#### **Fermilab OENERGY** Science



# **Recent studies of radiation damage effects in high-power accelerator target materials**

**Kavin Ammigan** (on behalf of HPT R&D group and RaDIATE Collaboration) Joint Experimental-Theoretical Physics Seminar 18 December 2020

#### **Overview**

#### Radiation damage effects in beam-intercepting materials (windows/targets)



- Challenges of high-power target facilities
- Need for material research to support future accelerator upgrades and facilities
- Approach to studying radiation damage in materials
- Recent results that will inform material selection and future research

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# High-Power Targetry Challenges



#### **The NuMI Beamline**



#### § **Current target facilities (2004 - ~2025**)

- NuMI-MINOS target (400 kW)
- § NuMI-NOvA target (700 kW)
- NuMI-NOvA AIP target (1000 kW)
- Beryllium primary beam window
- § **Future target facilities: LBNF-DUNE (2028–)**
	- Beam power:  $1.2 2.4$  MW
	- Beryllium primary beam window
	- § Titanium target containment window

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#### **Neutrino targets - MINOS**







#### **Neutrino targets – NOvA AIP**

#### NOvA AIP Target (1 MW)





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### **Future Neutrino Target**

#### LBNF-DUNE (1.2 MW)





- Titanium target containment windows
- Helium gas cooled graphite core







#### **Beam-intercepting device failures we do not want…**



MINOS NT-02 target failure: radiation-induced swelling (FNAL)



Be window embrittlement (FNAL)



NOvA MET-01 target fin fracture (FNAL)



MINOS NT-01 target containment water leak (FNAL)



Horn strip line fatigue failure (FNAL)





ISOLDE target (CERN) Target containment vessel cavitation (ORNL - SNS)<br> **Example:**<br> **Example:** 

# **High Power/Intensity Targetry Challenges**

#### Material behavior and targetry technologies



#### Additional neutrino beams challenges

- Primary beam handling and instrumentation
- Accuracy and consistency of beam inputs
- Focusing elements
- Beam-based alignment
- Secondary beam instrumentation
- Hadron production

Thermal Shock and Radiation Damage most cross-cutting challenges facing high power target facilities

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### Motivation for Material R&D



## **Motivation for Target Materials R&D**

- In recent past, major accelerators facilities have been limited in beam power by target and window survivability (J-PARC MLF, NuMI/MINOS)
- Next generation multi-MW accelerator target facilities will present even greater challenges (LBNF 1.2/2.4 MW, proposed Neutrino Factory 4 MW)
- Develop robust beam-intercepting devices to maximize physics benefits of future experiments.
	- Avoid compromising particle production efficiency by limiting beam parameters
	- Accurate component lifetime prediction and reliable operation

"*Realizing a multi-MW proton source to provide neutrino beam intensities at Fermilab beyond PIP-II project will require significant further R&D on targets and focusing systems, concentrated on tolerance of materials to radiation effects of intense beams*", HEPAP report, 2015.

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#### **Nu HPT R&D Materials Exploratory Map**



10x increase in accumulated proton fluence expected in future multi-MW facilities

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#### **Radiation Damage Disorders Microstructure**



*Verlag GmbH & Co. KGaA, 2010, pp. 1–61.* 

#### Microstructural response:

- o Creation of transmutation products
- o Creation and agglomeration of point defects
- o Segregation (precipitation) or depletion of point defect sinks
- o Atomic displacements (cascades): **Displacements Per Atom (DPA)** = Average number of stable interstitial/vacancy pairs created





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## **Radiation Damage Effects**

#### • **Displacement in crystal lattice** (expressed as Displacements Per Atom, DPA)

- Embrittlement
- Creep
- Swelling
- Fracture toughness reduction
- Thermal/electrical conductivity reduction
- Coefficient of thermal expansion
- Modulus of Elasticity
- Accelerated corrosion
- Transmutation products
	- H, He gas production causes void formation and embrittlement
- **Very dependent upon material and irradiation conditions (temperature, dose rate, particle energy/type)**



S. A. Malloy, et al., Journal of Nuclear Material, 2005. (LANSCE irradiations)



#### **Radiation Damage Effects**





D.L. Porter and F. A. Garner, J. Nuclear Materials, **159**, p. 114 (1988)

Void swelling in 316 Stainless Steel tube exposed to reactor dose of 1.5E23 n/cm2



#### **Thermal shock (stress waves)**

- Localized area of compressive stress generated due to fast expansion of material surrounded by cooler material
	- 1 MW target: 250 K in 10 µs (2.5 x 107 K/s)
- Stress waves move through the target at sonic velocities
- Plastic deformation, cracking and fatigue failure can occur



Iridium target tested at CERN's HiRadMat facility



Ta-rod after irradiation with 6E18 protons in 2.4  $\mu$ s pulses of 3E13 at ISOLDE (photo courtesy of J. Lettry)



Simulation of stress wave propagation in Li lens (pbar source, Fermilab)



#### **Stress wave example: T2K window**

#### Example: T2K Ti beam window





#### **Material response dependent on:**

- o Specific heat (temperature jump)
- o Coefficient of thermal expansion (strain)
- o Modulus of elasticity (stress)
- o Flow stress behavior (plastic deformation)
- o Strength limits (yield, fatigue, fracture toughness)



Initial stress wave amplitude

- Heavy dependence on material properties
	- But material properties dependent upon radiation damage
- Cyclic stress loading environment can lead to fatigue failure

 $c = \left| \frac{E}{A} \right|$  $\rho$ Elastic wave speed

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## **Radiation Damage in accelerators**



Effects from low energy neutron irradiations do not equal effects from high energy proton irradiations. Table compares typical irradiation parameters.

Cannot directly utilize data from nuclear materials studies!





a D | A T E Collaboration

**Radiation Damage In Accelerator Target Environments**

#### Broad aims are threefold:

- To generate new and useful materials data for application within the accelerator and fission/fusion communities
- To recruit and develop new scientific and engineering experts who can cross the boundaries between these communities
- To initiate and coordinate a continuing synergy between research in these communities, benefitting both proton accelerator applications in science and industry and carbon-free energy technologies

#### radiate.fnal.gov





### Radiation Damage Studies



# **High Energy Proton Irradiation**

High energy proton irradiation of candidate materials



- Multi-material irradiation campaign at BNL's BLIP facility organized by RaDIATE collaboration
- Post-Irradiation Examination (PIE) conducted at participating institutions with 'hot cell' facilities

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## **BNL's BLIP facility**

- Unique facility for material irradiation in tandem with medical isotope production
- High energy protons: 66 200 MeV with 165 µA peak current





#### **BLIP experimental set-up**





## **Complex experimental design for BLIP irradiation**



Capsule optimized to deliver precise proton energy/flux

Energy degradation example and Proton flux

for downstream isotope production

Computational Fluid Dynamics and Finite Element Analysis to accurately evaluate cooling heat transfer coefficient, irradiation temperature and structural integrity of capsule during irradiation



Dose rate evaluation for post-irradiation handling and shipment









#### **Alternative irradiation method: low energy ion irradiation**

#### HIT: **H**igh Fluence **I**rradiation Facility of the University of **T**okyo









Other ion irradiation facilities include:

- Michigan Ion Beam Laboratory (MIBL)
- Notre Dame Nuclear Science Laboratory (5U)
- IRRSUD facility (France)



## **HE proton irradiation**

- **High energy, high fluence, large volume irradiations**  $\odot$
- **Closely replicate HEP target environment and provide 'bulk' samples for analysis**  $\odot$

#### **LE ion irradiation**

- **Greatly accelerated damage rates (several DPA in hours**)  $\odot$ 
	- Efficient way to screen candidate materials
- Significantly lower irradiation cost  $\odot$

- **Long irradiation times (mon** 
	- $-$  HE irradiations (100 1000 (>1 DPA)
- **Difficulties of Post-Irradiation Examination**  $\bigoplus$ 
	- Activated samples requires remote handling techniques
	- Dedicated microscopy and testing equipment modified for use in 'hot cells'

 $\frac{1}{200}$  and time to reach re and irradiation techniques Still need HE proton irradiations to correlate and validate results

**ation (PIE in 'normal' lab)**  $\odot$ 

**letration (0.5 – 100 µm)**  $\oplus$ 

- Needs advanced micro-mechanics and meso-scale testing
- **Does not reproduce gas (H and He) production**  $\Theta$ 
	- Requires advanced triple-beam irradiation



#### Recent Results – Titanium Alloys



## **Titanium alloy studies**

- Key beam-intercepting material at J-PARC/FNAL to realize high power (1.2 MW+) operation
	- J-PARC: primary beam window, Optical Transition Radiation (OTR) foil material
	- FNAL: LBNF target containment windows

#### BLIP 2017-2018 irradiation





**Titanium capsules**



Fensile+Micro **Bend-fatique** 



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### **BLIP 2017/2018 Irradiation Summary**

- BNL BLIP irradiation executed in 3 phases with different target box configurations
	- Over 200 specimens in 9 capsules
	- 3 capsules containing Titanium specimens



181 MeV rastered beam 3 cm diameter footprint with 154 µA avg. current x 55 days 4.6 x 1021 POT

#### Comparable to MW facility operation

Past tensile data on proton-irradiated Titanium alloys only up to 0.3 DPA



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#### **Electron Microscopy of Ti-6Al-4V**



Ishida, T. et al., Journal of Nuclear Materials, 541, 2020.

- $\cdot$   $\alpha$ -phase and  $\beta$ -phase with different crystal structures co-exist.
- The  $\omega$ -phase is a very fine precipitate within the  $\beta$ -phase, with a specific relationship to the β-phase orientation.

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• Uniform elongation after irradiation (~0.1%)



Ishida, T. et al., Journal of Nuclear Materials, 541, 2020.<br> **In the Contract of Automater Materials, 541, 2020.**<br> **In the Contract of September 19th of Nuclear Materials, 541, 2020.** 



#### **TEM Studies**

Transmission Electron Microscopy observation of the β-phase in the Ti-6Al-4V alloy



Ishida, T. et al., Journal of Nuclear Materials, 541, 2020.

• In the electron beam diffraction pattern shown in the inset, distinct spots appear and grow in size with increasing proton beam exposure as indicated by the red arrows

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This is the  $\omega$ -phase that evolved upon proton beam exposure  $\rightarrow$  embrittlement

## **Ti alloy tensile testing (DS Ti2)**

#### *Preliminary results*



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• After 0.95 DPA, radiation-induced hardening in Ti-6Al-4V Gr23 is significantly higher than in Ti-15-3 STA

#### **LE ion irradiations and nano-indentations at HIT**



Dose rate: 9.5 x 10-4 DPA/s

Shimazu-DUH-211S type Indenter : Berkovich (115°)







Er : Elastic modulus of composite of indenter and specimen

$$
H_{IT} = \frac{F_{max}}{A_p(\boldsymbol{h_c})}
$$

 $E_r = \frac{S\sqrt{\pi}}{2\sqrt{A_p(h_c)}}$ 

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The indentation modulus  $E_{IT}$  of the specimen material is related to the reduced elastic modulus Er and the intender modulus Ei:

$$
E_{IT} = \frac{1 - (v_s)^2}{\frac{1}{E_r} - \frac{1 - (v_i)^2}{E_i}}
$$
 Ei,  $\nu$ i : Elasti  
of indenter  
Vs: Poisson's

ic modulus, Poisson's ratio ratio of specimen,



### **Ti-6Al-4V and Ti-15-3 nano-indentation results**

#### *Preliminary results*



- Radiation hardening of Ti-15-3 was significantly smaller than that of Ti-6Al-4V
- Hardness of Ti-15-3 (Solution Treatment, w/o Aging) stays the same within the statistical effort after the irradiation to 10 DPA

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#### **Fatigue Performance of Proton-Irradiated Ti-6Al-4V**



- Unique fatigue testing machine for irradiated specimen testing
- Cyclic load frequency 15 Hz
- Stress range: 375 MPa 1,250 MPa
- Automatically shuts off when sample fails
- Designed for operation in 'hot cell'







S.Bidhar et al., RaDIATE2019



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### **Ti-6Al-4V Fatigue Performance**





- 1<sup>st</sup> High Cycle Fatigue data on irradiated Ti-6Al-4V has been obtained
- Fatigue strength at 10 million cycles reduced from 750 MPa to 635 MPa at RT for 0.24 DPA
	- ~8M pulses/year for 1.16 s repetition cycle

S.Bi[dhar et al: https://meetings.triumf.ca/indico/event/81/session/6/contrib](https://meetings.triumf.ca/indico/event/81/session/6/contribution/18)ution/18

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## **Ti alloy key findings**

- Prominent embrittlement of dual-phase Ti-6Al-4V due to radiation-induced ω-phase production in β-phase
- Precipitation-strengthened metastable β-phase Ti alloy Ti-15-3 with ST condition is radiation damage tolerant up to 10 DPA or more, though not stable for temperature between  $250 - 400$  °C (w-embrittlement)
	- Confirmed by both HE and LE irradiations
- Ti-15-3 alloy with two-step ageing (ST2A) show promise for operation up to 300 C
	- Acceptable for 1.3 MW beam window with max. temp  $\sim$  250  $\degree$ C
- 1<sup>st</sup> high cycle fatigue data obtained for irradiated Ti-6Al-4V

Ishida, T. et al., "Tensile behavior of dual-phase titanium alloys under high-intensity proton beam exposure: Radiation-induced omega phase transformation in Ti-6Al-4V", Journal of Nuclear Materials, 541, 2020. "Why does Titanium Alloy Beam Window Become Brittle After Proton Beam Exposure", Press Release, November 6, 2020

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### Recent Results – Beryllium



#### **Radiation Damage Studies of Beryllium**

#### NuMI Be window



#### **Transition from transgranular to grain boundary/mixed mode fracture**

- Non-irradiated beryllium mainly transgranular cleavage
- Grain-boundary fracture may be caused by strengthening of the matrix or "weakening" of GB



120 GeV proton beam

1.54 x 1021 POT Up to 0.5 DPA

 $T \cong 50$  °C



Kuksenko et al., J. of Nuclear Materials, 490, pp. 260-271, 2017 S. Kuksenko, RaDIATE Collaboration Meeting, 2019

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#### **NuMI Be Window Hardness Measurements**



S. Kuksenko, RaDIATE Collaboration Meeting, 2019



- **Significant hardening is observed in the proton-irradiated Be even at 0.1 DPA**
- **Hardening increases with irradiation to higher doses (at least up to 0.5 DPA)**
- **Hardness of the irradiated Be is less anisotropic**

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#### **Be Micro-cantilevers fracture tests**



Micro-cantilevers were fabricated by Focused Ion Beam. Cantilevers were pre-notched so that the fracture properties of grain boundaries and basal cleavage plane, in both as-received and irradiated states, can be compared.

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#### **Be Micro-cantilevers fracture tests**





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S. Kuksenko, RaDIATE Collaboration Meeting, 2019

- More plastic deformation in as-received cantilevers oriented for GB fracture
- Almost no plasticity in cantilevers after irradiation in both GB and (0001) cleavage cantilevers
- Fracture load of both grain boundary and cleavage cantilevers increased significantly after irradiation



#### **Ion irradiation of Be**

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### **Irradiated Be tensile testing (in progress)**

- Various Be grade specimens irradiated at BLIP up to **0.06 DPA**
	- PF-60, S-65F, S-200F, S-200FH
- Tensile testing in hot-cells ongoing at PNNL at RT and 300 °C









Be specimens assembled in BLIP capsule





## **Beryllium Key Findings**

- Radiation induces **significant hardening and fracture mechanism change** of beryllium even at **0.1 DPA**
- **Irradiation at 200**°**C shows less radiation-induced hardening**
- Preliminary tensile tests reveal **significant hardening** of S-65F grade at 0.06 DPA
- **Complete loss of ductility** in pre-notched cantilevers observed after proton and He ion irradiation at 50°C irradiation.



### Recent Results – Graphite



### **X-ray Diffraction – Swelling in BLIP Irradiated Graphite**

- X-ray diffraction at NSLS-II (BNL) on irradiated graphite shows lattice  $\Box$ swelling
- Lattice swelling data from reactor studies agrees relatively well with data from HE proton irradiation!



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#### **Neutron Irradiated Graphite Dimensional Changes**



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#### **X-Ray Diffraction of NuMI Graphite Fin**

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## **Modeling of graphite swelling predicts failure**





#### **Progress to date**

- **Fermilab HPT R&D group and RaDIATE Collaboration grew to 14 institutions and over 70 participants**
- **Completed Proton Irradiation Studies on Graphite, Beryllium and Titanium**
	- Informed design of LBNF target cooling system and graphite grade
	- Will inform design of Be beam windows for LBNF
	- Will inform design and grade of Ti windows for both LBNF and T2K
	- Benefits many facilities including LBNF, T2K, BDF at CERN, FRIB and HL-LHC
- **Using low energy ion irradiation for informing designs at much higher DPA for future facilities**
	- Fast screening to down-select material grades/alloys
	- Correlation and validation of results with HE proton irradiations
- **Completed two thermal shock experiments at CERN's HiRadMat facility**
	- Enabled prediction of stress and deformation of Be beam windows or targets
	- Brings together radiation damage and thermal shock on Be, Graphite and Ti alloys

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# **Ongoing & Future Work**

- Completing **Post-Irradiation Examination** of BLIP 2017/2018 HE proton irradiation
- Planning next **RaDIATE multi-material irradiation** at BLIP in 2024
- Continue **examination of retired targets and windows** (NOvA graphite target)
- **Ion irradiation** at even higher doses and elevated temperatures
- Develop **alternative thermal shock and radiation damage methods**
	- Reduce cost and duration of R&D cycles
- Planning next **thermal shock experiment** at CERN's HiRadMat facility in 2022
- **Modeling of He bubble** nucleation and growth in irradiation Beryllium
- Continue **opportunities with students** (4 PhD students and 1 post-doc)
- Develop **novel targetry materials**: nanofiber mats and high-entropy alloys











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### **Summary**

- **High power accelerators require beam-intercepting components that are capable of stable operation under challenging conditions.**
	- Currently operating accelerator facilities have been limited in beam power due to target survivability issues
	- Planned multi-MW accelerator upgrades and new facilities will present even greater challenges
- **Targets, beam windows, and other beam intercepting devices will experience extreme conditions in next generation multi-MW facilities**
	- Lattice displacements & transmutation
	- Dynamic thermal stresses produced by pulsed beam
- **R&D by the global targets community via RaDIATE is progressing well**
	- High-energy proton irradiations and low-energy ion irradiations to study radiation-damage effects
	- In-beam thermal shock tests of irradiated material specimens tests both thermal shock and radiation damage in single experiments



# Back-up slides



#### **World First: Response of Irradiated Materials to Thermal Shock Induced By High Intensity Proton Beam**

- HRMT43-BeGrid2: Expose irradiated & non-irradiated specimens to high intensity beam pulses to compare thermal shock response
	- Irradiated specimens included Beryllium, Graphite, Silicon, Titanium, SiC-coated Graphite, Glassy Carbon specimens from BLIP
	- First unique test with radiation damaged materials with actual high-intensity proton beam-loading condition







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#### **HRMT-43 Experiment Set-up**

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#### **Successful Beam Exposure at CERN**





#### **Accelerator Targets Beyond High Energy Physics**

- **Spallation Neutron Sources**  $\bullet$ 
	- $-$  SNS at ORNL (1.4+ MW)
	- ESS in Sweden (4 MW)
	- $-$  ISIS in UK (0.16 MW)
	- $-$  MLF at J-PARC (1+ MW)
	- SINQ at PSI, Switzerland
- **Rare Isotope Beams**  $\bullet$ 
	- FRIB at MSU
	- ISAC at TRIUMF (Canada)
	- ISOLDE at CERN
	- RIKEN in Japan
	- RAON in S. Korea
- **Medical Isotope Production**  $\bullet$ 
	- Several facilities around the world
- **Accelerator Driven Sub-critical Reactors**  $\bullet$





#### **Development of New Targetry Materials**

An ultimate objective is to develop new materials specifically addressing the requirements of future target facilities. Some progress is being made in exploring some of the newer materials and forms of material that have been developed.

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- Glassy carbon (BeGrid2 material, CERN/FNAL)
- Molybdenum graphite (RaDIATE BLIP run material, CERN)
- Metal foams (BeGrid2 material, FNAL)
- 3-D Printed Ti alloy (RaDIATE BLIP run material, FRIB)
- Nano-fiber mats (BeGrid2 material, FNAL LDRD)
- High-Entropy Alloys (UW-M collaboration)



