Novel Materials R&D for Next-Generation Accelerator Target Facilities

DOE SC Early Career Research Program (2022-2027)

Kavin Ammigan
G. Arora, S. Bidhar, F. Pellemoine (FNAL); A. Couet, N. Crnkovich, I. Szlufarska (UW-Madison); W. Asztalos (IIT)

RaDIATE Collaboration Meeting

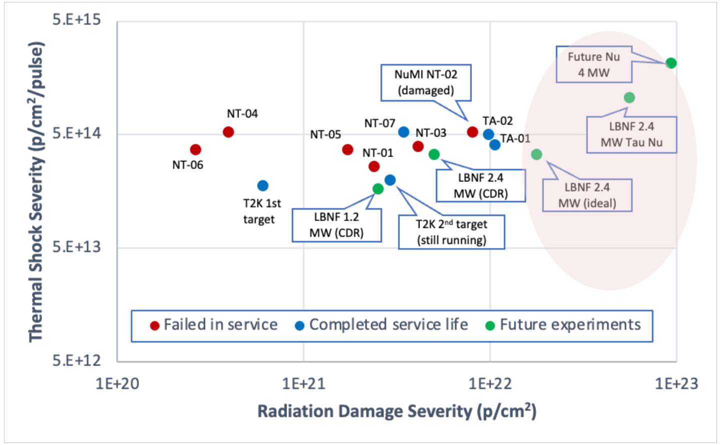
26-30 June 2023

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

Neutrino HPT R&D Materials Exploratory Map

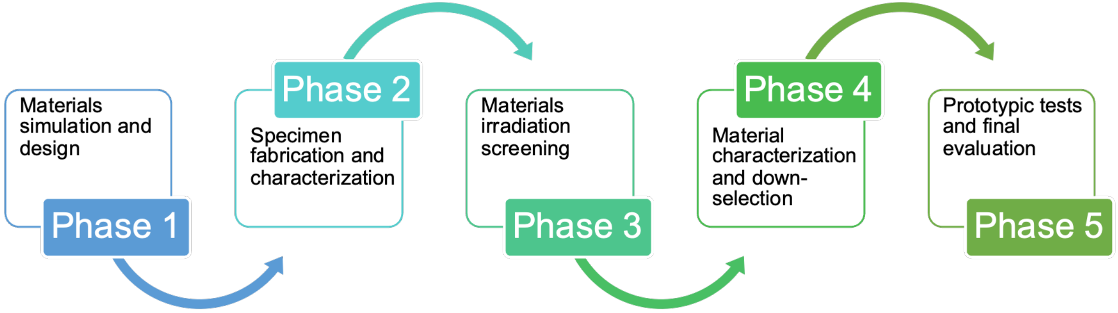


~10x increase in accumulated proton fluence and power density

- 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)



Personnel	Labor (FTE)				
	Year 1	*Year 2*	*Year 3*	*Year 4*	*Year 5*
PI (Kavin)	0.5	0.5	0.5	0.5	0.57
Postdoc (Abe)	0	1	1	1	0.5
Staff Engineer (Sujit)	0.19	0.11	0.08	0.1	0.17
Senior Technician	0.04	0.09	0.04	0.03	0.03
Machinist	0.05	0.05	0.04	0	0
Drafters	0.03	0.03	0.03	0	0
Scientist	0.02	0.02	0.02	0.04	0.11

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

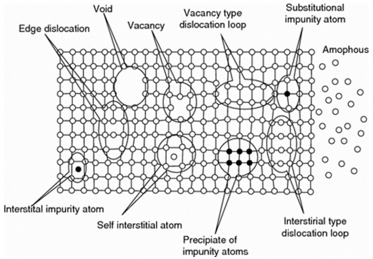
Radiation damage tolerance

✓ 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



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

Thermal shock tolerance

Reduce stress magnitude, stress wave velocity and fatigue life

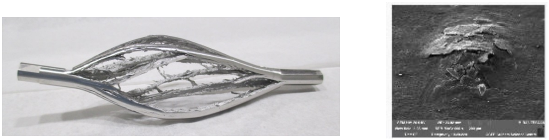
✓ Inherent ability to absorb and dampen stress waves

✓ Low density

✓ Low elastic modulus

✓ Low coefficient of thermal expansion

✓ High specific heat



Iridium (left) and Sigraflex (right) targets tested at CERN's HiRadMat facility

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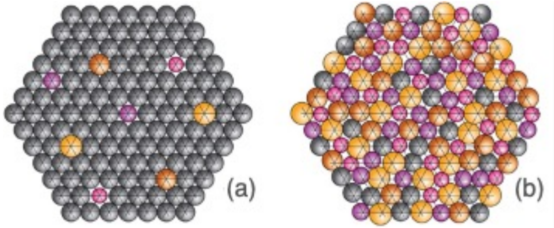
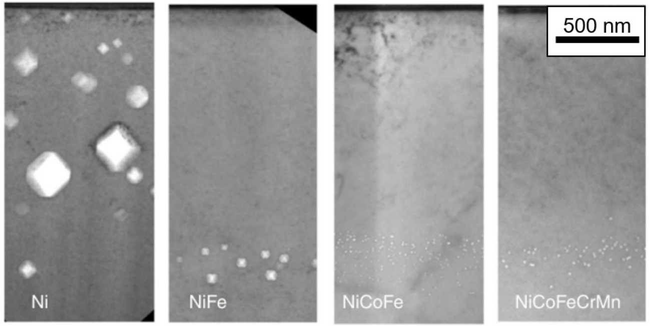
High-Entropy Alloys (HEAs)

- 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



(a) Conventional alloy, (b) High-entropy alloy
(Miracle & Senkov, 2016)



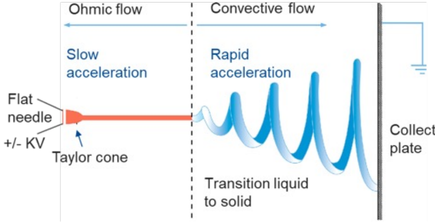
Void swelling shown to be less pronounced in more compositionally complex alloys upon heavy-ion irradiation
3-MeV Ni^{+} ions to 5 x 10^{16} cm^{-2} at 773 K
(Lu et al., Nature Com., 2016)

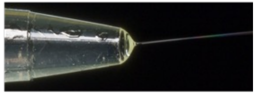
Large lattice distortion due to multiple elements of different sizes

- ✓ Sluggish diffusion of atoms, point defects and interstitial loops
- ✓ Reduction in segregation and defect clustering
- ✓ Promotion of recombination within the displacement cascade
- ✓ And more…

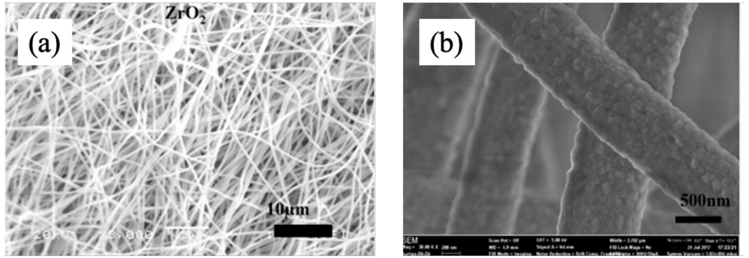
Electrospun Nanofiber Materials

- 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**





Electrospinning technique and Taylor cone.



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

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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

$$\mathrm{TRI} = \frac{R_\mathrm{M} c_p X_\mathrm{g}}{\bar{E}(1-\nu)\bar{\alpha} C_\mathrm{R} \rho^n} \cdot \left(\frac{T_\mathrm{m} c_p X_\mathrm{g}}{C_\mathrm{R} \rho^n} - 1 \right)^m$$

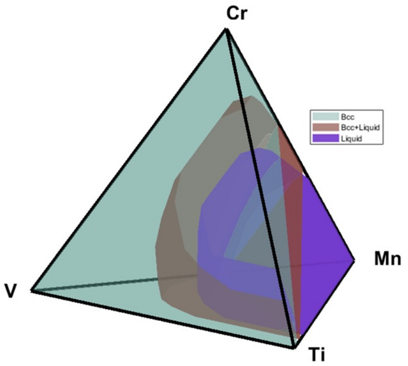
Bertarelli, 2014

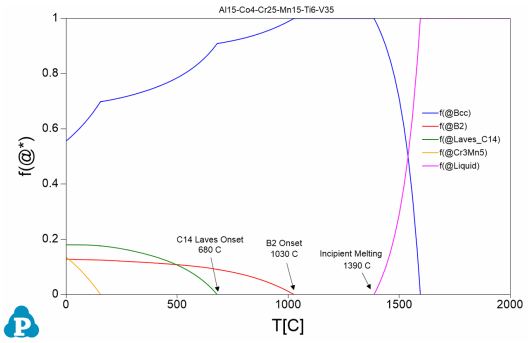
$$F.O.M = \frac{Y_S(1-\nu)k}{E\alpha\rho^2}$$

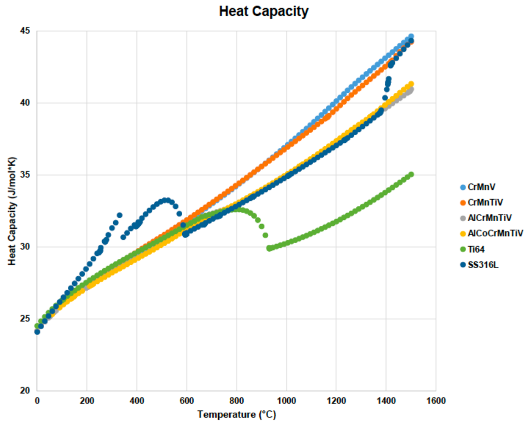
Ahmad et al., 2014

CompuTherm FOR MATERIALS DESIGN

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







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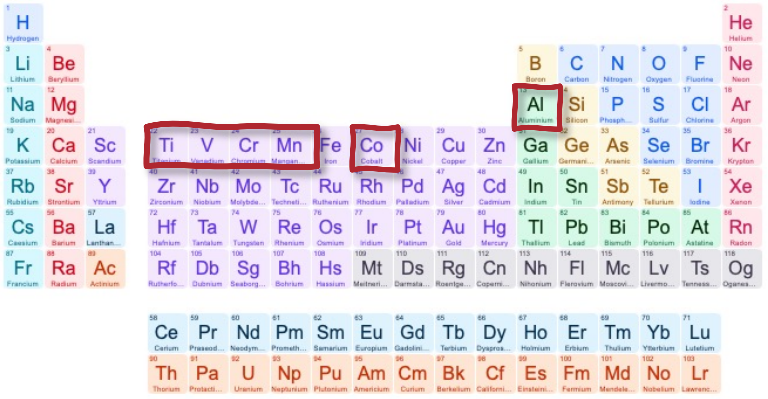
HEA Development

- **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



CrMnV
Equimolar BCC single phase

↓

CrMnTiV
Impurity getter

↓

AlCrMnTiV
BCC phase stabilizer

↓

AlCoCrMnTiV
Semi-coherent secondary phase

Preliminary HEA Synthesis

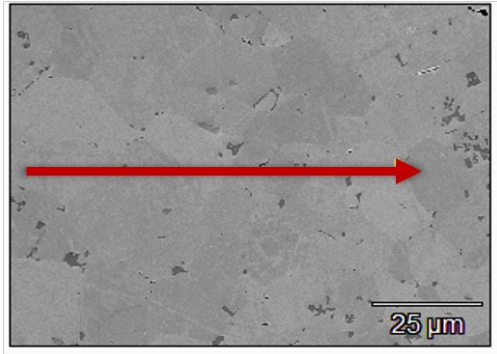
4 HEAs

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

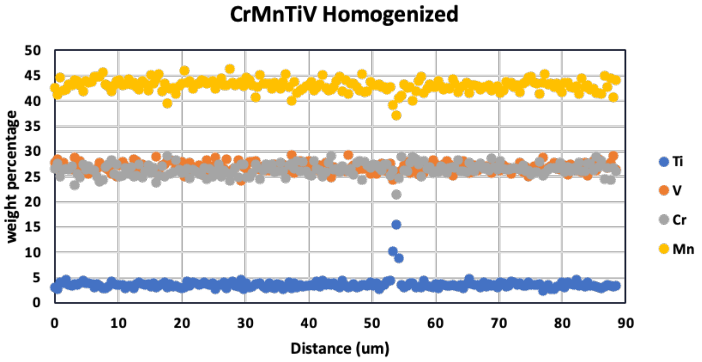
Sectioned arc-melted ingots (UW-Madison)

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

College of Engineering
UNIVERSITY OF WISCONSIN–MADISON



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



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

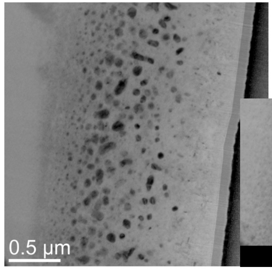
HEA plate fabrication and ongoing characterization

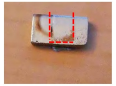
N. Crnkovich
G. Arora

- Procured plates from external vendor (Sophisticated Alloys)

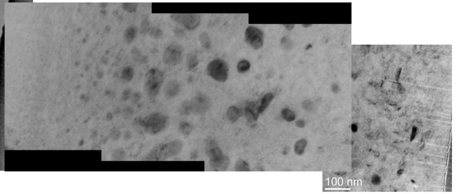
- NSUF RTE awarded to irradiate the 4 HEAs at University of Wisconsin Ion Beam Laboratory
  - V ions at 3.7 MeV
  - Up to 100 DPA, 500 C

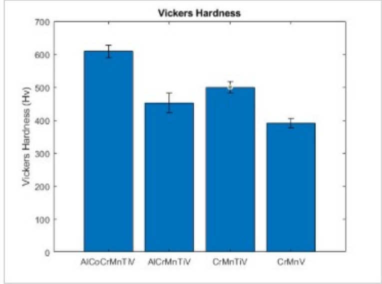
- TEM analyses ongoing (N. Crnkovich)

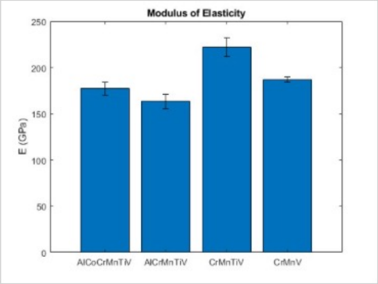


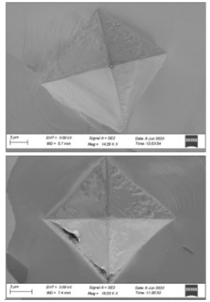


CrMnTiV HEA specimen irradiated to 100 DPA (UW-Madison, 2022)



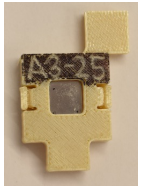






- Indentation measurements and validation of DFT elastic constant calculations
- No cracks observed at corners of indents (evidence of some ductility)

- HEAs survived high-intensity beam pulse at CERN's HiRadMat facility
- Impending PIE at MRF



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High Throughput CALPHAD simulations

N. Crnkovich
G. Arora

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

- **HEA compositions selected**
  - Al10-Co4-Cr25-Mn26-Ti1-V34
  - Al10-Co4-Cr27-Mn21-Ti4-V34

    Effect of Ti concentration as an impurity getter
  - Al16-Co2-Cr25-Mn30-Ti1-V26
  - Al16-Co4-Cr25-Mn30-Ti1-V24

    Effect of Co as B2 phase enhancer
  - Al20-Co1-Mn27-Ti2-V50
  - Al12-Co3-Cr6-Mn27-Ti2-V50

    Effect of Al concentration and removing Cr
  - Al18-Mn30-Ti2-V50
  - Al20-Co2-Mn26-Ti2-V50

    Effect of removing Co, with Al as the potential B2 phase enhancer

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

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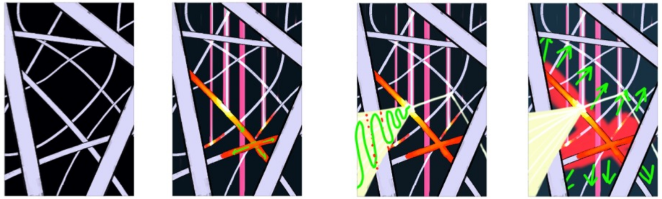
Electrospun Nanofiber Development

- 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**

W. Asztalos



1. Start!

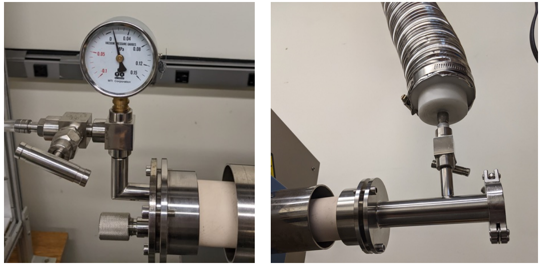
2. Beam hits: fibers instantly heat and conduction starts

3. Radiation is emitted from hot fibers

4. Helium around fibers heat, starts convection

(a) (b) (c)

(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

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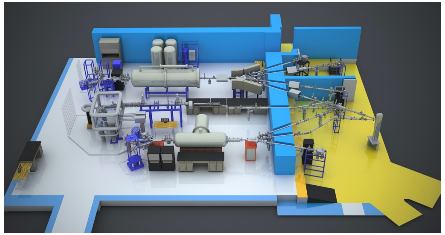
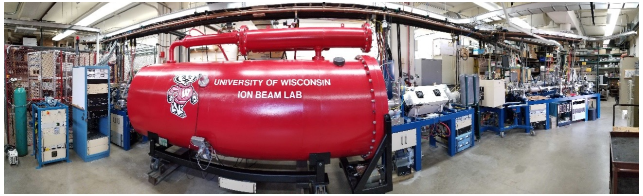
Materials Irradiation Screening

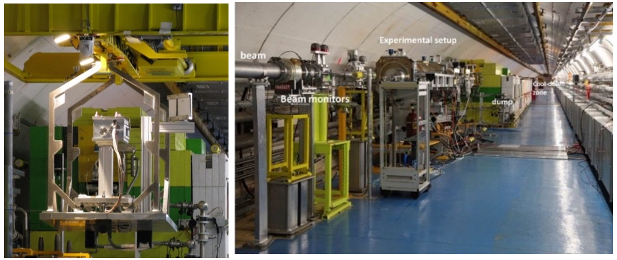
- **Low-energy (LE) ion irradiations** to emulate radiation damage effects and screen/downselect materials
  - 25 – 1000 C
  - 0.1 – 1 DPA
  - 500 – 2500 He appm/DPA
- **Thermal shock test**: CERN's HiRadMat facility or alternative method (Fermilab's A2D2 facility?)
- **Pre/Post-irradiation material 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)



Michigan Ion Beam Laboratory (MIBL) with triple-beam irradiation capabilities



Wisconsin Ion Beam Laboratory (IBL)



CERN's HiRadMat facility for single-shot thermal shock tests

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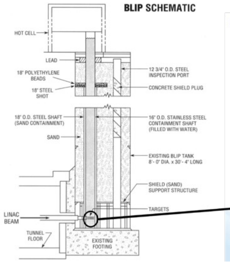
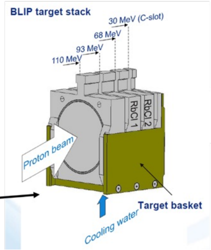
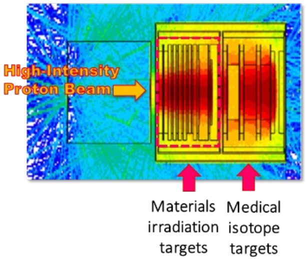
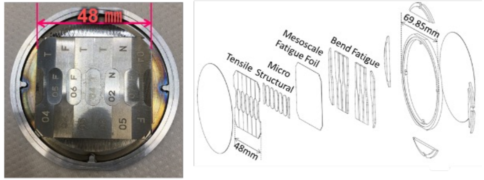
Prototypic Irradiations and Evaluation

- 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

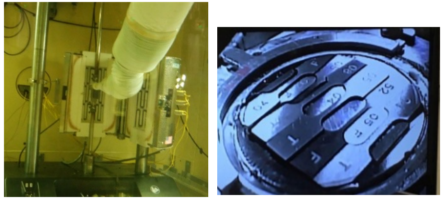








- PIE in hot cells due to activation of material specimens
  - Bulk thermal and mechanical property testing
  - Microscopy and microscale testing (TEM, nanoindentation, etc.)



Tensile testing in PNNL hot cells

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

Current Collaborations

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

- **University of Wisconsin-Madison**
  - A. Couet, N. Crnkovich, M. Moorehead, I. Szufarska, et al.

- **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 sett up PIE equipment (DSC, dilatometer, DIC)

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

RaDIATE Collaboration
Radiation Damage In Accelerator Target Environments

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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W. Asztalos

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Kavin Ammigan

RaDIATE Collaboration Meeting

29 June 2023