Roll-to-Roll Manufacturing Science and Applications: From Ideal Materials to Real-World Devices

R2RAMM multi-lab collaboration

Poster presentation, June 11, 2019

R2RAMM multi-lab collaboration
Greg Krumdick

- Collaboration Lead: Claus Daniel (ORNL)

- Argonne National Laboratory Lead: Gregory Krumdick
- Lawrence Berkeley National Laboratory Lead: Vince Battaglia
- National Renewable Energy Laboratory Lead: Michael Ulsh
- Sandia National Laboratory: Randy Schunk
- Oak Ridge National Laboratory Lead: David Wood

U.S. DOE Advanced Manufacturing Office Program Review Meeting
Arlington, VA

Overview

This poster is one section of R2R AMM DOE laboratory collaboration. Overall project information will be given at the oral presentation session

"Roll to Roll Consortium"
- by Claus Daniel (Collaboration Lead), June 12, 2019

Timeline

- Project start date: October 2019
- Projected end date:
  - Core Projects: September 2021
  - CRADAs: October 2019*
- Project completion for FY19
  - Core Projects: 30%
  - CRADA Projects: ~80%

Barriers*

- Continuous processing
- Registration and alignment challenges
- Materials compatibility
- Stoichiometry control
- Availability of materials data

* AMO MYPP for FY 2017-2021, June 2017 draft, section 3.1.8

FY 19 Budget

- Core lab work: $3,000K per year
- CRADA work: $1,150K plus $1,150K non-federal cost share

Partners

- ORNL, ANL, NREL, LBNL, SNL
- Navitas Systems
- Fisker, Inc.
- SolarWindow Technologies, Inc.
- Proton OnSite*

* Pending contract award

Project Objectives

- Continuing collaborative lab program begun in FY16, the objectives of FY19 project are further advancing materials scale up and processing science, creating impacts on a wider range of applications, such as functional coatings, filtration applications, flow batteries for grid applications, fuel cell membranes, PGM-free catalysts, electrodes for electrochemical CO_2 reduction concepts, and water manufacturing
- Explore and execute new methods and technologies to engage with industry and other R2R stakeholders that will enable pre-commercial, low TRL activities

FY 2018

MATERIALS AND PROCESSES DEVELOPMENT FOR WATER DESALINATION

BATTERY AND FUEL CELL MATERIALS DEVELOPMENT AND CHARACTERIZATION

Technical Innovation

- Resin wafer technology is capable of providing >35% energy efficiency for water desalination compared to 15% for the current state-of-the-art technologies

Limitations:

- Current resin wafer fabrication is time-consuming, inefficient, costly, and may contribute to inconsistent quality

Proposed Approach:

- Develop continuous R2R manufacturing capability that increases production capacity and quality control
- Improve separations energy efficiency and decrease processing costs for water purification
- Critical Innovations:
  1. Scalable R2R manufacture of resin wafer material for water purification
  2. 90+ wafers were produced at different conditions
  3. Conductivity of wafer made with SAAL was greater in comparison to conductivity of manually produced wafers
  4. Reduced processing time and cost for multiple industrial applications besides water purification, e.g., chemicals purification, biochemical separations and recovery

Technical Approach

SAAL for Resin Wafer Fabrication

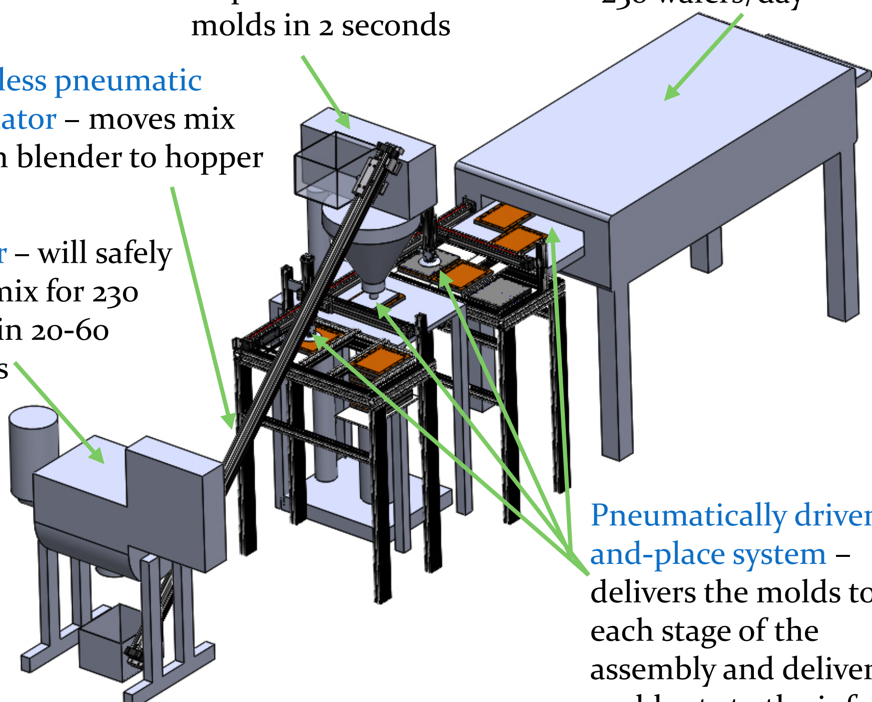
Semi-automated assembly line (SAAL) design can significantly reduce the time and labor cost to produce wafers necessary to support small and large cooling water systems

Dispenser – will dispense and fill molds in 2 seconds

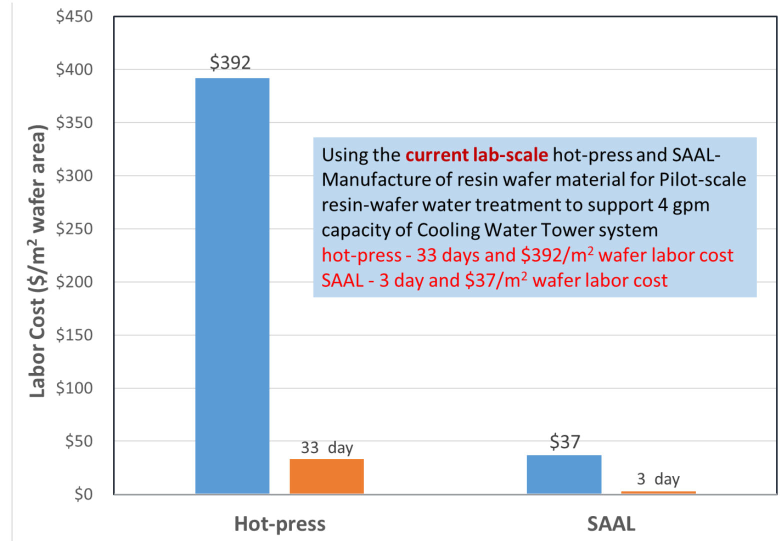
Infrared conveyor oven – makes up to 230 wafers/day

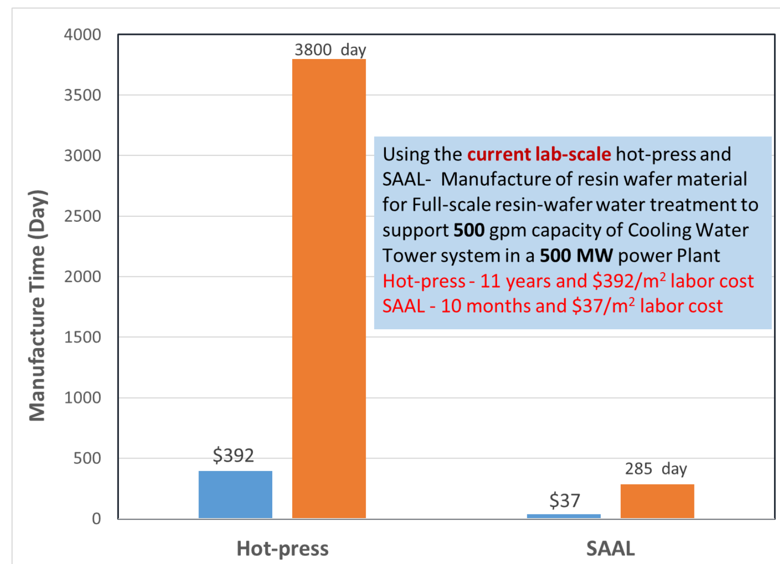
Rodless pneumatic actuator – moves mix from blender to hopper

Blender – will safely blend mix for 230 wafers in 20-60 seconds



Pneumatically driven pic-and-place system – delivers the molds to each stage of the assembly and delivers the mold sets to the infrared oven





Results and Accomplishments

- Built and implemented semi-automated assembly line (SAAL)
- Demonstrated SAAL for resin wafer fabrication

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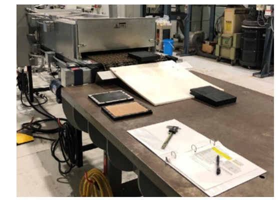

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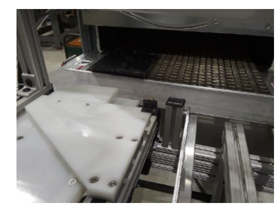

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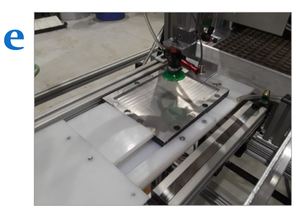

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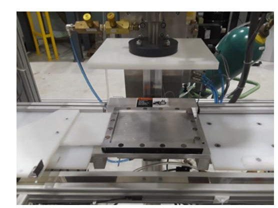


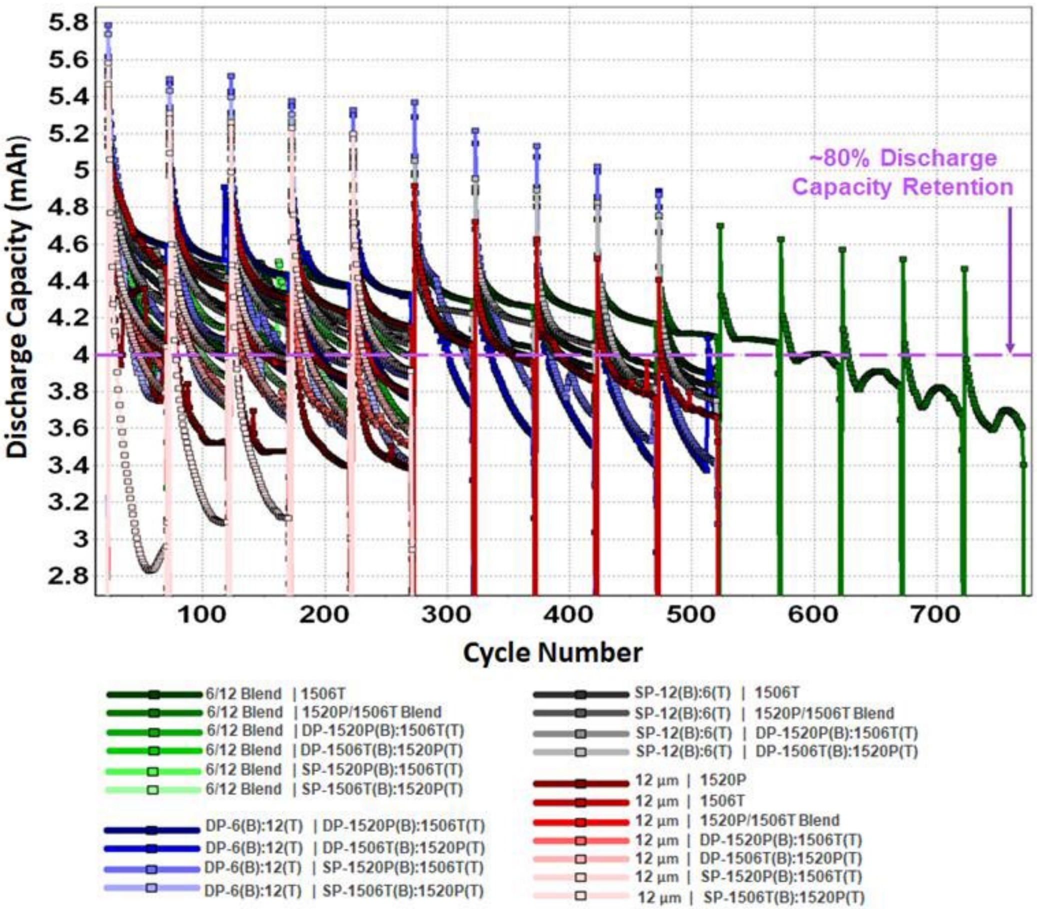
a) semi-automated wafer assembly system including b) mixer, c) bucket, d) pneumatic lift, e) vacuum cups, f) dispenser, g) moving belt, h) infrared oven, i) heat-cured wafers

Results and Accomplishments

Battery - Structured Anode Study

Best anodes: blend of small and large graphite particles; large graphite particles

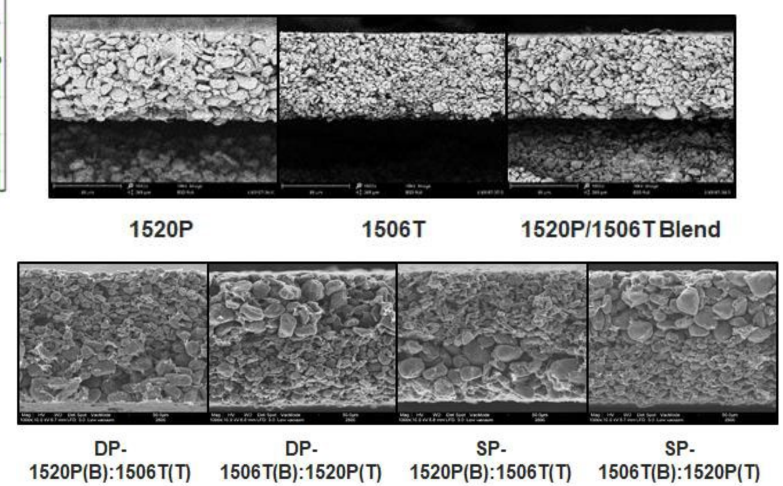
Discharge capacity of cathode active material from representative coin cell sets



For more battery results, refer to poster presentation of Michael Ulsh (NREL lead)

“Roll-to-Roll Manufacturing Science and Applications: Accelerate R2R Materials Manufacture for Energy Storage and Generation”, June 11, 2019

Micrograph of pristine anodes and structured combinations

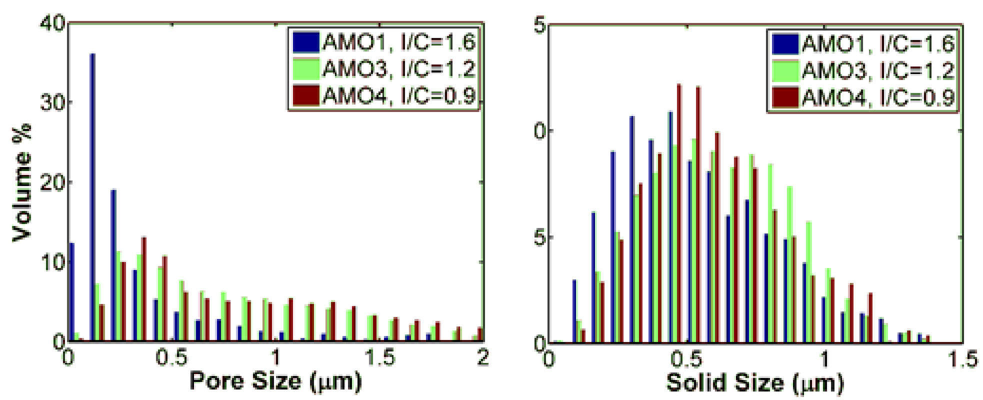


Results and Accomplishments

Full Cell – Nano- and Micro- X-Ray Tomography of Coated Electrodes

Nano-CT study indicates that reactant transport is mostly through the big cracks for the high ionomer content sample and less uniformly distributed than that for the lower ionomer content samples

(a)



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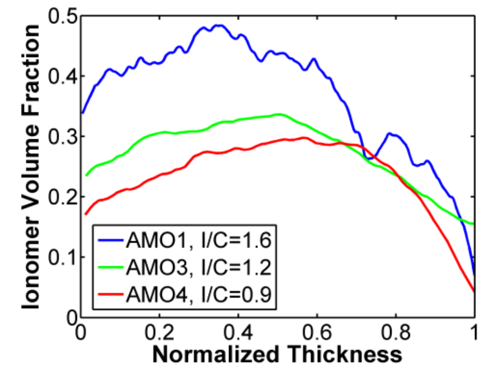

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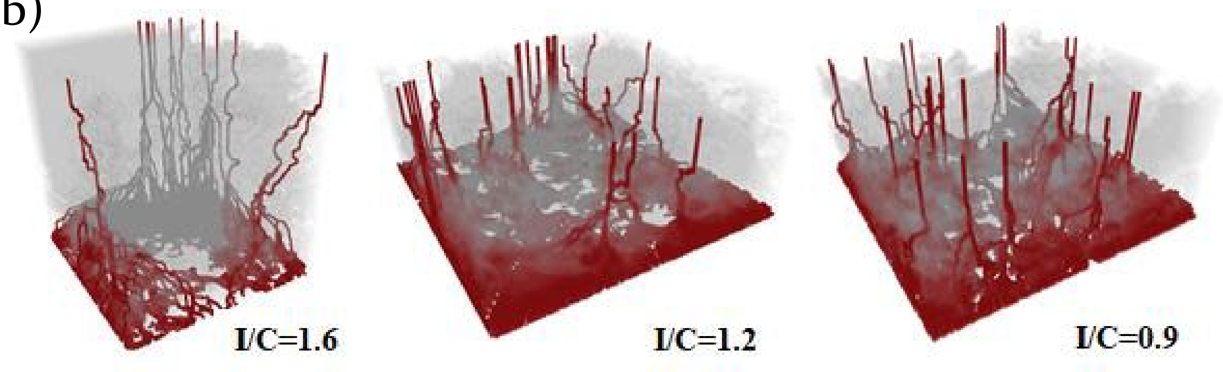


Analysis of GDEs (Gas Diffusion Electrodes) for (a) pore size and solid size distributions of three NREL GDEs with I:C ratios of 1.6, 1.2, 0.9, with (b) the shortest path tortuosity of the GDEs, and for (c) ionomer volume fraction through the thickness of the GDEs (0 thickness is at the membrane interface and 1 is at the GDL interface)

FY 2019

ROLL-TO-ROLL ELECTROSPINNING AS A PLATFORM TECHNOLOGY FOR ADVANCED MEMBRANE MANUFACTURING

Project Objectives

- Develop scalable materials and membranes manufacturing based on R2R electrospinning (ES) technology to enable high-throughput, energy-efficient, and cost-effective production
- Synergize efforts from multiple DOE laboratories to innovate roll-to-roll technologies and its application to multiple application domains
- ANL:
  1. ES recipe development for battery electrolytes, oxide fuel cell electrodes, and functional water filtration membrane applications
  2. Scalable R2R electrospinning design and demonstration
  3. Material and ink characterization using synchrotron-based x-ray techniques, such as small angle x-ray scattering (SAXS)
- NREL:
  1. ES process-window studies with initial focus on energy conversion membranes and active layers
  2. Ink characterization
  3. Transfer key formulations to ANL for R2R scale-up
- ORNL:
  1. ES recipe development for battery electrolytes, oxide fuel cell electrodes, and functional water filtration membrane applications
  2. Scalable R2R ES design and demonstration

Technical Innovation

- Multilayered coating and advanced membranes in demand for a large number of applications, such as ion transport media, electrochemical active layers, catalytic matrices, and water filtration media

Limitations:

- Current materials fabrication and coating process is time-consuming, inefficient, and costly

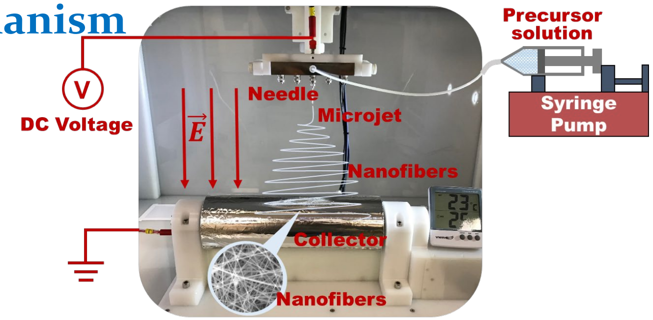
Proposed Approach:

- Develop continuous R2R manufacturing capabilities that are high-throughput, energy-efficient, and cost-effective
- Synergize efforts from multiple DOE laboratories to demonstrate R2R manufacturing on multiple energy materials and membranes
- Critical Innovations:
  1. Scalable R2R ES technique for materials fabrication for multilayered coating
  2. Modeling and simulation of drying/curing process for multilayered coating
  3. Physics, methodologies, and equipment for multilayered coatings
  4. Demonstration of advanced coatings for new applications: battery electrolytes, fuel cell electrodes, and water filtration membranes

Technical Approach

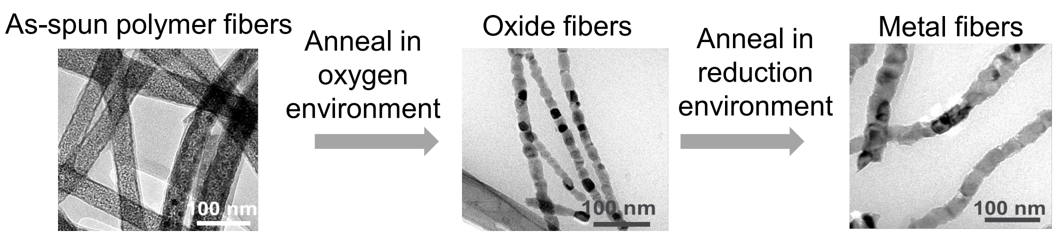
Electrospinning platform technology for nanofiber and non-woven materials fabrication

Electrospinning Mechanism

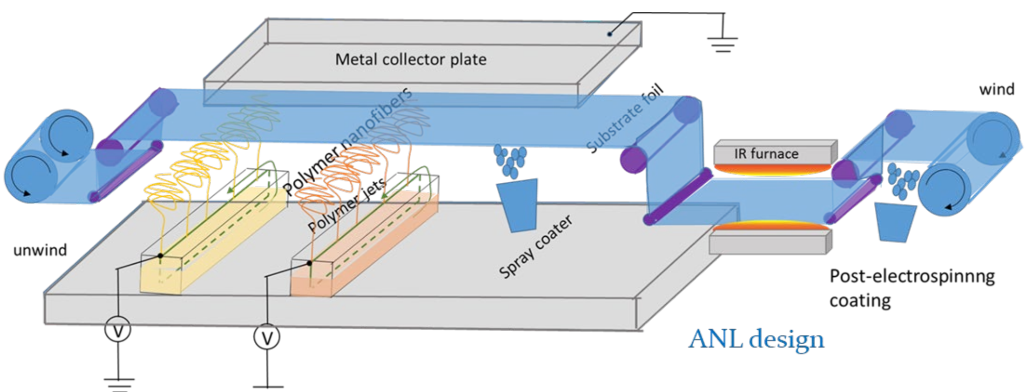


- Electrospinning draws charged polymer solutions through a thin tip under electric field to form nanofiber 3D network
- Thermal treatment converts polymer fiber to oxide or metal fibers

Oxide and metal nanofiber fabrication process



R2R electrospinning, in-line coating and thermal treatment for high throughput membrane manufacturing



ANL design

Technical Approach

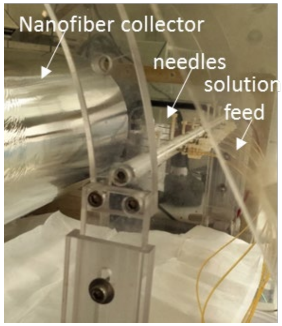
Small-scale electrospinning capability

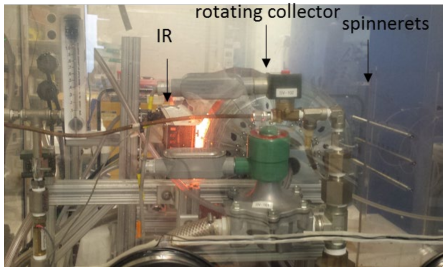
Gen 1. regular electrospinning

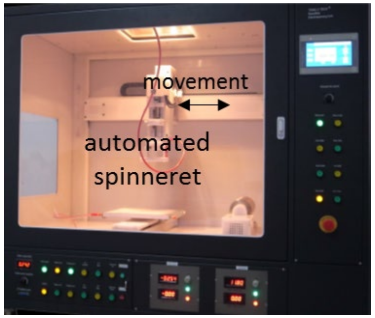
Gen 2. Spinning-annealing all-in-one

Gen 3. Semi-automated

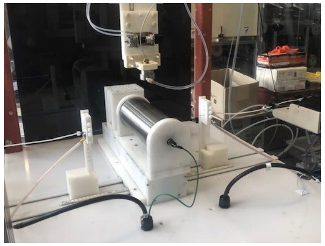
@ ANL



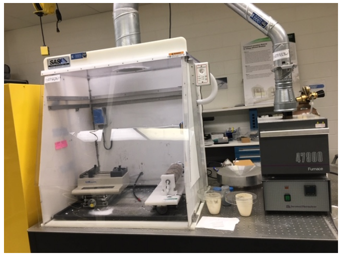




@ NREL



@ ORNL



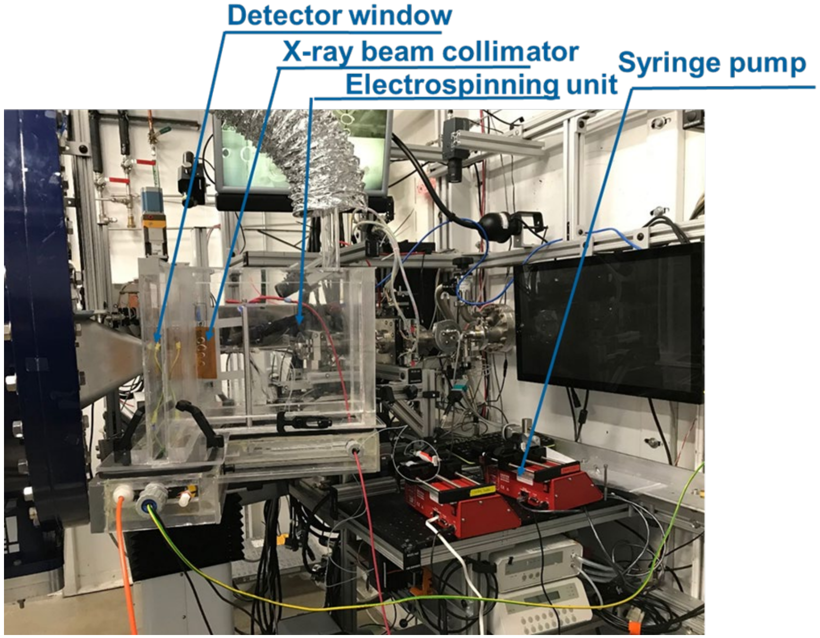
Capabilities:

- Temperature and RH controlled enclosure
- Dual spinning needles with integrated spray
- Coaxial spinning needles
- Multiple spinnerets to mimic production conditions
- Various types of collectors-rotating drum, wheel, mandrel, disk, flat-panel
- Spinning and annealing all-in-one system for oxide and ceramic materials fabrication and scale-up
- Computer control panel and automated spinneret arm for high precision process control

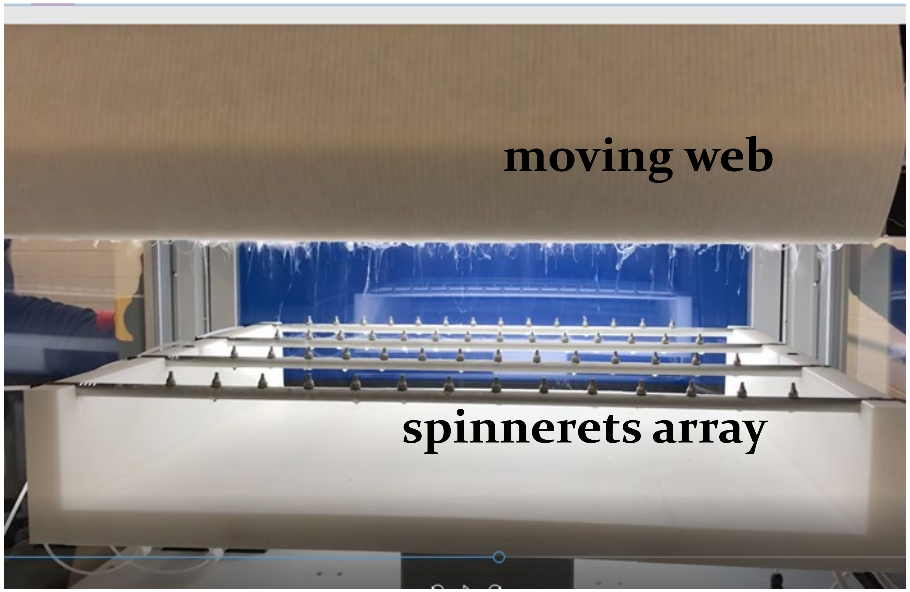
Results and Accomplishments

- Implemented R2R electrospinning capability
- Demonstrated *in-situ* small angle x-ray scattering (SAXS) study of electrospinning process at beamlines

In-situ SAXS of electrospinning at beamlines



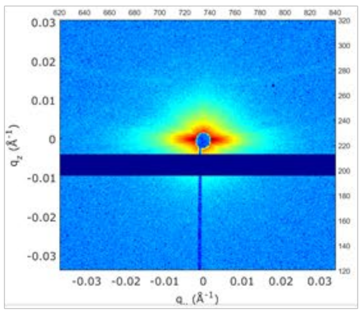
ANL R2R electrospinning module

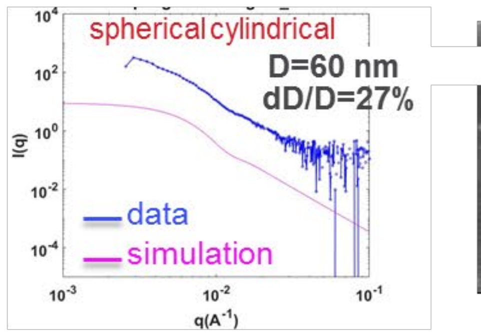


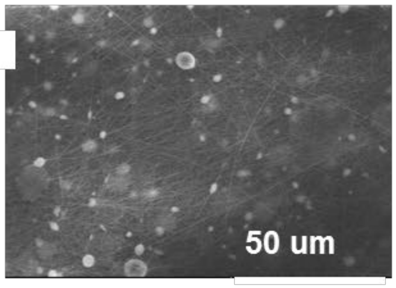
Results and Accomplishments

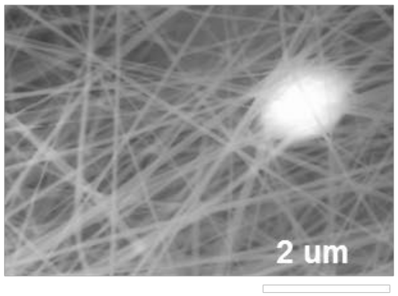
Demonstrated SAXS capability of capturing electrospun nanofibers of different morphologies and characteristics

Sample #1 Electrospinning from low viscosity precursor solution, deposit 60 sec, x-ray exposure 5 sec

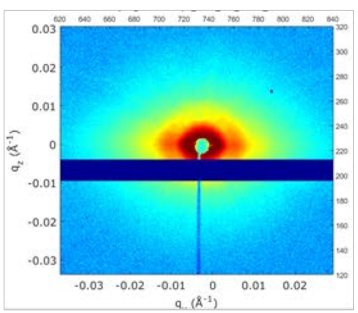


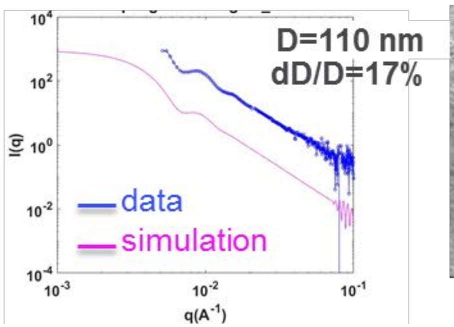


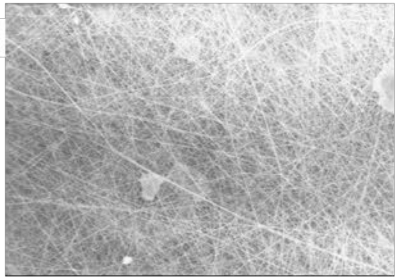


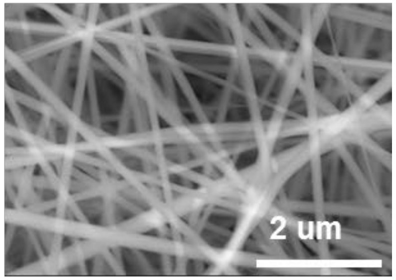


Sample #2 Electrospinning from high viscosity precursor solution, same process conditions





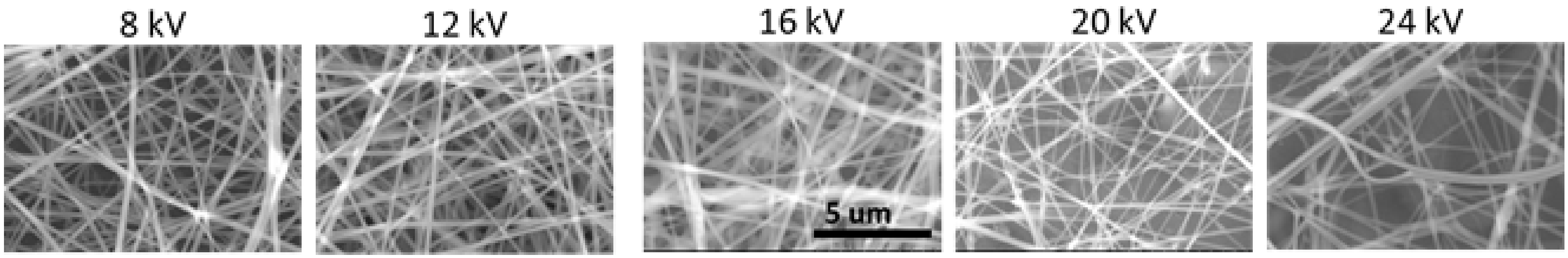




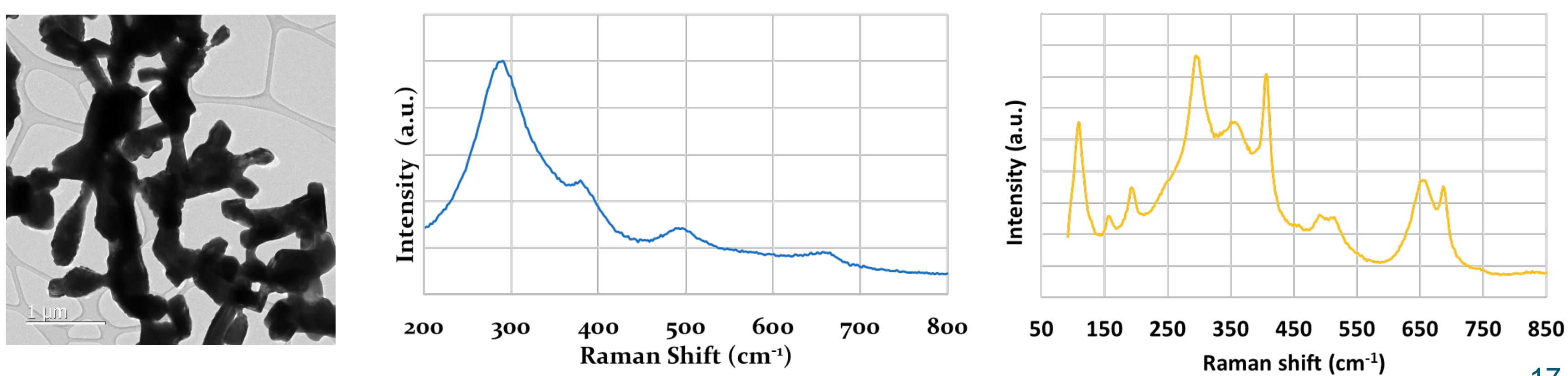
Results and Accomplishments

Developed electrospinning recipe for fabrication of $Li_7La_3Zr_2O_{12}$ (LLZO), for solid-state lithium battery electrolyte applications

SEM images of as-spun LLZO precursor nanofibers synthesized at different voltages



TEM image (left) and Raman profiles of cubic (middle) and tetragonal (right) phase LLZO nanofibers annealed at 900 °C



Results and Accomplishments

Aqueous solution

Precursors used
Zirconium nitrate
lithium nitrate
lanthanum nitrate
Polyvinylpyrrolidone

Dissolved in water and acetic acid
Feed rate: 0.16 mL/h
Voltage: 18 kV
Working distance: 13 cm

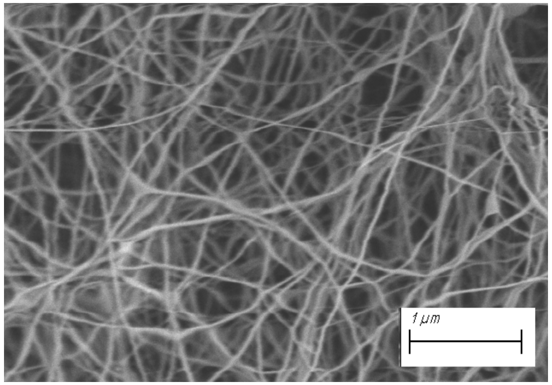
Non-aqueous solution

Precursors used
Zirconium isopropoxide
lithium nitrate
lanthanum nitrate
Polyvinylpyrrolidone

Dissolved in DMF and acetic acid
Feed rate: 0.16 mL/h, 1.38 mL/h
Voltage: 10 kV, 18kV
Working distance: 15 cm

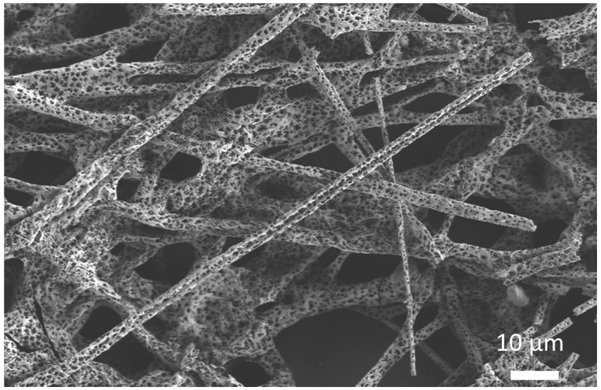
$Li_7La_3Zr_2O_{12}$ nanofibers

Water-based fibers

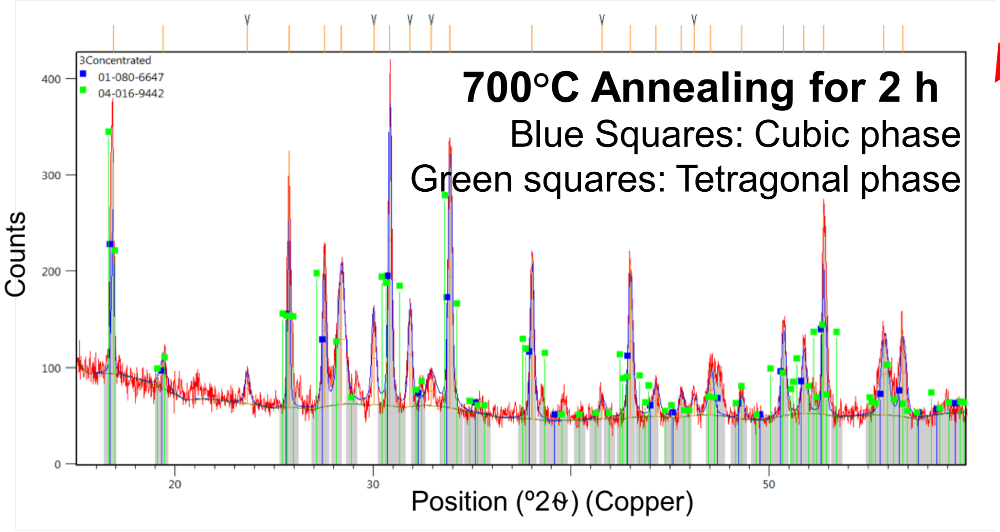


50-100 nm
Feed rate = 0.16 mL/h

DMF-based fibers



1-5 μm
Feed rate = 1.38 mL/h

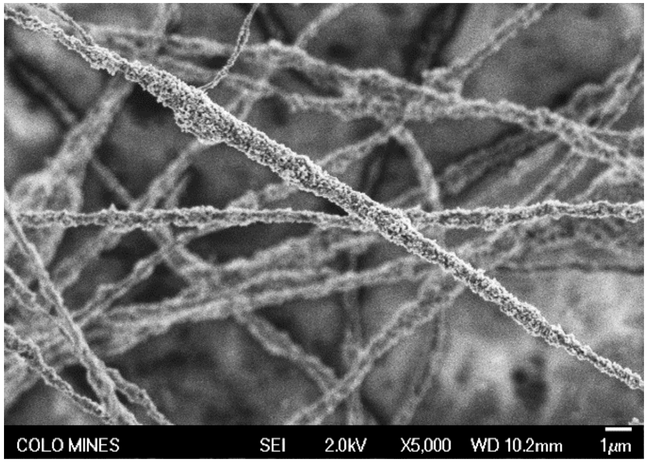


Results and Accomplishments

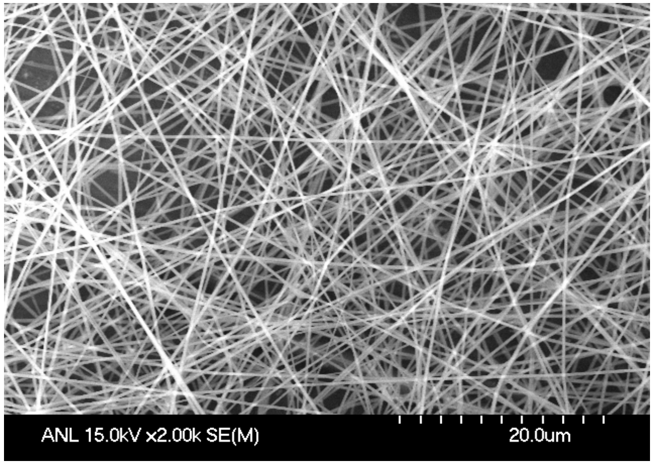
Developed electrospinning recipe for fuel cell materials

SEM images of as-spun nanofibers of polymer-based (left) and oxide-based (right) fuel cell materials

Polymer-based



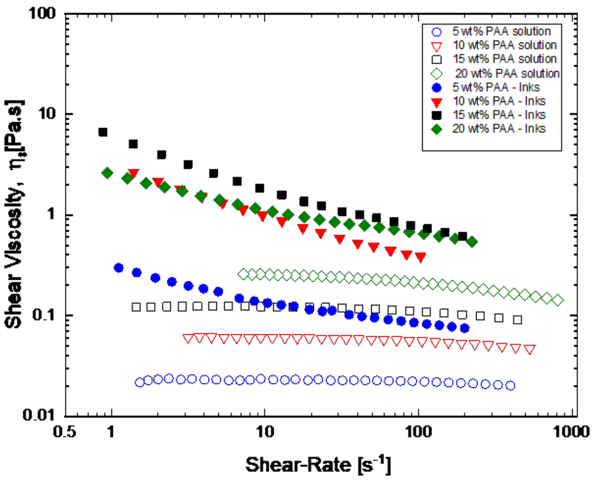
Oxide-based
(nominal composition $La_{0.6}Sr_{0.4}Co_{0.2}Fe_{0.8}O_3$)

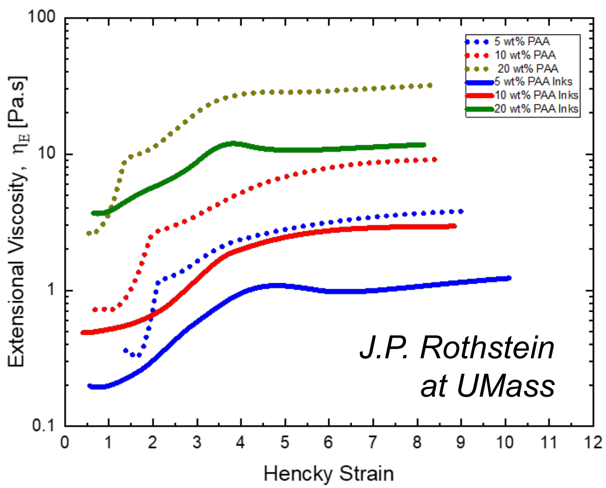


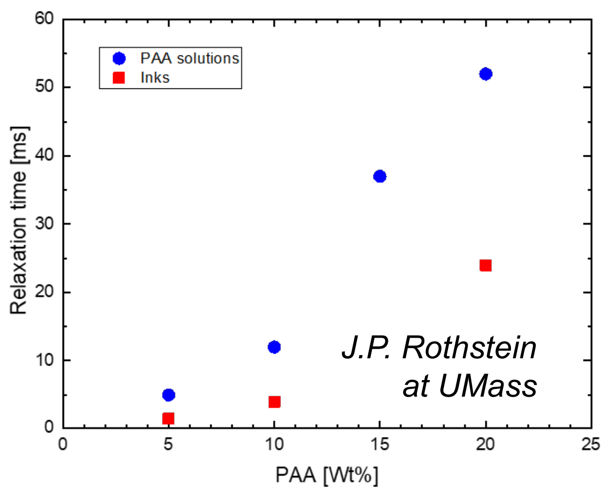
Results and Accomplishments

Shear and extensional rheology of PAA solutions and particle-containing inks

- Shear: Increased viscosity with particle addition indicates polymer-particle interactions at low shear
- Extensional: However, in extension, these interactions are reducing the number of polymer chains available to form the filament, thus reducing extensional viscosity
  - Extensional viscosity ~100x shear viscosity
  - Relaxation time increases with increasing PAA wt% and decreases with particle addition







Shear (left) and extensional (center) viscosity, and relaxation time (right) of PAA & PAA + particle inks