNL over the decades has maintained infrastructure for macroscopic characterization (hot cells, etc.)

Availability of the NSLS-II synchrotron with high energy X-rays and the commissioned techniques at the beamlines provide an excellent means of micro-characterizing BLIP irradiated materials.

While currently Electron Microscopy has been integrated into the characterization process only for unirradiated materials under extremes (at Center of Functional Nanomaterials facility)

**Protons** and other **ions** as surrogates to emulate the damaging effects of fast neutrons have been used at BNL . Also, BNL Linac/BLIP has provided to-date modest means to increase availability of **fast neutrons** to test materials for fast reactors

Proposal is moving forward at BNL to utilize the 200 MeV Linac and the 300  $\mu\text{A}$  peak current it can deliver after its upgrade (peak current achieved to-date 200+  $\mu\text{A}$ ) for usable fast neutron spectra for fission and fusion materials

MRE facility will allow for more through testing of irradiated materials.



# Questions?