

February 2021

EFCOG 2021 – NSR&D Subgroup Meeting

NSRD-20: Functional Testing of Novel MTC HEPA Filtration Media



 **Physical & Life Sciences** **GlobalSecurity**

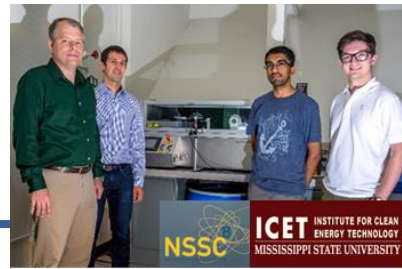
James Kelly
Ceramics & Polymers Engineering
Materials Engineering Division



LLNL-PRES-819452

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

Acknowledgements

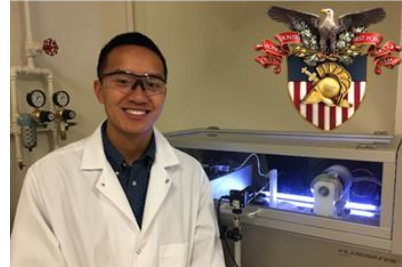


■ LLNL

- Mark Mitchell
- Howard Wong
- Danny Laycak
- Dr. Jeff Haslam
- Dr. Lauren Finkenauer
- Dr. Andrew Hoff
- Dennis Luong
- Dr. Patrick Campbell
- **Dr. Maira Ceron**
- Uday Mehta
- **Alyssa Troksa**
- Hannah Eshelman
- Erik Brown
- Dr. Swetha Chandrasekaran
- Dr. Nick Teslich
- **Michael Ross**
- **Brian Deemer**
- **Christina Santa-Lucia**

■ Cal Poly

- Prof. Mayer
- Prof. Harding
- Josh Clemons
- Nick Brodine
- Delaney Fitzsimmons
- **Leo Taranta-Slack**
- **Daniel Freeman**
- **Hunter Brooks**



■ 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)

- Prof. Chintalapalle
- Nanthakishore Makeswaran

■ MSIPP

- Trent Malone

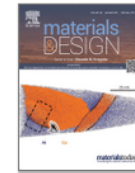
■ ANL, Nanoscience, Dipole

Accomplishments




- Alyssa Troksa *et. al.* published on feedstock development



Materials & Design
Volume 198, 15 January 2021, 109337



3D-printed nanoporous ceramics: Tunable feedstock for direct ink write and projection microstereolithography

Alyssa L. Troksa ¹, Hannah V. Eshelman ¹, Swetha Chandrasekaran, Nicholas Rodriguez, Samantha Ruelas, Eric B. Duoss, James P. Kelly, Maira R. Cerón  , Patrick G. Campbell 

- James Kelly *et. al.* filed patent applications
 - US Patent App. 16/739,830
 - US Patent App. 16/799,493

Goal: Improve safety of DOE nuclear facilities while also reducing operational costs

Destroyed filter bank after a fire



Water damage to filters following a fire

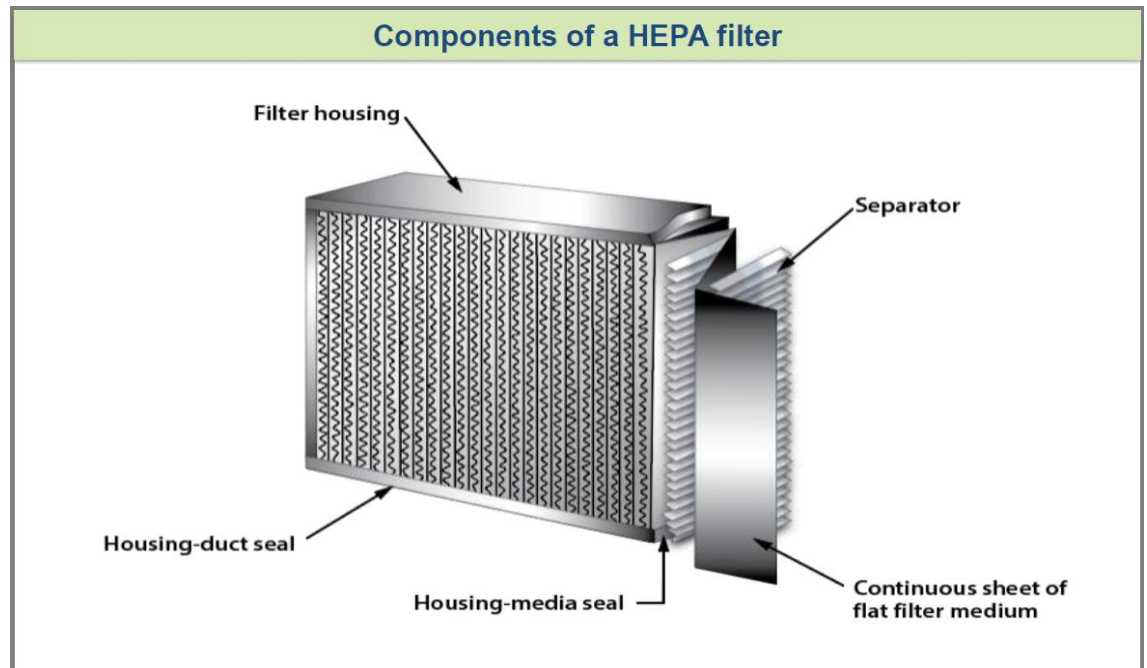


Ceramic filters can simplify and reduce the cost of safety- and filter-support systems for operations

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**



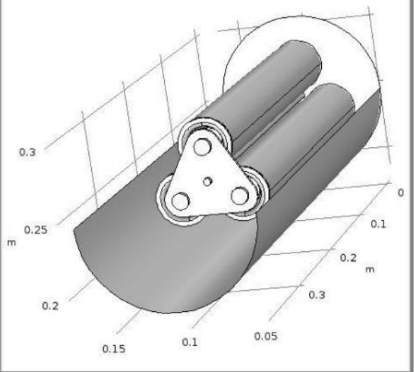
Susceptible to thermal and water damage, limited burst strength

LLNL Ceramic Filter Development

Filter prototype of patented design



Filter tubes with media overwrap



Ceramic Filters
(M. Mitchell)

Ceramic HEPA Filters

Ceramic Prefilters

HEPA Overwrap
(J. Haslam)

MTC Media
(J. Kelly)

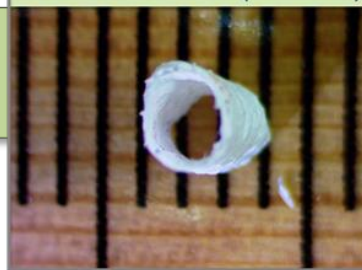
Advanced Manufacturing
(M. Ceron)

Sealants
(A. Hoff, M. Ross)

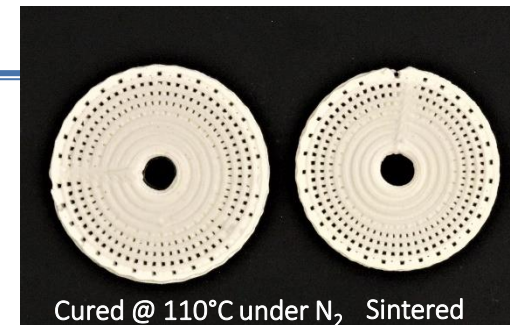
Tube length (mm-scale)



Thickness/diameter (mm-scale)



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



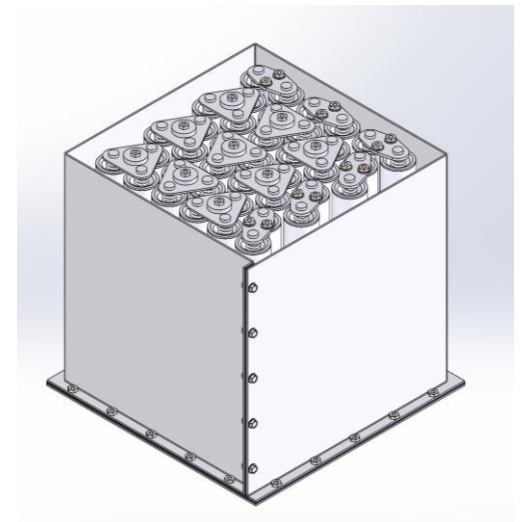
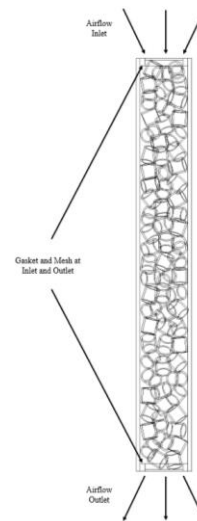
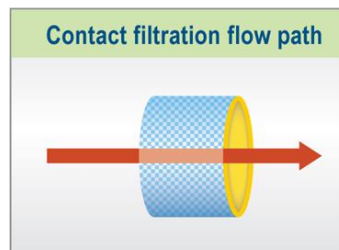
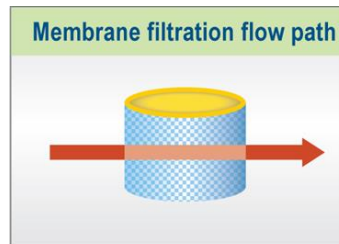
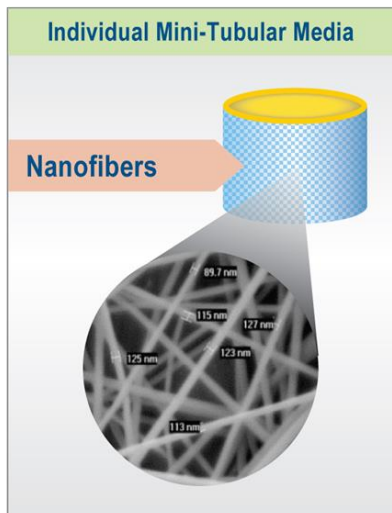
Cured @ 110°C under N₂ Sintered

Impact on DOE Guidance

Informs ASME AG-1 Code on Nuclear Air and Gas Treatment Section FO Ceramic Media Filters (in development)

■ Variations

- Framed vs. unframed
- Application temperatures
- Type A – Overwrap design (ceramic media on ceramic supports)
- **Type B – Unsupported media (no support structures)**
- Type C – Monoliths



MTC Filter Development

NSRD-12 (100% complete), NSRD-20 (90% complete), NSRD-31 (15% complete)

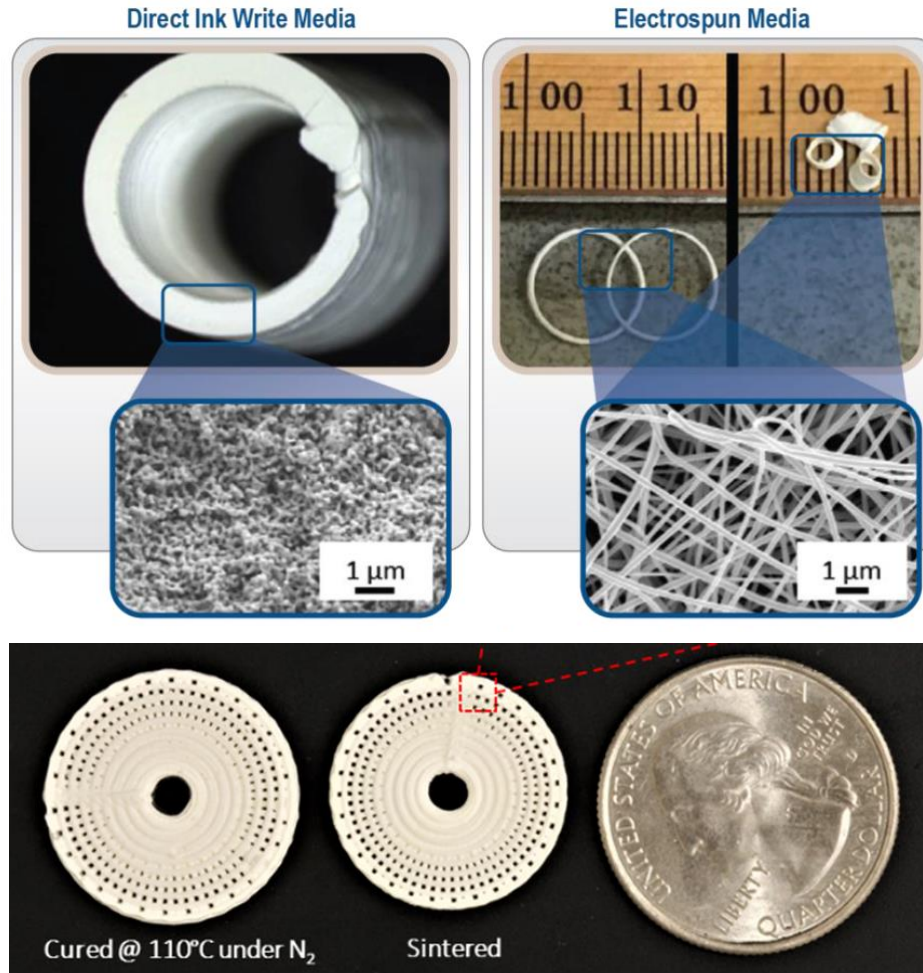


Project	Objectives
NSRD-12	Demonstrate MTC media can reduce dP, MTC media process development
NSRD-20	Evaluate alternative manufacturing methods, test MTC filter prototypes
NSRD-31	Pilot-scale production of MTC media, improve filter integration strategies

DOE NSR&D has been instrumental support for early-stage R&D needed to facilitate transitioning to other funding mechanisms (e.g., technology commercialization funding)

Analysis of alternative manufacturing methods

- Most efficient production rate at lab-scale
- Enables rapid testing of engineering prototypes
- Greater design freedom
- Can provide support to electrospun media
- Low dP
- **Need to improve filtration efficiency**



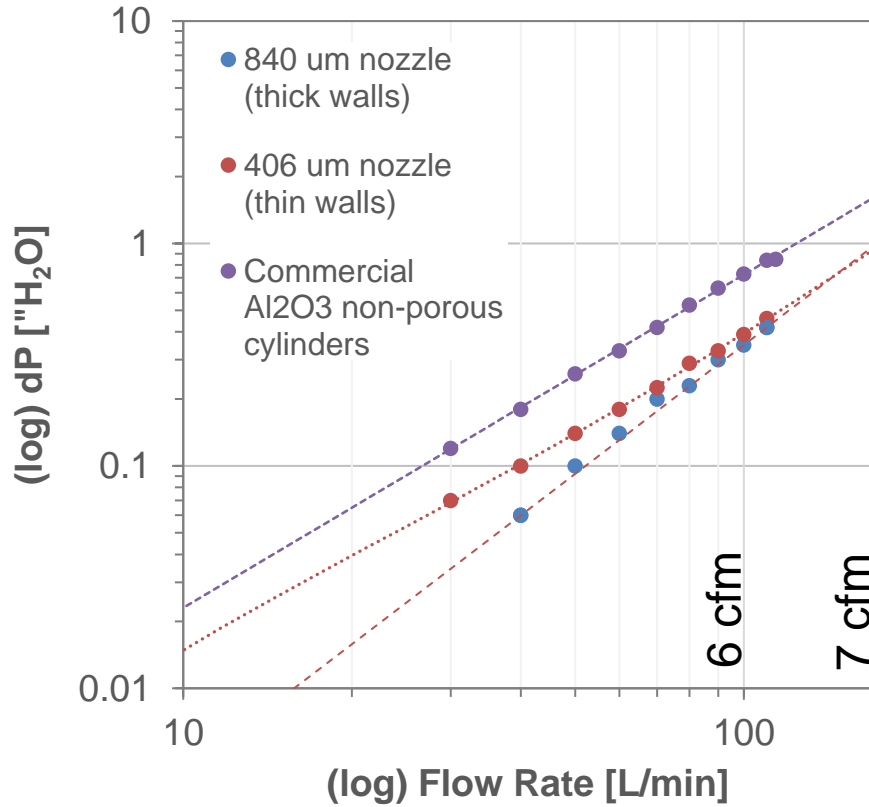
- Provides best filtration efficiency
- **Lab-scale spinning is inefficient and needs to be scaled**
- **Post-processing needs to be automated**



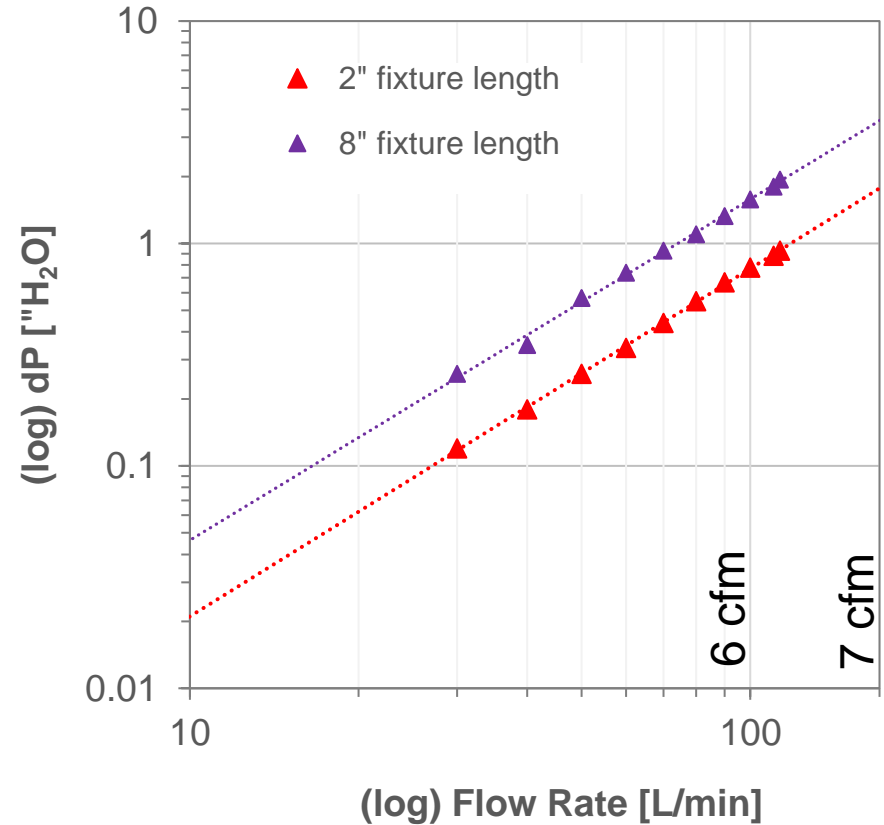
Direct-ink-write (advanced)

- **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-ZrO₂ mini tube media** on pressure drop (in 2" test fixture)



Effect of **test fixture length** on pressure drop of 3Y-ZrO₂ mini tube media

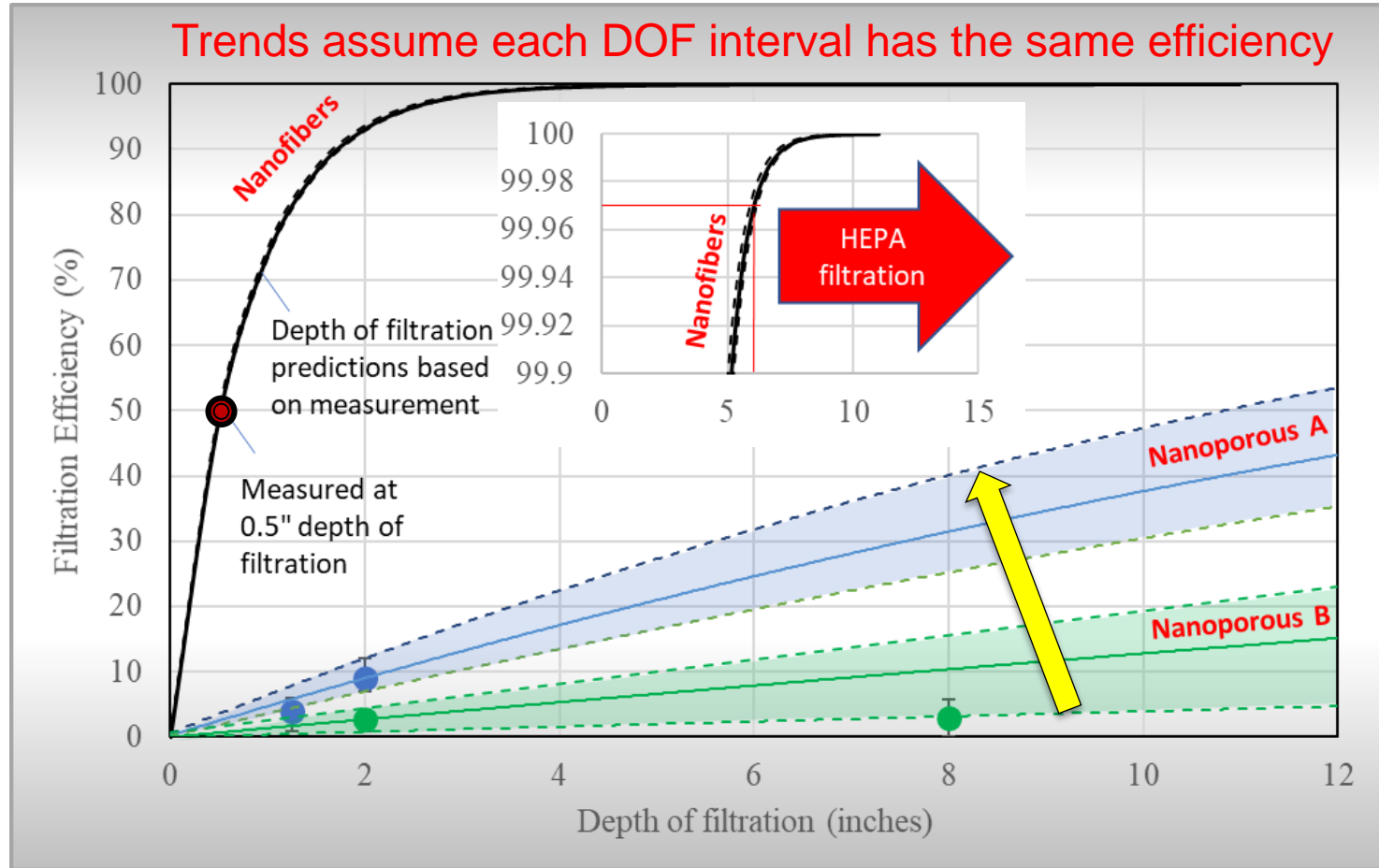


Early trends: porosity decreases dP, smaller diameter and larger DOF increases dP



Direct-ink-write (advanced)

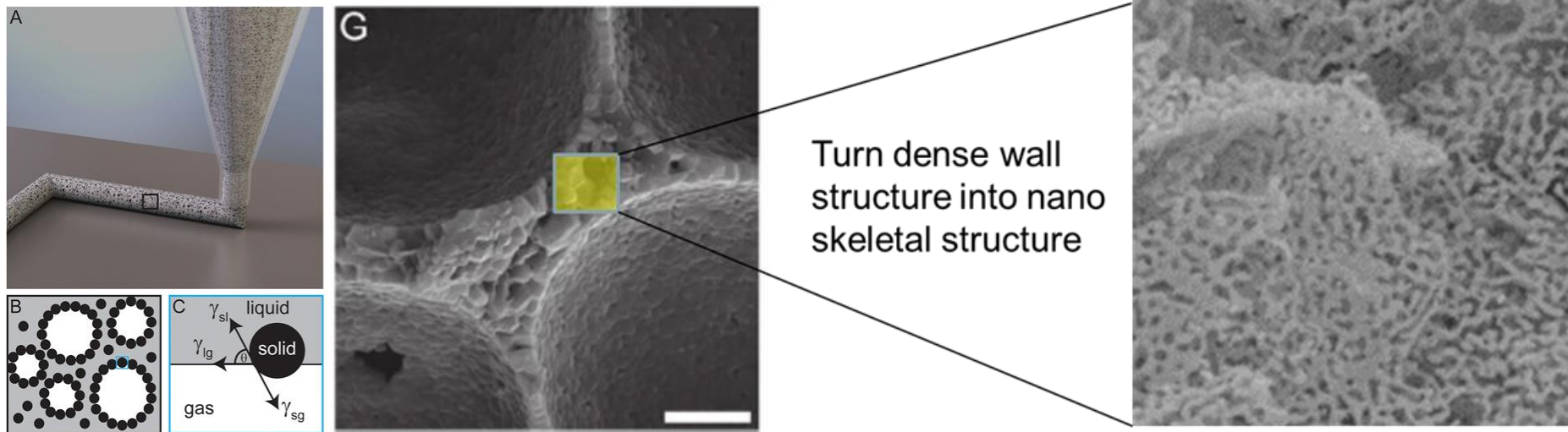
- **Most efficient production rate at lab-scale; enables engineering prototypes**
- Testing of engineering prototypes produced excellent dP results, better than conventional non-porous ceramic tubes, but efficiency was comparable to non-porous tubes indicating microstructure with nano and micro porosity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



Need to improve filtration performance of media prepared by DIW

Current efforts to improve DIW (NSRD-31)

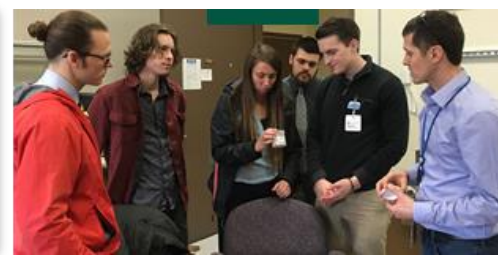
- Using dual pore formers and resulting in nano, micro, macro porosities to improve performance
- E.g., concept based on Muth *et. al.* 2017 (Harvard, MIT)



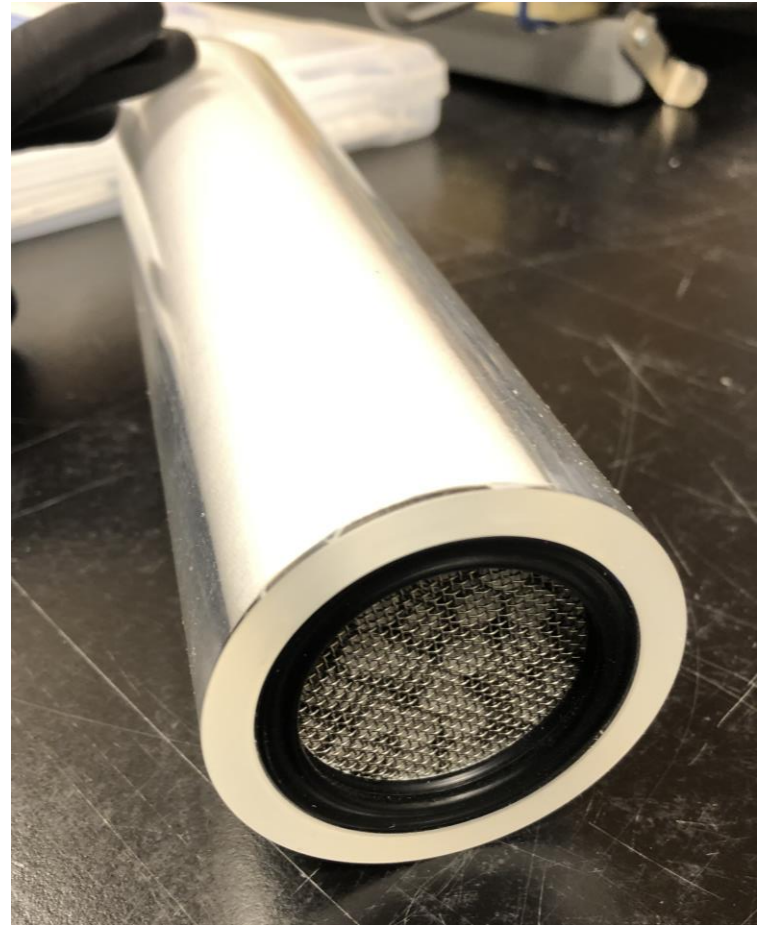
Additional porosity scales expected to improve performance

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)



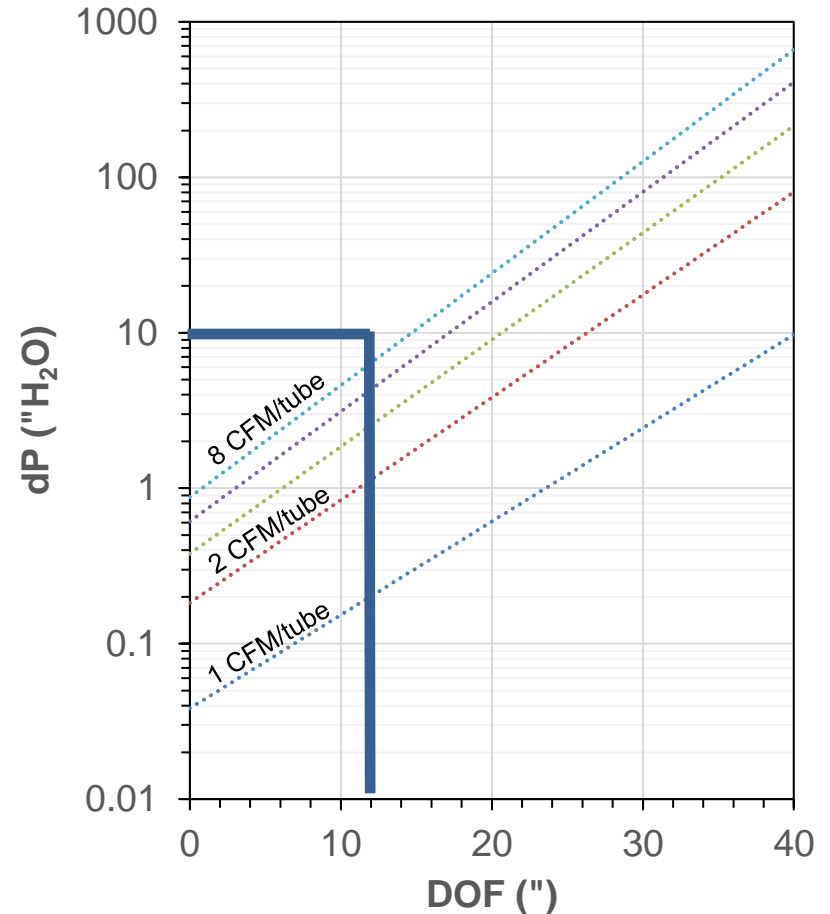
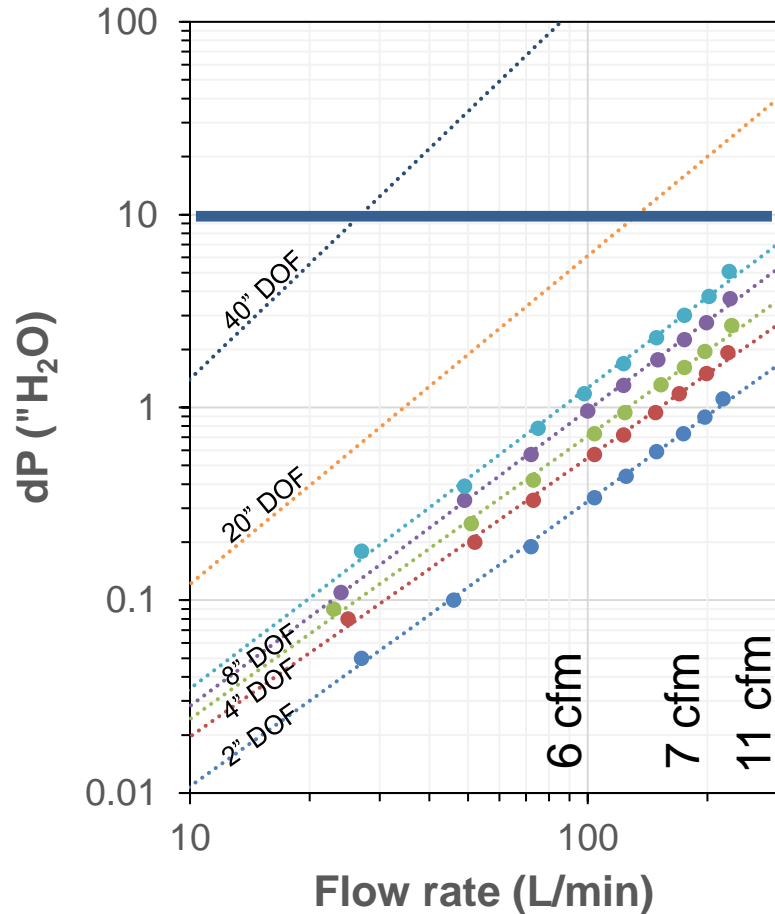
- MTC Surrogate Filter
- Fiberglass HEPA media formed into ¼" tubes
- Up to 10" DOF
- Enables further exploration of design principles
- Enables testing basic assumptions on filtration efficiency (each DOF interval has the same efficiency of remaining aerosol)



Built surrogate filter using conventional HEPA media to test performance assumptions

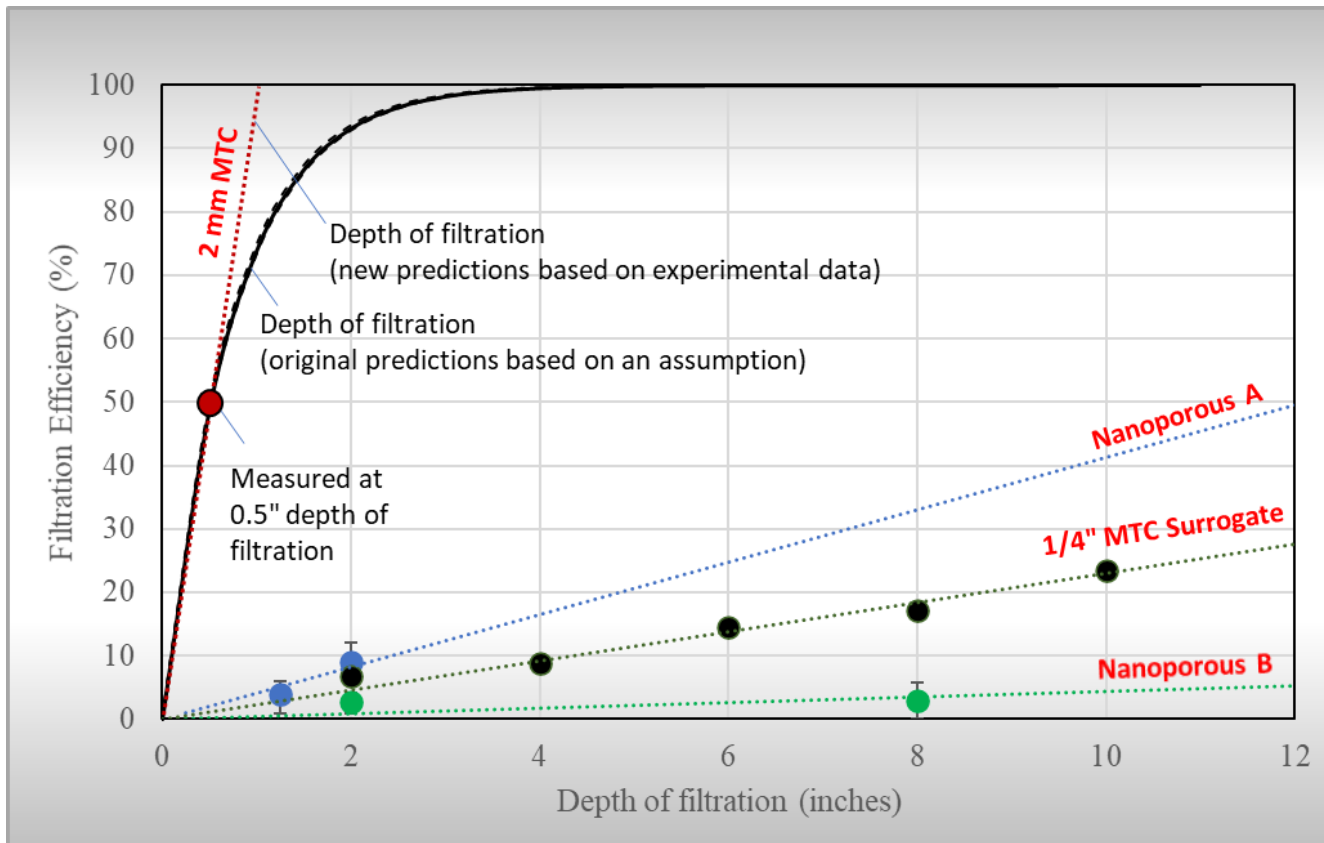


MTC Surrogate Filter Test Results



Indication of design trends, first dP prediction set within retrofitting requirements

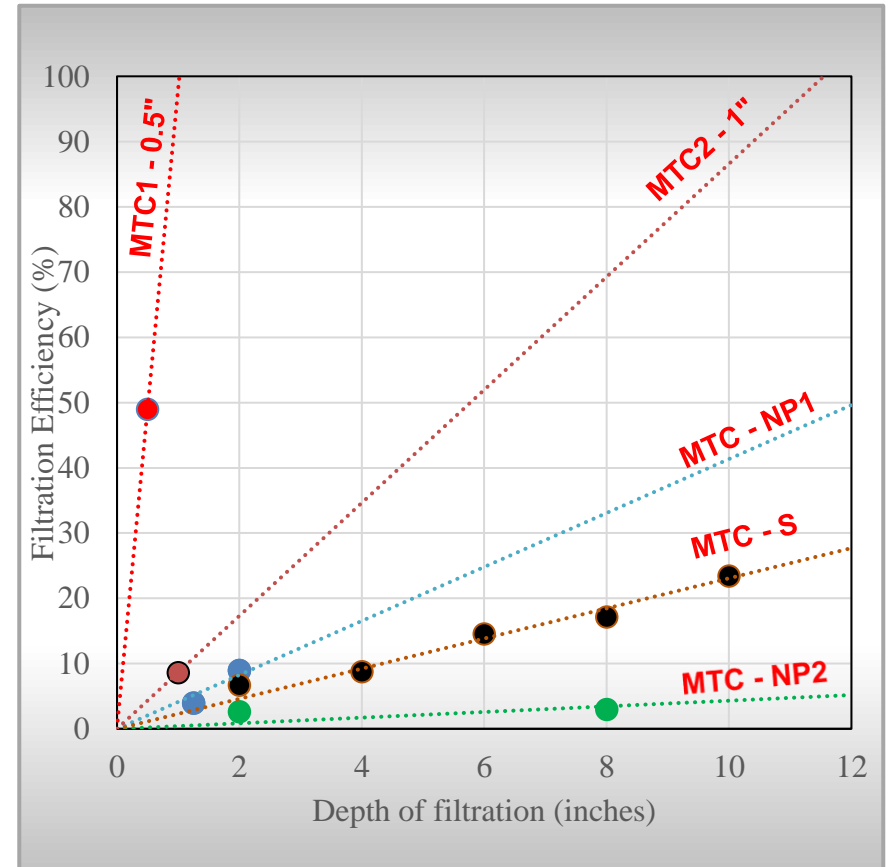
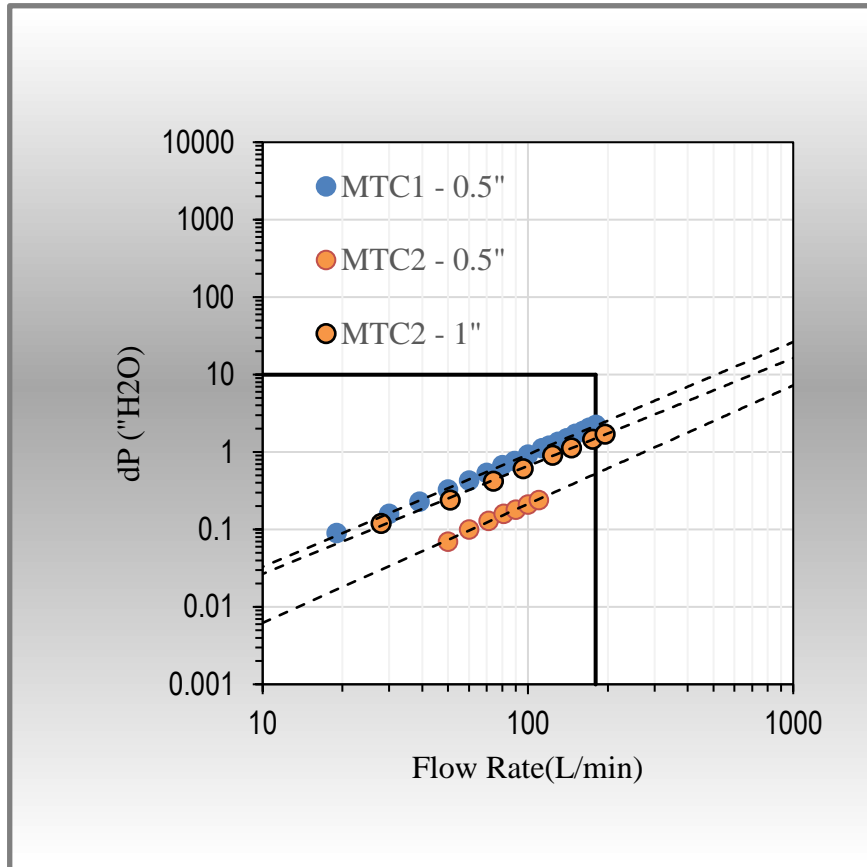
MTC Surrogate Filter Test Results



- Initial assumptions were too conservative (FE appears to be linear with DOF)
- Only need 1-2" DOF of MTC media for HEPA filtration
- Alternatively, can modify MTC media to further reduce dP

Will be measuring a MTC filter with 1-2" DOF in the near future

MTC filter tests



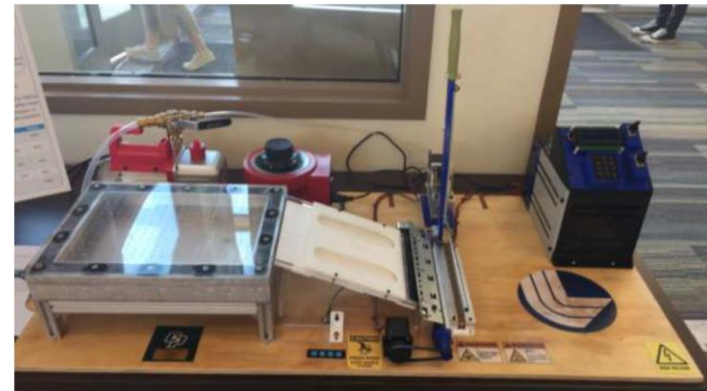
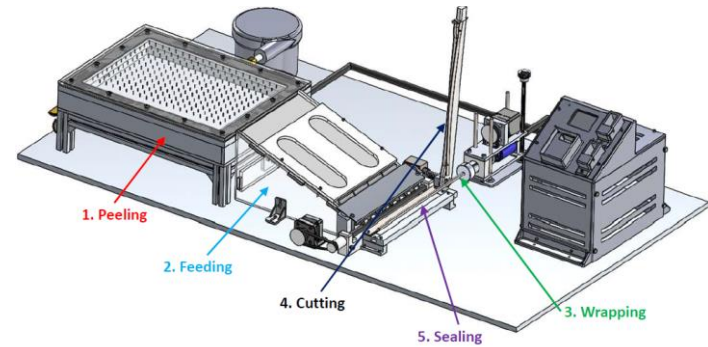
Smaller nanofiber MTC are most favorable for HEPA filtration

Current efforts on scaling (NSRD-31 activities)

R2R Electrospinning



Automating post-processing

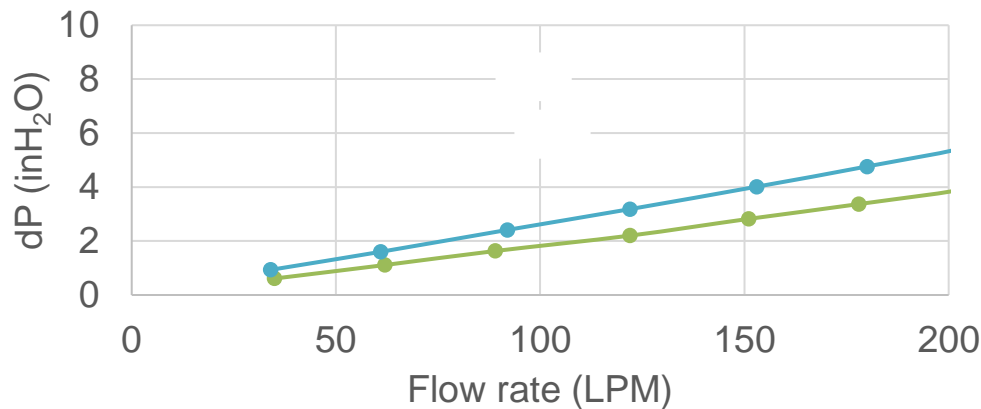


Currently working towards eliminating competing bottlenecks

Related Projects

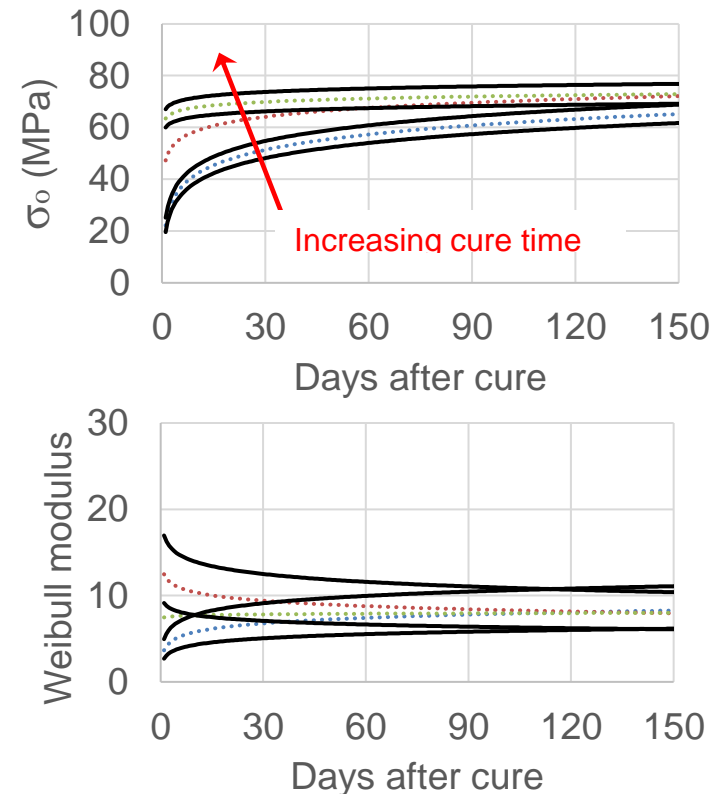
- Filter/scrubber combination

Flow rate (LPM)	Efficiency before 500°C exposure	Efficiency After 500°C exposure
56	99.96 ± 0.1	99.78 ± 0.1
183	99.98 ± 0.1	99.89 ± 0.2



Filter performance demonstration after a simulated fire

- High-temperature sealants



Baseline mechanical properties of sealants to evaluate aging

Summary and Outlook

- Several MTC-based filters have been constructed and tested
 - Still establishing design principles to optimize dP and FE
 - Important for retrofiting
- Pressure drop reductions
 - MTC geometric factors
 - Permeability (e.g., solid wall vs. porous wall)
- Pressure drop increase
 - Flow rate
 - DOF
- Filtration efficiency
 - Appears linear with DOF
 - Prior estimate of 6" DOF to achieve HEPA filtration with MTC media expected to be too conservative
 - Update to assumption based on measured data of MTC surrogate filter suggest 1-2" DOF is required (or gives more design margin to control dP)
- Need to scale and automate processes



**Lawrence Livermore
National Laboratory**