



## Novel Materials for Next-Generation Accelerator Target Facilities

**Kavin Ammigan**

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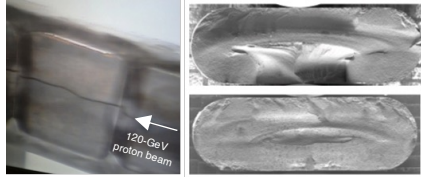
15<sup>th</sup> International Particle Accelerator Conference (IPAC'24)

22 May 2024

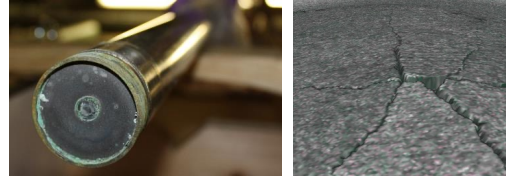
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# Motivation for Exploring Novel Beam-Intercepting Materials

Particle-production targets, beam windows, absorbers, ...

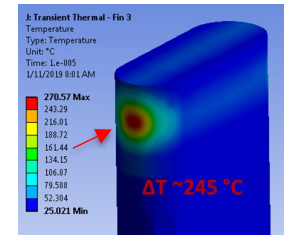
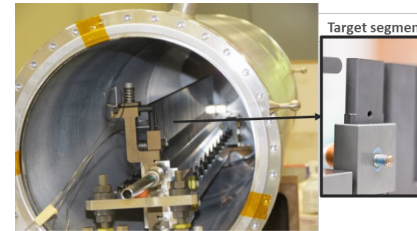
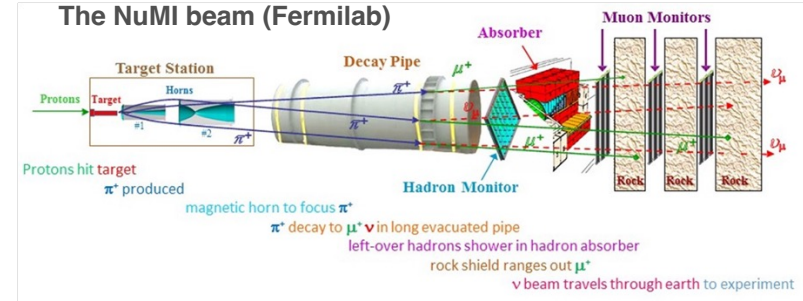


MINOS NT-02 target failure along beam path: radiation-induced swelling (FNAL)



Primary beryllium window embrittlement (FNAL)

- Target components are constantly bombarded by high-energy high-intensity beams
  - Operate under extreme conditions, enduring beam-induced **thermal shock** and **radiation damage**
  - Need to withstand millions of beam pulses
- Conventional materials are reaching their limits, and restricting the scope of experiments

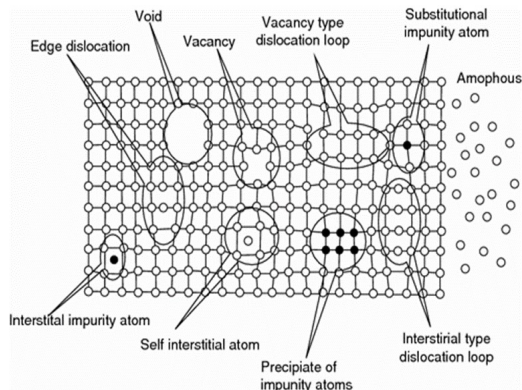


NOvA 1-MW target. Peak temperature rise  $\sim 245$  °C from 10- $\mu$ s beam pulse ( $2.5 \times 10^7$  K/s)

# Impacts of Beam-Induced Radiation Damage and Thermal Shock

**Radiation Damage:** disruption of lattice structure of the material upon irradiation

Expressed by Displacements Per Atom (DPA)

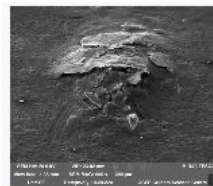


Dimensional changes after irradiation, D.L. Porter and F. A. Garner, J. Nucl. Mater., 159, p. 114 (1988)

- Hardening and embrittlement (loss of ductility)
- Bulk swelling (dimensional changes)
- Fracture toughness reduction
- Thermal conductivity reduction
- Coefficient of thermal expansion increase
- Transmutation and gas production
- ...

**Thermal Shock:** Localized energy deposition in the material induces dynamic stress waves

- Caused by short pulsed beams
- Cyclic loading environment can lead to **fatigue failure**



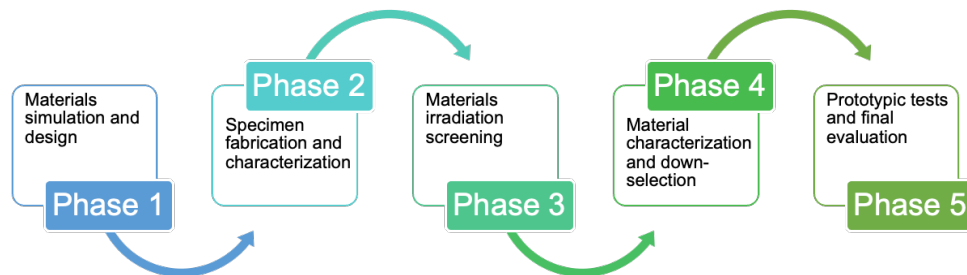
Sigrflex graphite (left) and Iridium (right) targets tested at CERN's HiRadMat facility with high-intensity single-shot beam pulse

# Looking Beyond Conventional Materials

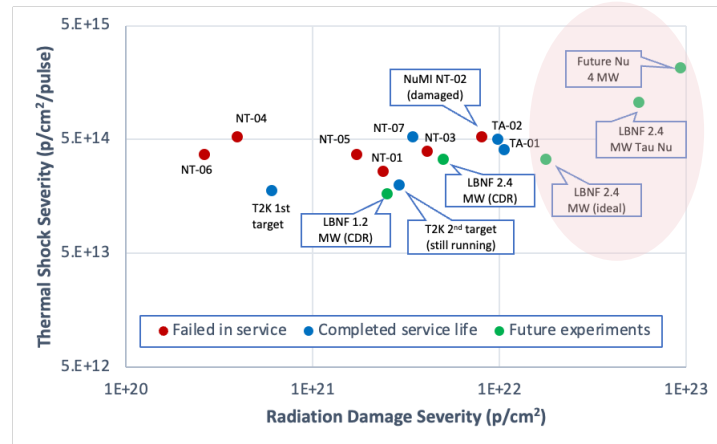
To advance the state-of-the-art of beam-intercepting materials

- Enable future multi-MW accelerator target facilities
  - LBNF/DUNE 2.4 MW, Mu2e-II, Future Muon Collider (4 MW+), ...
- Maximize particle production efficiency, improve reliability and operation lifetimes

## High-Entropy Alloys and Nanofiber Materials Development Program



## Neutrino HPT R&D Materials Exploratory Map

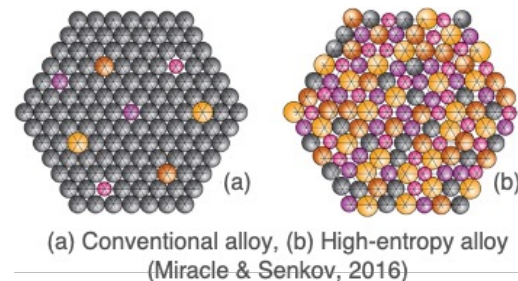


- Expect >10x increase in accumulated proton fluence and power density



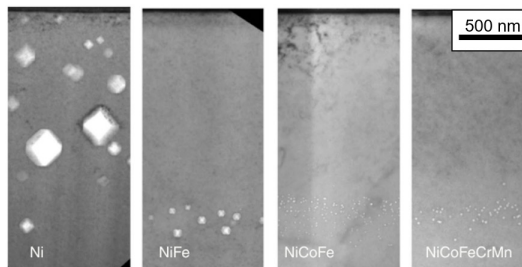
# Why High-Entropy Alloys (HEAs)

- Unlike conventional alloys, HEAs contain multiple principal elements in roughly equal proportions
- Offer large composition space to explore new alloy systems with tunable properties

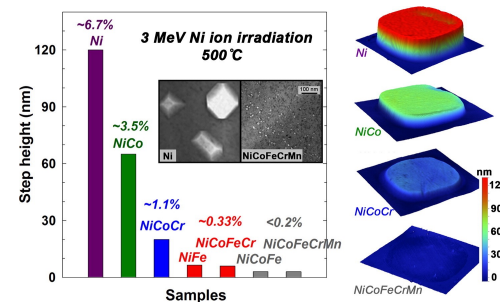


## HEAs have shown promising properties and remarkable radiation damage resistance

- High-temperature strength
- High specific strength
- Improved fatigue and fracture properties
- Corrosion and oxidation resistance
- **Enhanced radiation tolerance**
  - Greater phonon scattering, sluggish diffusion of atoms/defects, broad migration energies, and more...



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



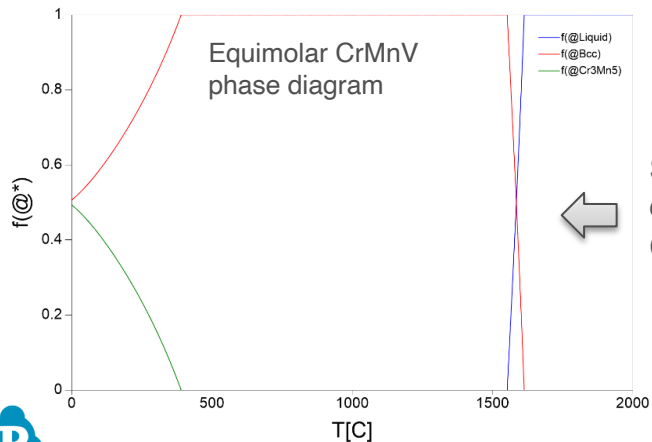
Swelling of increasingly complex alloys under ion irradiation, Jin et al., Scripta Materialia 119 (2016)

## HEAs for Accelerator Beam Windows

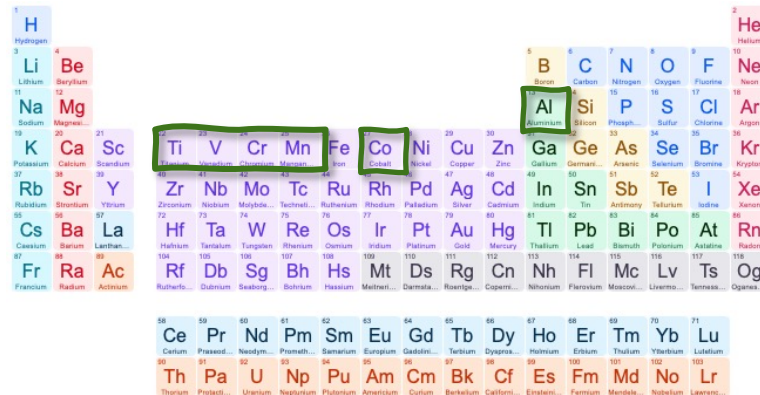
## Elemental selection considerations

- Light-weight elements (low density)
- Primarily a single-phase
- Minimal activation

## HEA design approach: CALPHAD simulations to predict phase diagrams of the multicomponent alloy system



Single body-centered-cubic phase predicted by CALPHAD (400 – 500 °C)



CrMnV (Barron et al., 2020)  
Equimolar BCC single phase

CrMnTiV  
Impurity getter

AlCrMnTiV  
BCC phase stabilizer

AlCoCrMnTiV  
Semi-coherent secondary phase

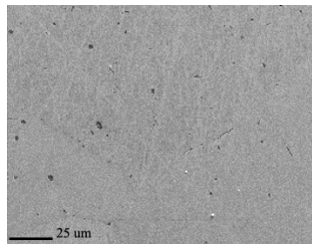
# HEA Synthesis & Characterization



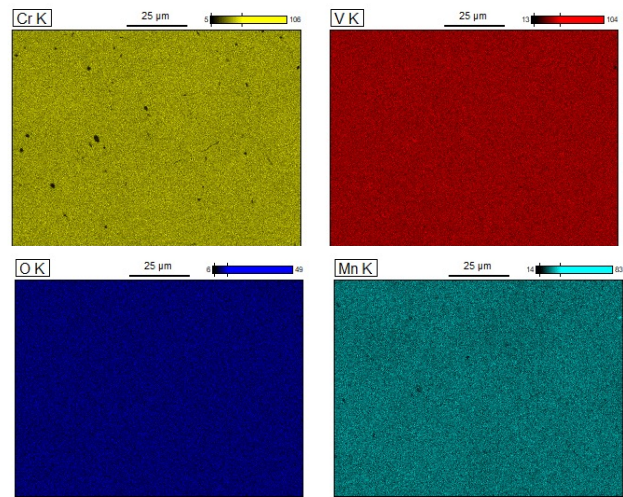
Sectioned arc-melted ingots (UW-Madison)



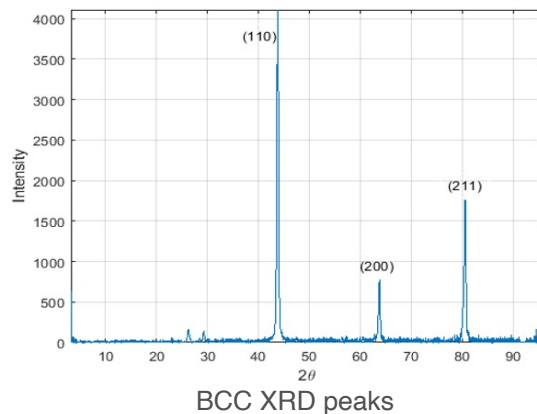
Larger plates fabrication by Sophisticated Alloys Inc.



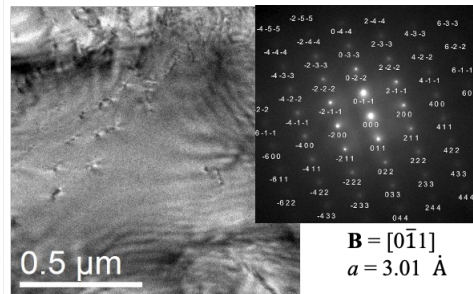
SEM-EDS maps of CrMnV alloy



- Plates underwent heat treatment to promote single phase
  - HIP under Ar at 1200 °C, 15 ksi for 4 hrs
  - Homogenized at 1200 °C for 48 hrs
- Achieved target composition within 1 at%
- SEM-EDS confirmed homogeneity
- XRD confirmed BCC single phase



BCC XRD peaks

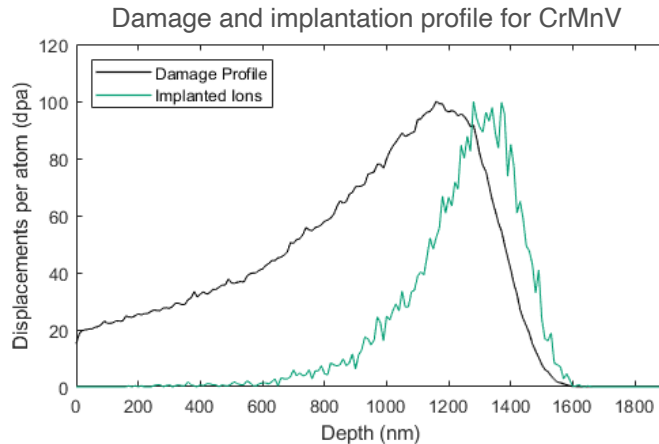


$$\mathbf{B} = [0\bar{1}1]$$
$$a = 3.01 \text{ \AA}$$

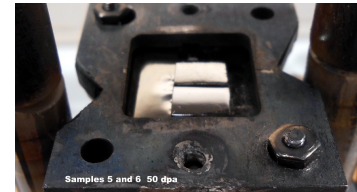
N. Crnkovich, UW-Madison

# Evaluating Radiation Damage with Low-Energy Ion Irradiation

- **Ion irradiation:** fast, inexpensive, no activation
- Study radiation-induced effects in the material
  - Voids, dislocation loops, elemental segregation
  - Phase stability
  - Hardening and embrittlement through micromechanical testing methods



Wisconsin Ion Beam Laboratory (IBL)



Samples in holder prior to irradiation

## CrMnV HEA and Ti-6Al-4V irradiation

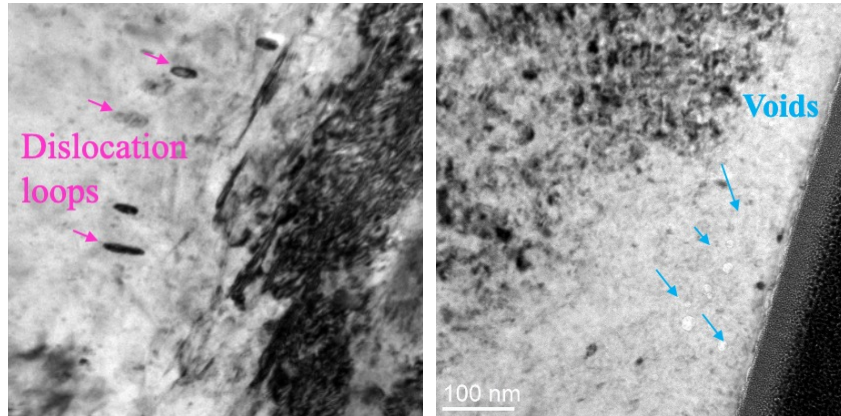
- 3.7 MeV  $V^{2+}$  ions at 500 °C
- 50 and 100 DPA at  $2.5 \times 10^{12}$  ions/cm<sup>2</sup>/s



# Post-Irradiation Radiation-Induced Microstructural Defects

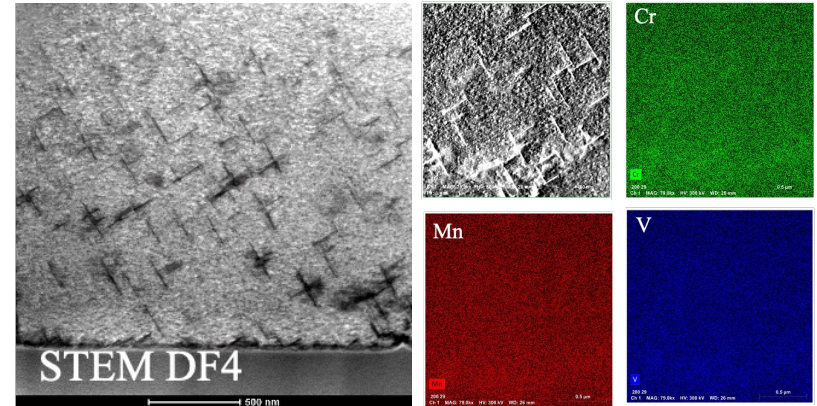
TEM analysis of CrMnV and Ti-6Al-4V samples at 50 DPA

Ti-6Al-4V at 50 DPA



- Void formation and dislocation loops observed in irradiated region

CrMnV at 50 DPA

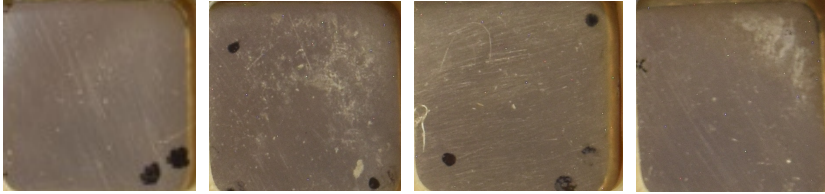


N. Crnkovich, UW-Madison

- Needle morphology observed, as in unirradiated condition, but no discernible segregation along needles
- No voids or dislocation loops observed

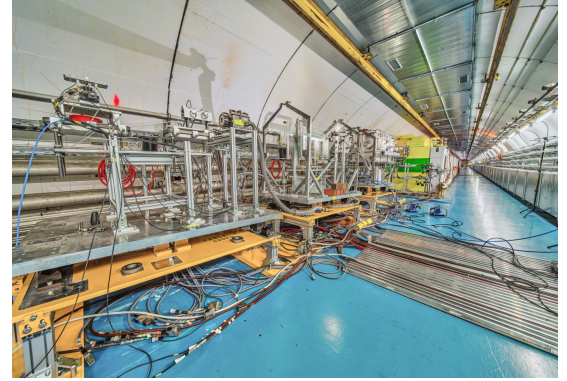
# HEAs Exposed to High-Intensity Pulses at CERN's HiRadMat Facility

- Thermal shock test of several HEA specimens
  - Single-shots:  $7 \times 10^{12}$  protons/pulse, 440 GeV,  $\sigma$ : 0.25 mm
  - $\Delta T \sim 1200^\circ\text{C}$  in 8  $\mu\text{s}$

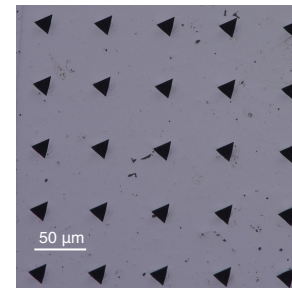
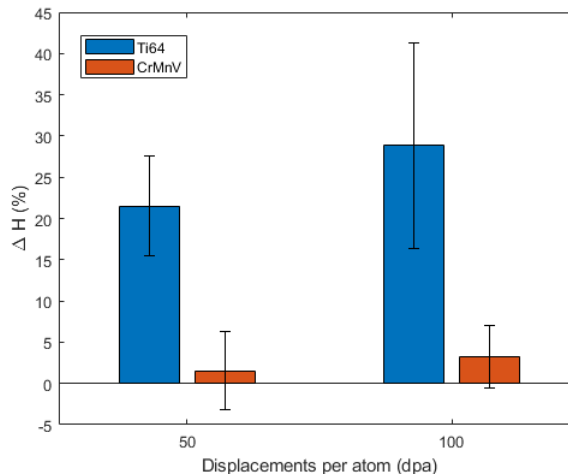
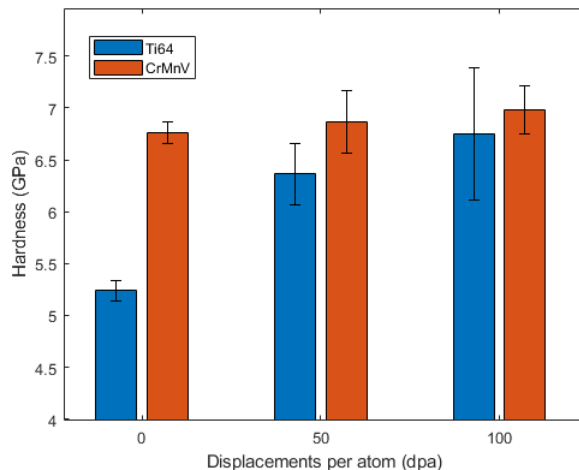


- No visible damage to HEA specimens
- Ensuing profilometry and SEM will assess plastic deformation

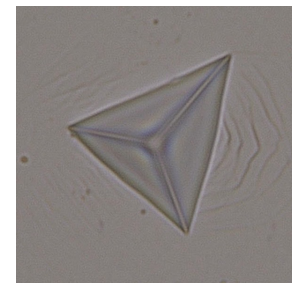
Delamination of  
graphite foil



# Minimal Hardening of CrMnV HEA up to 100 DPA



Berkovich tip  
nanoindentation  
matrix on sample

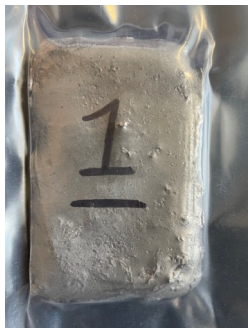


No observable cracks  
at corners of indents  
indicate ductility

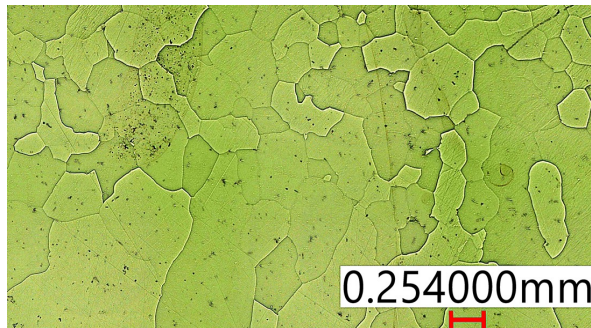
- Less than 5% hardening of CrMnV at 100 DPA
- Hardening of up to 30% in Ti-6Al-4V at 100 DPA
  - Likely due to observed irradiation-induced voids and dislocations from TEM analysis

# HEA Design Refinement

- Systematic compositional space search using high-throughput CALPHAD simulations
  - ~120,000 composition explored/optimized
  - Broaden BCC single phase region
  - Reduce concentration of impurities
  - Introduce semi-coherent B2 phase and reduce density



New batch of optimized HEAs



AlCoCrMnTiV HEA, Grain size  $\approx 150 - 500 \mu\text{m}$

CrMnV (Barron et al., 2020)  
Equimolar BCC single phase

CrMnTiV  
Impurity getter

AlCrMnTiV  
BCC phase stabilizer

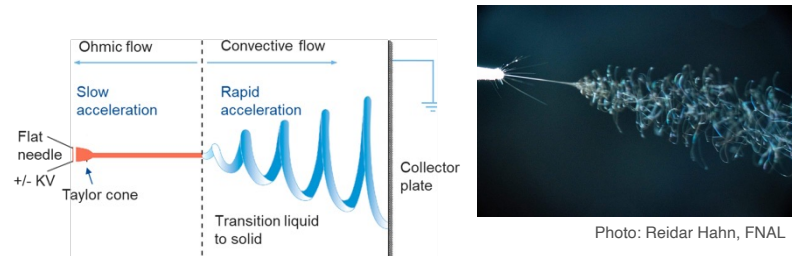
AlCoCrMnTiV  
Semi-coherent secondary phase

Material characterization,  
irradiation and post-irradiation  
characterization underway

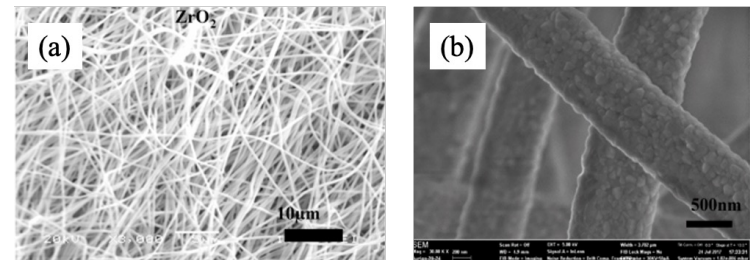


# Electrospun Nanofiber Materials

- Intrinsically tolerant to both thermal shock and radiation damage
- Nanofiber continuum is discretized at the microscale but continuum at meso-scale
  - More uniform heating - fiber diameter orders of magnitude smaller than typical beam spot sizes
  - Fiber discontinuity mitigates stress wave propagation
- Evidence of radiation damage resistance due to nanopolycrystalline structure of nanofibers
  - Large number of grain boundaries acts as defect sinks



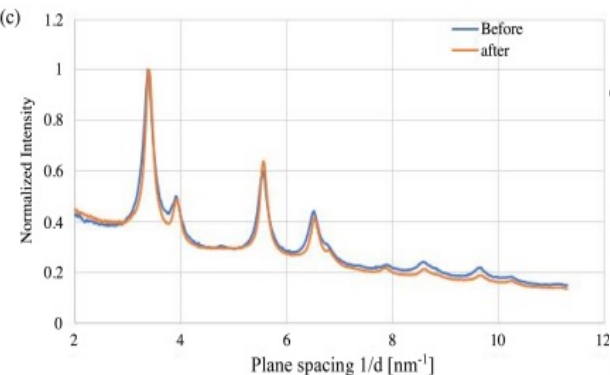
Electrospinning technique and Taylor cone.



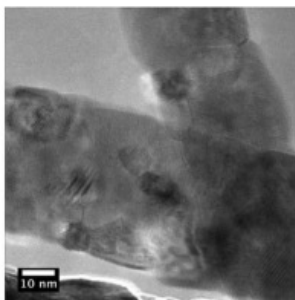
SEM images of Zirconia nanofibers produced at Fermilab, (a) bulk nanofiber mat, (b) single nanofibers revealing polycrystalline grains (Bidhar et al., PRAB, 24, 2021)

# In-Beam Tests of Ceramic Nanofibers

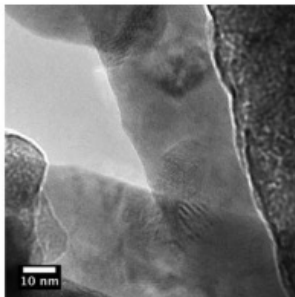
## Ion irradiation (in-situ TEM)



TEM before irradiation



TEM after irradiation



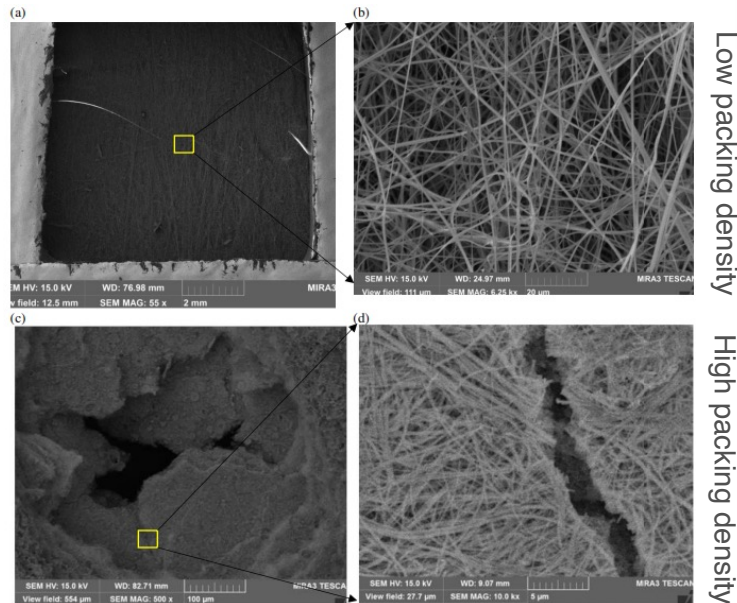
IVEM (ANL)  
S. Bidhar, PRAB, 24, 2021

- 1 MeV  $\text{Kr}^{2+}$  ion
- $3.25 \times 10^{15}$  ions/ $\text{cm}^2$ , 5 dpa
- **No measurable change in lattice parameter**
- **No defects formation**
- Initial evidence of radiation damage resistance

## Thermal shock test (HiRadMat)



**HiRadMat**  
High-Radiation to Materials

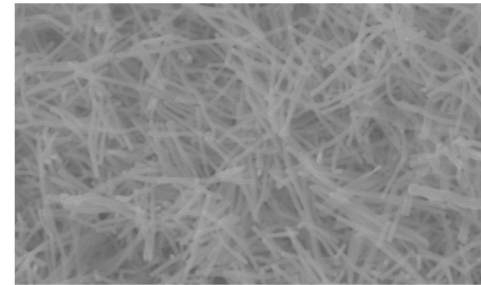


440 GeV protons,  $\sigma$ : 0.25 mm,  $1.21 \times 10^{13}$  protons in 4  $\mu\text{s}$

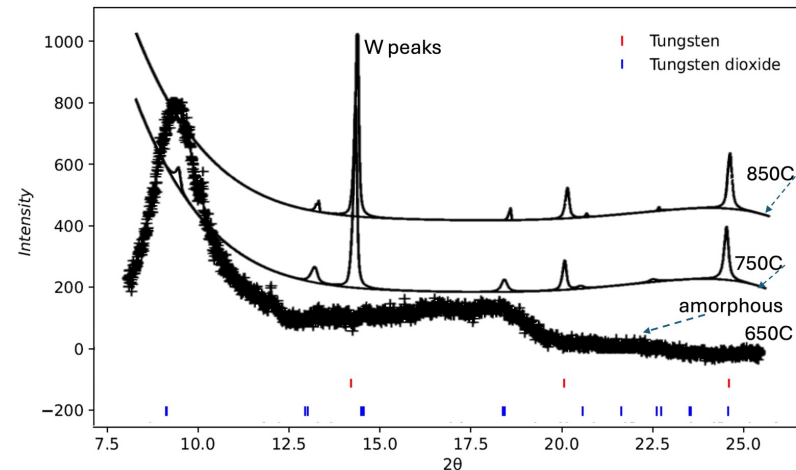
- Observed damage in higher packing density samples (W. Asztalos, Paper TUPS44)

# Tungsten Nanofiber Production

- High-density material to compensate for the reduced effective density in nanofiber form (W nanofiber  $\sim 2 \text{ g/cm}^3$ )
- Primary challenge is reducing oxide content after heat treatment of W-doped polymer nanofiber sample (several iterations)
- **In-situ XRD finally showed heat treatment at 850 °C in forming gas (95%N<sub>2</sub>, 5%H<sub>2</sub>) produced 99%W-1%WO<sub>2</sub>**
- Currently planning in-situ ion irradiation to assess radiation damage behavior



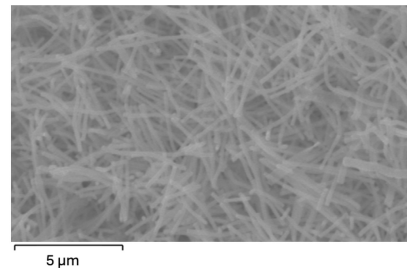
SEM of W nanofibers showing 0.5 μm fiber diameter



In-situ XRD of tungsten nanofibers during heat treatment (IMSERC facility – Northwestern University)

# Conclusions

- Initial HEA synthesis and tests demonstrate promising radiation damage tolerance
- Success in fabricating highly pure Tungsten Nanofibers
- Iterative novel material development process
  - Refining composition and microstructure (heat treatment)
  - Microstructural characterization and bulk property testing before and after irradiation
  - Complementary DFT and MD modeling of radiation damage effects to guide material design
  - Irradiation and evaluation under prototypic beam conditions (high-energy proton beams)
- Our goal is to develop robust target materials to enable next-generation multi-MW accelerator target facilities





# Acknowledgements

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