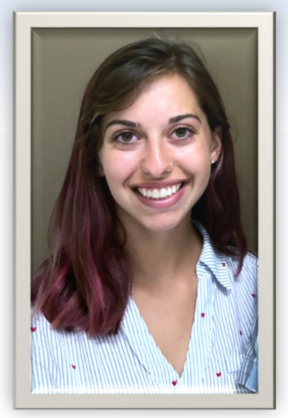
Electrospinning Nanofibrous Ceramic Filter Media for Nuclear Ventilation Systems



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ISNATT INTERNATIONAL SOCIETY FOR NUCLEAR AIR TREATMENT TECHNOLOGY
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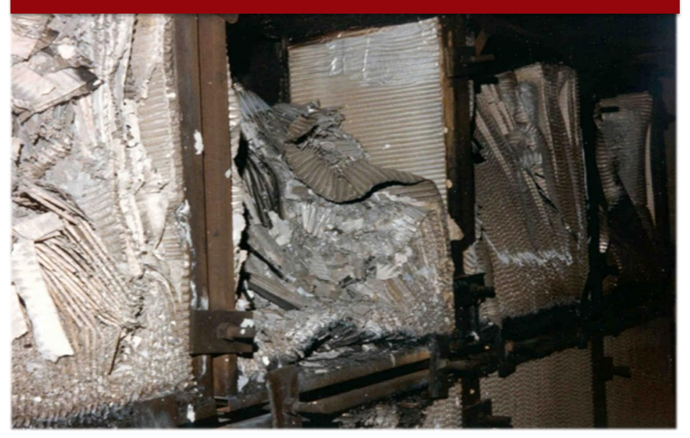
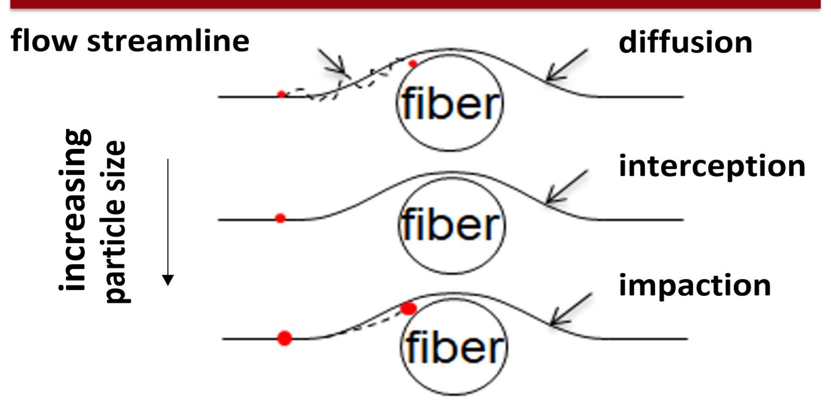
Background

- The Defense Nuclear Facilities Safety Board (DNFSB) highlighted the need for High Efficiency Particulate Air (HEPA) filter R&D
- Ceramic filters can likely eliminate reliance on expensive safety support systems to mitigate against loss-of-filtration during and after an accidental fire scenario
- Filters require large surface area, porosity, and low pressure drop

Water damage to filters following a fire



Filtration mechanisms

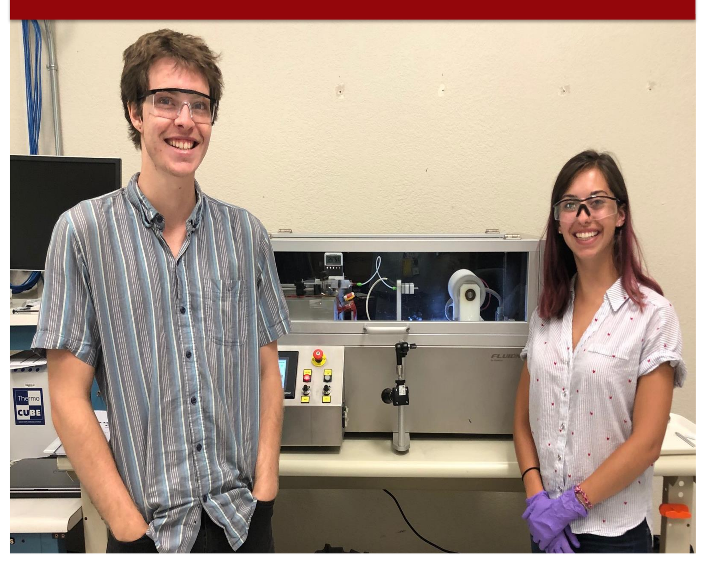


Electrospinning Filter Media

- Precursor solutions are made with polymers, solvents, and salts
- **Ga_2O_3** and **Al_2O_3-4YSZ composite** precursor solutions are characterized and electrospun to generate nanofiber filter media
- An electric field is created between the emitter and collector causing fiber ejection, fiber stretching, fiber whipping, evaporation of solvent, and deposition of pre-ceramic nanofibers onto the collector

Electrospinner emitter (left) and collector (right)

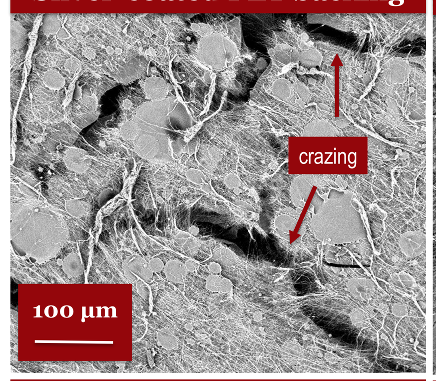
Electrospinning apparatus



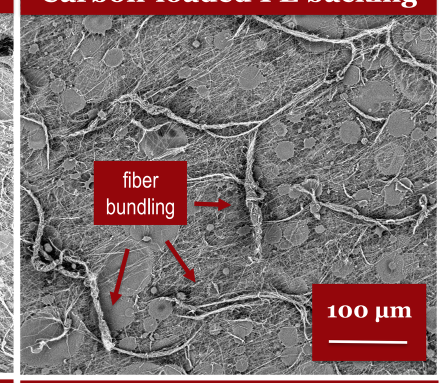
- Viscosity, surface tension compete with conductivity, electric field strength to determine degree of fiber stretching and fiber diameters
- Solution characterization is performed using a rheometer, conductivity probe, and surface tensiometer
- Current efforts include exploration of coaxial solvent sheath flow to minimize gelling, reduce defects in bulk fiber mat, and use equipment for unattended runs

Electrospinning Optimization

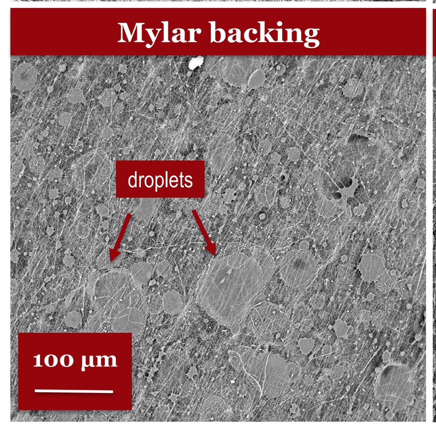
Silver-coated PET backing



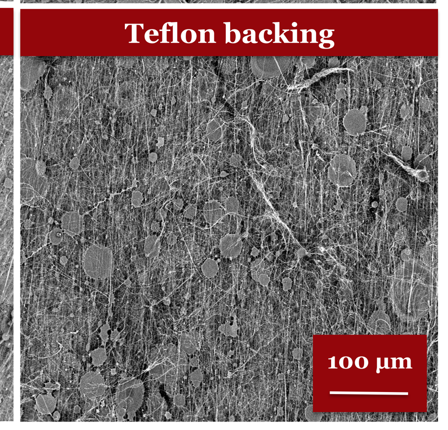
Carbon-loaded PE backing



Mylar backing

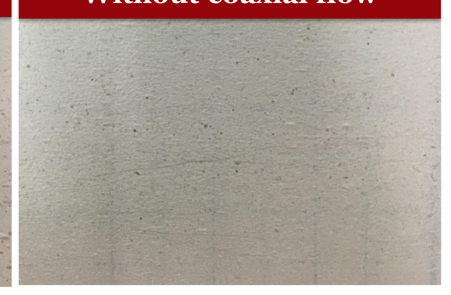


Teflon backing



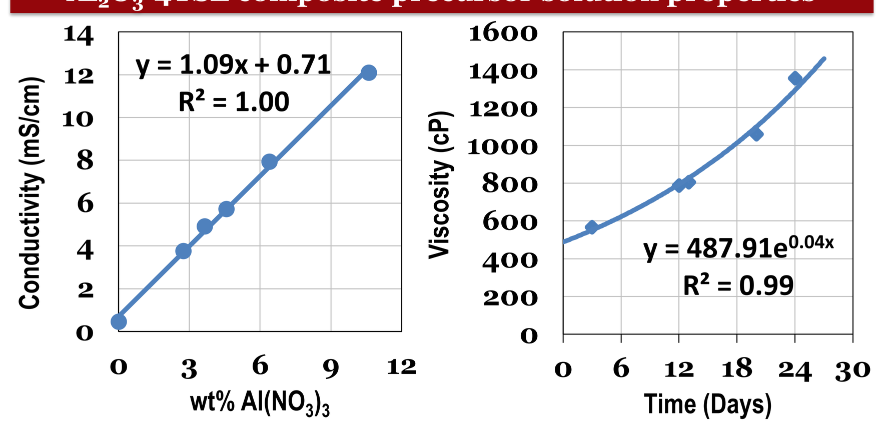
With coaxial flow

Without coaxial flow



- Fiber mats show three types of microstructural defects; droplets, bundling, and crazing
- Coaxial spinning currently introduces more droplets during prolonged runs, requiring further optimization of the sheath solution
- Solution viscosity exponentially increases with time, conductivity is linear with precursor concentration, and surface tension is constant

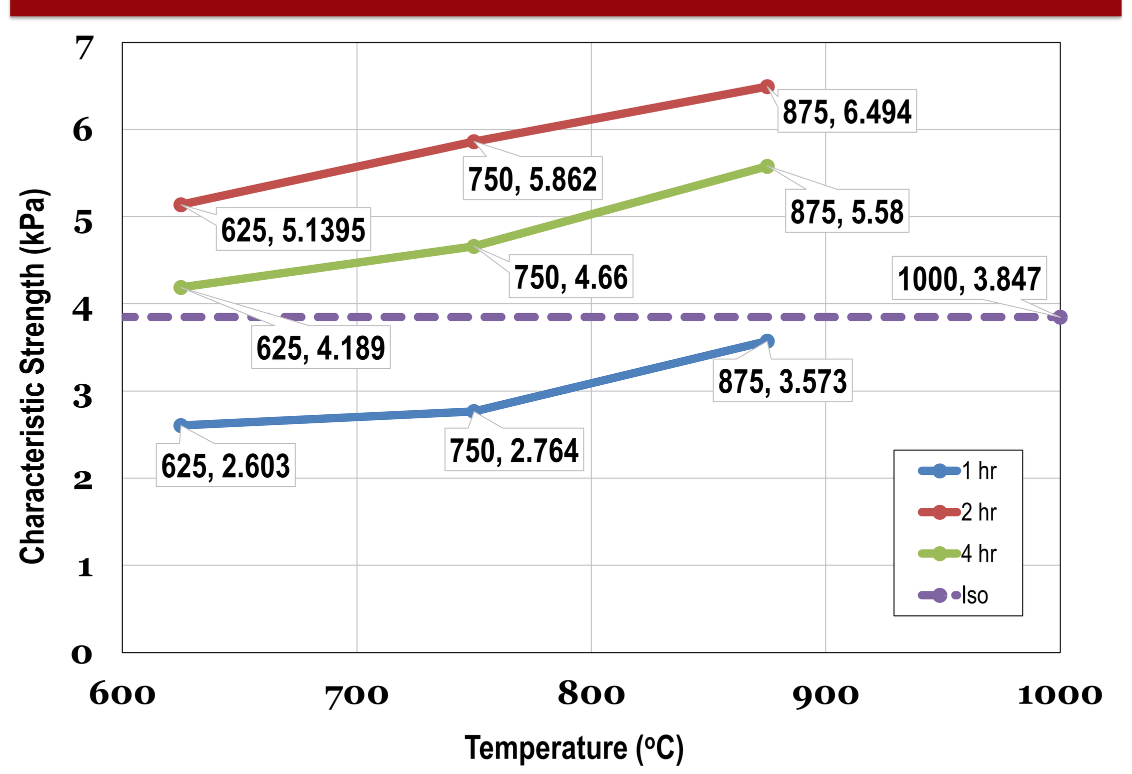
Al_2O_3-4YSZ composite precursor solution properties



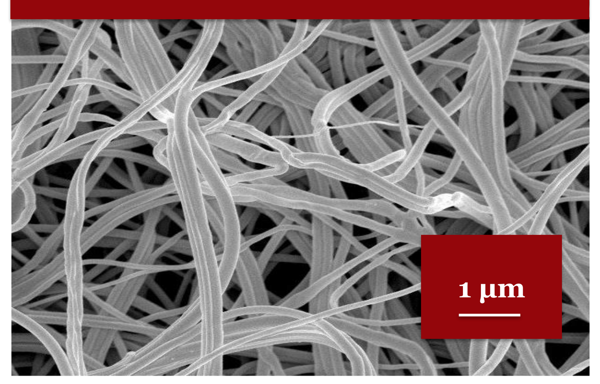
Thermal Conversion to Ceramic

- Pre-ceramic tubes undergo heat treatment to pyrolyze polymer binder and transform salt precursors to ceramic nanofibers
- Fibers undergo severe (~ 60 to 70%) volume shrinkage upon heating
- Strength of the fiber mat can be dependent on heating rate, temperature, time, and porosity (correlates to number of fibers resisting a load)
- Two-step sintering strengthens the fiber mat compared to isothermal sintering (see LLNL-TR-778420, *Optimizing Electrospun Ceramic Nanofiber Strength Through Two-Step Sintering*)

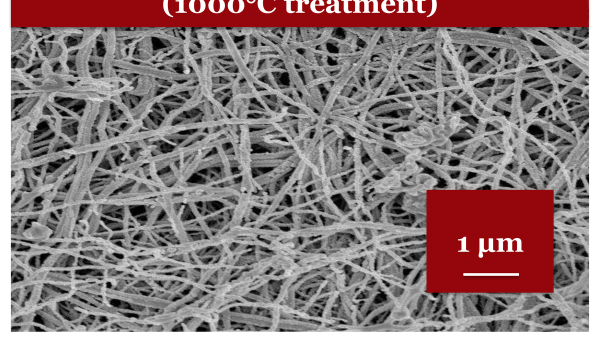
Fiber mat strength for two-step sintering vs. isothermal sintering



Pre-ceramic nanofibers



Ceramic nanofibers (1000°C treatment)



Tubular Media Fabrication

- Nanofibrous sheets are formed into tubular structures by hand using a Peel-Roll-Seal process
- Optimization of substrate used during electrospinning and vacuum oven is necessary for peeling – best substrates: mylar and carbon-loaded PE backing
- This stage is key to assess when converting from lab- to pilot-scale production because it will likely require automation; automated equipment is now at LLNL (see LLNL-TR-779817, *Automation of Mini-Tube Production of Electrospun Nanofibers*)

Peeling of nanofiber mat

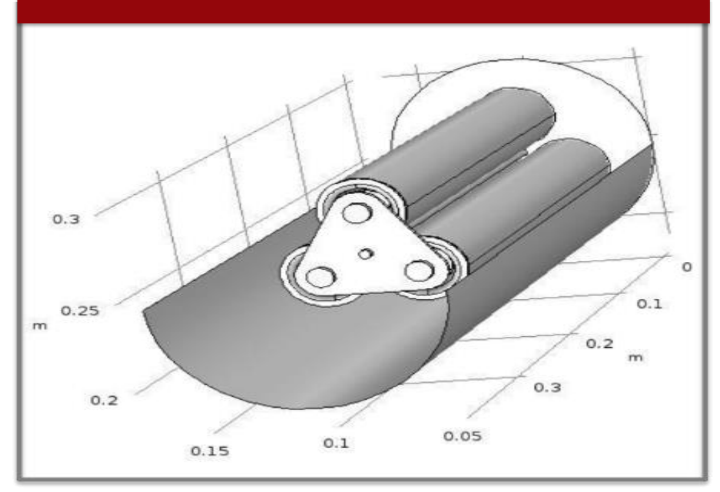
Pre-ceramic MTC filter media

Future Work

- Electrospun media is time consuming to fabricate, these processing improvements will facilitate new methods to scale up production.
- Optimizing formulations and heat treatments for maximum fiber mat strength and fracture toughness improves structural properties
- Integration of ceramic nanofiber media into LLNL filter designs will be needed
- Alternative approaches to make larger area filter media are being investigated which benefit from the recent process improvements

Filter prototype design

Filter tube with media overwrap



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