Radiation Damage Experiments Update from the RaDIATE Collaboration

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on behalf of the RaDIATE Collaboration

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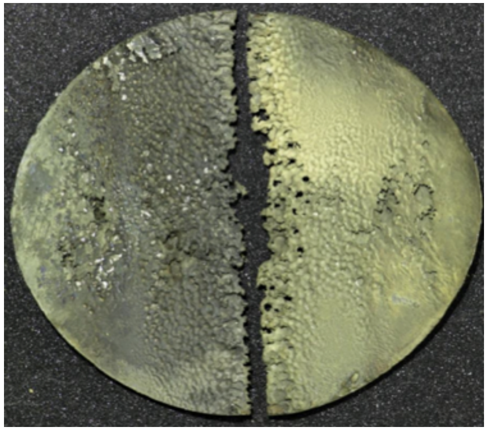
Motivation

- Recent major accelerator facilities have been limited in beam power and operation time by target and window survivability
- Future high power, high intensity accelerators will present even greater challenges
- Understanding the causes of radiation-enhanced failure mechanisms is critical to enabling the science from future facilities

Cracked NuMI target, FNAL

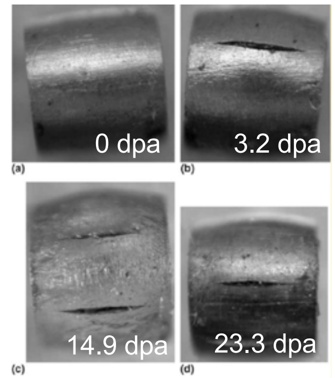
NuMI-MINOS target cooling water leak and radiation-assisted corrosion, FNAL



Cavitation-induced erosion of SNS target vessel, ORNL

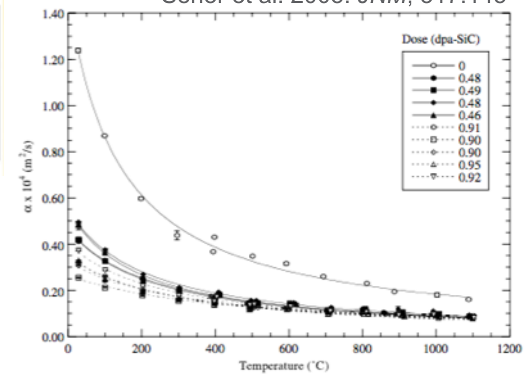
Radiation Damage Effects

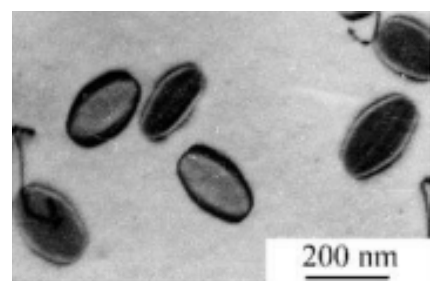
- Irradiation can degrade a variety of material properties
  - Hardening and embrittlement
  - Swelling
  - Radiation-enhanced creep
  - Irradiation-assisted corrosion and stress corrosion cracking
  - Transport property degradation
- Microstructural effects are responsible for property degradation
  - Isolated point defects
  - Clusters and dislocation loops
  - Gas production and transmutation
  - Voids and bubbles
  - Radiation-induced segregation
  - Loss of phase stability



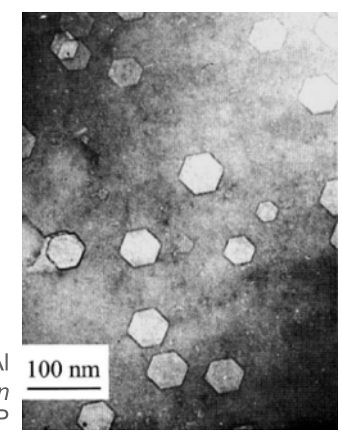
Swelling of W after proton irradiation
Malloy et al. 2005. *JNM*, 343:219

Degradation of thermal diffusivity in β-SiC
Senor et al. 2003. *JNM*, 317:145





Ni irradiated at 500°C
Kiritani. 1994. *JNM*, 216:200



Voids in irradiated Al
Jenkins and Kirk. 2001. *Characterization of Radiation Damage by TEM*, IOP

Irradiation Contributes to Other Failure Mechanisms

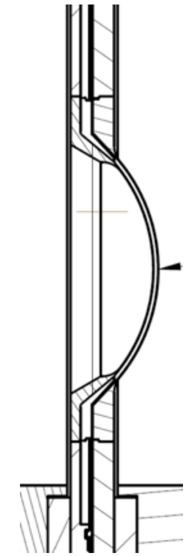
- Thermal shock (stress)
  - Rapid heating and thermal expansion of material at the beam location, surrounded by cooler material, creates a sudden localized compressive stress
  - Stress waves travel through the material causing plastic deformation, cracking, etc.

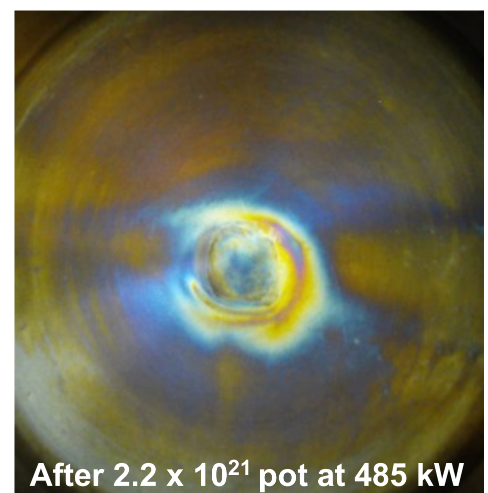
(Photo courtesy J. Lettry, ISOLDE)

Ir rod after a single proton pulse (1.27 x 10^{12} pot)
Torregrosa et al. 2016. *CERN-EN-2016-004*

Example Application of Interest

- T2K Ti-6Al-4V beam window at J-PARC
  - Window performance is dependent on a variety of material properties
    - Thermal expansion
    - Elastic modulus
    - Plastic deformation
    - Tensile strength
    - Fatigue strength
    - Fracture toughness
  - Radiation damage affects all of these properties to some degree
    - Most target/window materials of interest have limited proton irradiation data
    - Reactor irradiation data are of limited utility because of differences in H/He production rates and transient temperature effects compared to high-power accelerators
  - Anticipating future performance, particularly at higher beam power, requires more data and improved understanding of these effects





After 2.2 x 10^{21} pot at 485 kW

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RaDIATE Collaboration

- The Radiation Damage In Accelerator Target Environments Collaboration was founded in 2012 by five organizations led by FNAL and STFC
- Membership has grown to 14 organizations in six countries
- Primary purpose is to bring together the target design and radiation materials science communities to improve understanding of radiation effects on accelerator-relevant materials at accelerator-relevant conditions with the ultimate goal of improving target performance and lifetime

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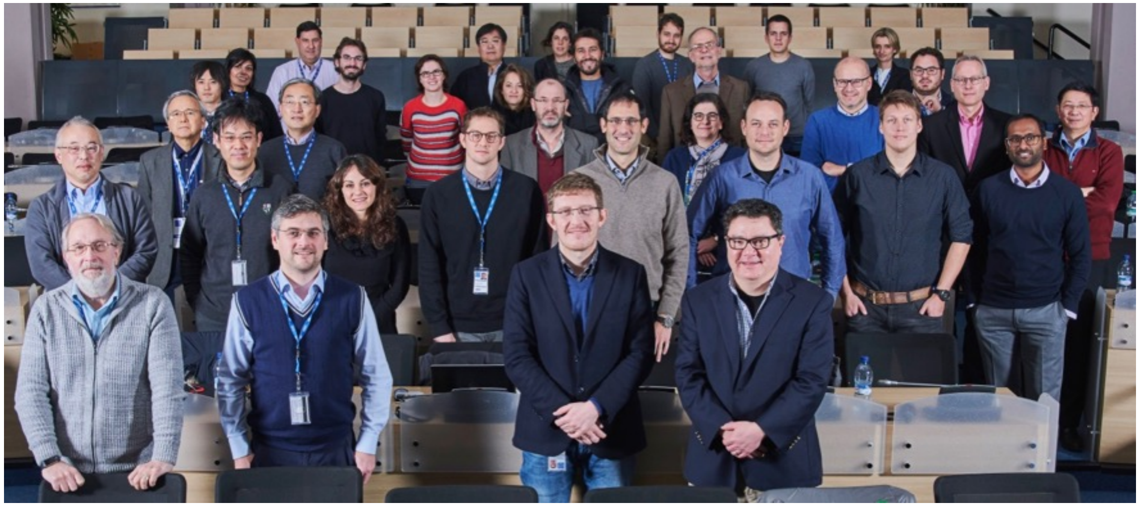
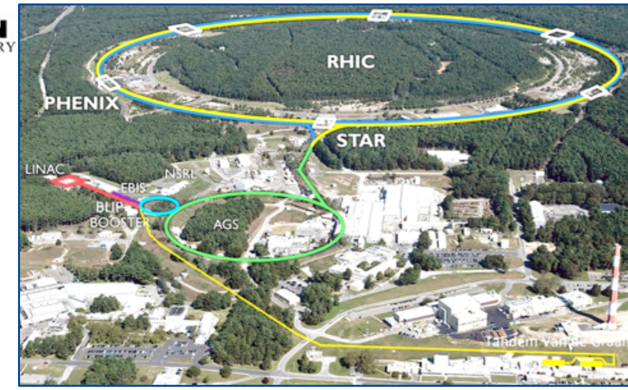


Attendees at the 5th Annual RaDIATE Collaboration Meeting, December 2018, Hosted by CERN in Geneva

Principal RaDIATE Activities

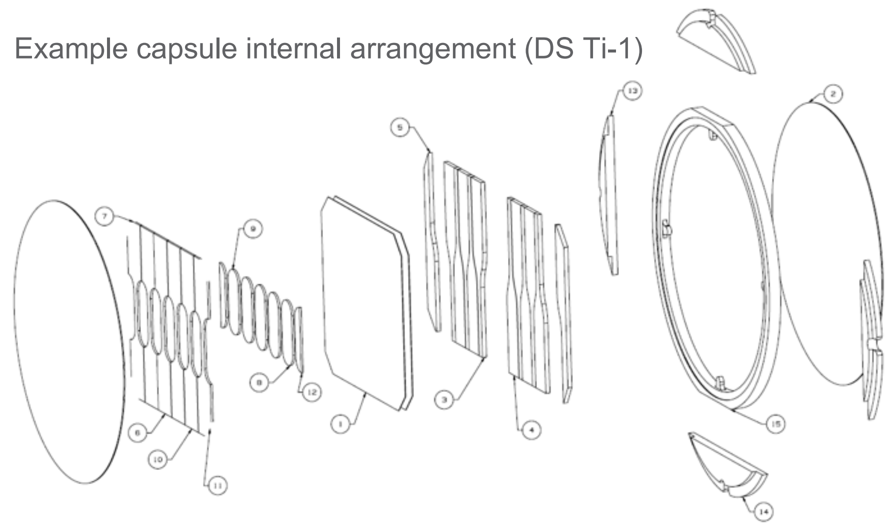
- Proton irradiation of materials for beam-intercepting devices at the Brookhaven Linac Isotope Producer (BLIP)
  - Ti-base alloys for vacuum windows
  - Additively-manufactured Ti-base alloys for beam dump vessel
  - Be for advanced neutrino targets
  - SiC-coated graphite for oxidation-resistant targets
  - Ir, Si, Mo-TZM and CuCrZr for antiproton targets and beam dumps
  - Al-base alloys for spallation source beam windows
- Post-irradiation examination and testing performed at PNNL, Fermilab and CCFE
- BeGrid2 experiment using BLIP-irradiated samples for thermal shock testing as part of HRMT43 at CERN

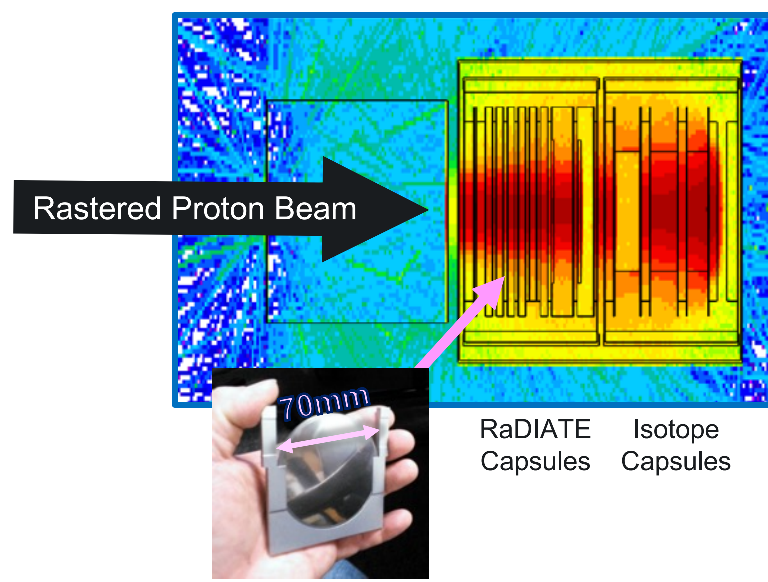


BLIP Irradiation Capsules

- A total of 9 separate capsules (>200 samples) were irradiated at BLIP
  - Three separate irradiation runs during 2017-18
  - 181 MeV/154 μA incident rastered proton beam
  - 0.7-4.6 x 10^{21} pot
  - Each capsule designed to achieve specific temperatures relevant to the applications for those samples
- Four of the nine capsules experienced sample or capsule damage during or after irradiation that limited post-irradiation testing opportunities

Example capsule internal arrangement (DS Ti-1)

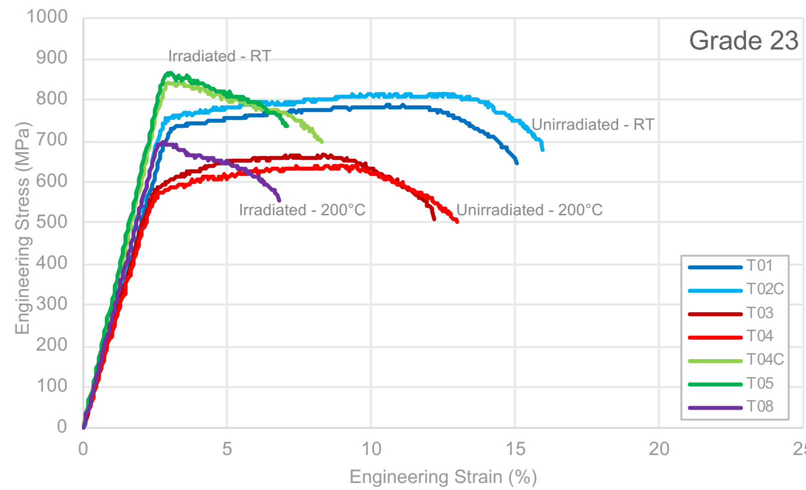


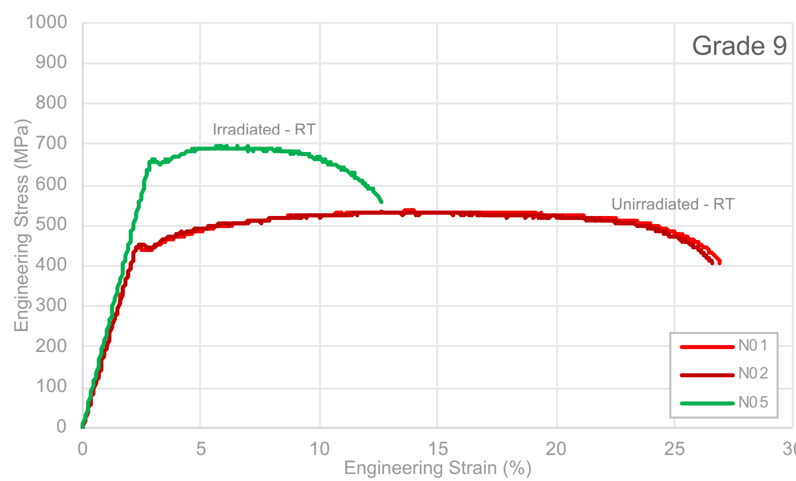


RaDIATE Capsules
Isotope Capsules

Ti-Base Alloy Tensile Testing

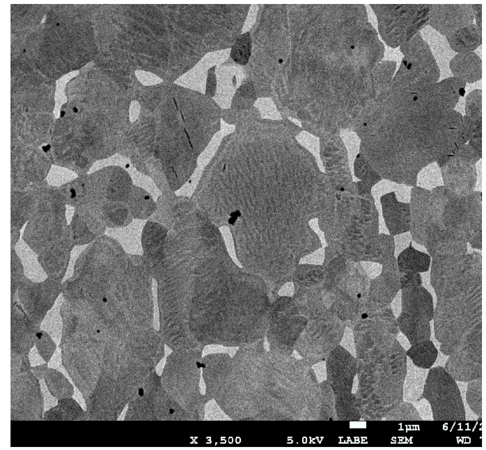
- Irradiated samples received ~0.25 dpa
- Distinct radiation hardening observed for each of the four grades
  - Ti-6Al-4V
    - Grade 5 – Standard commercial grade
    - Grade 23 – Extra low interstitials (ELI)
    - Grade 23-F – ELI, alternate processing
  - Ti-3Al-2.5V
    - Grade 9 – Standard commercial grade
- Ti-3Al-2.5V retains some uniform elongation after irradiation while Ti-6Al-4V grades have essentially none
- Ti-6Al-4V grades stronger than Ti-3Al-2.5V but less ductile
- Additional irradiated (1.0-1.5 dpa) tensile tests currently underway

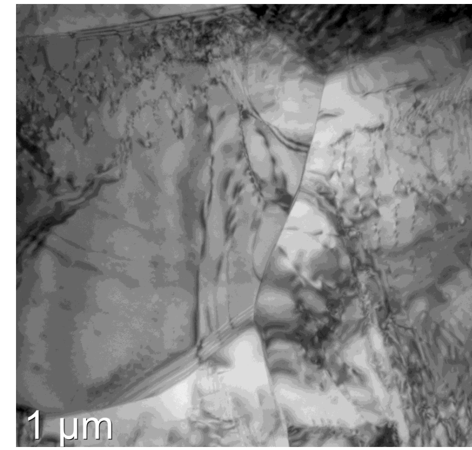


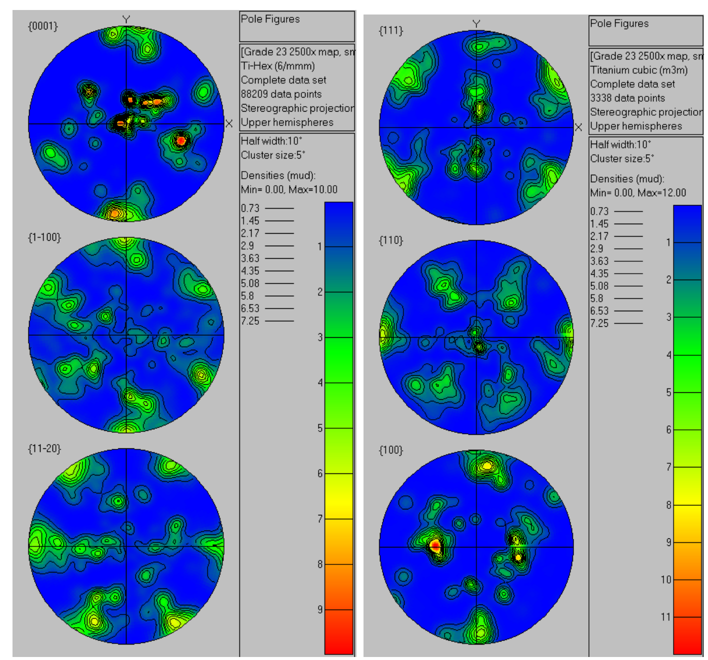


Ti-Base Alloy Microscopy

- Grade 23 Microscopy on unirradiated samples
  - Scanning electron microscopy reveals composition contrast related to two-phase ($\alpha+\beta$) microstructure with elongated grains due to thermomechanical processing
  - Transmission electron microscopy reveals moderate dislocation density caused by thermomechanical processing
  - Electron backscatter diffraction (EBSD) successfully indexed both phases and revealed that both exhibit strong inter-related texture
- Irradiated sample microscopy currently underway
  - Differences in microstructure will provide insight into radiation damage mechanisms that can be related to macro-scale property degradation

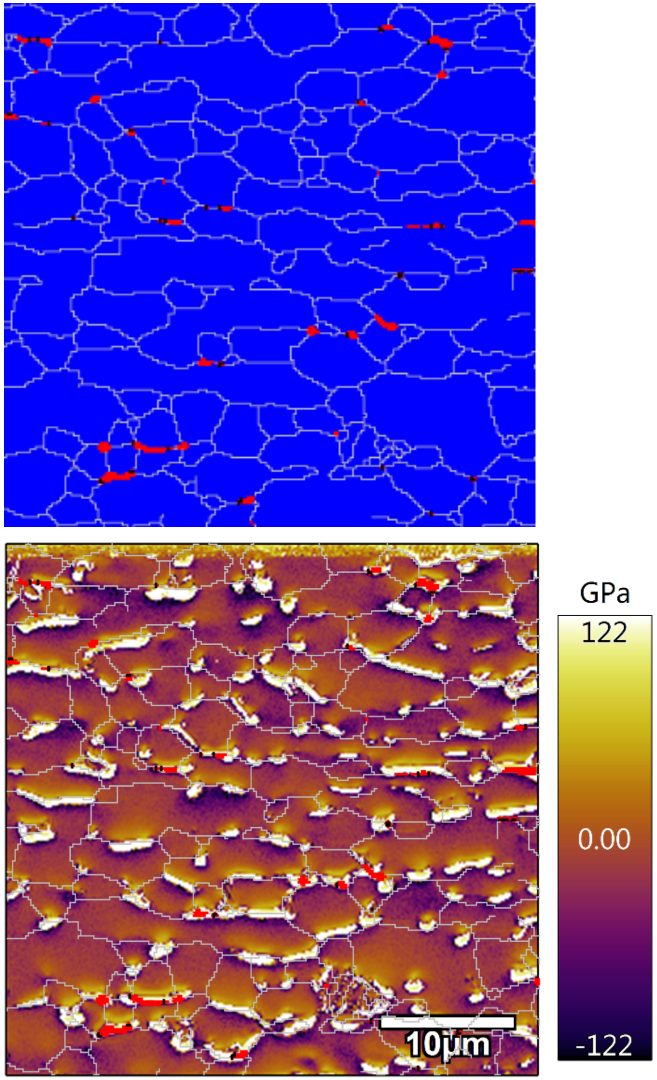






Ti-Base Alloy Microscopy

- Correlated EBSD and AFM measurements on unirradiated and irradiated samples
  - Allows quantitative correlation of individual grain nanohardness to grain orientation
    - Analysis still pending
  - Differences in nanohardness or relationship to grain orientation between unirradiated and irradiated will supplement insight gained from microstructural studies
  - Based on AFM results, it appears not all of the β-Ti grains were resolved and indexed by EBSD
    - Good correlation between the indexed β-Ti phase regions and the AFM nanohardness results
  - Significant relative hardness contrast between α-Ti and β-Ti phases

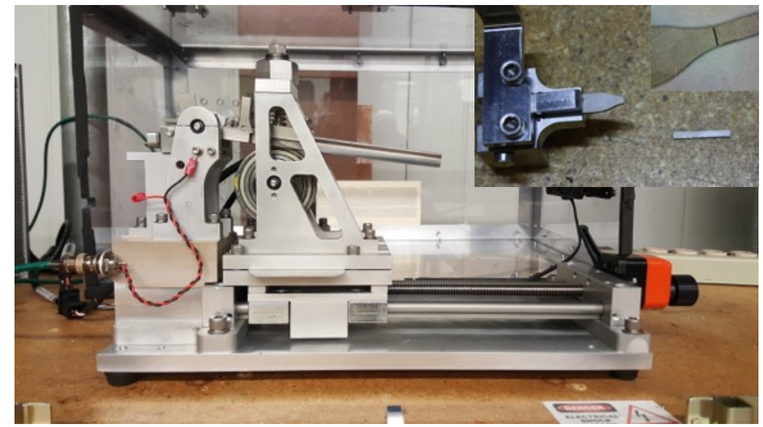


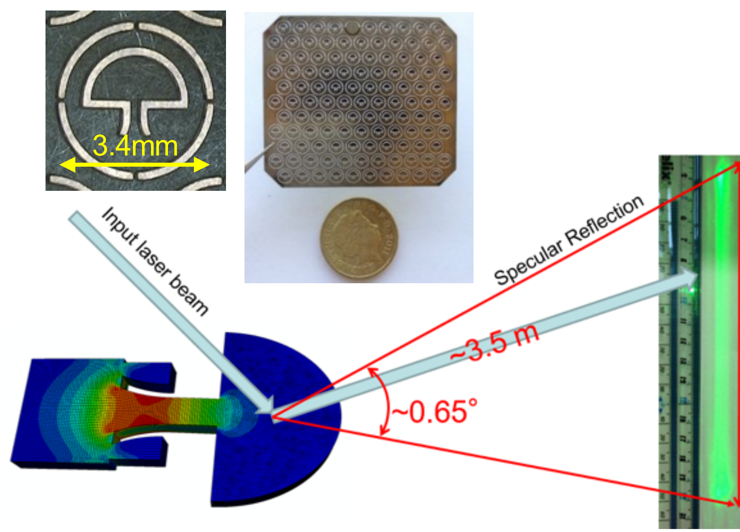
Irradiated Grade 5 (sample F05) EBSD (top) and AFM (bottom)

Ti-Base Alloy Fatigue Testing

- Macro-scale fatigue testing at Fermilab
  - Developed and refined a fatigue testing apparatus using a cantilever-type specimen
  - Demonstrated on unirradiated samples
  - Irradiated fatigue samples recently shipped from PNNL to Fermilab
- Meso-scale fatigue testing at CCFE, in collaboration with University of Oxford and STFC
  - Uses mm-scale samples cycling at very high frequency (kHz) coupled with optical measurement of deflection
  - Originally developed and demonstrated on unirradiated samples at University of Oxford
  - System for irradiated sample testing currently being installed at CCFE in collaboration with STFC
  - Irradiated samples to be shipped from PNNL to CCFE in fall 2019

Fermilab





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Be Tensile Testing

- Unirradiated and irradiated (0.06 dpa) tensile testing recently completed
  - PF-60 and S-65F grades tested at RT and 400°C
    - Primary difference between the two grades is manufacturing method
  - Distinct differences in yield behavior observed between the two grades
  - Detailed data analysis still underway

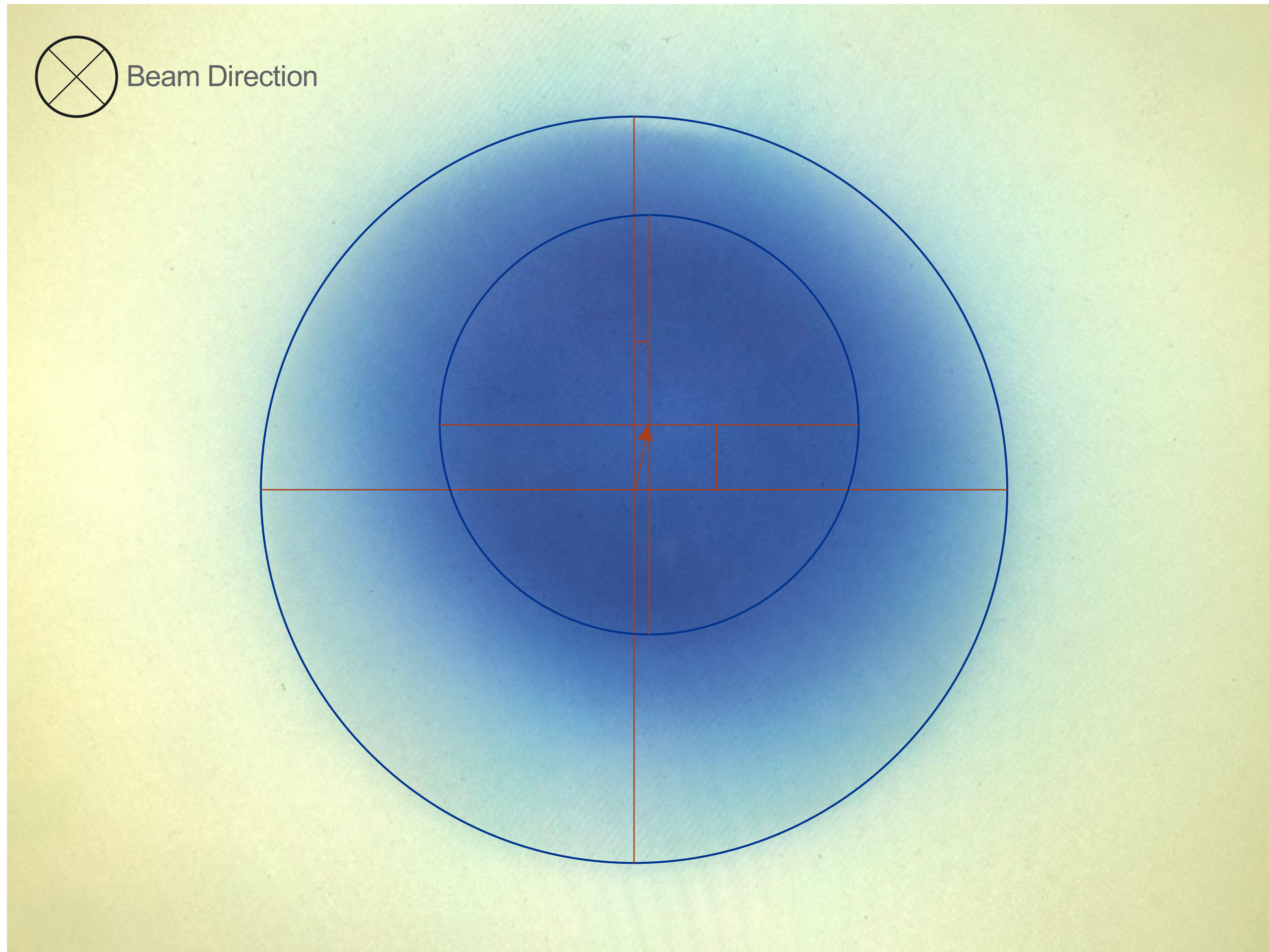
Load frame and furnace in the hot cell at PNNL

One layer of Be (PF-60) tensile samples during assembly of the irradiation capsule

Be sample loaded in the tensile testing fixture and mounted in the load frame

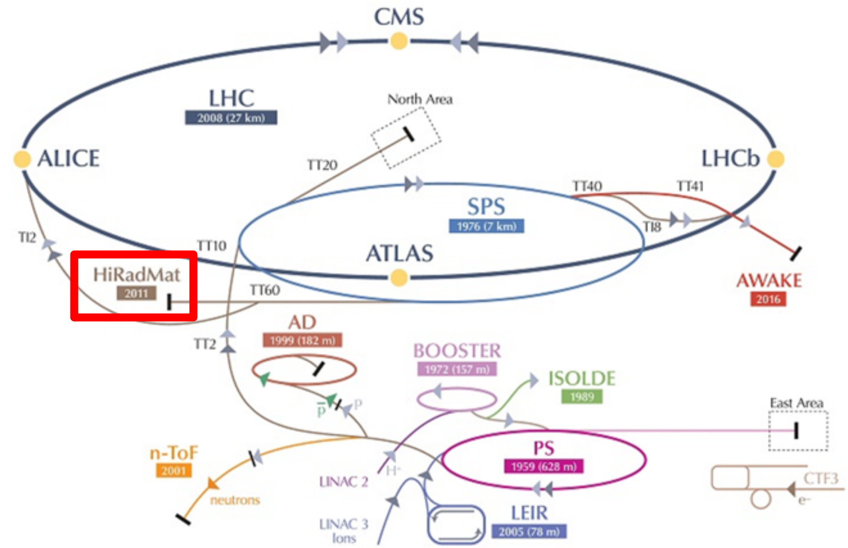
Be Capsule Film Dosimetry

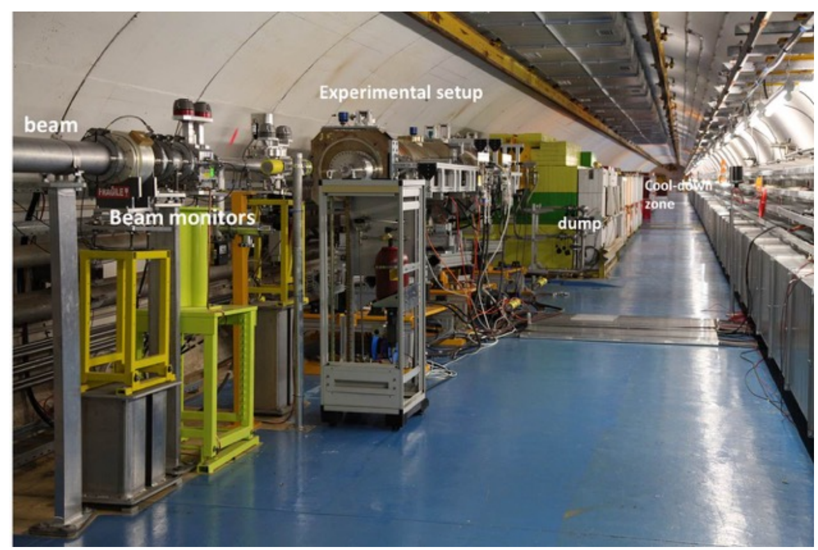
- Performed post-irradiation beam diagnostics using gamma-sensitive film to locate beam spot relative to capsule face
- Center of high dose region appears to be offset
  - ~5 mm upward
  - ~1 mm right
- This information can help refine estimates of radiation damage (dpa) and irradiation temperature for individual samples



Thermal Shock Testing

- Thermal shock testing takes place in the HiRadMat facility at CERN
  - Used to evaluate tolerance of materials to thermal shock
  - Single pulse experiments at challenging beam conditions
  - BeGrid2 (HRMT43) experiment in 2018 included previously-irradiated samples for the first time
  - Builds off successful BeGrid(HRMT24) experiment in 2015 that studied four grades of Be

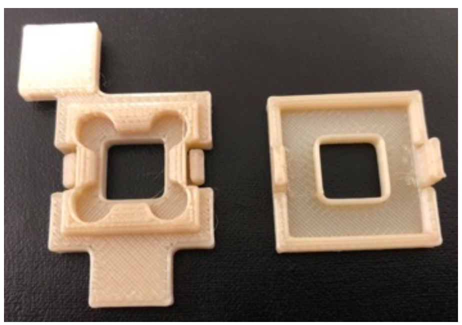


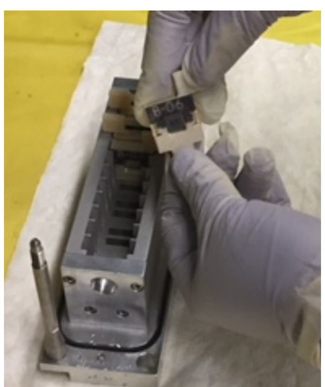


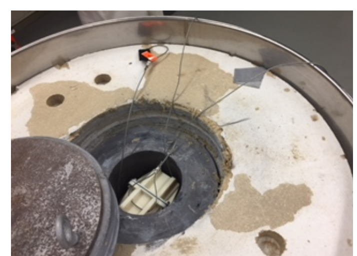
Beam Parameters	
Beam energy	440 GeV
Max bunch intensity	1.2 x 10^{11} ppb
No. of bunches	144, 216 or 288
Max pulse intensity	3.5 x 10^{13} ppp
Pulse length	7.2 μs
Gaussian beam size	0.25 mm (1σ)
Peak proton fluence	9.5 x 10^{15} p/cm^2

Thermal Shock Testing

- The BeGrid2 experiment included Be, Si, Ti, glassy carbon, graphite, SiC-coated graphite, foam (C, SiC) and electro-spun nanofiber mat (SiO_2, ZrO_2) samples
  - Loaded into holders and experiment fixtures design by FNAL
  - Unirradiated samples and fixture arrays prepared at FNAL
  - Irradiated samples and fixture arrays prepared at PNNL





Thermal Shock Testing

- The sample arrays were shipped to CERN and then loaded into the experiment frame
- The thermal shock beam pulses were successfully delivered to the samples in October 2018
- The samples will be shipped to PNNL and CCFE for post-irradiation examination and testing

Summary

- The RaDIATE Collaboration is pursuing a variety of technically challenging and unique experiments to enable design and operation of advanced accelerators
  - Broad engagement between the target design and radiation materials science communities
- Post-irradiation testing of the samples irradiated at BLIP in 2017-18 is underway
  - Several of the materials were exposed to unprecedented dpa values
  - The data will be a significant contribution to knowledge of beam-intercepting device irradiation performance
    - Irradiation-induced swelling/shrinkage
    - Mechanical properties including tensile, fatigue and bend
    - Microstructural characterization including SEM, TEM, EBSD and AFM
- In-beam thermal shock testing at CERN's HiRadMat facility completed in 2018
  - First-ever exposure of previously-irradiated materials
  - Post-irradiation examination and testing still to come

Upcoming RaDIATE Meeting

- The 6th Annual RaDIATE Collaboration Meeting will be hosted by TRIUMF
  - Vancouver, BC, Canada
  - Week of 9 December 2019
  - Details forthcoming soon
- Participation is welcome
- For further information, please contact
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