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Electrospun nanofiber materials for high power target applications

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Outline

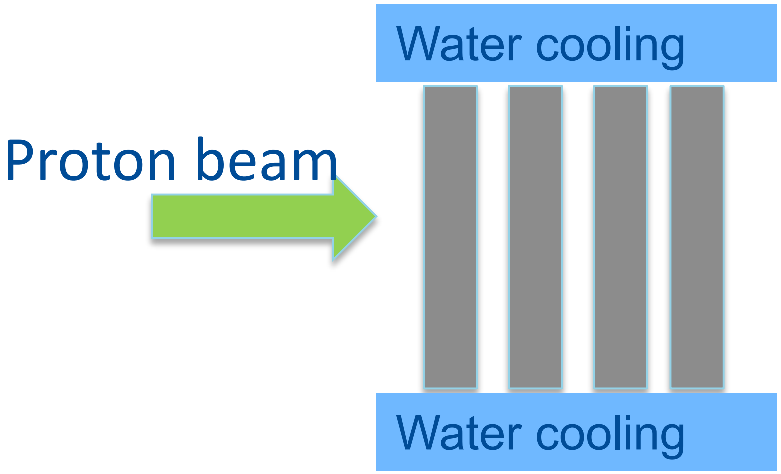
- Background & Objectives
- Electrospinning process
- In-house electrospinning unit
- Candidate target materials manufacturing
- Single nanofiber mechanical characterization
- Future plans and summary

Background- Target material

- Demand of multi-MW high performance particle production targets
  - LBNE 2.3MW proton beam, $1.6X10^{14}$ 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



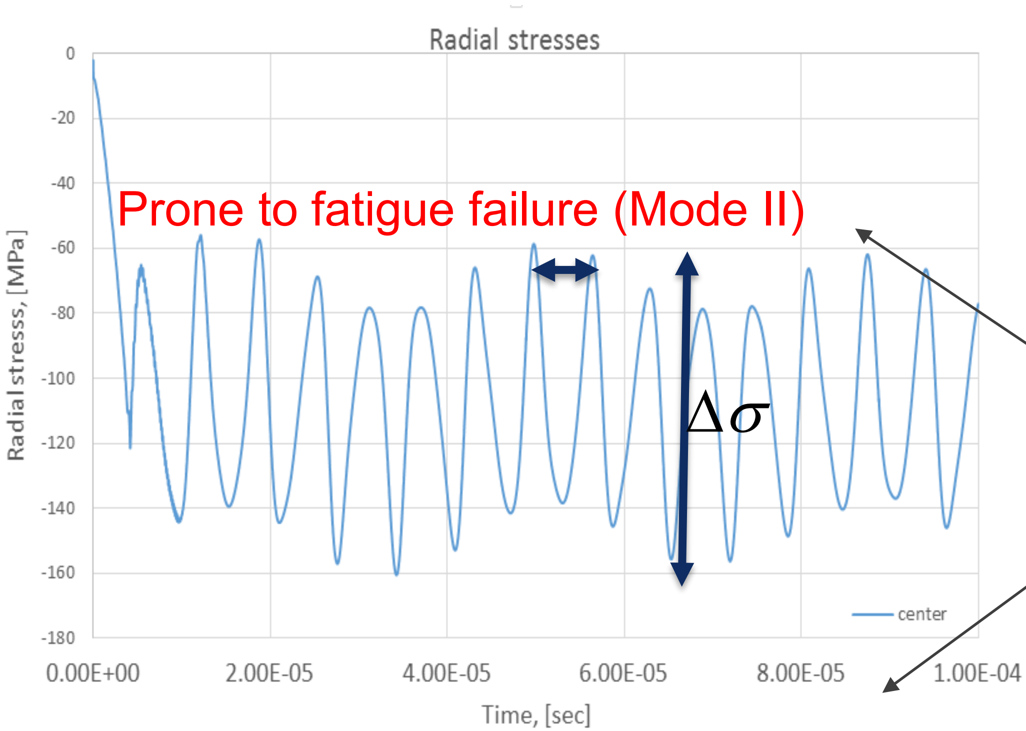
Compressive strength 345MPa
Tensile strength : 60~140 MPa
Endurance Limit : 20 MPa

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

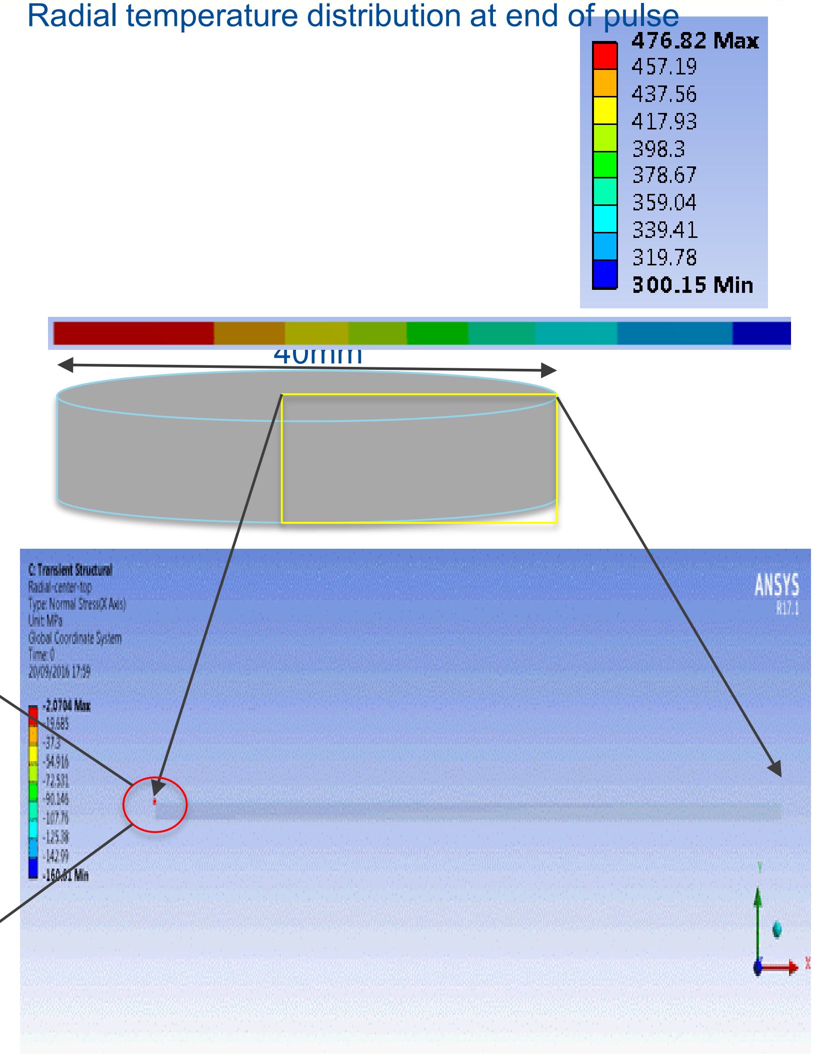
Can it perform satisfactory at higher energy??

HPT Issues -Stress wave

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



Radial temperature distribution at end of pulse



Reduce amplitude, decrease wave speed

T2K window

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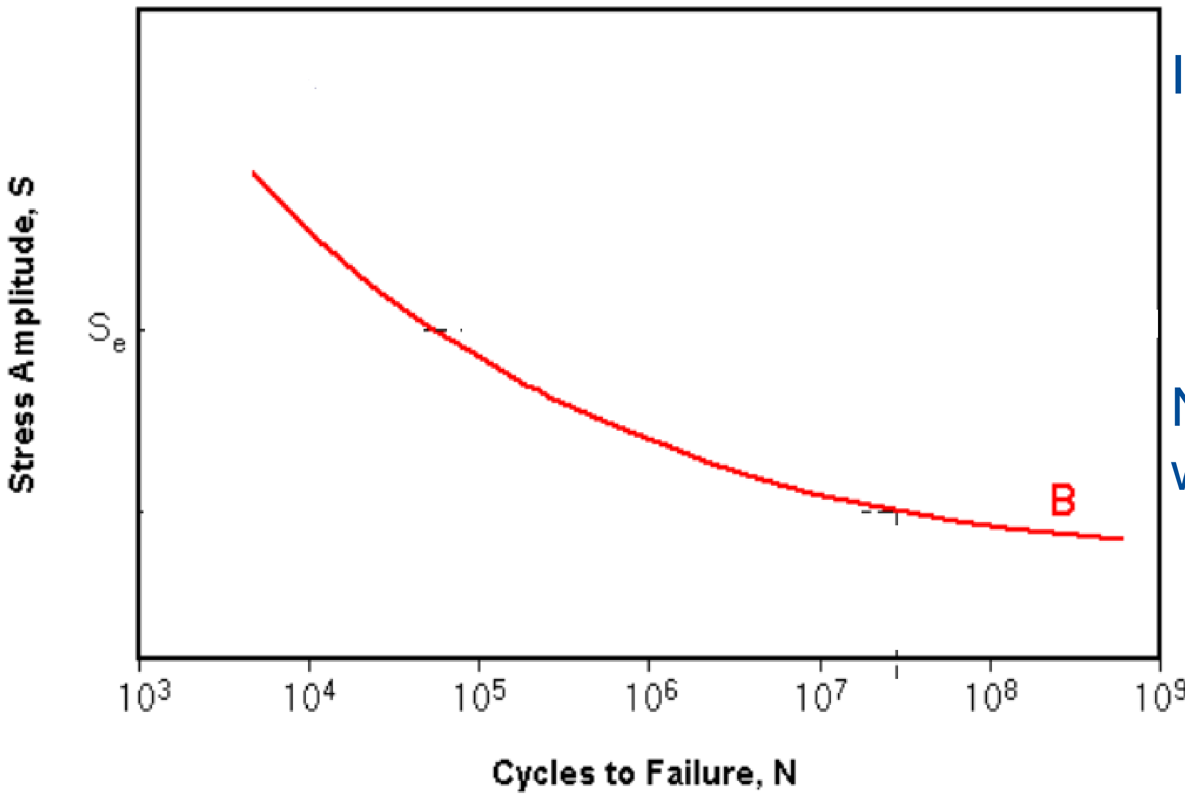
Fatigue life



Initial stress wave amplitude

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

temperature gradient

Number stress cycles ~Elastic wave speed

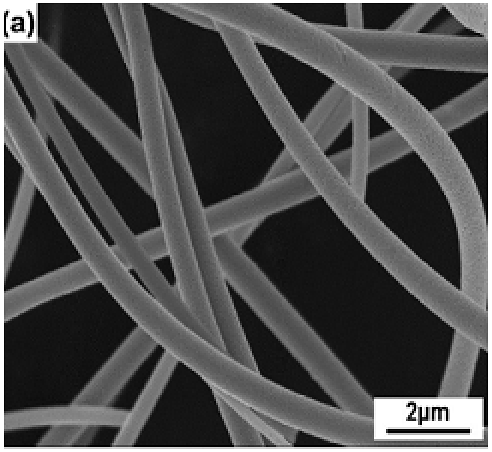
$$c = \sqrt{\frac{E}{\rho}}$$

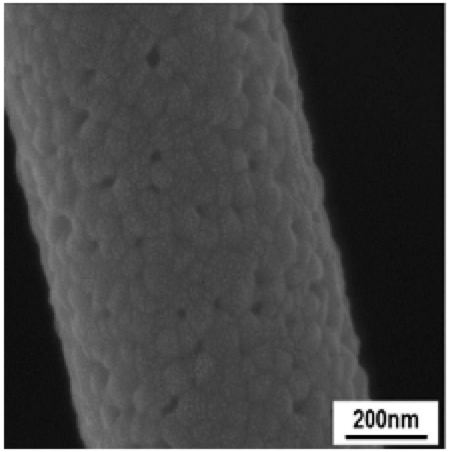
Possible by microstructure design

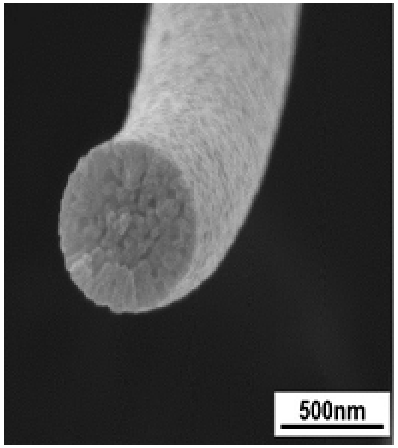
Sinuous Target -Candidate Microstructure

Design Microstructure to mitigate issues

Electrospun nano-fiber→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:

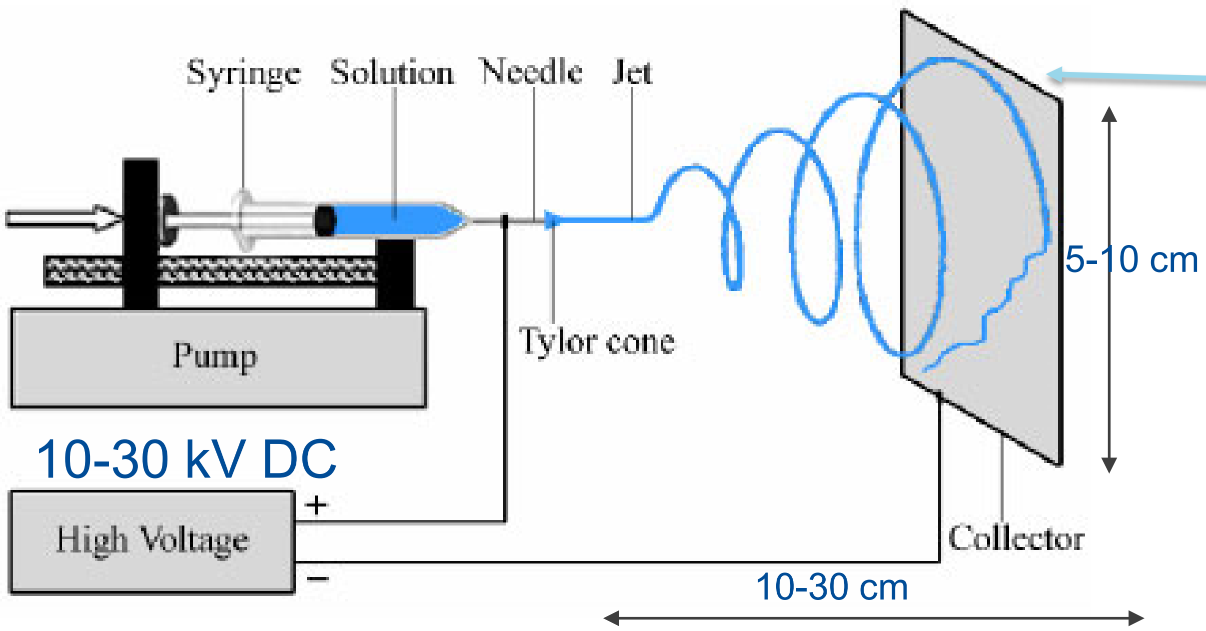
- Widely used for polymer nanofibers
- Limited research in ceramics/metal nanofiber
- Low density (long target)

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



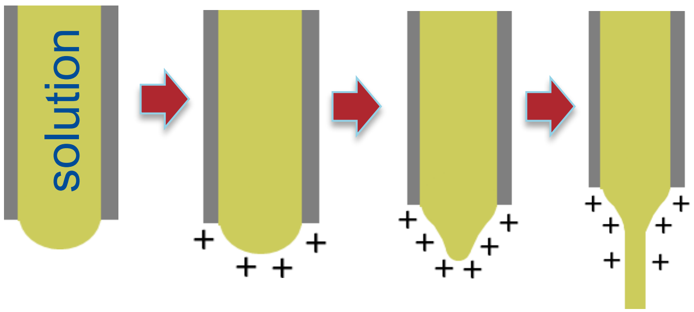
Collector

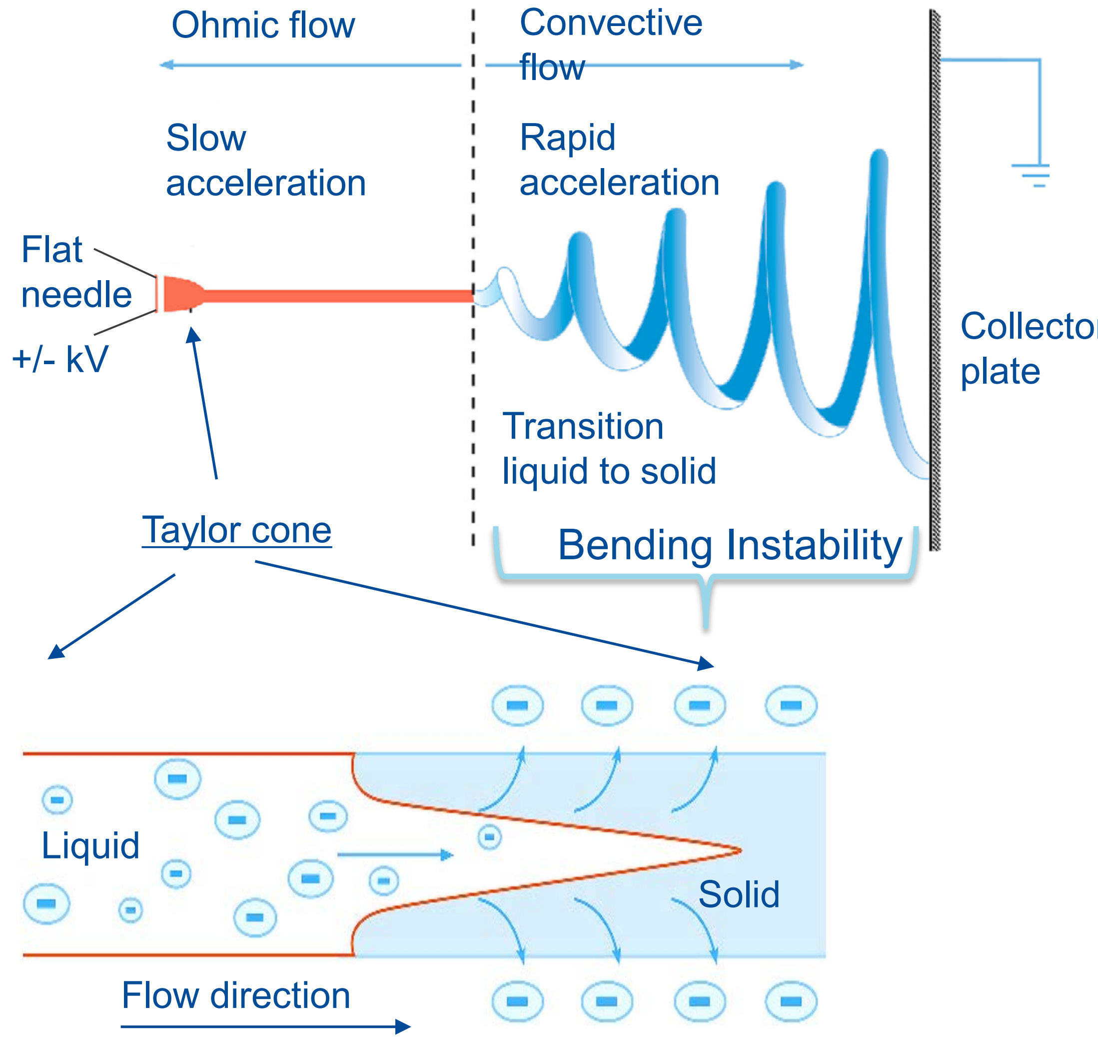
- Fixed type
- Rotating drum type

Process carried out at room temp. and atm. pressure

Electrospinning Principle

Jet Initiation stages





Electrostatic repulsion> surface tension

- Droplet is stretched
- Jet elongated by whipping action

Ceramics Nanofiber Fabrication

Inorganic precursor(inorganic compound+solvent)

+

Polymer solution(polymer+solvent)

+

Salt additives (surfactants)

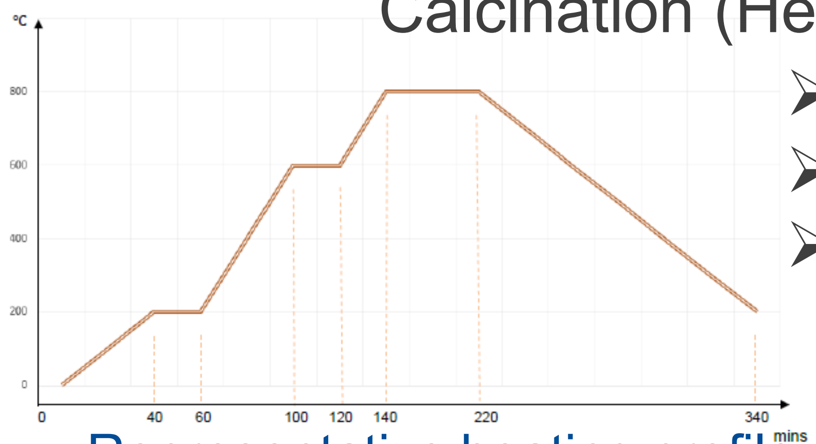
↓

Solution for electrospinning→polymer-ceramic nanofiber

↓

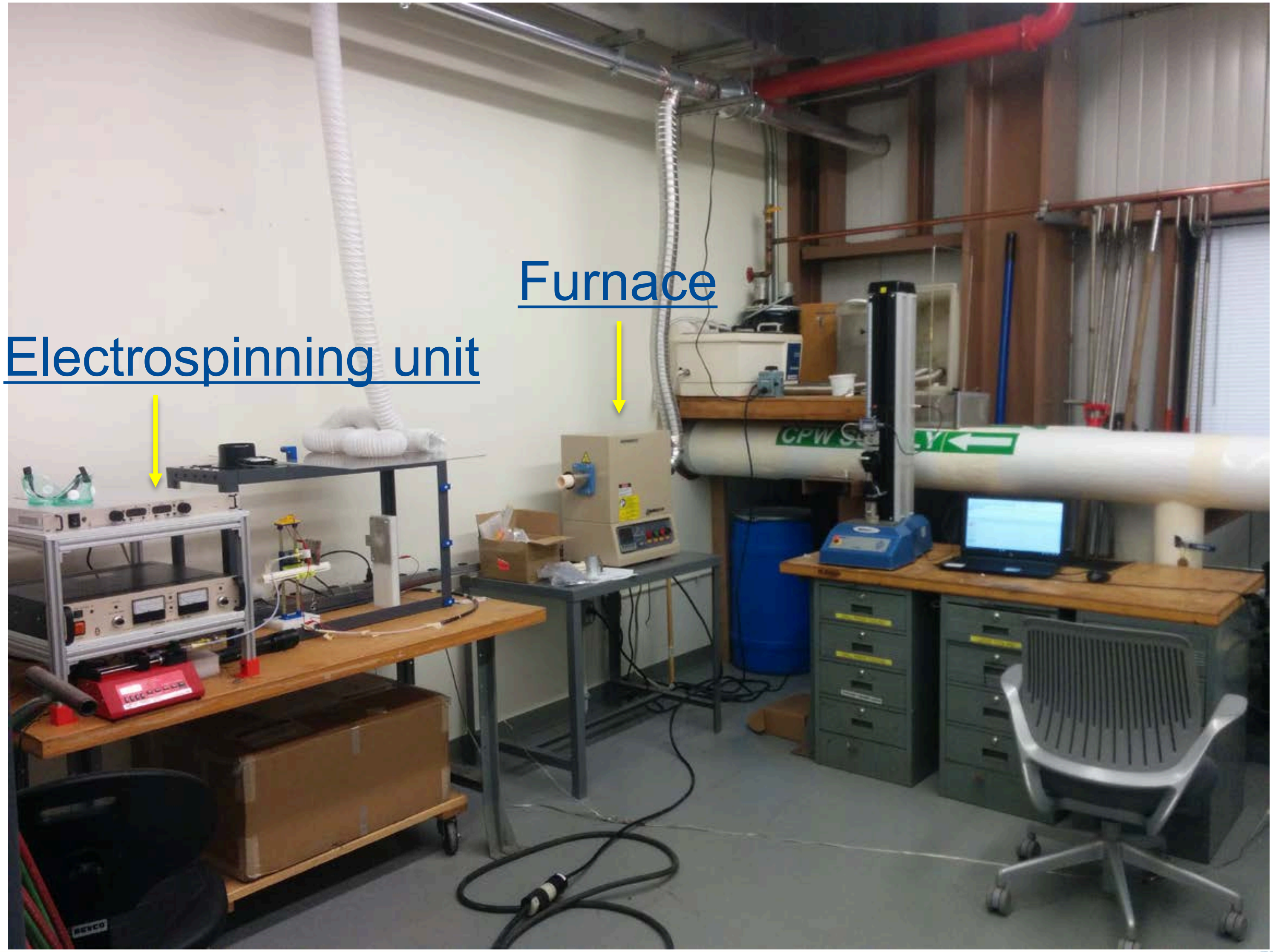
Calcination (Heat treatment)

- Vaporize polymer
- Promotes crystal growth
- Bonding

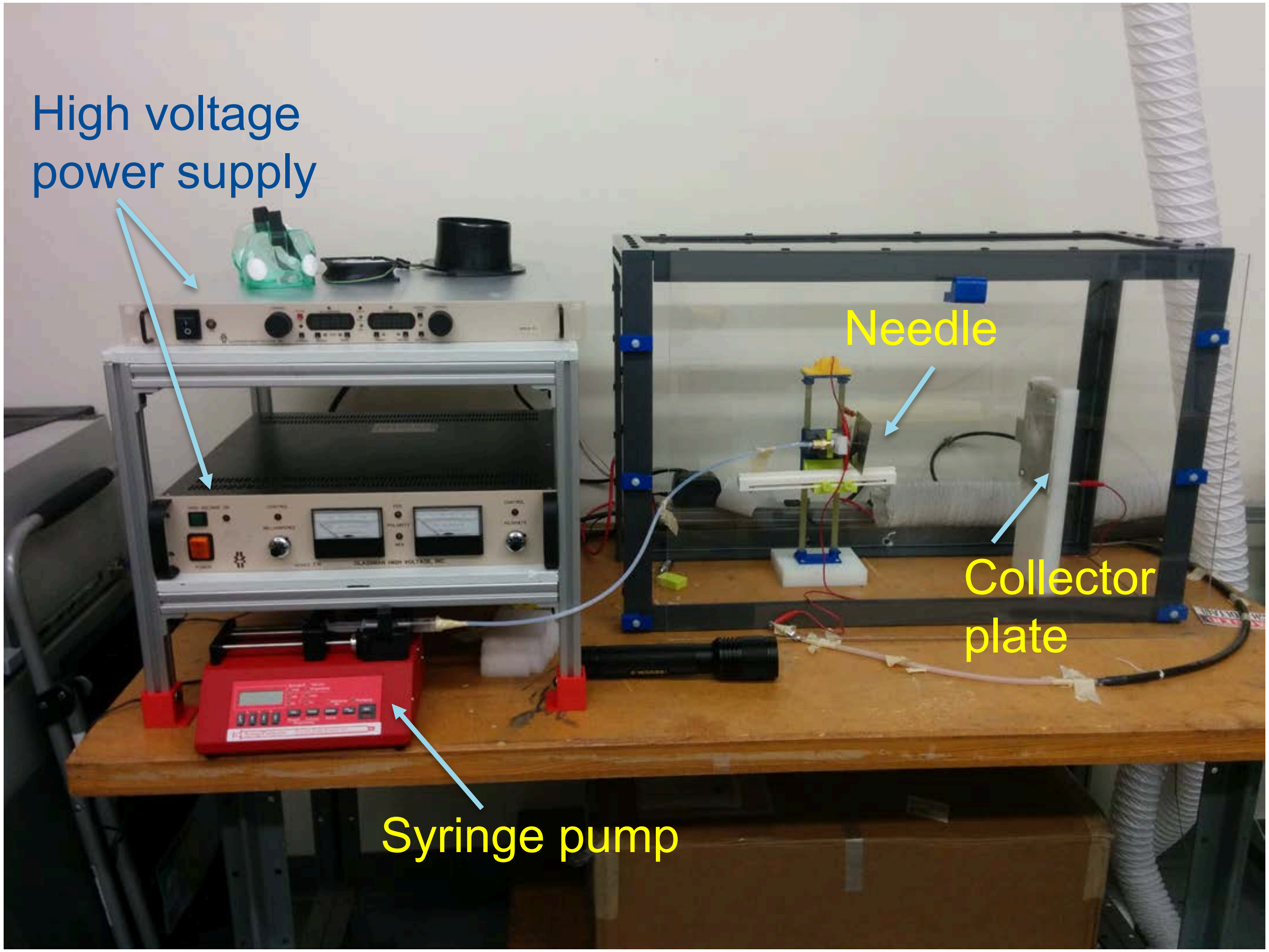


Representative heating profile

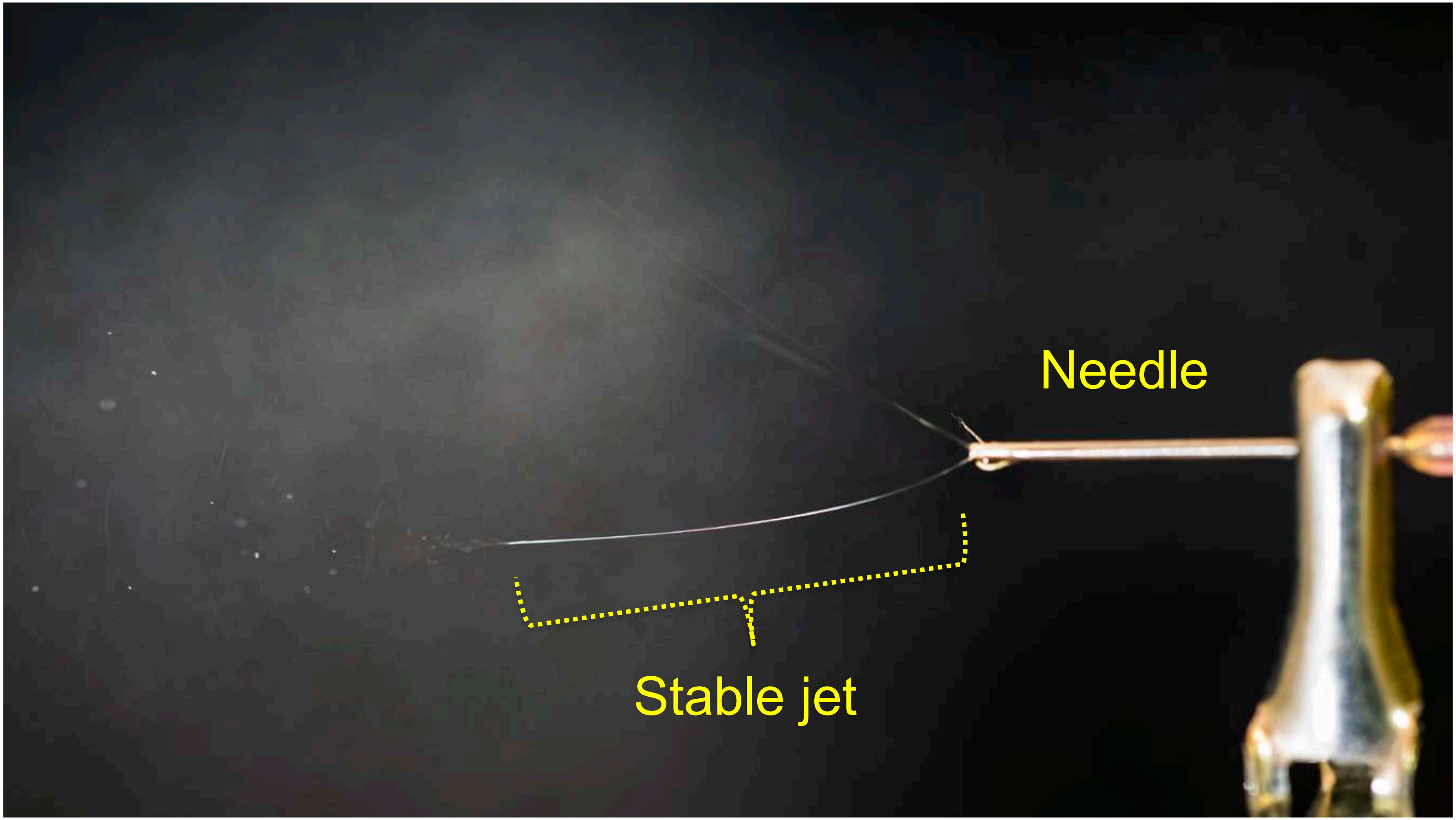
Lab Set-up



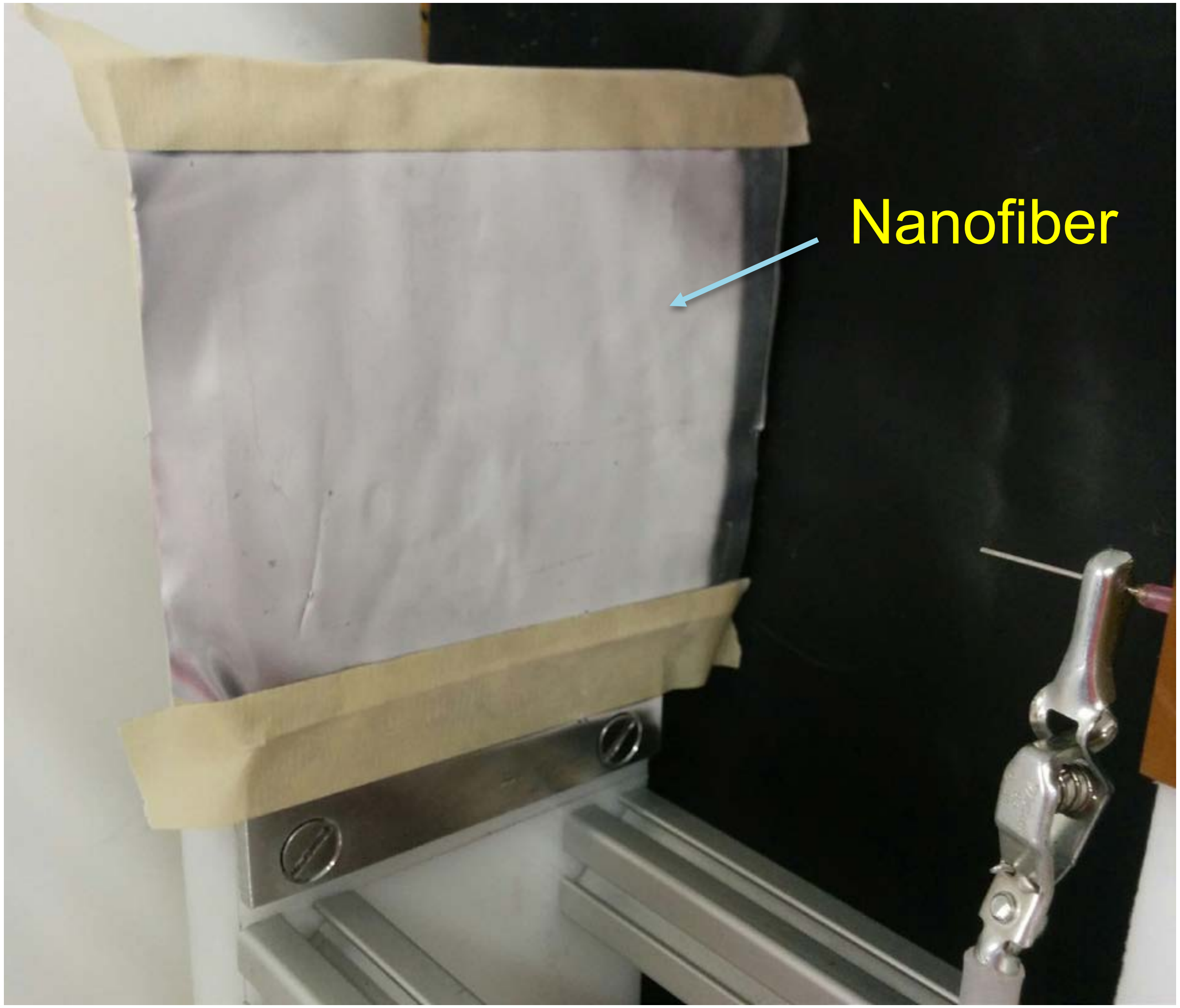
Electrospinning Set-up



Electrospinning Jet



Nanofiber Mat – Random Orientation



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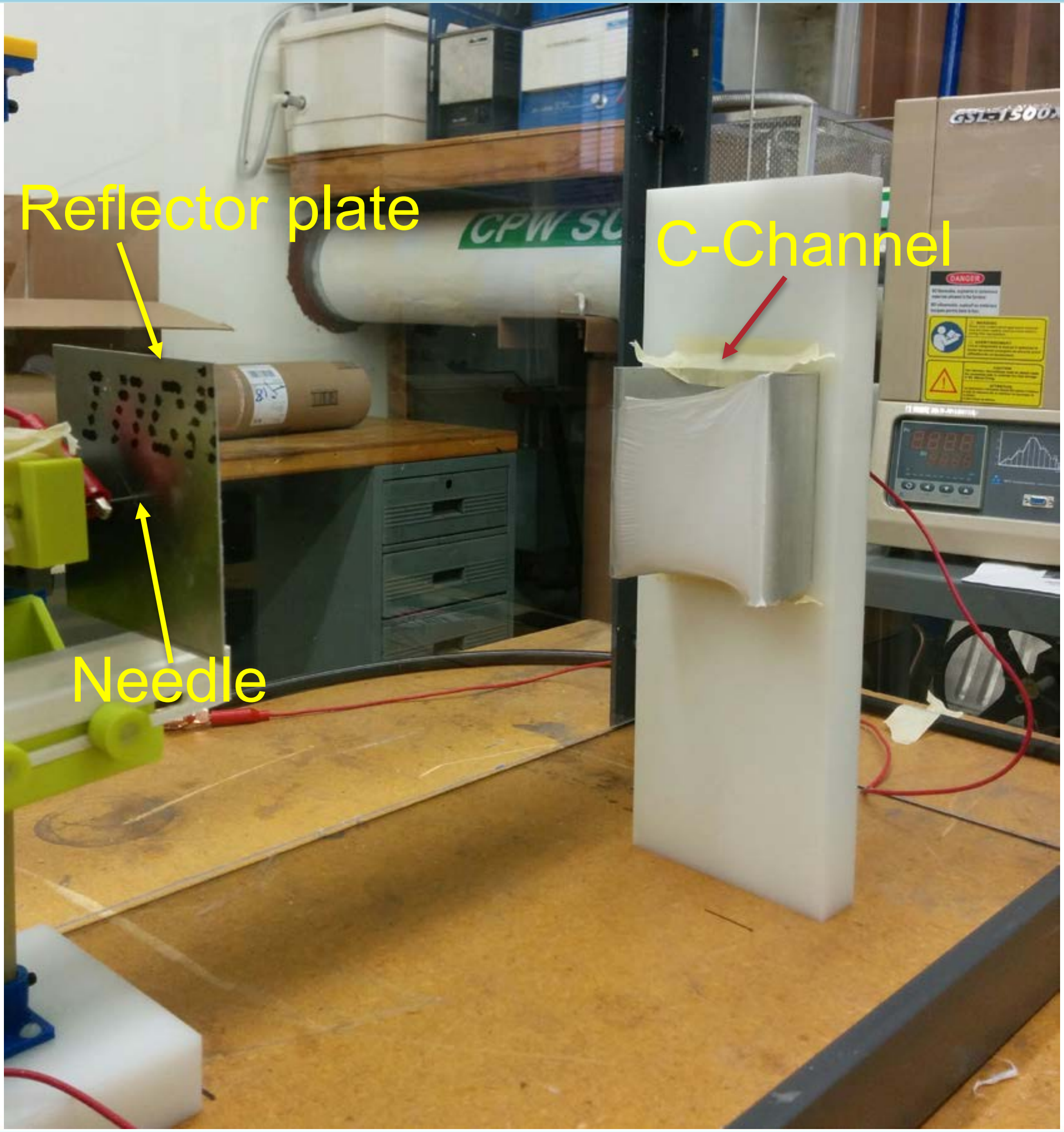
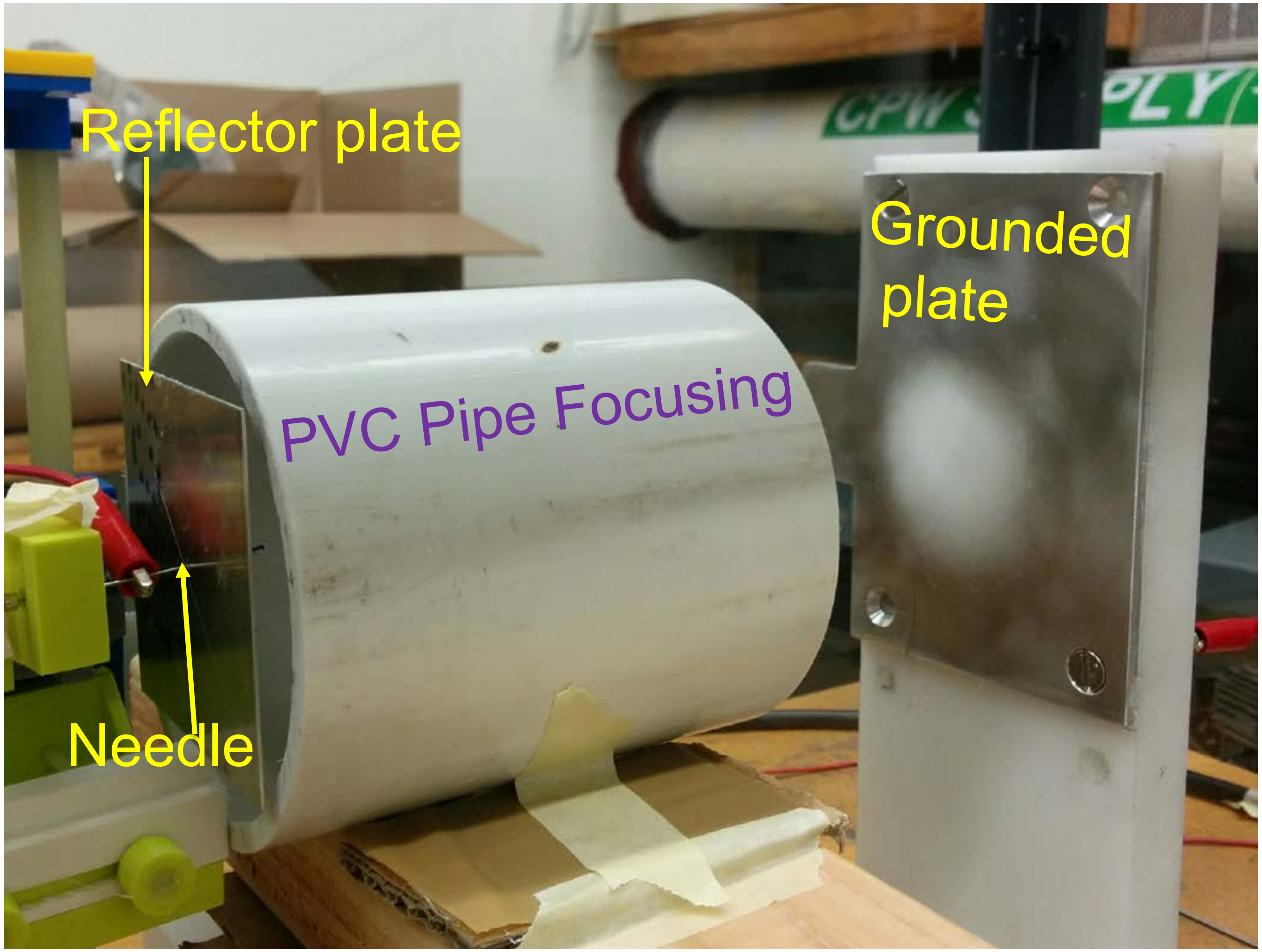
Nanofiber Mat- Aligned



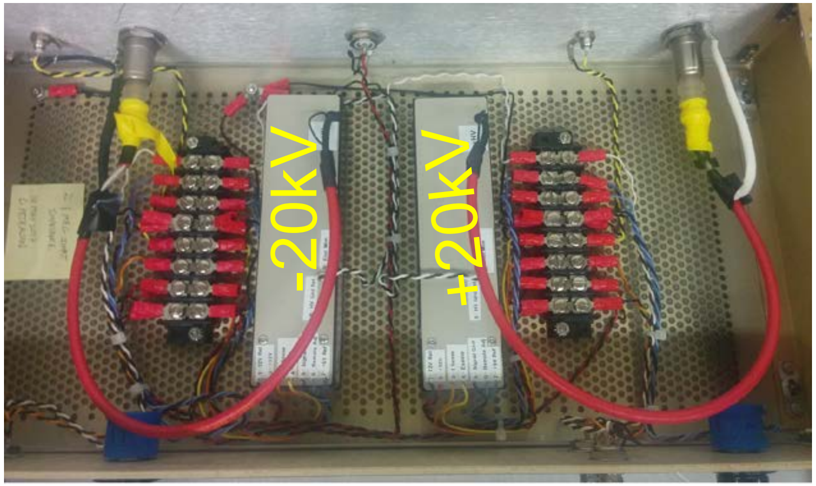
Focused Deposition



Improvised Compact Power Supply

120 Watt, 120VAC in

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 → Aluminum 2,4-pentadionate+Aceton
- Zirconia →Zirconium Carbonate +Acetic Acid
- WO_3 → Ammonium meta-tungstate + D.I. Water
- TiO_2 → Titanium Isopropoxide

Done

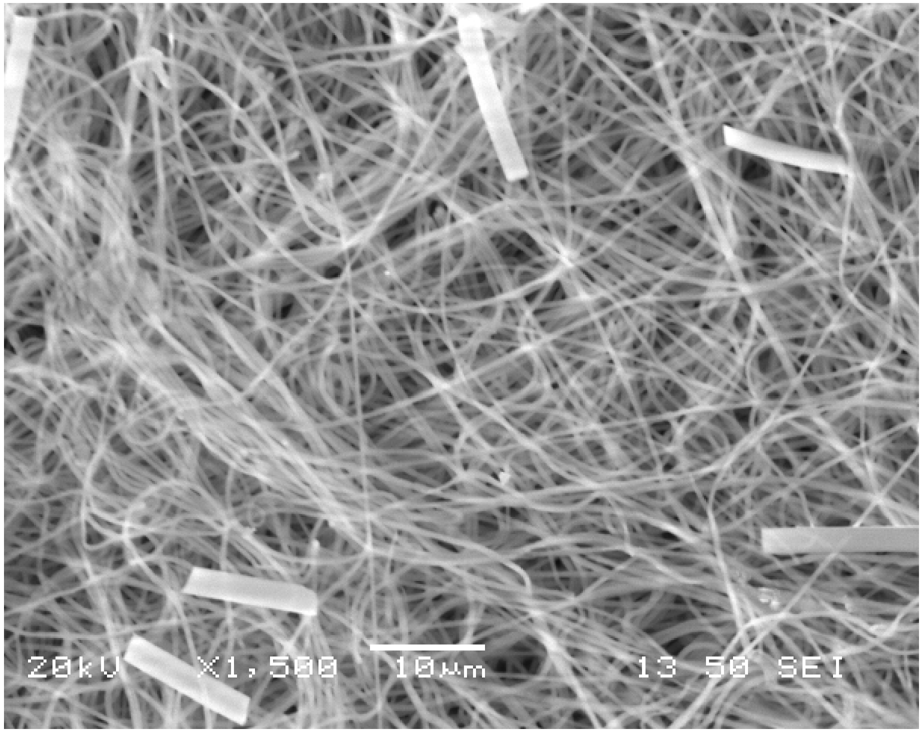
Carbon-nanotube Composite

- CNT-Alumina
- CNT-Zirconium

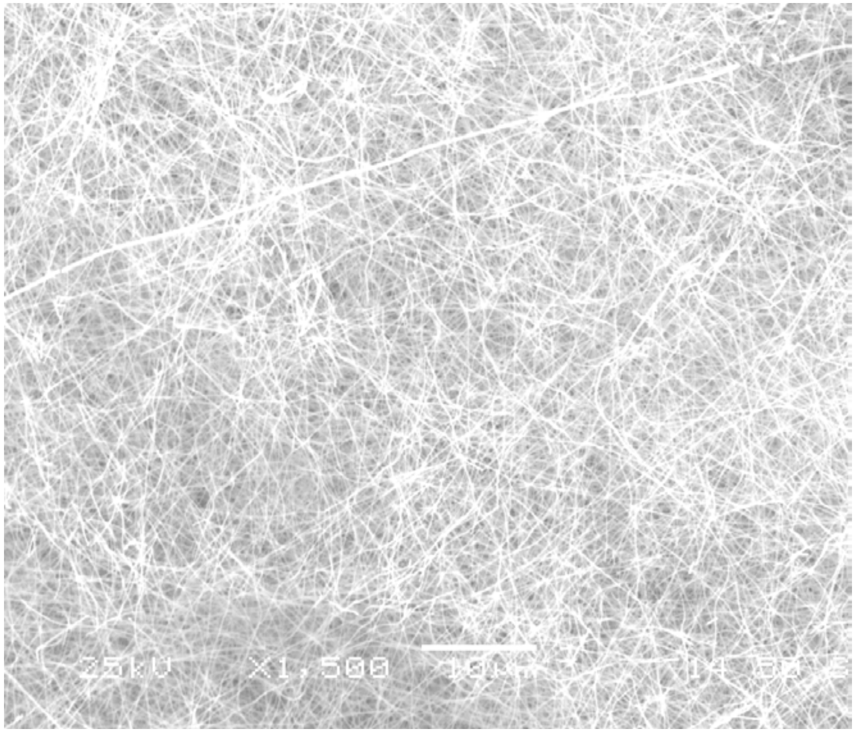
Proposed

Alumina Nanofiber

As spun

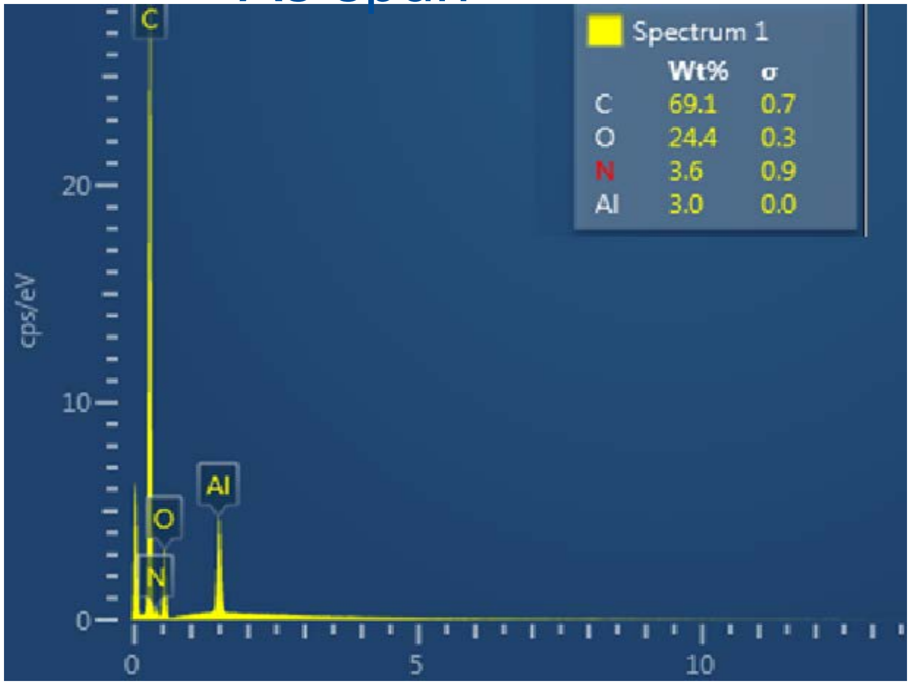


After heat treatment

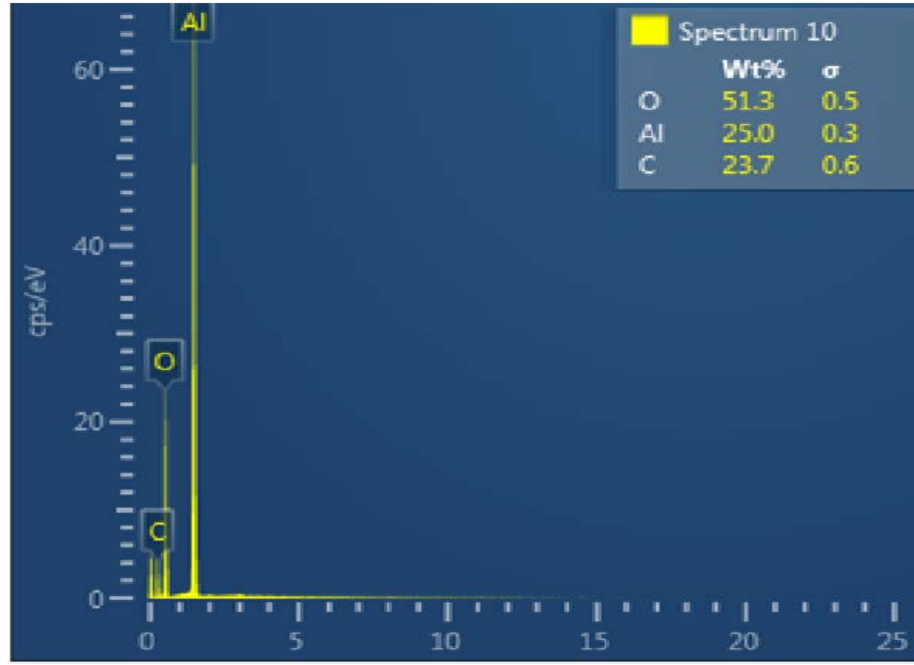


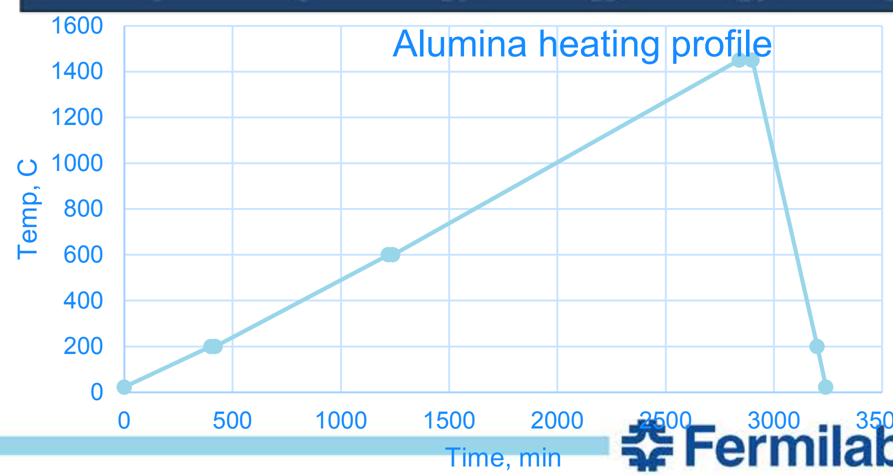
EDS Mapping Alumina Nanofiber

As spun



After heat treatment

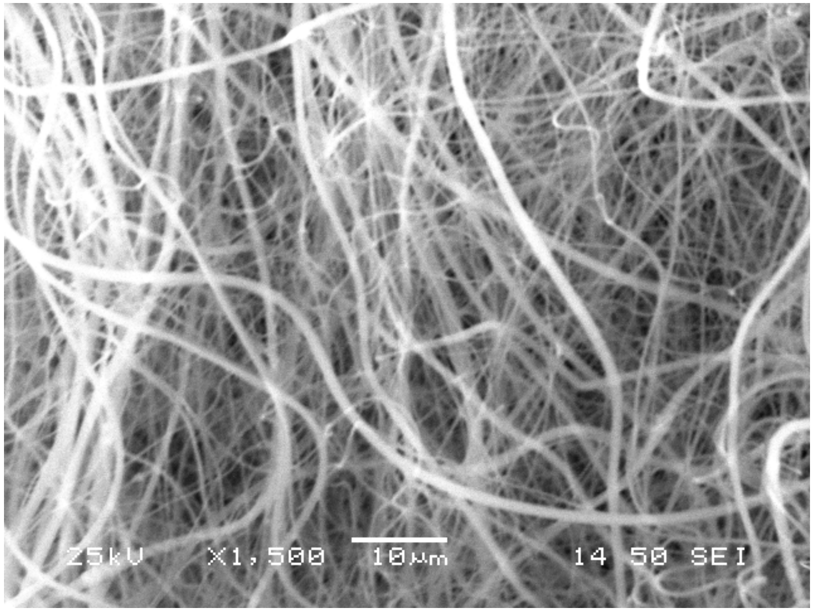




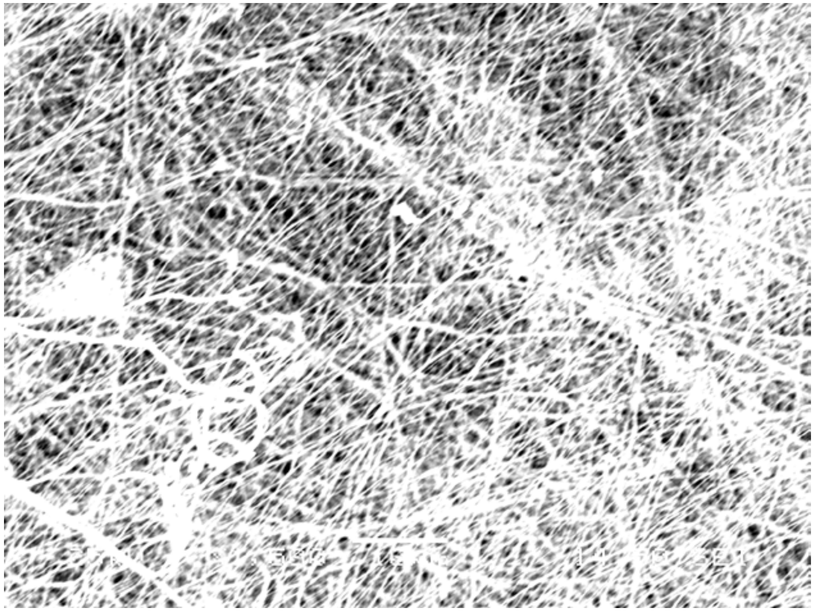
Theoretical Al wt% in Al_2O_3 is 53%
Achieved in actual 25%

Titania(TiO2) Nanofiber

As spun

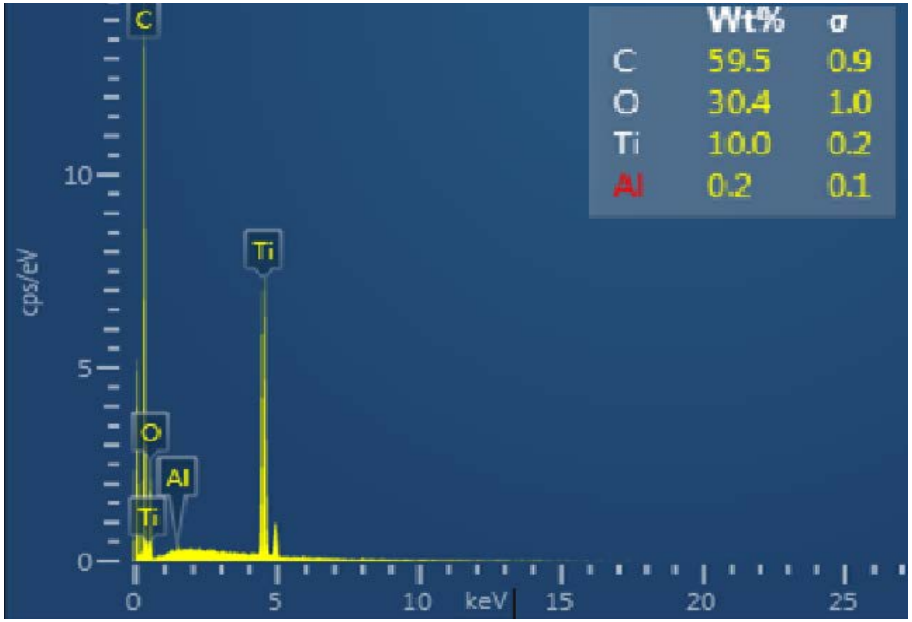


After heat treatment

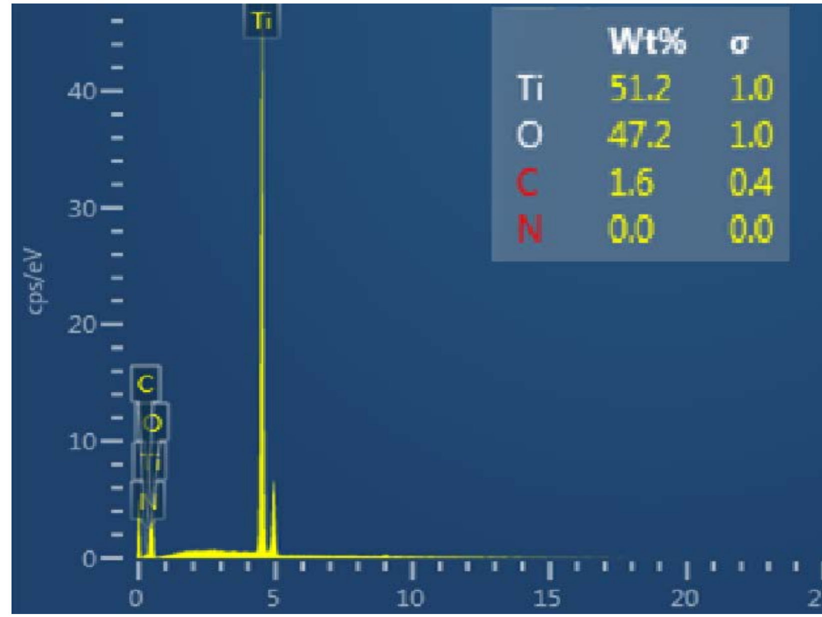


EDS Mapping TiO2

As spun

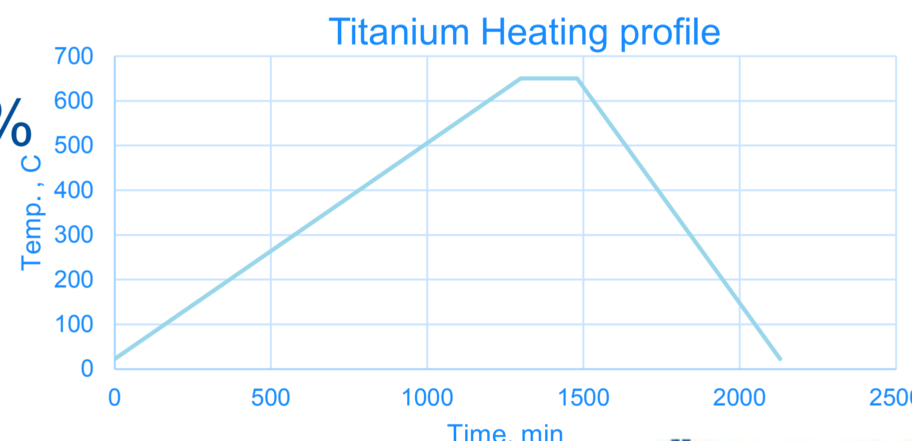


After heat treatment



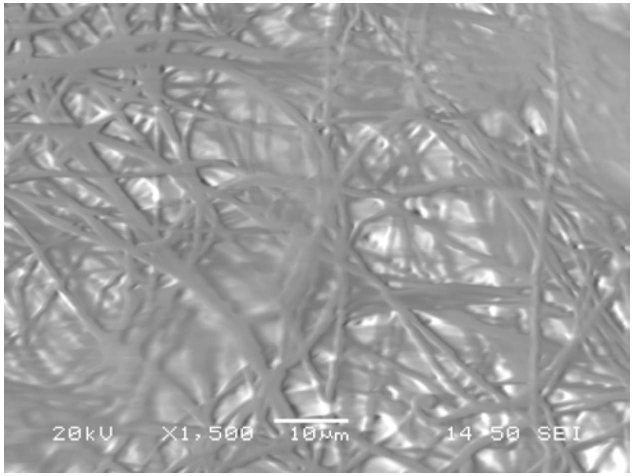
Theoretical Ti wt% in TiO2 is 60%

Achieved in actual 51%

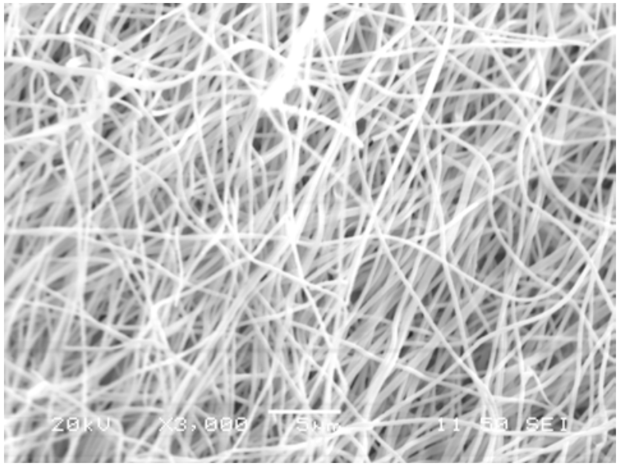


Tungsten Oxide (WO3) Nanofiber

As spun



After heat treatment



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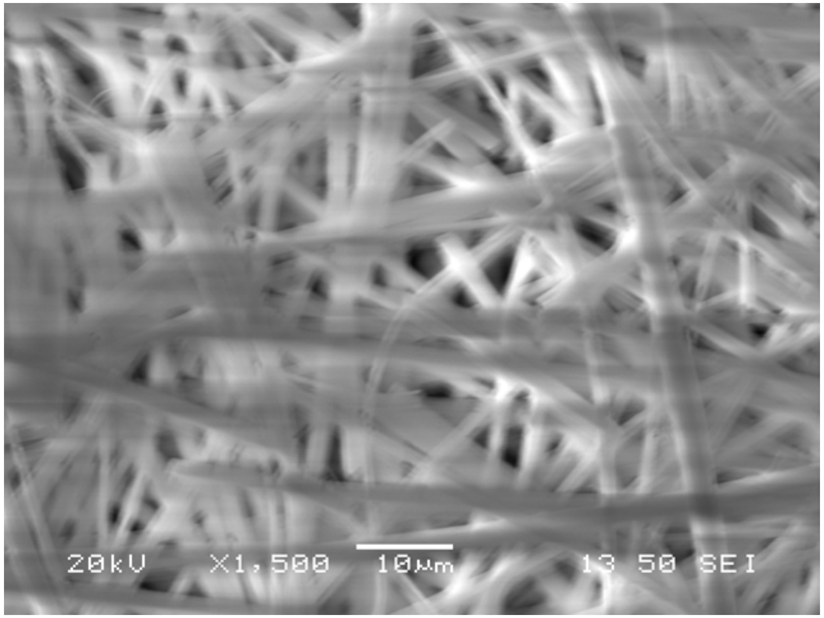
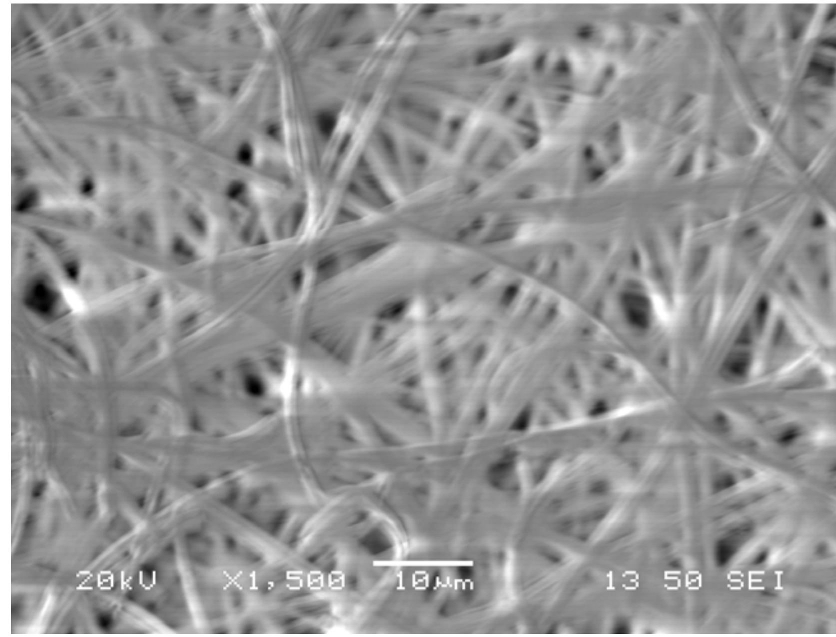
Zirconia Nanofiber

As spun



1.2 gm

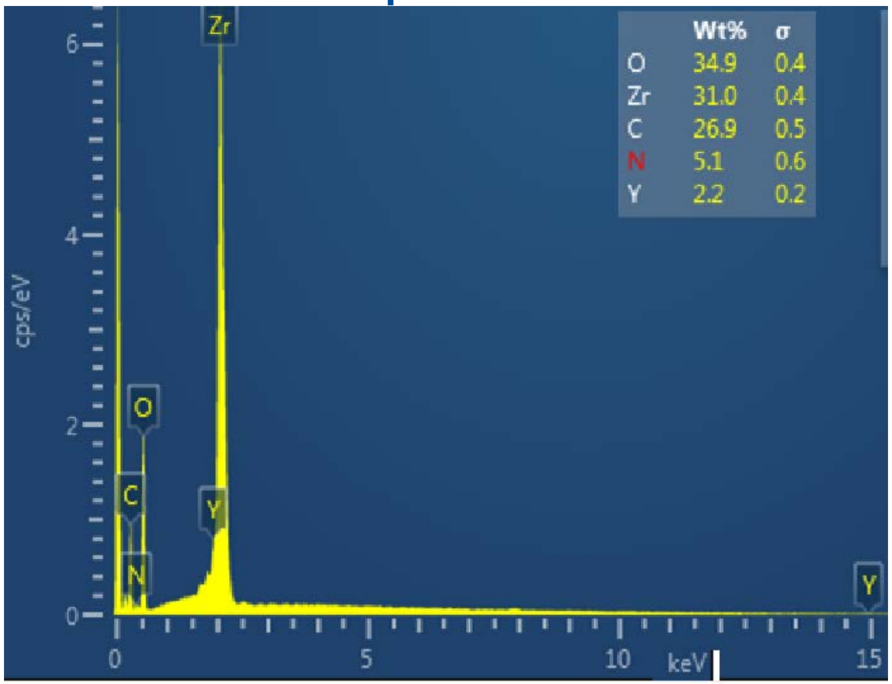
After heat treatment



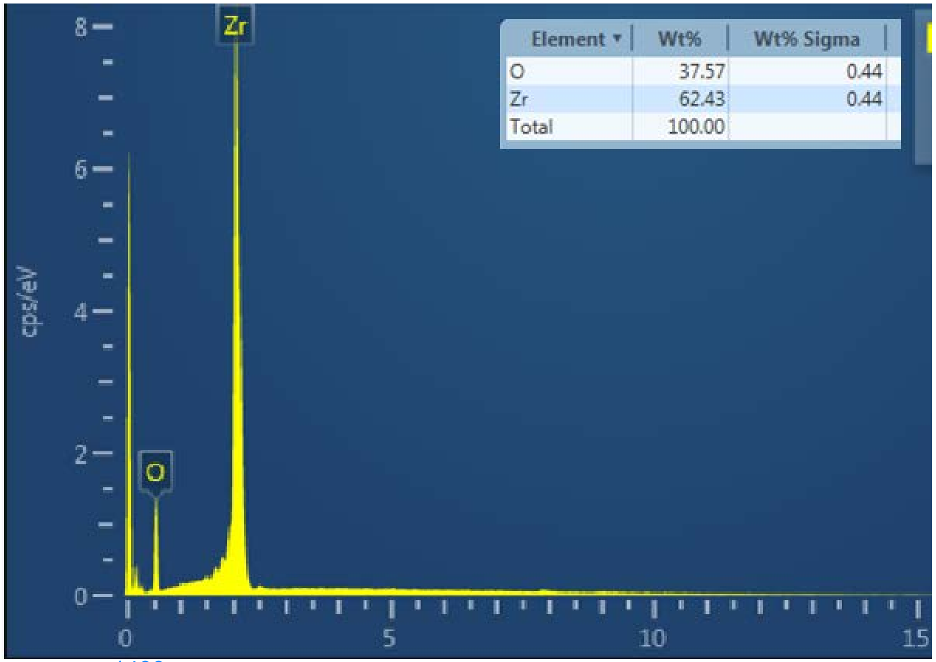
0.5 gm

EDS Mapping- Zirconia Nanofiber

As spun

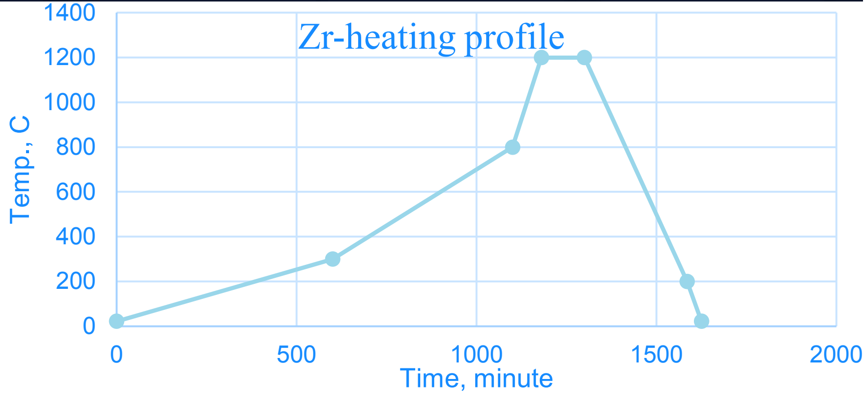


After heat treatment



Theoretical Zr wt% in ZrO_2 is 74%

Achieved in actual 62%



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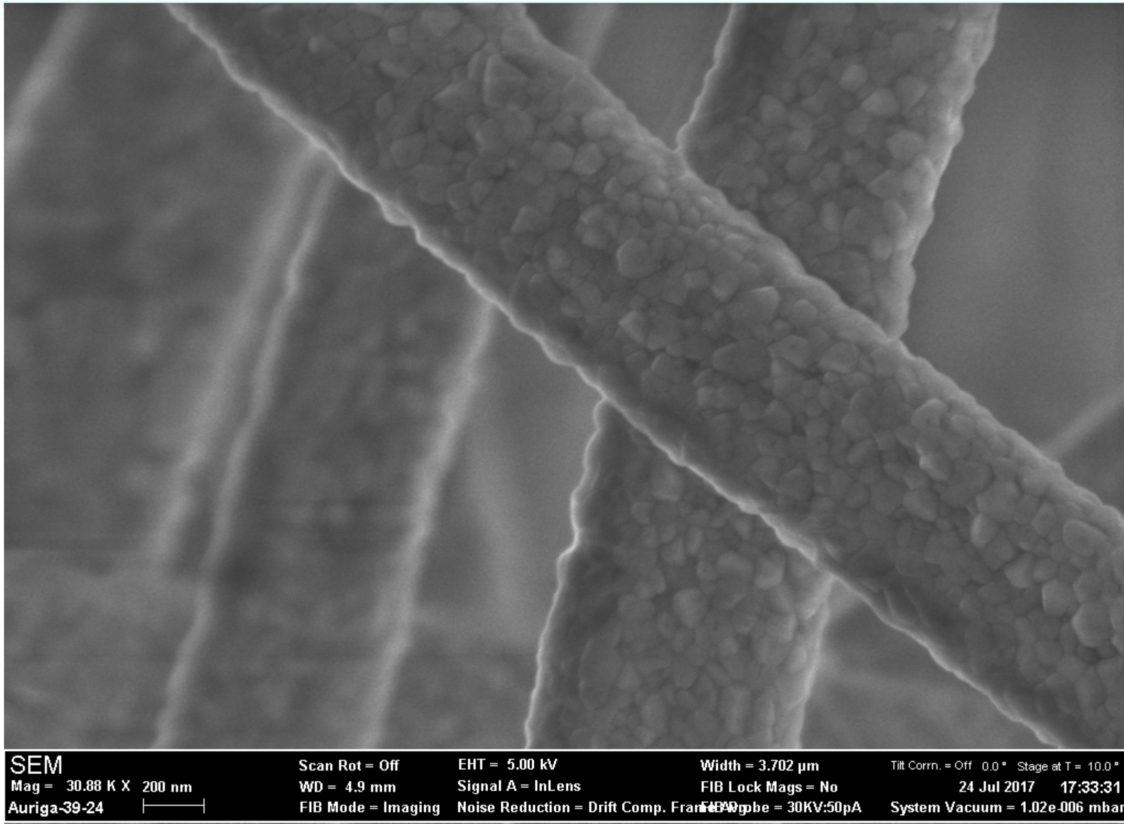
Zirconia Nanofiber- Yttrium doped



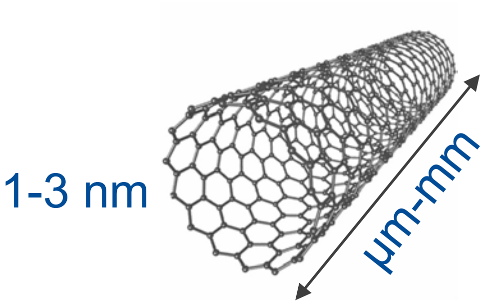
Improve thermal shock resistance by Yttrium doping

Improve radiation resistance

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

 *Sci Rep. 2015; 5: 7746.

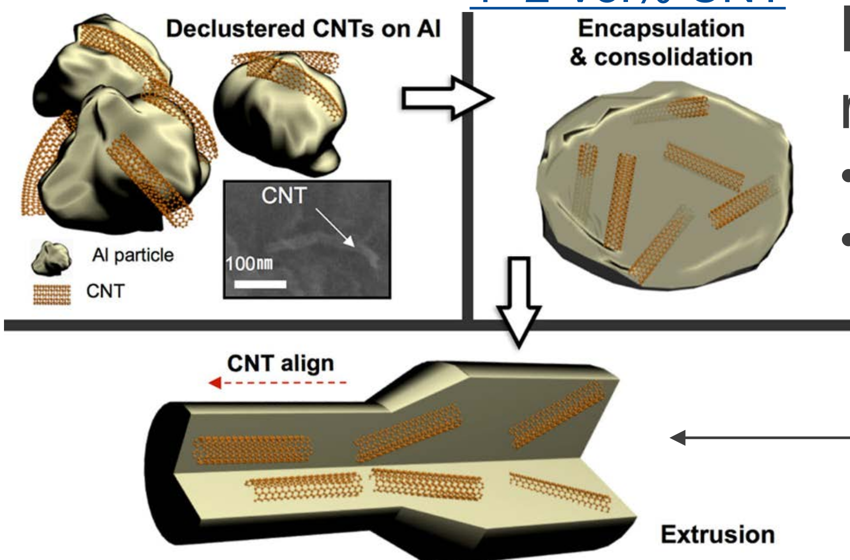
CNT-Ceramics Nanofiber Composites



SWCNT

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

1~2 Vol% CNT



MIT-research*

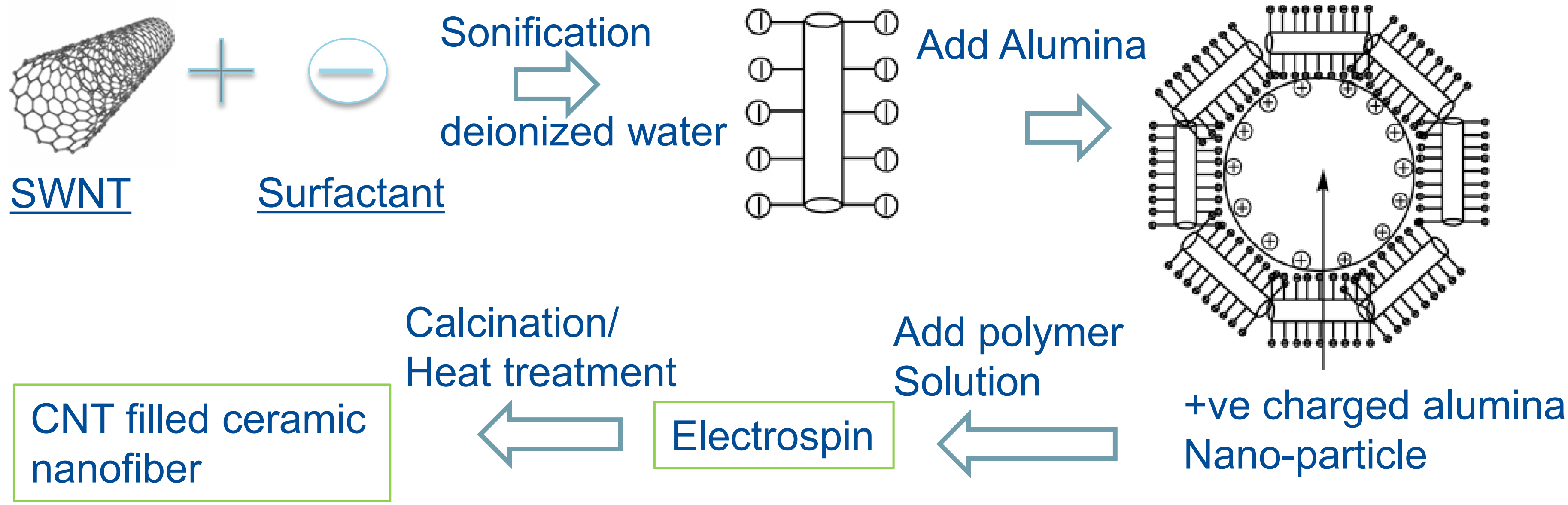
Protects Aluminum metal against radiation damage~70 DPA

- 1-D transport network for He to escape
- Reduce embrittlement by 5-10 times.

Bulk not nanofiber!!

*http://news.mit.edu/2016/carbon-nanotubes-improve-metal-longevity-under-radiation-0302

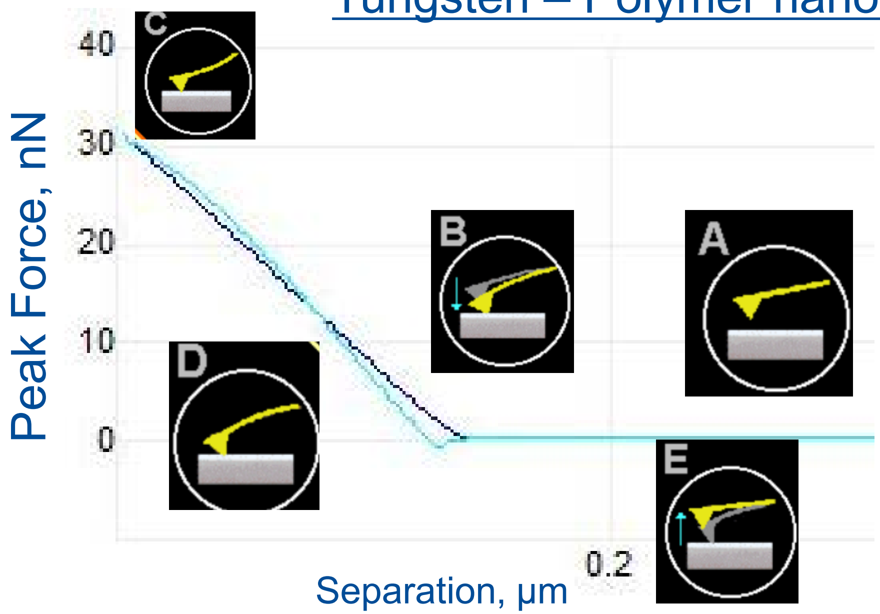
CNT-Zirconia/Alumina Proposed Nanofiber



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

Nano-mechanical mapping – Atomic Force Microscopy

Tungsten – Polymer nanofiber

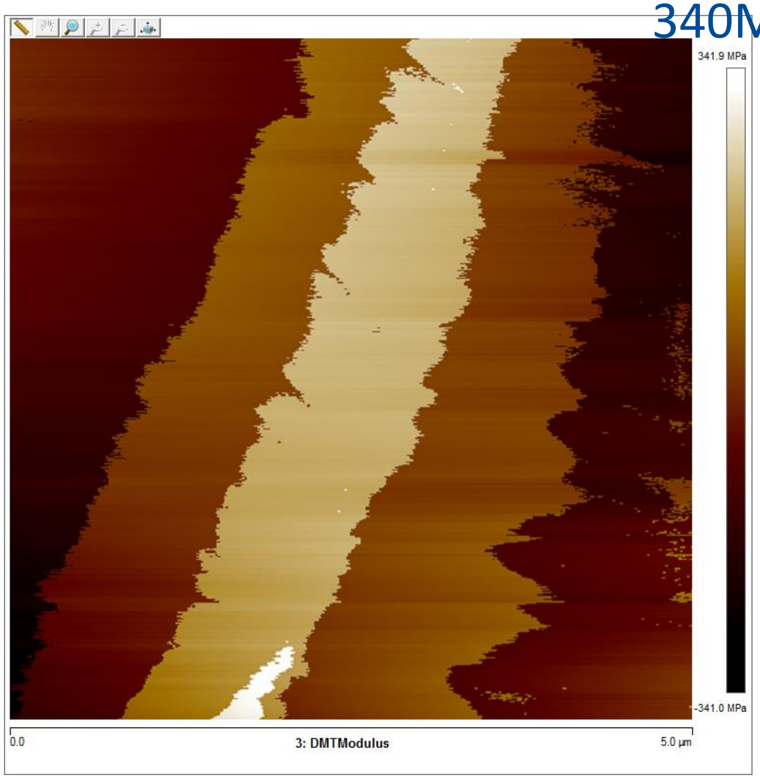


Tip Deflection in contact mode

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

Elastic Modulus map

340MPa



Nanofibers fixed to substrate using double sided tape
Soft substrate compared to nanofiber

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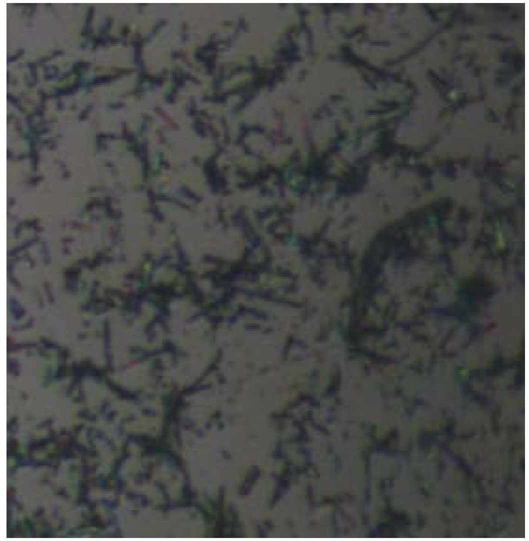
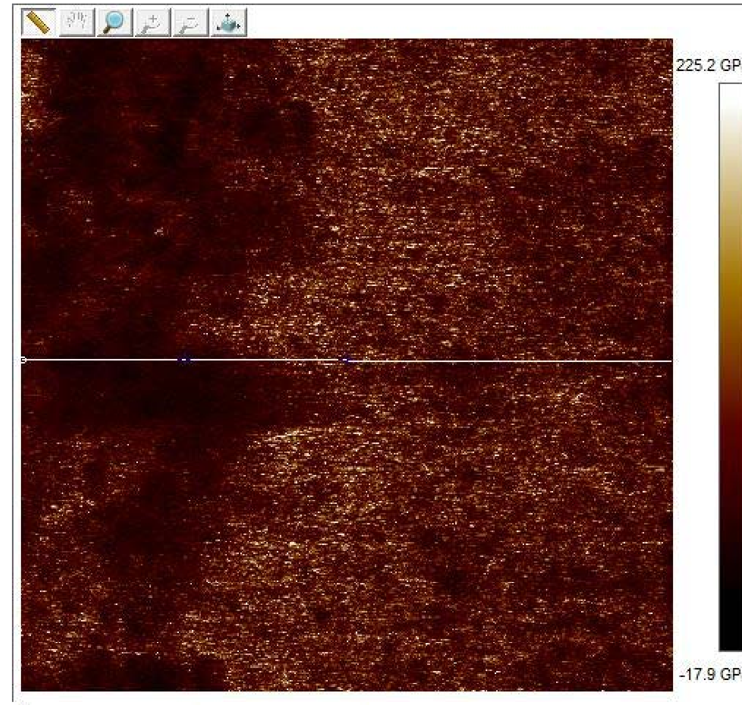
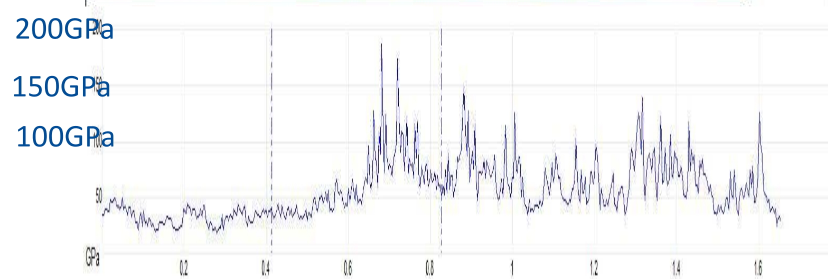
Nano-mechanical mapping – Atomic Force Microscopy



Elastic Modulus map

225GPa





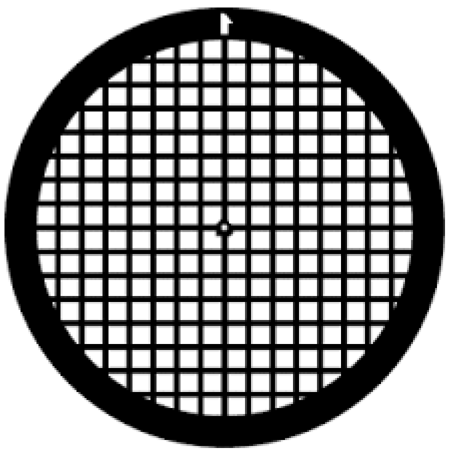
Nanofiber solution casted on harder smooth mica substrate

Average Elastic modulus ~ 100GPa

Mechanical characterization

<u>Fracture strength single nanofiber</u>

1. 3 point Bending test on TEM Grid



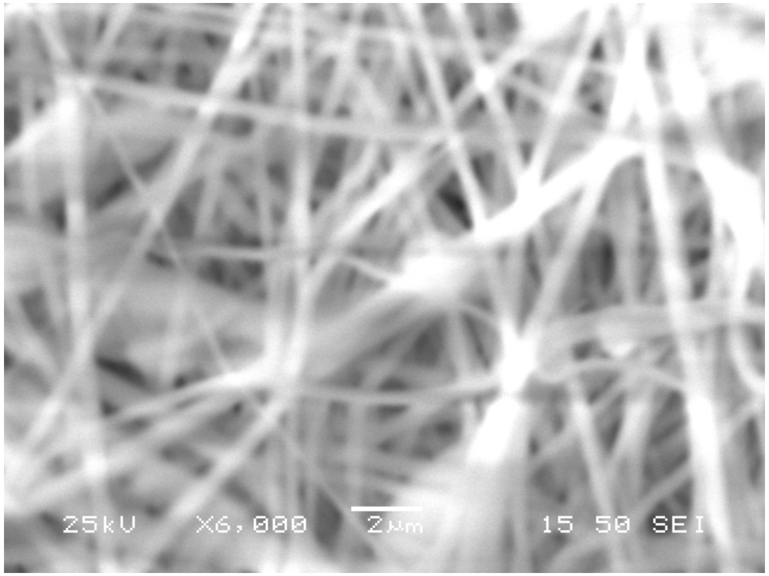
<u>Set up</u>

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

2. Nano-indentation using AFM tip

<u>Macro testing electrospun nanofiber mat</u>

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

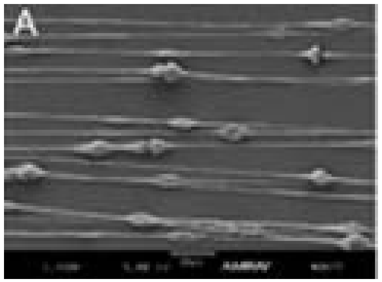
- Expose nanofiber mat to HiRadMat test
  - Single fiber radiation damage study
- Improve ductility of ceramic nanofiber
  - Fabricate ceramics-CNT composite.
  - Heat treatment profile
- Physical properties before and after radiation.
  - Damage modeling

Thank You for Your Attention!!

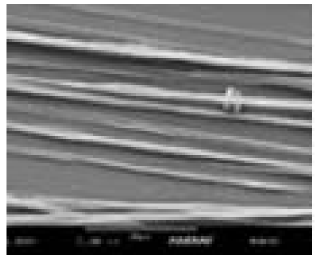
Parameters

- Molecular weight of polymer
- Solution properties (Viscosity, conductivity, surface tension)
- Concentration, electrical potential, flow rate
- Distance between needle and collector
- Needle gage
- Ambient temperature, humidity, air flow

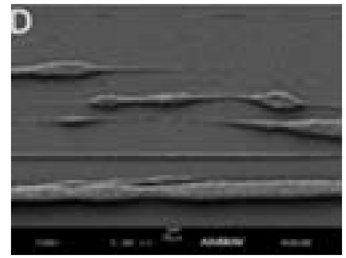
Problems



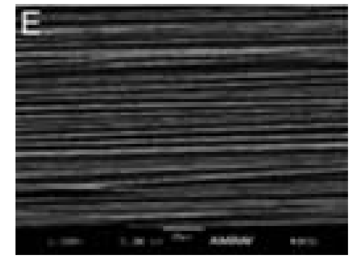
Beading



Ribbons



Porous globe

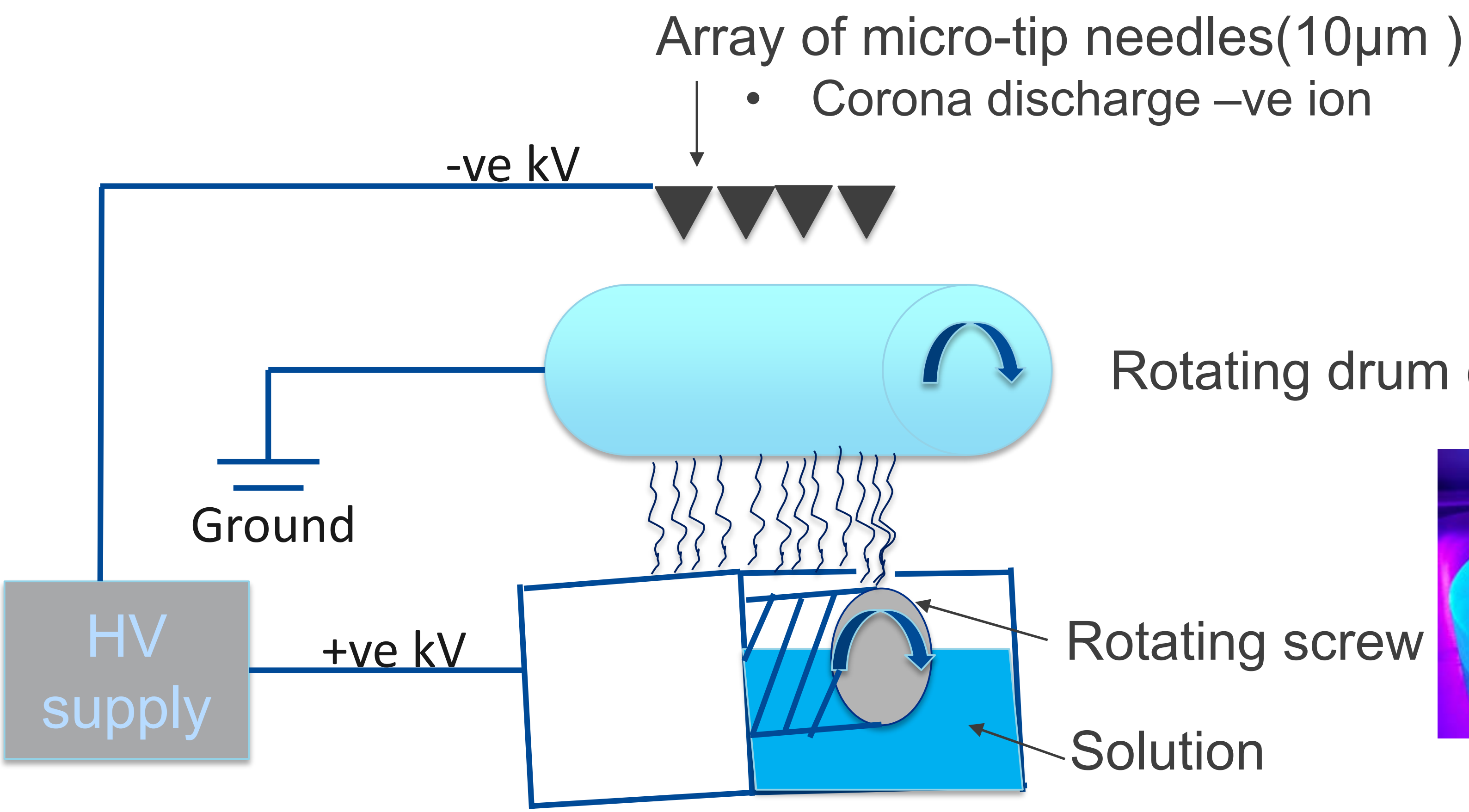


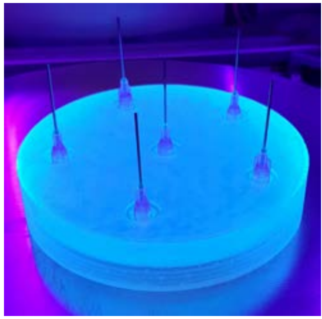
Cylindrical fiber

Troubleshoot

- Increase flow rate
- Increase polymer concentration or solvent with high evaporation
- Salt additives (Surfactant)
- Adjust distance between needle and collector
- Porosity→evaporative characteristic of solvent

Mass Production-Needleless (In progress)





- Charged nanofiber prevents thicker mat formation
- Opposite charged ion neutralize nanofiber and promote thicker mat