Radiation Damage Experiments Update from 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
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 $2.2 \times 10^{21}$ pot at 485 kW
The RaDIATE Collaboration was founded in 2012 by five organizations led by FNAL and STFC. Membership has grown to 14 organizations in six countries. The 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.
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
  - $0.7 \times 4.6 \times 10^{21}$ 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
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
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

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
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<tbody>
<tr>
<td>Beam energy</td>
<td>440 GeV</td>
</tr>
<tr>
<td>Max bunch intensity</td>
<td>$1.2 \times 10^{11}$ ppb</td>
</tr>
<tr>
<td>No. of bunches</td>
<td>144, 216 or 288</td>
</tr>
<tr>
<td>Max pulse intensity</td>
<td>$3.5 \times 10^{13}$ ppp</td>
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<tr>
<td>Pulse length</td>
<td>7.2 $\mu$s</td>
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<tr>
<td>Gaussian beam size</td>
<td>0.25 mm ($1\sigma$)</td>
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<tr>
<td>Peak proton fluence</td>
<td>$9.5 \times 10^{15}$ p/cm$^2$</td>
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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

This process was continued until an array box was completely loaded. Then the array box was closed and the cognizant scientist verified by photograph the array box labels. The array box was loaded into the shipping drum and this process was repeated until all samples were loaded into sample holders and sample holders loaded into the shipping drum. This process is demonstrated in the collage below.
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
    o Irradiation-induced swelling/shrinkage
    o Mechanical properties including tensile, fatigue and bend
    o 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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Acknowledgements

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