NSRD-20 Background and Status Update

Novel Mini-Tubular Ceramic (MTC) HEPA Filtration Media for Nuclear Facility Ventilation Systems

Dr. James Kelly et al.
Ceramics & Polymers Engineering
Materials Engineering Division

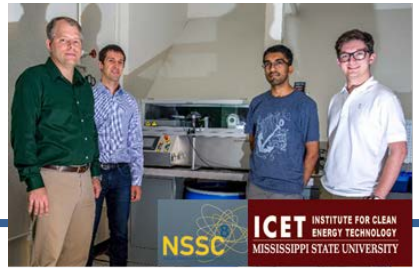
EFCOG Nuclear & Facility Safety Workshop
February 2020

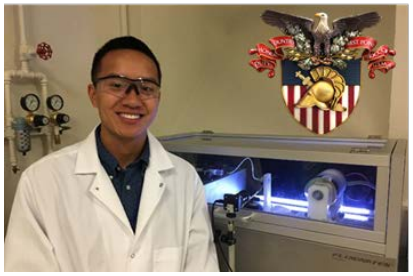
LLNL-PRES-805409

This work was performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under contract DE-AC52-07NA27344. Lawrence Livermore National Security, LLC

Lawrence Livermore National Laboratory

Acknowledgements

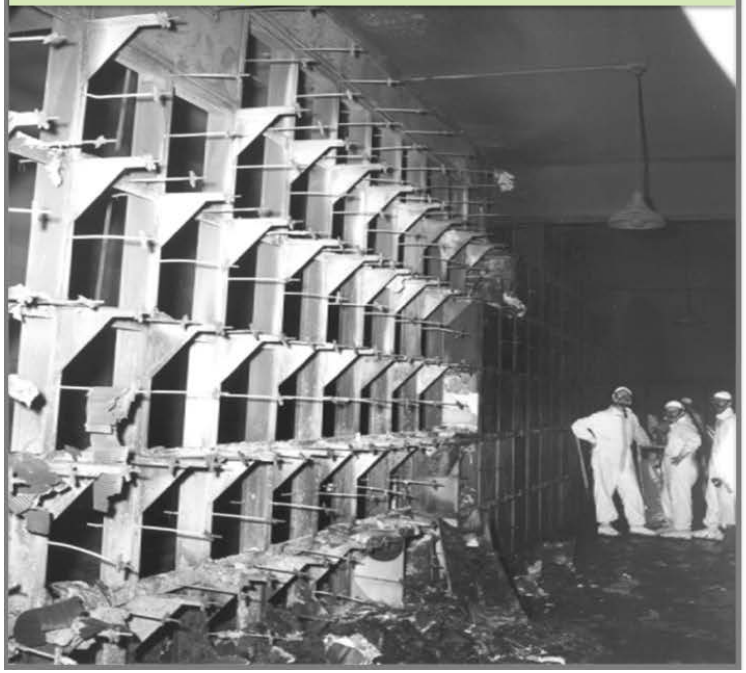




- LLNL
  - Mark Mitchell
  - Howard Wong
  - Danny Laycak
  - Dr. Jeff Haslam
  - Dr. Lauren Finkenauer
  - Dr. Patrick Campbell
  - Dr. Maira Ceron
  - Uday Mehta
  - Alyssa Troksa
  - Hannah Eshelman
  - Erik Brown
- Cal Poly (Profs. Mayer & Harding)
  - Josh Clemons
  - Nick Brodine
  - Brian Deemer
  - Delaney Fitzsimmons
  - Michael Ross
- MSU ICET (Prof. Cho)
  - Brandon Bogle
  - Col. Ivan Beckman
- MARA (Barry Goldman)
  - Jack Bui, West Point
  - Jamie Maguire, Naval Academy
  - Wesley Russell, Coast Guard
- NSSC (Prof. Chintalapalle)
  - Nanthakishore Makeswaran
- MSIPP
  - Christina Santa-Lucia
  - Trent Malone

Goals: Lower Nuclear Facility Costs, Improve Safety

Destroyed filter bank after a fire



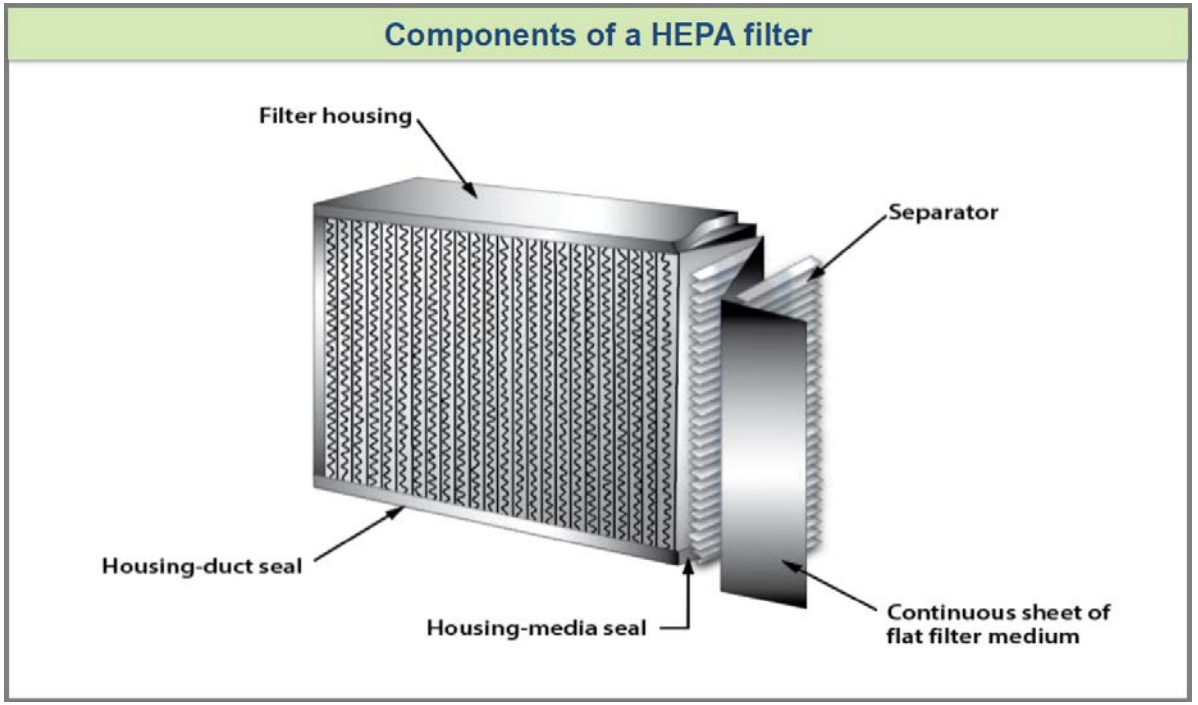
Water damage to filters following a fire

Ceramic filters perform at higher temperatures and are likely to eliminate reliance on credited fire suppression systems

Conventional Filters

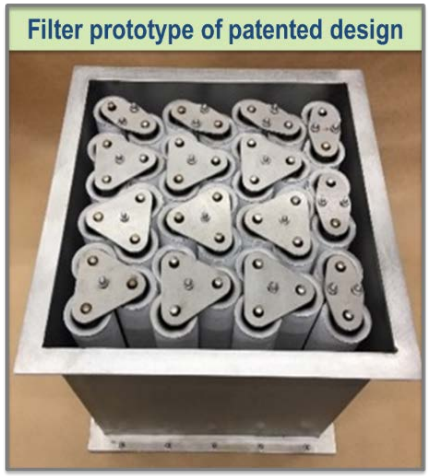
DOE Needs Analysis:

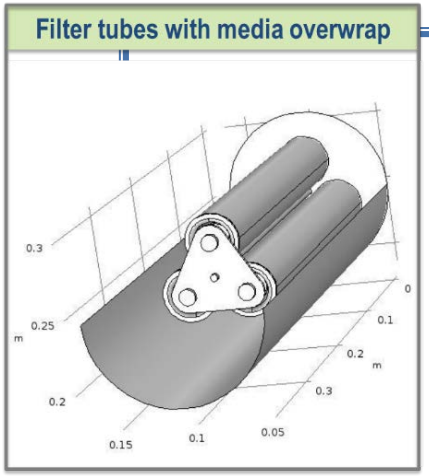
- 100% of knowledgeable nuclear air cleaning professionals believe HEPA filter media strength is very, or extremely, important
- 92% believe it is important to develop alternatives to current glass-fiber filters



Susceptible to thermal and water damage

LLNL Ceramic Filter Development





Ceramic Filters

Ceramic **HEPA** Filters

Ceramic **Prefilters**

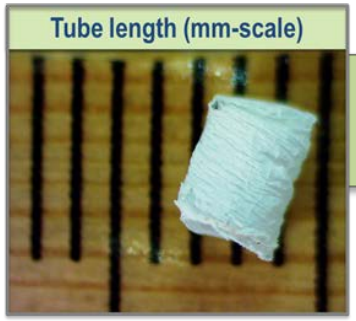
HEPA Overwrap (J. Haslam)

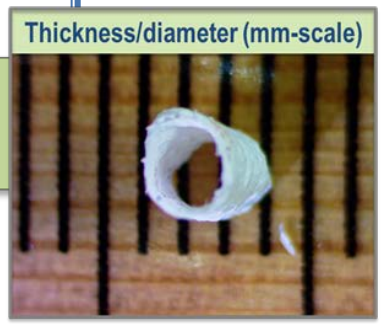
MTC Media (J. Kelly)

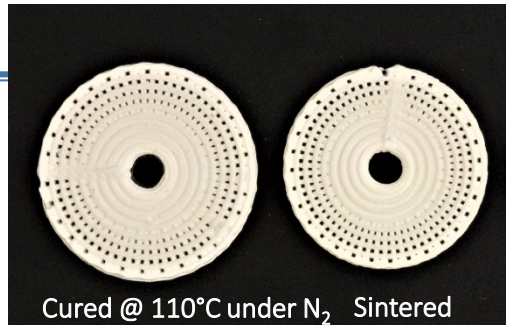
Advanced Manufacturing (M. Ceron)

Sealants (L. Finkenauer)

Survived prolonged exposure to elevated temperatures (500°C) with no statistical difference in filtration efficiency







MTC Media & Filter Development

Purpose: To improve the fire safety of DOE nuclear facilities, create advanced ceramic filtration components that can survive a fire, maintain performance requirements, and reduce costs

Benefit: Our engineering solution protects filters during a fire to simplify and reduce the cost of safety- and filter-support systems for operations

NSRD-12

- Demonstrate MTC media can reduce pressure drop
- Develop processes to produce MTC nanofiber filtration media

NSRD-20

- Prepare MTC media using different manufacturing approaches
- Test pressure drop and filtration efficiency of MTC filter elements

MTC Filter Development

FEASIBILITY STUDY

NSRD 12

MTC MEDIA

- *Lab-scale*
- *Proof-of-concept*
- *Process development*

ENGINEERING PROTOTYPES

NSRD 20

MTC FILTER ELEMENT

- *Lab-scale*
- *Design principles*
- *Analysis of alternatives*
- *Process optimization*
- *Tooling development*

FILTER PROTOTYPES

MTC FILTER

- *Scaling*
- *Automation*
- *Integration*
- *Certification*

MANU-FACTURING

- *Licensing*
- *Deployment*
- *Servicing*
- *Continuous Improvement*

Current Development Stage

Analysis of alternative manufacturing methods

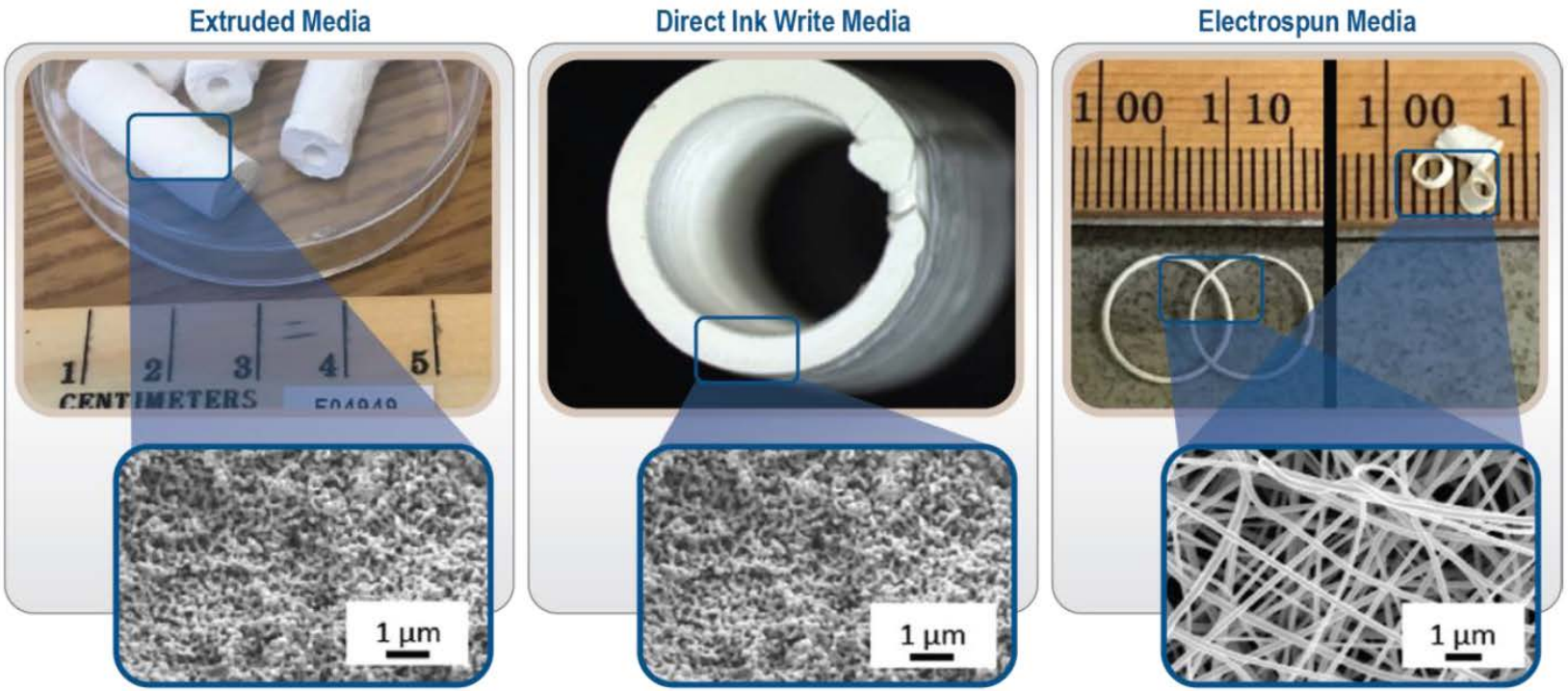
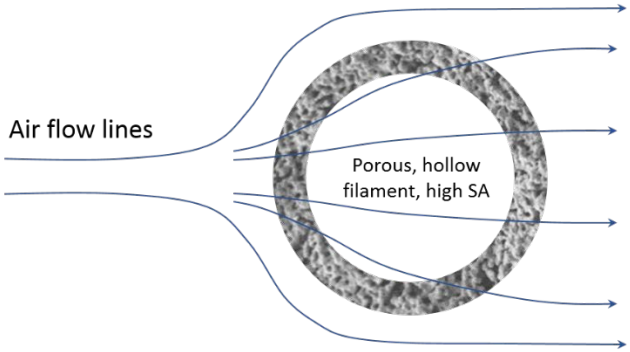


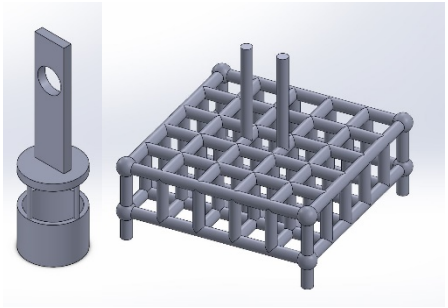
Figure 4. Macrostructure, microstructure, and nanostructure of novel ceramic mini-tubular filtration media with hierarchical architecture. The media is produced by three different fabrication processes (extrusion, direct ink writing, or electrospinning).

Extrusion (conventional)

- Although DIW is an additive manufacturing method based on extrusion, the DIW feedstock could not be readily adapted into a conventional extrusion process
- A pivot towards another conventional method – dip coating – in combination with additive manufacturing was used to coat sacrificial templates and demonstrate feasibility of the method; results showed significant development was required.

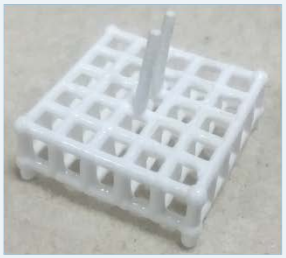
pivot



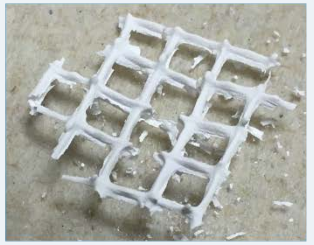


- coating speed influenced distortion, eliminating curling
- Thinner sacrificial template walls created elliptical distortion

- Applied lessons learned to lattice
- Cracked after burnout

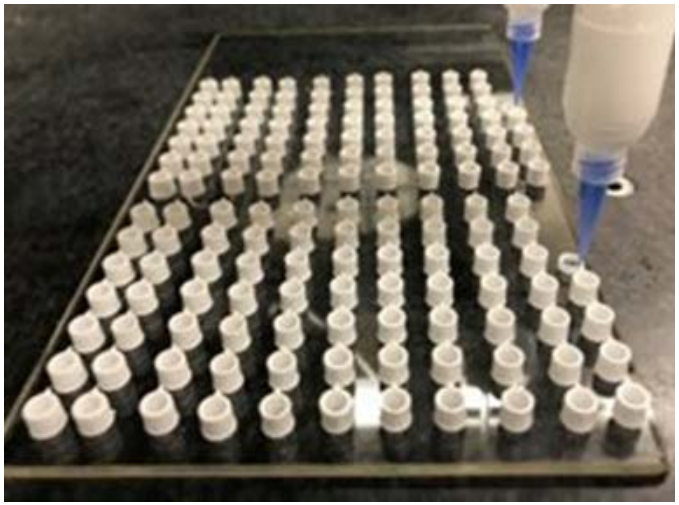


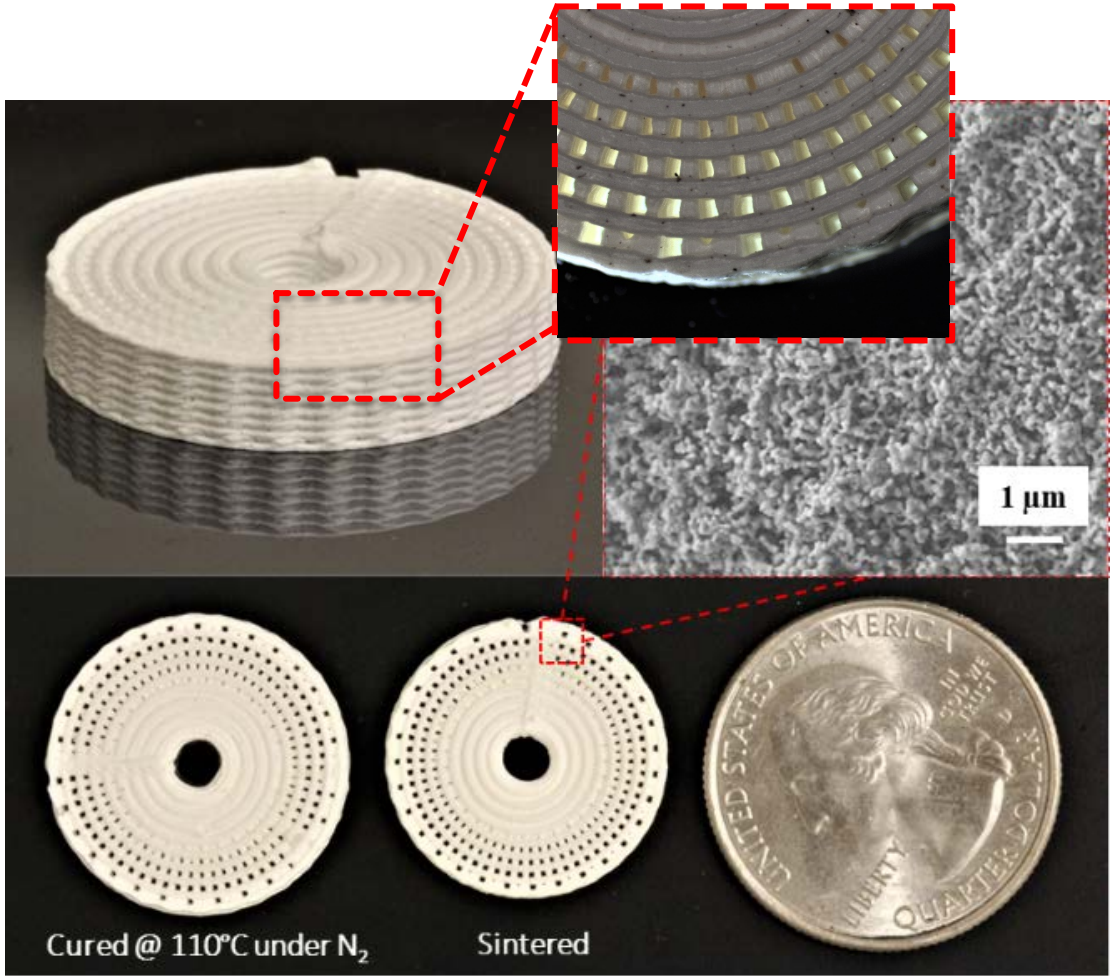
- Further modifications of thermal profiles enabled hollow tubes and struts from sacrificial templates



Direct-ink-write (advanced)

- **Most efficient production rate at lab-scale; enables engineering prototypes**
- Testing of engineering prototypes produced excellent dP results, better than conventional non-porous ceramic tubes, but efficiency was comparable to non-porous tubes indicating microstructure with nano and micro porosity is desired
- More elaborate parts were fabricated to show the versatility of printable features that may facilitate more effective design and filter integration strategies

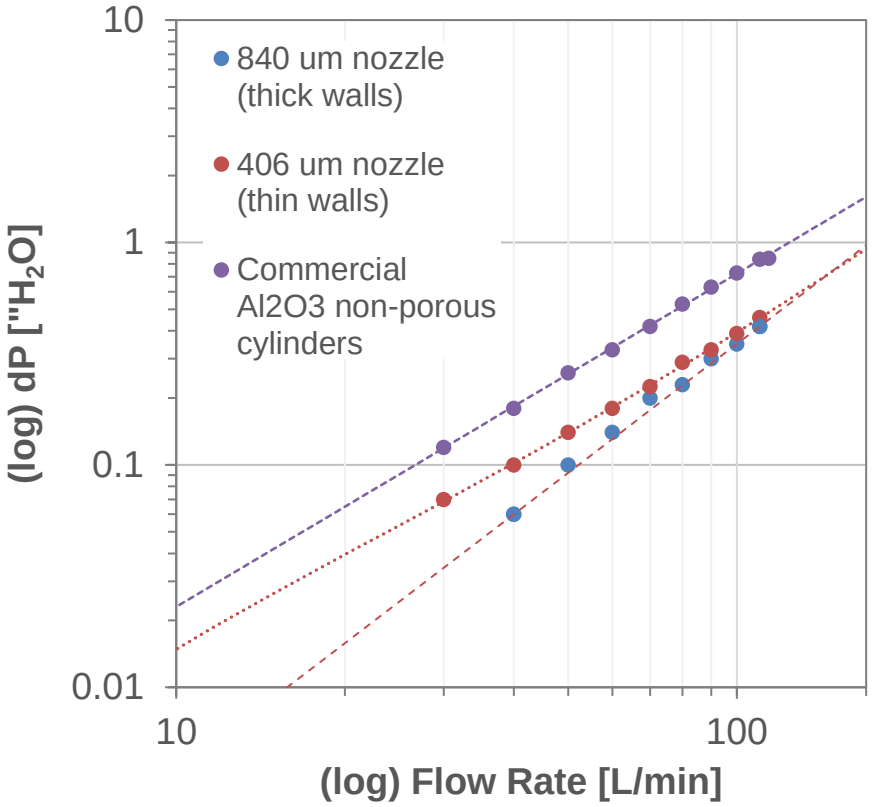




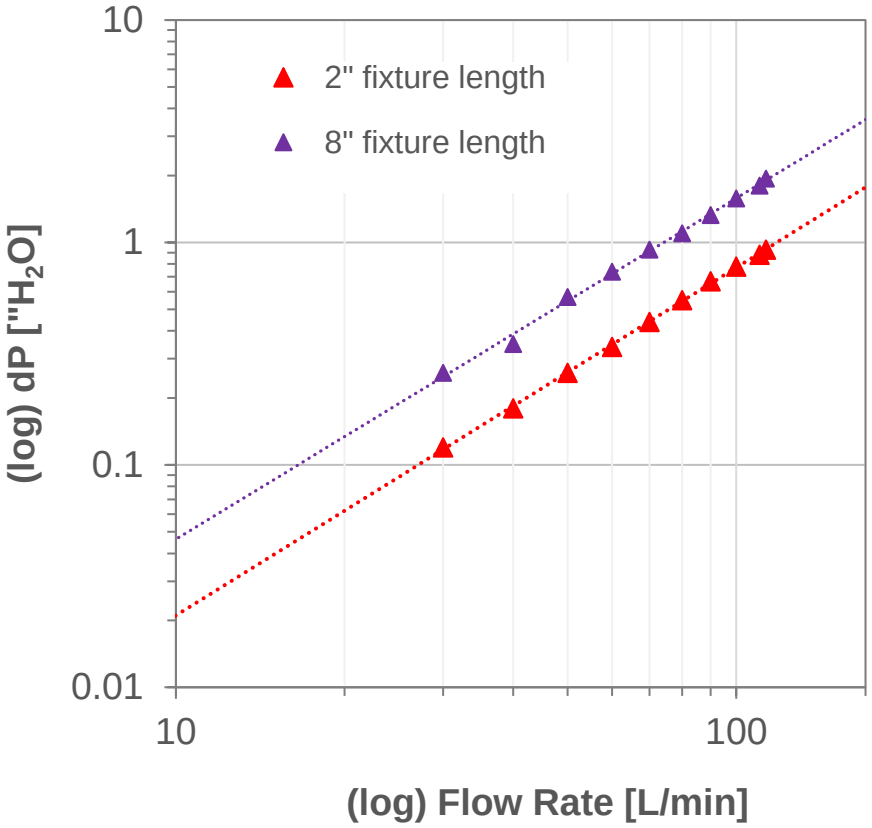
Direct-ink-write (advanced)

- **Most efficient production rate at lab-scale; enables engineering prototypes**
- Testing of engineering prototypes produced excellent dP results, better than conventional non-porous ceramic tubes, but efficiency was comparable to non-porous tubes indicating microstructure with nano and micro porosity is desired
- More elaborate parts were fabricated to show the versatility of printable features that may facilitate more effective design and filter integration strategies

Effect of **different 3Y-ZrO_2 mini tube media** on pressure drop (in 2" test fixture)



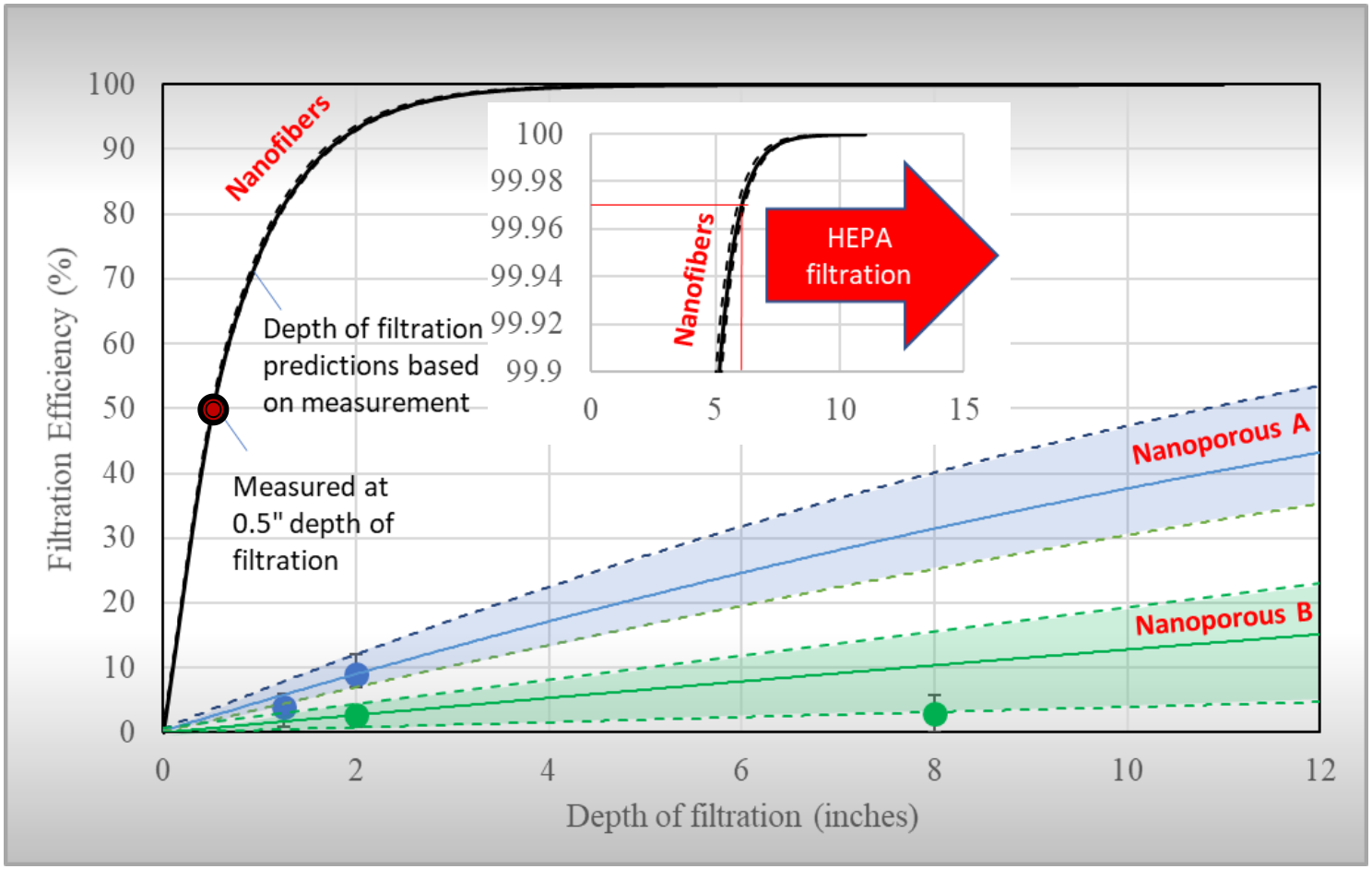
Effect of **test fixture length** on pressure drop of 3Y-ZrO_2 mini tube media



Porosity decreases dP, smaller diameter and larger DOF increases dP

Direct-ink-write (advanced)

- **Most efficient production rate at lab-scale; enables engineering prototypes**
- Testing of engineering prototypes produced excellent dP results, better than conventional non-porous ceramic tubes, but efficiency was comparable to non-porous tubes indicating microstructure with nano and micro porosity is desired
- More elaborate parts were fabricated to show the versatility of printable features that may facilitate more effective design and filter integration strategies

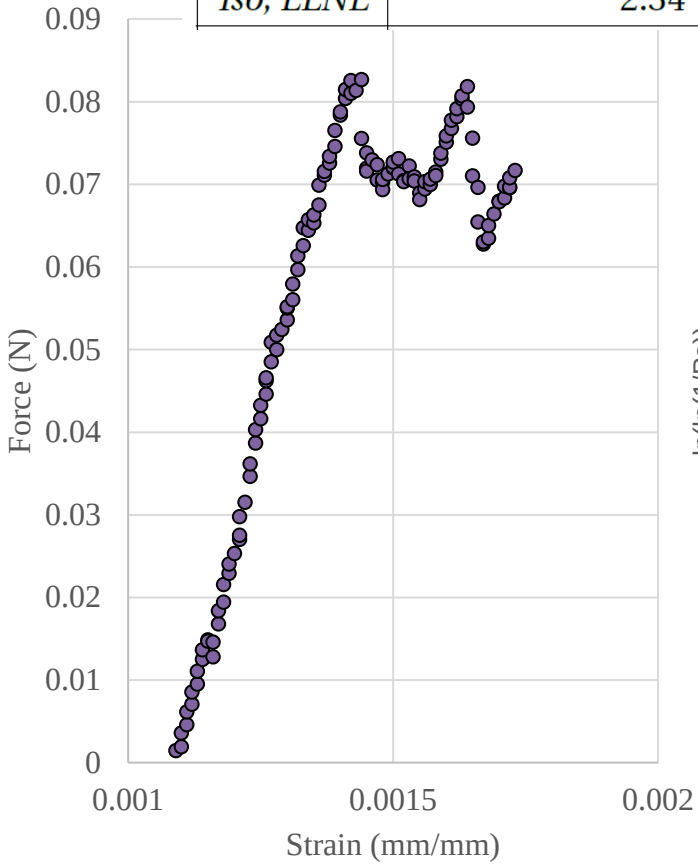


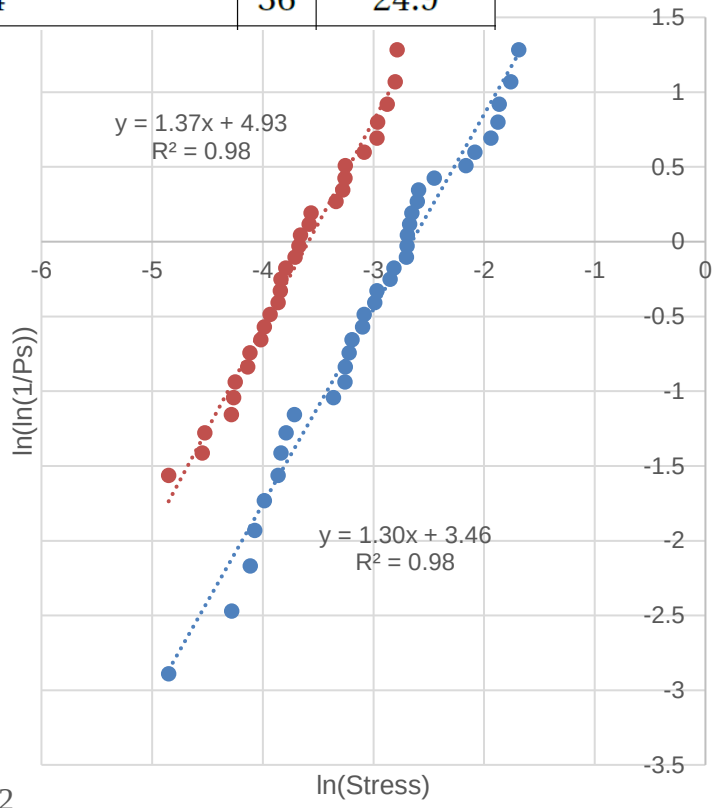
Nanofiber MTC media resulted in highest filtration efficiency, MTC size matters

Electrospinning (advanced)

- **Electrospun media currently result in the best filtration efficiency**
- MTC strength testing has been performed to optimize thermal treatments
- Enhanced formulation development produced tougher MTC media
- Efficient Prototype MTC forming equipment has been developed eliminating tedious manual operations
- Lab-scale electrospinning is inefficient and needs to be scaled
- Preliminary evaluation of commercial, pilot-scale electrospinning equipment demonstrates >10x gain in production efficiency from lab-scale is possible
- A patent application has been submitted (U.S. Patent Application No. 16739830)

Profile	Average Tube Diameter (mm)	N	$\overline{\sigma_y}$ (kPa)
Iso, LLNL	2.34	36	24.9

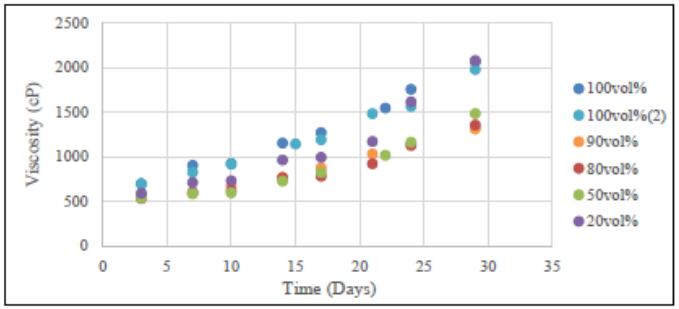


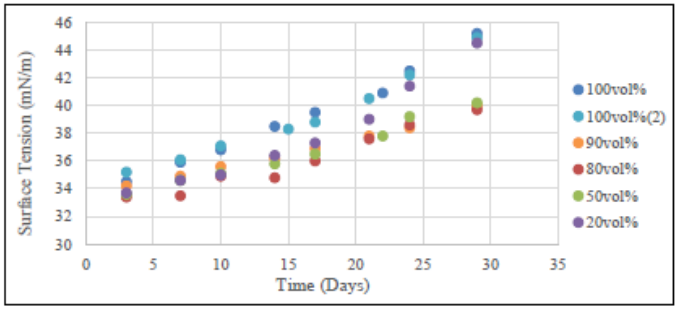


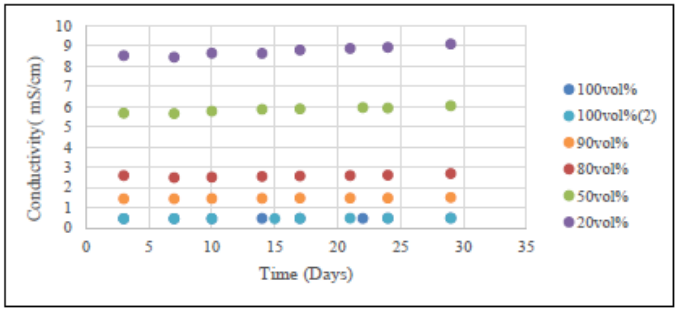
Developed strength testing protocols for Weibull analysis/thermal optimization

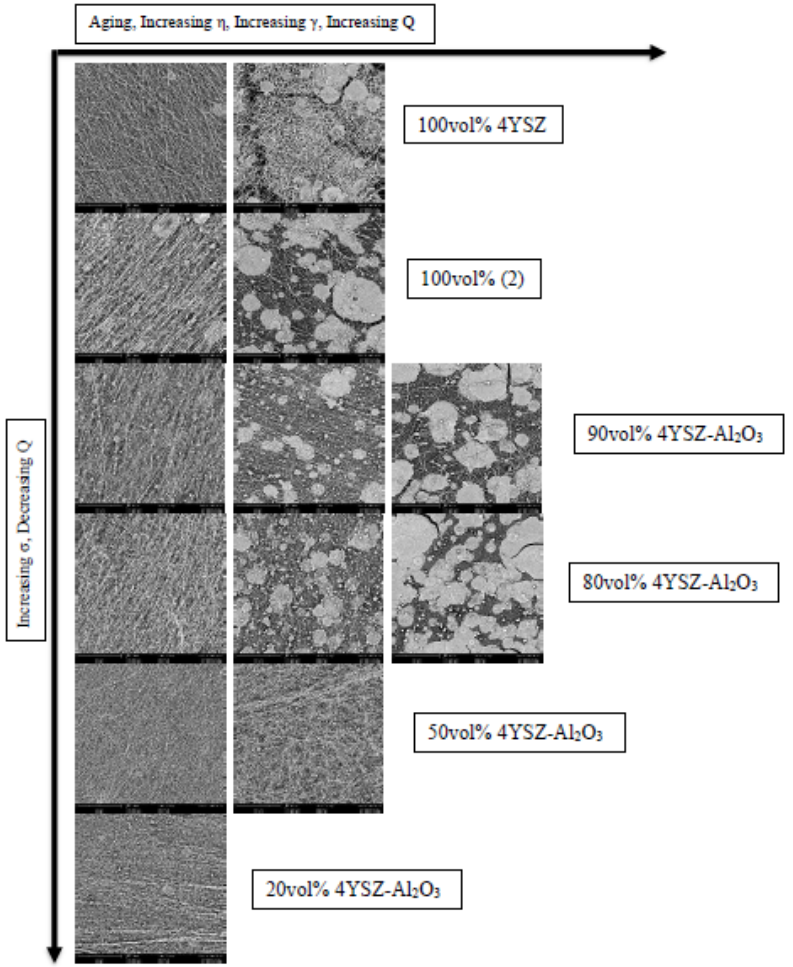
Electrospinning (advanced)

- **Electrospun media currently result in the best filtration efficiency**
- MTC strength testing has been performed to optimize thermal treatments
- Enhanced formulation development produced tougher MTC media
- Efficient Prototype MTC forming equipment has been developed eliminating tedious manual operations
- Lab-scale electrospinning is inefficient and needs to be scaled
- Preliminary evaluation of commercial, pilot-scale electrospinning equipment demonstrates >10x gain in production efficiency from lab-scale is possible
- A patent application has been submitted (U.S. Patent Application No. 16739830)





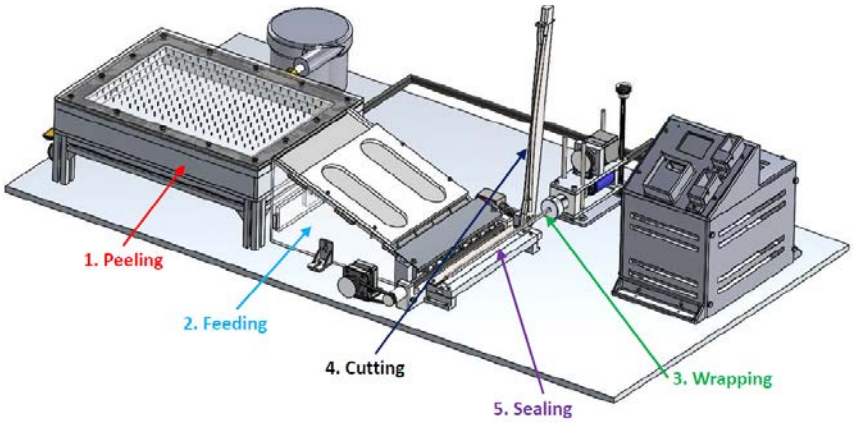




Formulation development for stronger and tougher MTC media

Electrospinning (advanced)

- **Electrospun media currently result in the best filtration efficiency**
- MTC strength testing has been performed to optimize thermal treatments
- Enhanced formulation development produced tougher MTC media
- Efficient Prototype MTC forming equipment has been developed eliminating tedious manual operations
- Lab-scale electrospinning is inefficient and needs to be scaled
- Preliminary evaluation of commercial, pilot-scale electrospinning equipment demonstrates >10x gain in production efficiency from lab-scale is possible
- A patent application has been submitted (U.S. Patent Application No. 16739830)



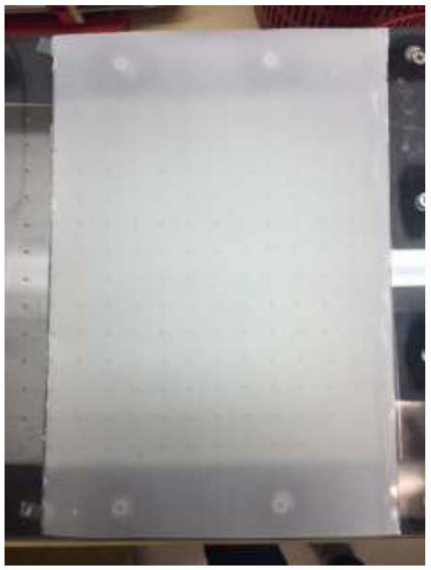


Figure 7.1a: Mesh Prior to Vacuum Table Use

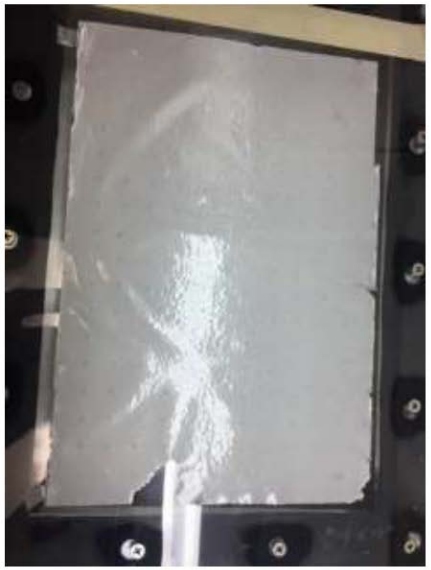
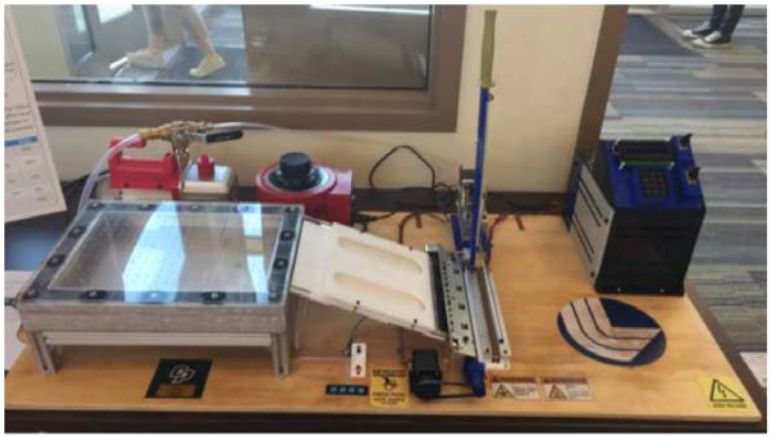
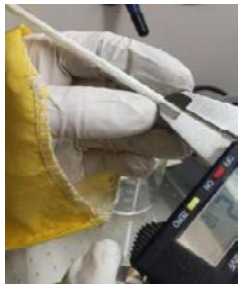


Figure 7.1b: Mesh After Vacuum Table Use

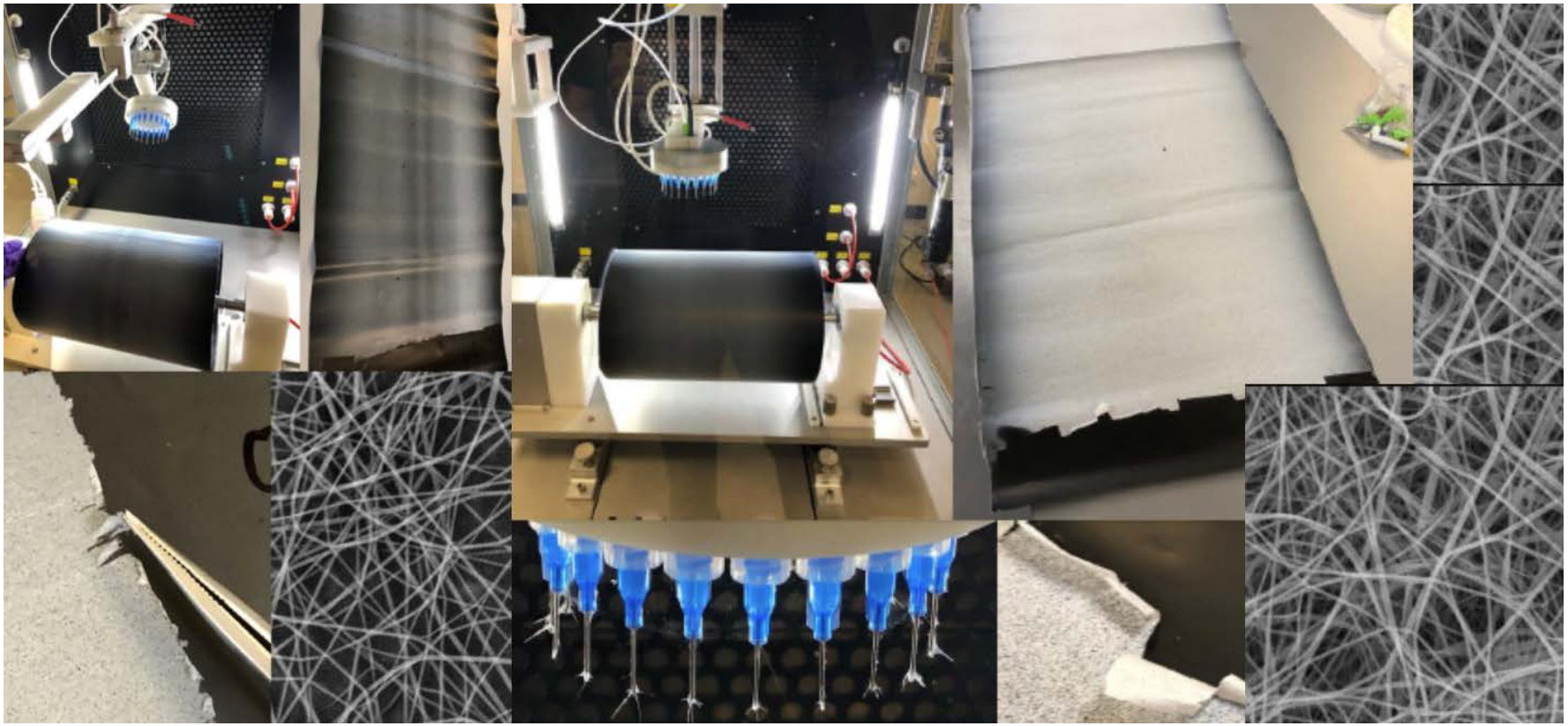




Developed tooling for peeling, feeding, cutting, rolling, and sealing MTC media

Electrospinning (advanced)

- **Electrospun media currently result in the best filtration efficiency**
- MTC strength testing has been performed to optimize thermal treatments
- Enhanced formulation development produced tougher MTC media
- Efficient Prototype MTC forming equipment has been developed eliminating tedious manual operations
- Lab-scale electrospinning is inefficient and needs to be scaled
- Preliminary evaluation of commercial, pilot-scale electrospinning equipment demonstrates >10x gain in production efficiency from lab-scale is possible
- A patent application has been submitted (U.S. Patent Application No. 16739830)



Currently exploring MTC nanofiber media production efficiency improvements; Identified 4 collaborators and initiated trials; demonstrated ~20x faster production

Summary and Outlook

Manufacturing Method	Significant Results
Extrusion (conventional)	• Although DIW is an additive manufacturing method based on extrusion, the DIW feedstock could not be readily adapted into a conventional extrusion process • A pivot towards another conventional method – dip coating – in combination with additive manufacturing was used to coat sacrificial templates and demonstrate feasibility of the method; results showed significant development was required.
Direct-ink-write (advanced)	• **Most efficient production rate at lab-scale; enables engineering prototypes** • Testing of engineering prototypes produced excellent dP results, better than conventional non-porous ceramic tubes, but efficiency was comparable to non-porous tubes indicating microstructure with nano and micro porosity is desired • More elaborate parts were fabricated to show the versatility of printable features that may facilitate more effective design and filter integration strategies
Electrospinning (advanced)	• **Electrospun media currently result in the best filtration efficiency** • MTC strength testing has been performed to optimize thermal treatments • Enhanced formulation development produced tougher MTC media • Efficient Prototype MTC forming equipment has been developed eliminating tedious manual operations • Lab-scale electrospinning is inefficient and needs to be scaled • Preliminary evaluation of commercial, pilot-scale electrospinning equipment demonstrates >10x gain in production efficiency from lab-scale is possible • A patent application has been submitted (U.S. Patent Application No. 16739830)

Improve filter efficiency; use as supports

Improve production efficiency


Lawrence Livermore
National Laboratory