Electrospun nanofiber materials for high power target applications

Sujit Bidhar

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Outline

• Background & Objectives
• Electrospinning process
• In-house electrospinning unit
• Candidate target materials manufacturing
• Single nanofiber mechanical characterization
• Future plans and summary
Background- Target material

• Demand of multi-MW high performance particle production targets
  – LBNE 2.3MW proton beam, $1.6 \times 10^{14}$ p/pulse
• Structural integrity over time
  – High temperature, thermal stresses, dynamic stresses
• Withstand radiation damage
  – Embrittlement, radiation corrosion, swelling
Current Target

- ANU/NOvA, 700KW → Graphite blocks

Proton beam

Water cooling

Compressive strength 345MPa
Tensile strength: 60~140 MPa
Endurance Limit: 20 MPa

Beam spot size << target dimension
Solid continuum → high local temperature gradient, thermal stress wave

Can it perform satisfactory at higher energy??
HPT Issues - Stress wave

T2K window
750kW
Spill width: 4.2 µsec
Gaussian beam
Sigma 4.2 mm

Prone to fatigue failure (Mode II)

Reduce amplitude, decrease wave speed

Radial temperature distribution at end of pulse

T2K window

Fermilab

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Fatigue life

Initial stress wave amplitude

$$\Delta \sigma_0 = \sqrt{\rho E \cdot \alpha \cdot \frac{\Delta T}{\Delta t}}$$

temperature gradient

Number stress cycles ~ Elastic wave speed

$$c = \frac{E}{\sqrt{\rho}}$$

Possible by microstructure design
Sinuous Target - Candidate Microstructure

Design Microstructure to mitigate issues

Electrospun nano-fiber $\rightarrow$ Sinuous target

Electrospun Zirconia nanofiber*

*Journal of Alloys and Compounds 649 (2015) 788e792
Advantages & Limitations

Advantages:
• Local damage of single fiber won’t affect target structural integrity as a whole
• Reduced thermal stress wave
  – Reduced temp. gradient
  – High surface area & gaps → effective local heat removal by passing He gas
• Customize material properties by mixing different materials

Limitations:
• Widely used for polymer nanofibers
• Limited research in ceramics/metal nanofiber
• Low density (long target)
Objective

Fabricate ceramic/metal, composite material nano-fiber with high strength, high density and alloying elements to reduce radiation damage.

- Set up in-house electrospun unit
- Fabricate ceramic composites nanofiber
- Micro-mechanic study single fiber mechanical characterization
Basic Electrospinning Set up

10-30 kV DC

Process carried out at room temp. and atm. pressure

Collector
- Fixed type
- Rotating drum type
Electrospinning Principle

Jet Initiation stages

- Electrostatic repulsion > surface tension
  - Droplet is stretched
  - Jet elongated by whipping action

Ohmic flow
- Slow acceleration
  - Flat needle
  - +/- kV

Convective flow
- Rapid acceleration
  - Transition liquid to solid
  - Bending Instability

Taylor cone

Flow direction
- Liquid
- Solid
- Collector plate

Fermilab
Ceramics Nanofiber Fabrication

Inorganic precursor (inorganic compound + solvent)

Polymer solution (polymer + solvent)

Salt additives (surfactants)

Solution for electrospinning → polymer-ceramic nanofiber

Calcination (Heat treatment)

- Vaporize polymer
- Promotes crystal growth
- Bonding

Representative heating profile
Lab Set-up

Electrospinning unit

Furnace
Electrospinning Set-up

- High voltage power supply
- Syringe pump
- Needle
- Collector plate
Electrospinning Jet
Nanofiber Mat – Random Orientation
Nanofiber Mat- Aligned

Reflector plate

C-Channel

Needle
Focused Deposition

- Reflector plate
- Needle
- PVC Pipe Focusing
- Grounded plate
Improvised Compact Power Supply

120 Watt, 120VAC in

-5kV DC out

60kV DC out

4 Watt, 6~12VDC in, +/-20kV DC out

- Much safe to use (120W→4W!)
- Mobile compact unit
  - Can be run on 9 or 12 V battery
Candidate electrospun nanofiber - Raw materials

Polymer solution
• PVP + Ethanol + Aceton

Metal/Ceramic
• Alumina $\rightarrow$ Aluminum 2,4-pentadionate + Aceton
• Zirconia $\rightarrow$ Zirconium Carbonate + Acetic Acid
• $\text{WO}_3$ $\rightarrow$ Ammonium meta-tungstate + D.I. Water
• $\text{TiO}_2$ $\rightarrow$ Titanium Isopropoxide

Carbon-nanotube Composite
• CNT-Alumina
• CNT-Zirconium

Proposed

Done
Alumina Nanofiber

As spun

After heat treatment
Theoretical Al wt% in Al₂O₃ is 53% Achieved in actual 25%
Titania(TiO2) Nanofiber

As spun  After heat treatment
EDS Mapping TiO₂

As spun

After heat treatment

Theoretical Ti wt% in TiO₂ is 60%
Achieved in actual 51%
Tungsten Oxide (WO3) Nanofiber

As spun

After heat treatment
Zirconia Nanofiber

As spun

After heat treatment

1.2 gm

0.5 gm
Theoretical Zr wt% in ZrO₂ is 74%  
Achieved in actual 62%

Theoretical Zr wt% in ZrO₂ is 74%  
Achieved in actual 62%
Zirconia Nanofiber- Yttrium doped

Improve thermal shock resistance by Yttrium doping

Improve radiation resistance

- More grain boundaries blocks dislocation, defect movements, defect recombination*.
- YSZ strong resistance to amorphization*

CNT-Ceramics Nanofiber Composites

Excellent mechanical properties
E : 1~5 Tpa
Tensile strength : 15-50 Gpa
Elongation % : 16%
High thermal conductivity (axial), insulator lateral

Protects Aluminum metal against radiation damage~70 DPA
• 1-D transport network for He to escape
• Reduce embrittlement by 5-10 times.

1~2 Vol% CNT

Bulk not nanofiber!!

CNT-Zirconia/Alumina Proposed Nanofiber

- Ceramic (Zirconia) will have high Z value
- CNT will enhance mechanical strength, provide protection against radiation damage

**Diagram Description:**
- SWNT + Surfactant → Sonification → deionized water → Add Alumina → Add polymer Solution → Electrospin → Calcination/Heat treatment → +ve charged alumina Nano-particle → CNT filled ceramic nanofiber
Nano-mechanical mapping – Atomic Force Microscopy

Tungsten – Polymer nanofiber

Elastic Modulus map

340 MPa

Peak Force, nN

Separation, µm

Tip Deflection in contact mode

\[ F - F_{adh} = \frac{4}{3} E^* \sqrt{R(d - d_0)^3} \]

Nanofibers fixed to substrate using double sided tape
Soft substrate compared to nanofiber
Nano-mechanical mapping – Atomic Force Microscopy

Elastic Modulus map

Nanofiber solution casted on harder smooth mica substrate

Average Elastic modulus ~ 100GPa
Mechanical characterization

Fracture strength single nanofiber
1. 3 point Bending test on TEM Grid

Set up
• Solution cast
• Fix to TEM grid using Ion beam (Pt tape)
• Press using diamond AFM tip

2. Nano-indentation using AFM tip

Macro testing electrospun nanofiber mat
Propose target shape

Alumina nano-fiber block

No major modification to current target holder
Customizable
Cheaper and scalable
Physical Properties Characterization (In progress)

- Raman spectroscopy:
  - disorderness, bond information
- Electron energy loss spectrometry (EELS):
  - sp2/sp3 ratio, atomic composition (low Z)
- Wide Angle X-ray Diffraction (WAXD):
  - lattice parameters, orientation, isotropy.
- Thermal analysis:
  - DSC and DMA: melting and glass transition temperature
Summary and Future Work

• Installation of a low cost electrospinning set up completed.
• Success in fabricating metallic and ceramic nanofiber.
• Physical properties of single nanofiber evaluation in progress.

Future work
• Expose nanofiber mat to HiRadMat test
  • Single fiber radiation damage study
• Improve ductility of ceramic nanofiber
  • Fabricate ceramics-CNT composite.
  • Heat treatment profile
• Physical properties before and after radiation.
  • Damage modeling
Thank You for Your Attention!!