Novel Materials R&D for Next-Generation Accelerator Target Facilities
DOE SC Early Career Research Program (2022-2027)

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RaDIATE Collaboration Meeting
26-30 June 2023
Novel Targetry Materials ECRP objectives

- Advance the state-of-the-art in targetry materials for future multi-MW accelerators
  - Enable and fully reap the physics benefits of next-generation multi-MW accelerator target facilities
  - Improve performance, reliability and operation lifetimes of beam-intercepting devices

- High-Entropy Alloys (HEAs) and Nanofiber Materials are two promising novel classes of materials for accelerator target applications

~10x increase in accumulated proton fluence and power density
- LBNF/DUNE 2.4 MW
- Mu2e-II
- Future Muon Collider and Neutrino Factory (4 MW+)
Novel Materials Early Career Research Program Plan

- **DOE SC program** to support early career researcher for 5 years ($2.5M)
  - Awarded in FY22 (Aug 2022 – July 2027)
- Proposed experimental program, coupled with complementary computations, to develop novel radiation damage and thermal shock resistance novel materials
  - **High-Entropy Alloys** (beam windows)
  - **Electrospun Nanofibers** (secondary particle production targets)
Looking Beyond Conventional Materials

- Develop radiation damage and thermal shock resistant HEAs and Nanofiber materials by targeting the inherent properties, microstructure and bulk properties of these materials.

Radiation damage tolerance

✓ Nanocrystalline materials: fine grains with high-density grain boundaries
  • GBs serve as defect sinks to absorb and annihilate irradiation-induced defects

✓ Phase stability
  • Prevents irradiation-induced grain coarsening and void swelling

Thermal shock tolerance

Reduce stress magnitude, stress wave velocity and fatigue life

✓ Inherent ability to absorb and dampen stress waves
✓ Low density
✓ Low elastic modulus
✓ Low coefficient of thermal expansion
✓ High specific heat

Dimensional change due to void swelling (Porter et al., 1988)

Iridium (left) and Sigraflex (right) targets tested at CERN’s HiRadMat facility
High-Entropy Alloys (HEAs)

- Alloys consisting of 3 or more principal elements in near equi-atomic ratios (large design space)
- Excellent inherent properties that include high-temperature strength, improved fatigue and fracture properties, good corrosion and oxidation resistance and enhanced radiation damage resistance

Void swelling shown to be less pronounced in more compositionally complex alloys upon heavy-ion irradiation 3-MeV Ni\textsuperscript+ ions to $5 \times 10^{16}$ cm\textsuperscript{-2} at 773 K (Lu et al., Nature Com., 2016)

Large lattice distortion due to multiple elements of different sizes
- Sluggish diffusion of atoms, point defects and interstitial loops
- Reduction in segregation and defect clustering
- Promotion of recombination within the displacement cascade
- And more…
Electrospun Nanofiber Materials

- Development of unique electrospinning nanofiber technology (Zwaska, Bidhar, 2015-)

- Intrinsically tolerant to both thermal shock and radiation damage

- Nanofiber continuum is discretized at the microscale to allow fibers to absorb and dampen thermal shock. Discontinuity prevents stress wave propagation.

- Evidence of radiation damage resistance due to nanopolycrystalline structure of ceramic nanofibers (Bidhar et al., PRAB, 24, 2021)

- ECRP will extend work to metallic (W) nanofibers

SEM images of Zirconia nanofibers produced at Fermilab, (a) bulk nanofiber mat, (b) single nanofibers revealing polycrystalline grains (S. Bidhar)
Materials Simulation and Design

- Determining compositions and in-beam response
  - CALPHAD simulations to understand the thermodynamics and phase diagrams of the alloy systems
  - MARS/FLUKA particle-matter interactions
  - Thermomechanical response from ANSYS FEA

- Thermal shock figures of merit for comparative assessment

CALPHAD showing body-centered cubic (BCC) phase of CrMnTiV at 1500 °C
HEA Development

- Beam window requirements
  - Minimize energy loss and multiple scattering → Low density
  - High thermal diffusivity, low CTE, ductility, and high strength
  - Low activation

- Extensive **high throughput** CALPHAD simulations to determine low density HEA design space
  - Maximize BCC phase range
  - High solidus temperature
  - Optimize physical/thermal properties

N. Crnkovich
G. Arora
Preliminary HEA Synthesis

4 HEAs
• Cr-Mn-V (1:1:1)
• Cr31-Mn31-Ti7-V31
• Al15-Cr20-Mn20-Ti10-V35
• Al15-Co4-Cr25-Mn15-Ti6-V35

Sectioned arc-melted ingots (UW-Madison)

HEA samples sealed in quartz under vacuum before heat treatment (UW-Madison)

• Achieved target composition within 1at%
• HEAs homogenous after heat treatments
• XRD confirmed single-phase BCC phase

A. Couet, M. Moorehead et al.
UW-Madison

CrMnTiV Homogenized

A. Couet, M. Moorehead et al.
UW-Madison
HEA plate fabrication and ongoing characterization

- Procured plates from external vendor (Sophisticated Alloys)

- NSUF RTE awarded to irradiate the 4 HEAs at University of Wisconsin Ion Beam Laboratory
  - V ions at 3.7 MeV
  - Up to 100 DPA, 500 C

- TEM analyses ongoing (N. Crnkovich)

  CrMnTiV HEA specimen irradiated to 100 DPA (UW-Madison, 2022)

- Indentation measurements and validation of DFT elastic constant calculations
- No cracks observed at corners of indents (evidence of some ductility)

- HEAs survived high-intensity beam pulse at CERN’s HiRadMat facility
- Impending PIE at MRF
High Throughput CALPHAD simulations

- Further optimization of HEA compositions to study effects of individual components

**HEA compositions selected**

- Al\textsubscript{10}-Co\textsubscript{4}-Cr\textsubscript{25}-Mn\textsubscript{26}-Ti\textsubscript{1}-V\textsubscript{34}
- Al\textsubscript{10}-Co\textsubscript{4}-Cr\textsubscript{27}-Mn\textsubscript{21}-Ti\textsubscript{4}-V\textsubscript{34}
- Al\textsubscript{16}-Co\textsubscript{2}-Cr\textsubscript{25}-Mn\textsubscript{30}-Ti\textsubscript{1}-V\textsubscript{26}
- Al\textsubscript{16}-Co\textsubscript{4}-Cr\textsubscript{25}-Mn\textsubscript{30}-Ti\textsubscript{1}-V\textsubscript{24}
- Al\textsubscript{20}-Co\textsubscript{1}-Mn\textsubscript{27}-Ti\textsubscript{2}-V\textsubscript{50}
- Al\textsubscript{12}-Co\textsubscript{3}-Cr\textsubscript{6}-Mn\textsubscript{27}-Ti\textsubscript{2}-V\textsubscript{50}
- Al\textsubscript{18}-Mn\textsubscript{30}-Ti\textsubscript{2}-V\textsubscript{50}
- Al\textsubscript{20}-Co\textsubscript{2}-Mn\textsubscript{26}-Ti\textsubscript{2}-V\textsubscript{50}

- Effect of Ti concentration as an impurity getter
- Effect of Co as B\textsubscript{2} phase enhancer
- Effect of Al concentration and removing Cr
- Effect of removing Co, with Al as the potential B\textsubscript{2} phase enhancer

- Order placed with Sophisticated Alloys Inc. and expect delivery later this month
- Vacuum arc melt + HIP + Vacuum Anneal
Electrospun Nanofiber Development

- Currently **upgrading electrospinning set-up** at Fermilab (S. Bidhar, FNAL)
  - Reconfiguration of heat treatment furnace for inert gas capability
  - New control box, needle and collector plate
- **Explore physical properties** (fiber diameter, fiber length, crystal structure, density, etc.) of Tungsten nanofibers
- **Multiphysics modeling**

![Image: Power supply and electrospinning set-up](image1)
![Image: High-temperature furnace](image2)
![Image: Roll-to-roll nanofiber fabrication technique](image3)

(a) Power supply and electrospinning set-up, (b) high-temperature furnace for nanofiber heat treatment, (c) roll-to-roll nanofiber fabrication technique (MI-8, FNAL)

- Furnace reconfiguration for heat-treatment of nanofibers in inert gas environment
- New control box
Materials Irradiation Screening

- **Low-energy (LE) ion irradiations** to emulate radiation damage effects and screen/downselect materials
  - 25 – 1000 C
  - 0.1 – 1 DPA
  - 500 – 2500 He appm/DPA

- **Thermal shock test**: CERN’s HiRadMat facility or alternative method (Fermilab’s A2D2 facility?)

- **Pre/Post-irradiation material characterization**
  - Characterize size and density of radiation-induced dislocation loops, point defect clusters, void swelling and segregation (XRD, SEM/EDS, FIB/TEM, XRD, AFM, ..)
  - Bulk property testing (tensile, CTE, specific heat, ..)
  - **Development of novel PIE techniques**
    - Transient Grating Spectroscopy (TGS)
    - Differential Scanning Calorimetry (DSC)
Prototypic Irradiations and Evaluation

- **Final phase**: prototypic beam irradiation of selected materials with high-energy high-intensity proton beams
  - BLIP facility at BNL, Fermilab beamlines or other facilities
  - CERN’s HiRadMat facility or other facilities

- PIE in hot cells due to activation of material specimens
  - Bulk thermal and mechanical property testing
  - Microscopy and microscale testing (TEM, nanoindentation, etc.)

- Final validation/evaluation (FOMs) and selection of novel materials

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Tensile testing in PNNL hot cells

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BLIP target station arrangement

Specimens layout in capsule
Current Collaborations

- **Fermilab**
  - G. Arora, S. Bidhar, F. Pellemoine

- **University of Wisconsin-Madison**

- **Illinois Institute of Technology**
  - W. Asztalos, Y. Torun

- **Students, Faculty and Postdoc involvement**
  - A. Burleigh joining Fermilab as the ECRP Postdoc on Aug. 7th
  - Joint Task Force Initiative Fellowship (JFTI) – possible Postdoc next month
  - DOE Visiting Faculty Program (VFP) – Summer 2023
  - Community College Internship (CCI) and Lee Tang UG internship - Summer 2023
    - 2 students helping set up PIE equipment (DSC, dilatometer, DIC)

- **Open for collaborations and engagement opportunities!**
Summary

• Innovate and advance state-of-the-art in targetry materials to support next-generation multi-MW accelerator target facilities
  
  • High-Entropy Alloys (beam windows)
  • Nanofiber materials (secondary particle-production targets)

• Iterative material development process where adjustments to the compositions and processing parameters will be made based on the results of property testing
  
  • Optimization of composition, microstructure and properties
  • Planning several experiments (irradiations and material characterizations)
  • Complementary DFT & MD modeling of radiation damage effects in HEAs

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29 June 2023