Fermilab Science Office of Science



High-Power Targetry R&D for Next-Generation Accelerator Target Facilities

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High-power targetry (HPT) challenges

Next gen. multi-MW particle production accelerators expect $\approx 10X$ proton fluence & power density increase

- Target survivability concerns have led several facilities to limit beam power NuMI-MINOS, FNAL (2010-11): faulty welds
 - Reduced beam power (-10% to -40%)
 - MLF, J-PARC (2015-16)
 - Early replacement of target/limited beam power SNS, ORNL (2013-14)
 - Reduced beam power (-15%) frequently
- Radiation damage and thermal shock are the primary material challenges
- HPT critical for maximizing particle production efficiency

Neutrino HPT R&D Materials Exploratory Map



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Radiation damage & thermal shock effects

D.L. Porter and F. A. Garner.

Radiation damage:

- Hardening/embrittlement/creep
- Lattice expansion/void formation/bulk swelling
- Fracture toughness reduction
- Thermal/electrical conductivity reduction
- Coefficient of thermal expansion
- Modulus of elasticity
- Accelerated corrosion



Pulsed beam thermal shock

- $E \text{ dep.} \rightarrow \Delta T \rightarrow \text{dynamic stress wave}$
 - 1 MW target: 250 K in 10 µs (2.5 x 10⁷ K/s)
 - Plastic deformation, cracking, fatigue failure

Material response dependent on:

- Specific heat (ΔT)
- Coefficient of thermal expansion (strain)
- Modulus of elasticity (stress)
- Flow stress behavior (plastic deformation)
- Strength (yield, fatigue, fracture toughness)

Sigraflex target tested at CERN's HiRadMat facility





Test Iridium target irradiated at CERN's HiRadMat



What We Want to Avoid...



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



Be window embrittlement (FNAL)



NOvA MET-01 target fin fracture (FNAL)



MINOS NT-01 target containment water leak (FNAL)



Horn stripline fatigue failure (FNAL)



ISOLDE target (CERN)



Target containment vessel cavitation (ORNL - SNS)

Neutrino Targets MINOS (400 kW)



•	Helium	
	atmosphere	

- Beryllium • windows
- Water cooled graphite core

		MINOS
Э	Graphite fins	47 x 20 mm x 6.6 mm
	Beam energy [GeV]	120
	p/pulse	3.37E+13
	Power [kW]	340
	σ [mm]	1.1
	Peak Temp. [°C]	330
	QS Temp [°C]	60
	POT	6.55E+20
	Peak dpa	0.63
	Peak He [appm]	2270

NOvA (0.7 – 1 MW)





1 MW ach

Peak dpa

Peak He [appm]

Helium • atmosphere Beryllium windows . Water cooled • aluminum pressing

plates

Graphite core ٠

nieved (3 hr)!						
	`	NOvA	AIP			
\$	Graphite fins	50 x 24 mm x 7.4 mm	50 x 24 mm x 9 mm			
	Beam energy [GeV]	120	120			
	p/pulse	4.90E+13	6.50E+13			
	Power [kW]	700	1000			
	σ [mm]	1.3	1.5			
3	Peak Temp. [°C]	670	1000			
	QS Temp [°C]	390	890			
	POT	1.10E+21	1.28E+21			

1.10

5580

0.96

3600



LBNF-DUNE (1.2 – 2.4 MW)

- Helium atmosphere
- Titanium target containment windows
- Helium gas cooled graphite core

	DUNE
Graphite fins	TBD
Beam energy [GeV]	60-120
p/pulse	7.50E+13
Power [kW]	1200-2400
σ [mm]	2.67
Peak Temp. [°C]	TBD
QS Temp [°C]	TBD
POT	2.54E+21
Peak dpa	0.73
Peak He [appm]	400

Graphite (purified)

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Research Approach - Prototypic irradiation to closely replicate material behavior in accelerator target facilities



- Analysis of in-beam accelerator components to directly test effects of high-energy, pulsed proton beams
- High-energy proton irradiation of material specimens at BNL-BLIP facility in partnership with the RaDIATE collaboration
 - Post-Irradiation Examination (PIE) at participating institution with hot-cell facilities (PNNL)
 - In-beam thermal shock experiment at CERN's HiRadMat facility including pre-irradiated (BLIP) and non-irradiated specimens

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• Low energy ion irradiation to achieve high damage levels on short timescales and without activation

He implantation of Beryllium



XX

UK Atomic Energy

NuMI target (NT-02) autopsy and failure analysis



- D, G-band broaden at beam center
- Second order 2D-band gradually disappear
- I(D)/I(G) ratio stops increasing at high damage levels
- higher level of damage → significant broadening
- Total pore volume decreased from ~ 12 vol.% to ~ 8 vol.%
- Reduction of pores greater than 0.1 µm³ (bulk swelling)





- Temperature gradient: ~370°C (beam center) ~60°C (cooled edge)
- Inert helium environment

M. Jiang et al., Carbon 213 (2023) 118181

201

High-Cycle Fatigue Testing of Irradiated Ti Alloys

Proton-irradiated fatigue life data crucial in evaluating component lifetime

- First high-cycle fatigue testing of irradiated Titanium at Fermilab
- Design of 3rd generation fatigue testing machine under development
- Complement prev. tensile tests on irradiated Ti









HRMT60 – RaDIATE experiment





~120 material specimens tested:

- Graphite, Ti alloys, Beryllium, Nanofiber mats, High-Entropy alloys, Sigraflex, TFGR tungsten, SiC/SiC composite
- Understand single-shot thermal shock response and limits
- Explore novel advanced materials
- Assess the performance of various grades of conventional materials
- Compare the behavior of non-irradiated to pre-irradiated materials
- Directly measure beam-induced dynamic effects to validate simulation

Preliminary results:

- Visual inspection and high-resolution pictures of specimens
- Most of the samples didn't show track of beam damage except nanofiber specimen
- Predicted through heat transfer simulations in nanofiber media.











Nanofibers as production targets

Inherent resistance to radiation damage/thermal shock

- Radiation tolerance
 - Nanopolycrystalline grain structure
 - Absorb defects
 - In-situ ion irradiation & TEM: Argonne National Lab IVEM to 5 DPA
 - No observed damage
- Thermal shock
 - Discrete at microscale
 - Reduced temperature gradient (1D)
 - Heat dissipation in gas (high surface area & porosity)
 - Absorb/dampen stress waves
 - Survived test CERN's HiRadMat facility

(Bidhar et al., PRAB, 24, 2021)









Zirconia nanofibers produced at Fermilab

- (a) Bulk nanofiber mat
 - b) Single nanofibers revealing polycrystalline grains





- Low packing density samples survived

- Holes, crack in higher density samples

Multiphysics Simulations of HRMT-43/60 Nanofiber Mats



Pressure distribution at end of beam heating



1. Start!



conduction starts



3. Radiation is emitted from hot fibers

4. Helium around fibers heat, starts convection

Porous Media Models: three modes of heat transfer

- Conduction: introduce effective thermal conductivity k(T)for nanofiber mat due to Bhattacharyya and Daryabeigi
- *Convection:* use Darcy's Law to simulate fluid flow, include energy equation in simulations
- Radiation: Mie Theory to solve scattering off infinite cylinder exactly, feed into model due to Lee



Will Asztalos (MS/PhD work, IIT) Rapid heating

OF TECHNOLOGY

ILLINOIS INSTITUT

- Pressure wave for high packing dens. Higher packing density
- Reduced airflow
- Greater pressure on fibers

Asztalos, W., et al. *arXiv preprint arXiv:2405.19496* (2024)

Pure W Nanofiber Fabrication

Initial SEM/EDS studies

- Presence of Oxygen (8 ~14 wt%)
- Also showed the presence of Carbon (black color)

Increased heat treatment temperature

- Reduced oxide formation
- Oxygen wt% dropped from 14% to 1.35%
- XRD analysis ongoing to evaluate crystal parameters, phase and structure





С

Ν

0

AI

Si

w

Total:

HEA properties and compositions

Microstructure to combat radiation damage

- Sluggish atom diffusion
- Reduce defect segregation, increase recombination
- Phase stability

CALPHAD: CALculation of PHAse Diagrams

- Broad range: single-phase BCC
 - B2 precipitates: strengthening
- Low $Z \rightarrow$ reduce density
 - Decreased energy deposition and scattering

4 Gen. 1 compositions

- Varying number of alloying elements:
 - CrMnV → AlCoCrMnTiV

8 Gen. 2 compositions

Equimolar BCC single phase

CrMnV

 Study effects of different relative atomic concentrations



Void swelling lessened in more complex alloys (3-MeV Ni⁺, 5 x 10¹⁶/cm², 773 K), Lu et al., Nature Comm., 2016



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



Impurity getter (<8%)

CrMnTiV

HEA homogeneity & impurity distribution



Energy Dispersive X-ray Spectroscopy (EDS)

- High degree of homogeneity observed in principal elements
- Ti working well as impurity getter
 - Captures C, N, O, S, predominantly at grain boundaries



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Nanoindentation studies

Gen. 1

e250

5.2

- Significant hardness increase with increased complexity (right)
 - Mitigated by heat treatment
- Signs of ductility

2.7

2.5

2.8

5.7

- Stiffer than Ti-64 (E = 110 GPa)
- Less stiff than Beryllium (E = 303 GPa)

2.6

N. Crnkovich, UW-Madison

College of Engineering NIVERSITY OF WISCONSIN-MADISON

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SEE: IWSMT16 presentation in October

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- Work being done in the framework of the RaDIATE Collaboration (<u>https://radiate.fnal.gov</u>)
 - 80+ participants from 20 institutions worldwide
- Funded by DOE Early Career Award (Kavin Ammigan)



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Bonus slides



The NuMI Beam



Target station able to operate with 1-MW

v beam travels through earth to experiment

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- Main injector proton beam (120 GeV/c) incident on
 1.2 m graphite target to create charged pions and kaons.
- Pions focused in horns decay into muons/muon neutrinos in decay pipe to produce neutrinos.
- Beam detectors downstream of the decay pipe monitor the neutrinos produced and residual charged particles for the experiments.

Radiation damage process

Beam-induced damage

- Incident proton displaces target atoms
 - Causes damage cascade
 - Accumulated damage measured as Displacements Per Atom (DPA)



From D. Filges, F. Goldenbaum, in:, Handb. Spallation Res., Wiley-VCH Verlag GmbH & Co. KG aA, 2010, pp. 1–61.

Microstructure effects

- Creation of point defects (interstitial/vacancy)
- Transmutation products (H and He production)



From V. Verma, K. Katovsky, Radiation Damage and Development of a MC Software Tool, in: Spent Nuclear Fuel and Accelerator-Driven Subcritical Systems, Springer, 2019.

Radiation damage bulk effects

Bulk effects:

- Hardening/embrittlement
- Creep
- Lattice expansion and bulk swelling
- Fracture toughness reduction
- Thermal/electrical conductivity reduction
- Coefficient of thermal expansion
- Modulus of elasticity
- Accelerated corrosion
- Void formation and embrittlement (due to transmutation)



Factor of 10 reduction in thermal conductivity of graphite after 0.02 DPA

D.L. Porter and F. A. Garner, J. Nuclear Materials, **159**, p. 114 (1988)



Void swelling in 316 stainless steel tube exposed to reactor dose of $1.5 \times 10^{23} \text{ n/cm}^2$



Thermal shock & stress waves

Pulsed beams: prompt E dep. $\rightarrow \Delta T \rightarrow$ dynamic stress wave

- Localized compressive stress
 - Fast expansion of material within cooler surrounding •
 - 1 MW target: 250 K in 10 µs (2.5 x 10⁷ K/s) •
- Stress waves move at sonic velocities
- Can result in
 - Plastic deformation •
 - Cracking
 - Fatigue failure

Sigraflex target tested at CERN's **HiRadMat** facility





Test Iridium target irradiated at CERN's HiRadMat



1 MW NuMI target simulation by K. Ammigan

Material response dependent on:

- Specific heat (ΔT)
- Coefficient of thermal expansion (strain)
- Modulus of elasticity (stress)
- Flow stress behavior (plastic deformation)
- Strength (yield, fatigue, fracture toughness)



Initial stress wave amplitude

Elastic wave speed

Nanofibers as production targets

Inherent resistance to radiation damage and thermal shock

- Radiation tolerance
 - Nanopolycrystalline grains \rightarrow absorb defects
- Thermal shock
 - Discrete at microscale
 - Reduced temperature gradient (1D)
 - Heat dissipation in gas (high surface area & porosity)
 - Absorb/dampen stress waves (discontinuity)





Photo: Reidar Hahn, FNAL

Electrohydrodynamic production of nanoscale fiber mats

- Electrostatic repulsion > surface tension
 - Droplet stretched
 - Jet elongated
- Zirconia nanofiber production in place
- Tungsten nanofibers under development







(c)

FNAL electrospinning setup: (a) Power supply and electrospinning setup, (b) high-temperature furnace for nanofiber heat treatment, (c) rollto-roll nanofiber fabrication technique



High entropy alloys (HEAs) for beam windows

- Alloy with 3+ principal elements
- Near equi-atomic compositions
- Primarily a solid-solution matrix with distorted crystal lattice (atomic size difference)
- Large composition space (adjustment of atomic ratios)





(a) Conventional Alloy, (b) High-entropy alloy Miracle and Senkov, Acta Materialia 122 (2017) 448-511

Many HEAs exhibit:

- Good ductility & hightemperature strength
- High strength to density ratio (specific strength)
- Fatigue, fracture, corrosion, oxidation resistance

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Oh et al., Nat Comm. 10, 2090 (2019) Youssef et al., Materials Research Letters, 95-99 (2015)

HEA radiation damage resistance

HEAs: microstructure to combat radiation damage

- Sluggish atom diffusion
 - distortion & size mismatch
 - Reduced segregation and defect clustering
 - Increased in-cascade recombination
- Phase stability
 - reduce grain coarsening and void swelling

Reduced defect segregation + increased recombination:

- Minimizes void formation & swelling (right, top)
- Reduces bulk swelling effects (right, bottom)
- Increasing # of elements → greater effects
 - vs. pure materials/traditional alloys
- Phonon scattering/migration energies



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



Simulations to determine HEA compositions

CALPHAD: CALculation of PHAse Diagrams

Select compositions with:

- Broad range: single-phase BCC
 - Increased ductility/machinability
 - B2 precipitates: strengthening
- Low $Z \rightarrow$ reduce density
 - Decreased energy deposition and scattering
 - Minimize beam loss in window

4 Gen. 1 compositions

- Varying number of alloying elements:
 - CrMnV → AlCoCrMnTiV

8 Gen. 2 compositions

Equimolar BCC single phase

CrMnV

 Study effects of different relative atomic concentrations



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Impurity getter (<8%)

CrMnTiV

HEA Synthesis

4 Gen. 1 HEA compositions

- Varying number of alloying elements:
 - CrMnV: Equimolar with single BCC phase
 - CrMnTiV: Ti as impurity getter
 - AICrMnTiV: AI to stabilize BCC phase
 - AICoCrMnTiV: Co for B2 precipitates



Sectioned arc-melted ingots (UW-Madison)

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

8 Gen. 2 compositions from Sophisticated Alloys to study effects of relative concentration

- 5 AICoCrMnTiV compositions to study:
 - Varied Ti concentration as impurity getter
 - Varied Co concentration \rightarrow secondary B2 phase
- Increased AI content without Cr
 - BCC phase stability
- Absence of Co
 - Al as B2 phase enhancer



Gen. 2 plate from Sophisticated Alloys

Grain structure and orientation

Electron backscatter diffraction (EBSD)

- Mapping to determine grain sizes
- + EBSD map shows grain sizes D ${\approx}150$ 500 ${\mu}m$ up to > 1mm
- Future study: nanoindentation orientation dependence



Helios 5 CX at FNAL IPF Z Color 9





 $AI_{12}Co_3Cr_6Mn_{27}Ti_2V_{50}$ 20X optical



EBSD Kikuchi pattern



Elemental composition: Gen. 1



(N. Crnkovich, UW-Madison)



Mechanical properties following irradiation

Minimal hardening of CrMnV (< 5% at 100 DPA)

- Hardening of up to 30% in Ti-6AI-4V at 100 DPA
- Likely due to observed irradiation-induced voids and dislocations from TEM analysis



Crystal lattice structure and spacing

TEM completed on Gen. 1 alloys using FEI Tecnai TF-30 TEM (Nick Crnkovich, UW-Madison)



CrMnV: single phase BCC with d = 0.301 nm

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Irradiation resistance testing

Post-irradiation TEM examination of Gen. 1 alloys (Nick Crnkovich, UW-Madison)

- 50 and 100 DPA irradiated specimens
- Comparison to Ti-64 reference
 material

Ti-64 (left): many voids/dislocation loops observed at 50 DPA

- \rightarrow not observed in CrMnV at 50 DPA (right)
- Needle morphology of CrMnV shows no chemical segregation



Crystal lattice structure and spacing

Transmission electron microscopy (TEM) at UW-Madison

Properties of interest

- Lattice spacing/expansion
 - Body centered cubic (BCC)
- B2 precipitates
 - Strengthening through dislocation pinning
- Transmission EDS
 - Composition of precipitates
- Irradiation induced defects





BCC

B2

Lindahl, B. B., Burton, B. P., & Selleby, M. (2015). Ordering in ternary BCC alloys applied to the Al–Fe–Mn system. Calphad, 51, 211-219.



Precipitate identification in Gen. 2 HEAs

Gen. 2 TEM ongoing at UW-Madison

- Pristine samples initially
- 0.15, 0.30, 0.45, 5, and 10 DPA in future

$AI_{10}Co_4Cr_{25}Mn_{26}Ti_1V_{34}$ shown to right

- 10 30 nm precipitates observed
- Indexing to confirm phases awaits higher resolution images



Diffractogram (FFT) of entire micrograph to right







RaDIATE collaboration created in 2012, with Fermilab as the leading institution. The collaboration has grown up to 20 institutions over the years.

Objective:

- Harness existing expertise in nuclear materials and accelerator targets
- Generate new and useful materials data for application within the accelerator and fission/fusion communities

Activities include:

- Analysis of materials taken from existing beamline as well as new irradiations of candidate target materials at low and high energy beam facilities
- In-beam thermal shock experiments

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