Roll-to-roll Advanced Materials Manufacturing Lab Consortium

Consortium Lead: Claus Daniel (ORNL)
Email: danielc@ornl.gov; Phone: 865-946-1544
National Renewable Energy Laboratory Lead: Michael Ulsh
Argonne National Laboratory Lead: Gregory Krumdick
Oak Ridge National Laboratory Lead: David Wood
Lawrence Berkeley National Laboratory Lead: Ravi Prasher

AIMCAL R2R Conference
Tampa, Florida
October 16, 2017

OAK RIDGE National Laboratory
Argonne NATIONAL LABORATORY
R2R
BERKELEY LAB
NREL
NATIONAL RENEWABLE ENERGY LABORATORY

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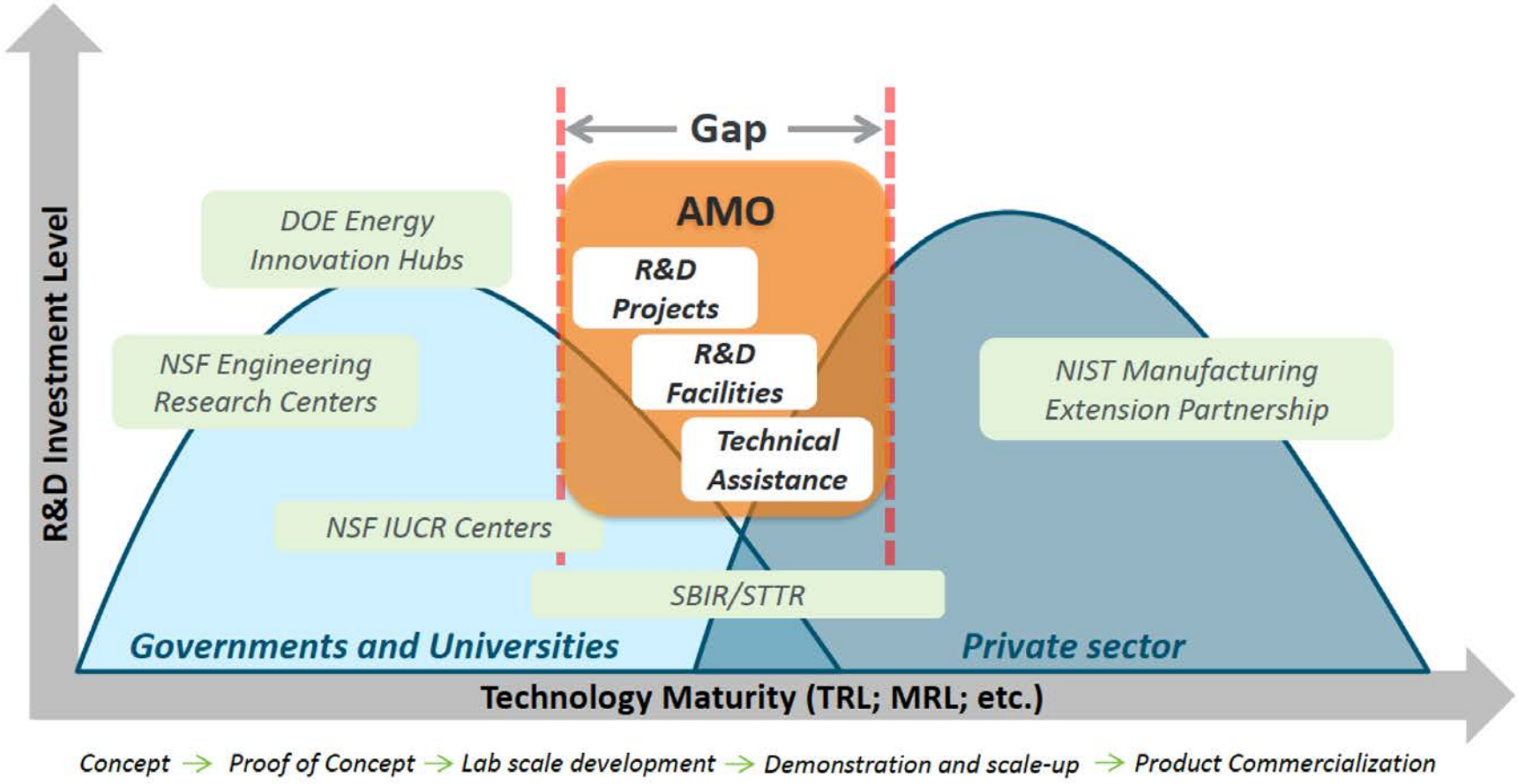
Outline

- Introduction to DOE Advanced Manufacturing activity
- R2R Advanced Materials Manufacturing Consortium
  - Goals
  - Approach
- Lab capabilities
- Opportunity to collaborate

Taking Materials to Products: A Big Challenge

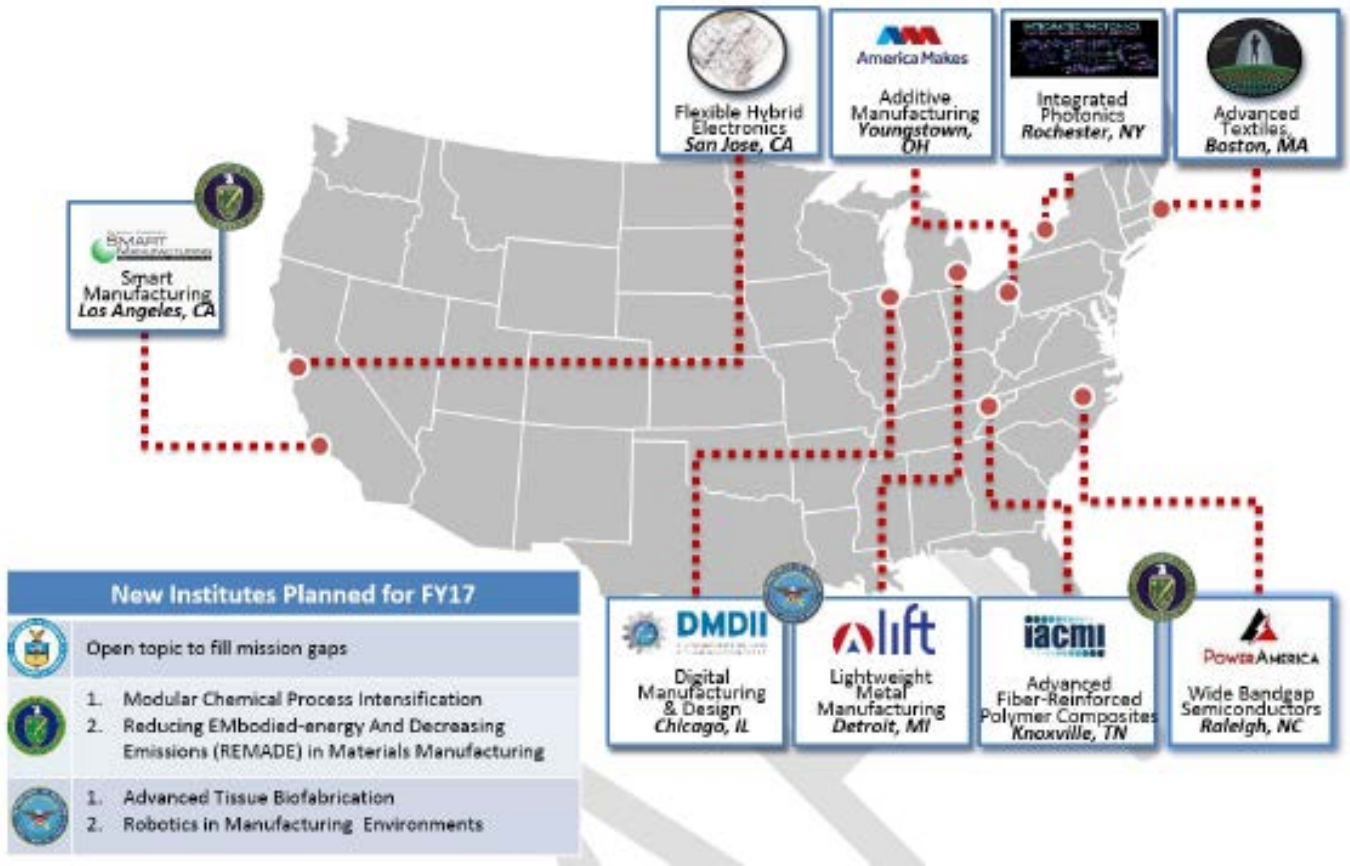


- The DOE Advanced Manufacturing Office (AMO) is interested in bridging the gap between R&D investment and private sector investment, especially regarding processing technologies

AMO mission and organization

- AMO supports the development of manufacturing technologies via:
  - R&D Consortia
  - R&D Projects
  - Technical Assistance

Mission: Catalyze research, development and adoption of energy-related advanced manufacturing technologies and practices to drive U.S. economic competitiveness and energy productivity.



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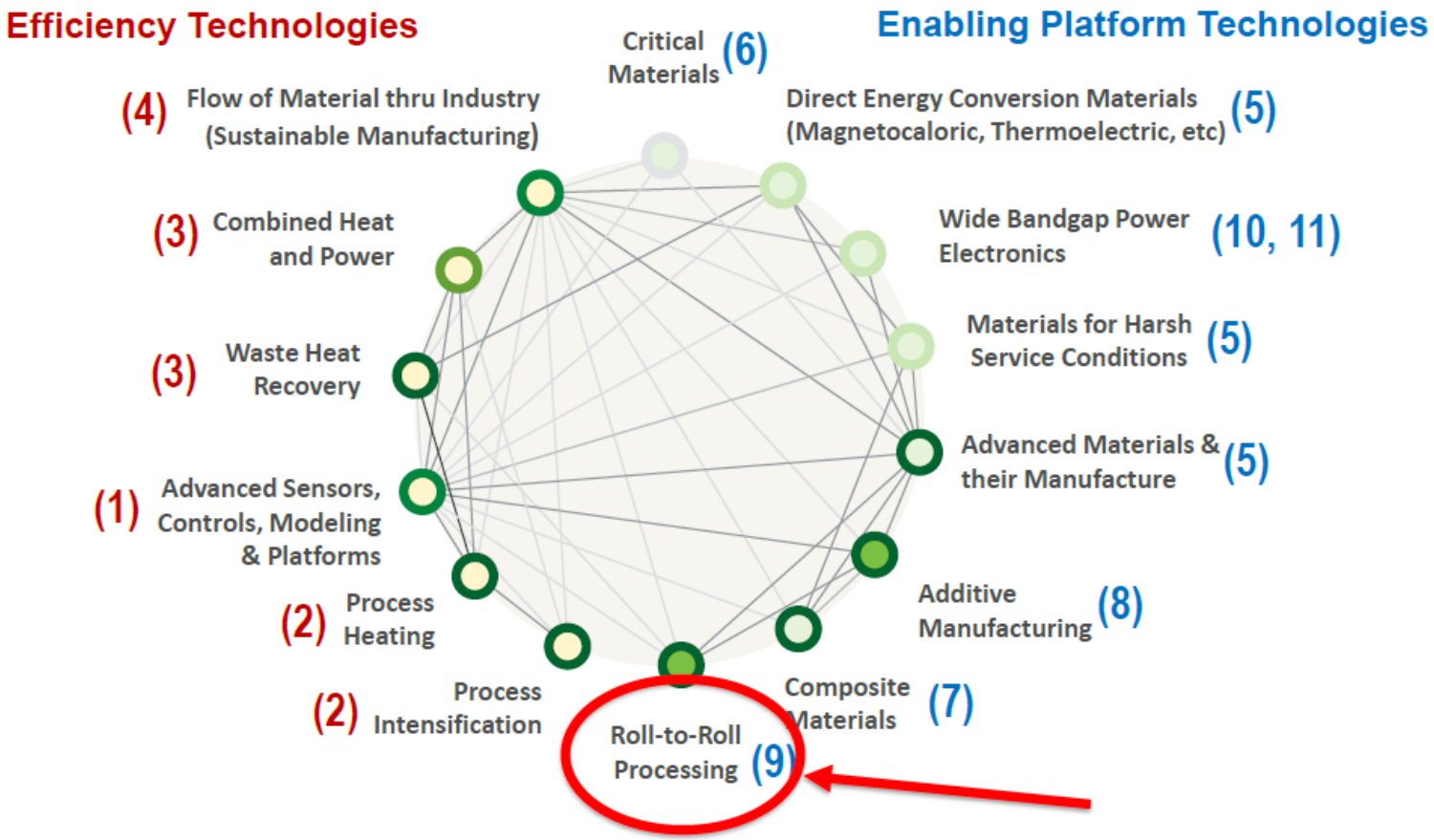
DOE Critical Manufacturing Technologies: R2R

- Roll-to-roll manufacturing is identified as a critical technology because of its relevance to many clean energy technologies
- DOE seeks to support R&D related to R2R manufacturing that industry can't or wouldn't support by itself



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Breadth of technologies related to DOE mission that are relevant to R2R

Solar PV Cell

Carbon Fibers

Light Emitting Diodes

Electro-Chromic Coatings

Membranes

EV Batteries

Multi-Material Joining

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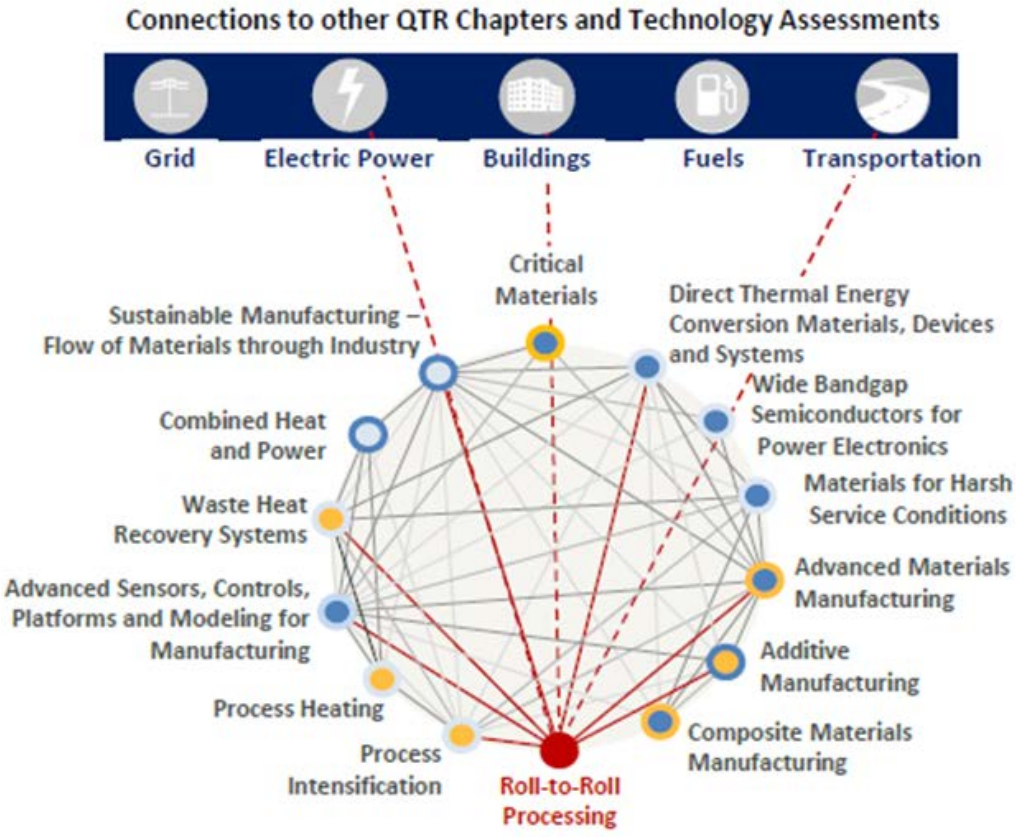
R2R evaluated in DOE quadrennial technology review



Key Extra-Chapter Connections

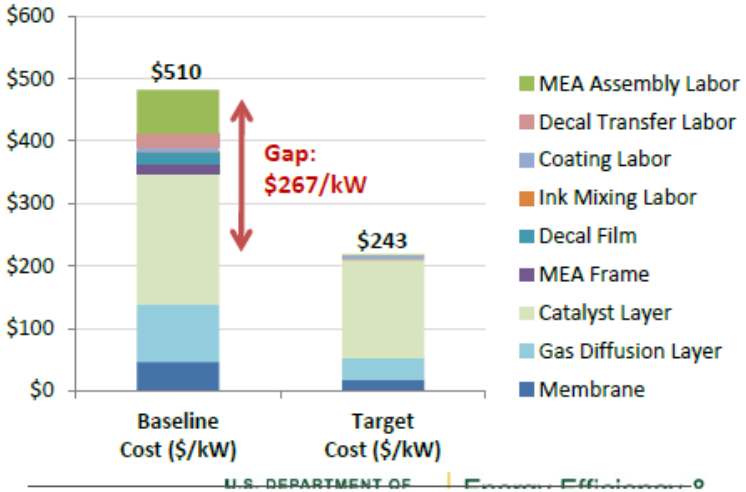
- **Electric Power:** *flexible solar panels*
- **Buildings:** *window insulation films*
- **Transportation:** *battery electrodes*

Ch. 6 – Roll-to-Roll Processing Technology Assessment

Scope

- Roll-to-roll (R2R) applications such as flexible solar panels, printed electronics, thin film batteries, and membranes
- Deposition processes such as evaporation, sputtering, chemical vapor deposition, and atomic layer deposition
- Metrology for inspection and quality control

Strategy for meeting cost targets for automotive fuel cell membrane electrode assembly using roll-to-roll processing techniques*



*Source: Manufacturing Fuel Cell Manhattan Project, presented by the Benchmarking and Best Practices Center of Excellence, Office of Naval Research, ACI Technologies, 2012.

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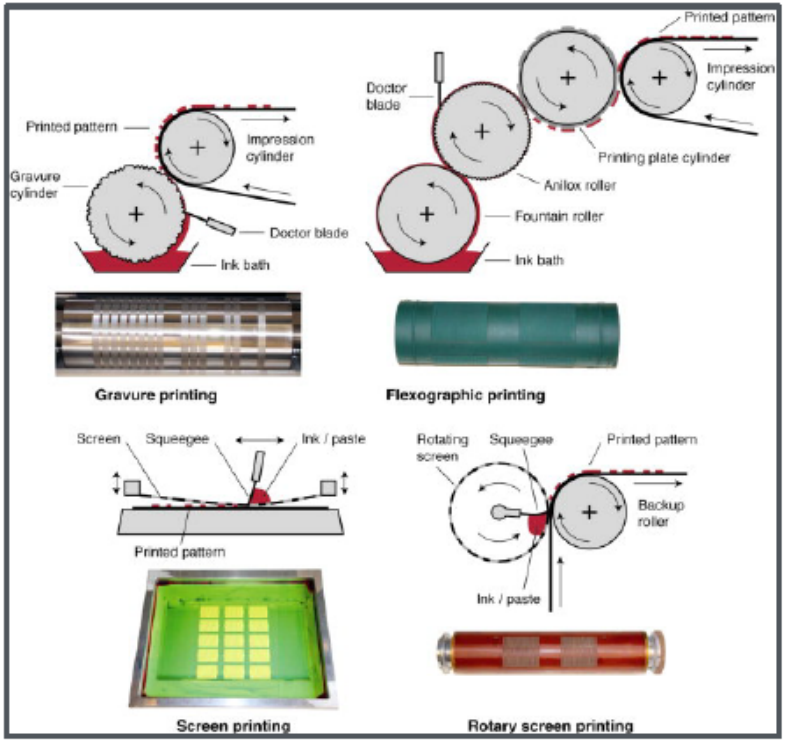
DOE motivation for R2R

Motivation

Ultra-high-quality, high-throughput, energy efficient roll-to-roll manufacturing processes that are cost-competitive (High Value Roll-to-Roll) can both **enable advanced clean energy applications**, particularly in the nanomanufacturing sector, as well as **capture significant energy savings** compared to traditional manufacturing processes.

Challenge

Although R2R processes have been around for more than forty years, rapid evolution of use, application and require dramatic improvements in quality, feature size, consistency, metrology and process modularity in order to maintain cost competitive. DOE is focused on identifying the key technologies and processes that will **unlock high-value roll-to-roll across a number of industries**.



R2R Processing diagrams for organic electronics/thin films

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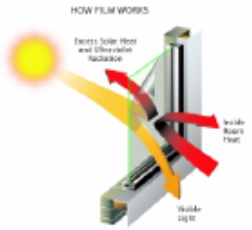
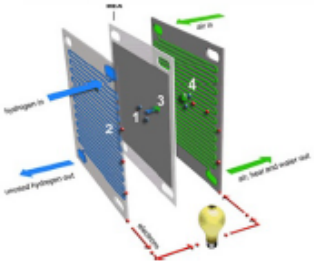
AMO R2R Workshop: Breakout topics and key research areas

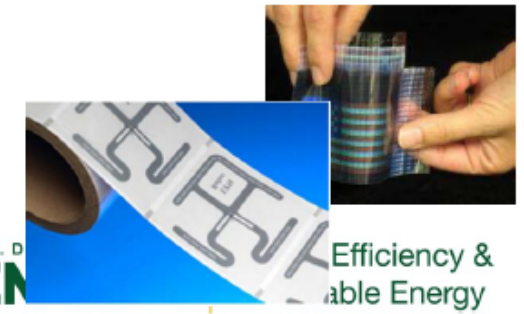
- Advanced Deposition & Processing
  - Low temperature processing
  - Multi-scale processes
  - Scalability of new processes
  - Printing dense and vertically integrated devices
  - In-line QC
- Metrology & Quality Systems
  - QC technique development
  - Integrations with process control
- Membranes & Substrates
  - Materials and substrates
  - Membrane design and fabrication
  - Metrology and process control
  - Scalability
- Transitioning to R2R
  - From batch
  - From plate-to-plate
- Continuous Processing
  - Scale-up and commercialization
  - Predictive modeling and computational tools
  - Multi-layer deposition
  - Process monitoring and control

AMO R2R Workshop: Applications

- Multilayer capacitors (MLC, i.e. NPO to XR7/Relaxer/etc.)
- Thick and thin-film substrates (Al2O3, AlN, Si3N4, SiC, GaN, MgO, ZrO)
- Thick-film sensor materials (temperature sensors, positioners, transducers, e.g. negative temperature coefficient thermistors, Piezoelectric/lead zirconate titanate (PZT), active/passive, selective gas)
- Fabric (clothing textiles, fiber reinforce mat/fiberglass/carbon/polymer)
- Anti-static, release, reflective and anti-reflective coatings (glass, MylarTM, polyethylene)
- Barrier coatings (thermal and environmental)
- Fuel cells (laminar solid oxide fuel cells (SOFC), proton exchange membranes (PEM), membrane electrode assemblies and gas diffusion media)
- Batteries (laminar Li ion, etc.)
- Flexible electronics for displays, heaters, sensors, circuit substrates, consumer appliances, etc.
- Metal ribbon (transformer "coils", etc.)
- Paper industry
- Chemical separation membranes (RO, catalyst)
- CIGS Photovoltaic (PV) and other flexible PV products





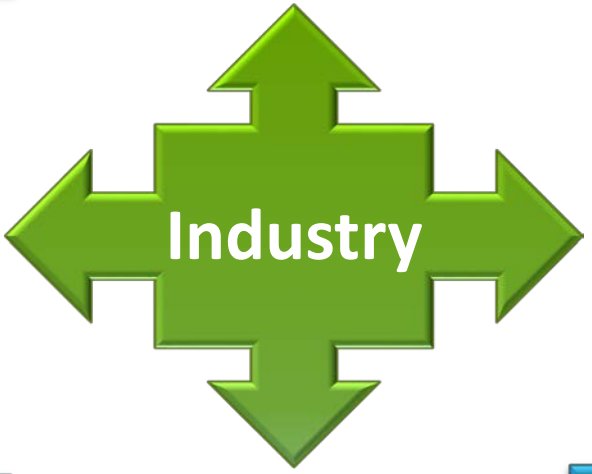


Why a National Laboratory consortium?

Materials synthesis
Device assembly and testing

OAK RIDGE National Laboratory

Materials processing
Materials characterization



Argonne NATIONAL LABORATORY

NREL
NATIONAL RENEWABLE ENERGY LABORATORY

Modeling and simulation of process conditions and resulting performance

BERKELEY LAB

Metrology of coatings
Understand morphology and porosity

- Utilization of combined national lab resources to solve complex problems

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Lab-Industry partnership created with Eastman Business Park

- Objective is to ultimately provide a scale-up path to industry partners who don't have access to pilot/production equipment

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DOE Goals for R2R Consortium

- Enabling R2R potential
  - Low manufacturing costs
  - Low energy processes
  - High volume production
  - High throughput due to thinner membranes
  - Compatible with many material platforms
  - Large areas
  - Varying feature sizes and dimensions
- Goals depending on technology area
  - Increase throughput by 5x and reduce production footprint
  - Reduce energy consumption by 2x
  - Increase production yield by 2x
  - Enable substantial shift of manufacturing to the United States by assisting in the development of a domestic supply chain

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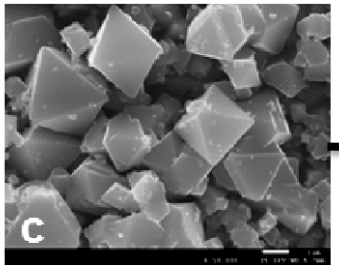
Consortium approach

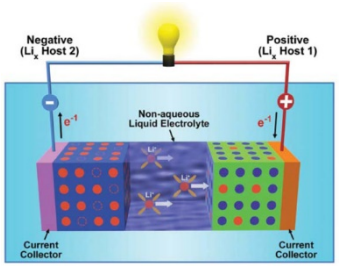
Synthesis

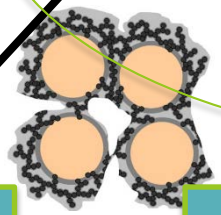


Processing Science

Mixing → Coating → Drying → Calen-dering

Device Physics





Colloidal interactions → Rheology, Temperature → Mechanical strength Contact resistance, porosity

How do we link materials processes to device performance?

- Elucidate particle-polymer-solvent interactions in active layer ink formulation and mixing, to improve coating throughput, uniformity and quality
- Understand the physics of scalable coating and drying/curing processes and how they affect active layer morphology and device performance
- Determine the impact that process-based defects in active layer and substrate materials have on performance and lifetime of devices
- Study material-excitation interactions to facilitate the development of real-time instrumentation and measurement of active layer uniformity and properties

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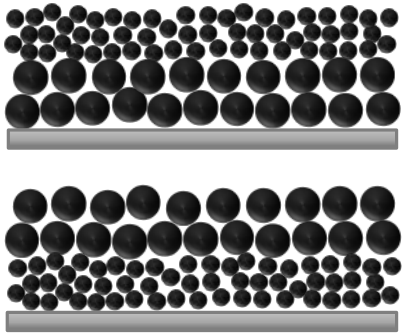
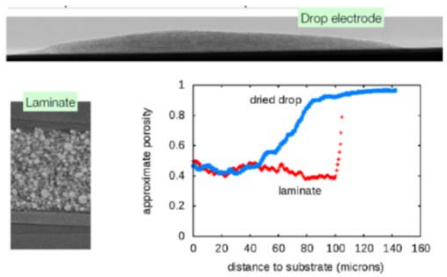
From theory to full device with understanding of defects and performance limitations

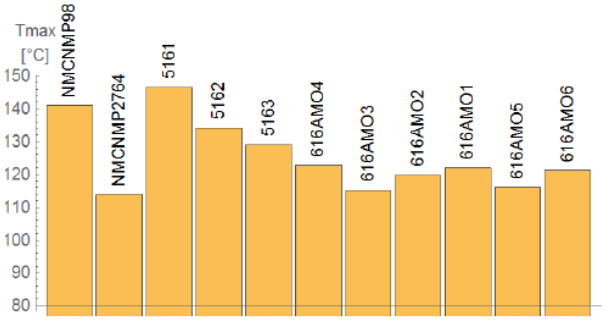
R2R processing and materials assumptions



Modeling, simulation, and in-situ observation



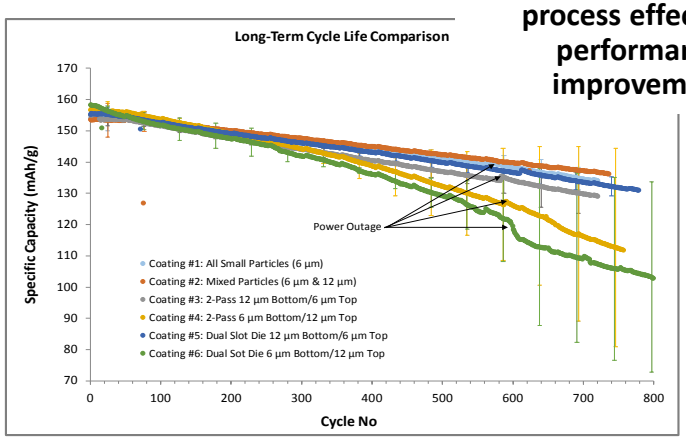
V. Srinivasan et al. J. ECS (2017)



Process validation
Defect and coating analysis

Device fabrication and testing

Understanding of process effect and performance improvement



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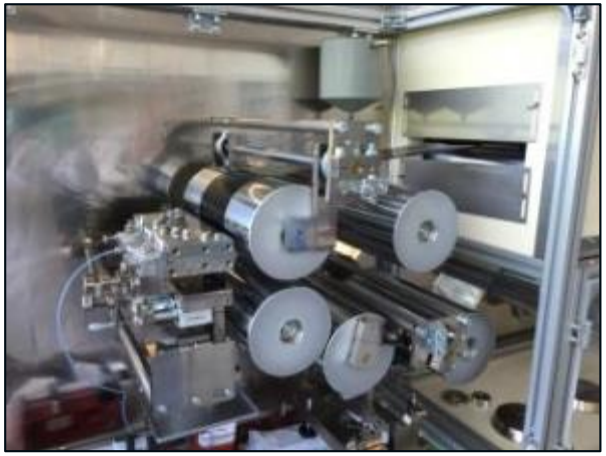
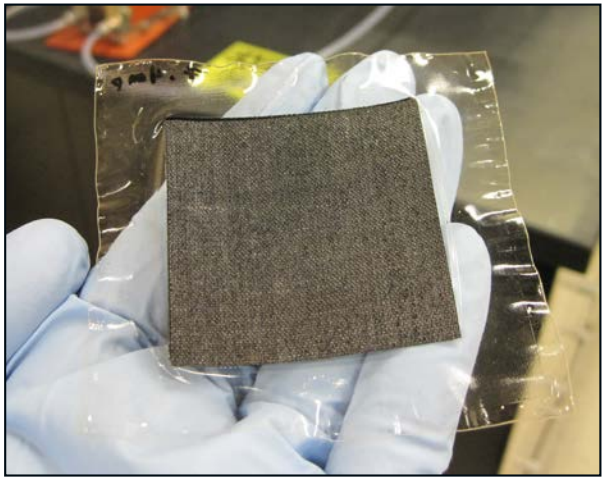
Leveraging and coordinating capabilities across the labs

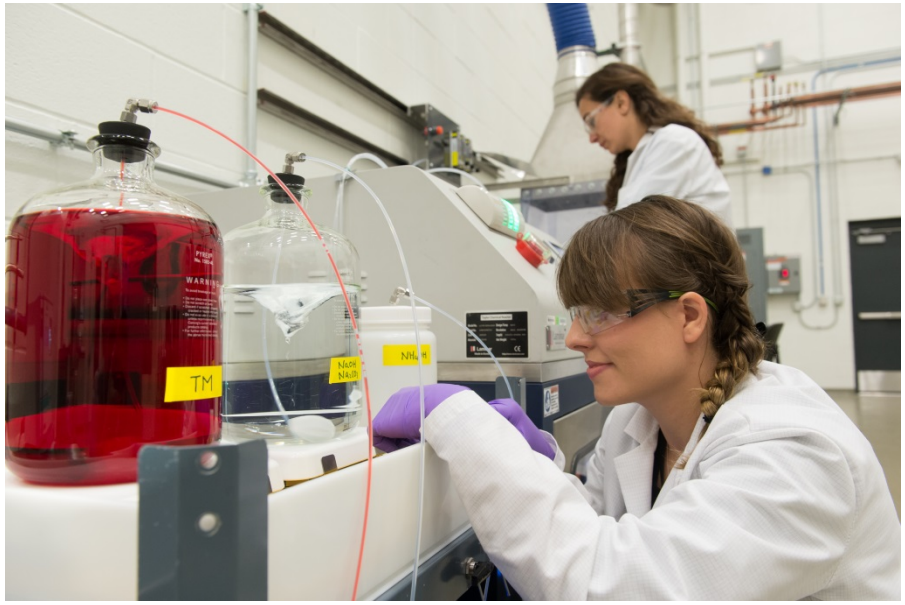
1. **[NREL]** Explore phase-separation and other single-coating-layer methodologies to achieve an ionomer-rich surface on the GDE electrode using slot and/or micro-gravure coating
2. **[ORNL]** Explore dual-slot coating of electrode/ionomer to achieve a similar structure
3. **[LBNL]** Develop and provide flow visualization and process modeling, under conditions relevant to the processes being explored by NREL and ORNL, of single and bi-layer electrode ink structures, with a focus on particle-ionomer interactions
4. **[ANL]** Provide USAXS characterizations of inks under different ultrasonic and shear mixing conditions
5. **[ANL]** Provide high-throughput exploration of ink synthesis parameter space, as necessary, based on initial formulation studies at NREL and ORNL
6. **[ANL]** Provide nano- and/or micro- x-ray tomography of coated electrodes
7. **[ORNL]** Provide high-resolution microscopy of coated electrodes
8. **[NREL]** Make MEAs from the GDE sheets made in 1 and 2 using standard methods
9. **[ORNL]** Explore roll lamination of GDE sheets (made in 1 and 2) and membranes using the calender
10. **[NREL, ANL]** Test hot-pressed and calendered MEAs for performance

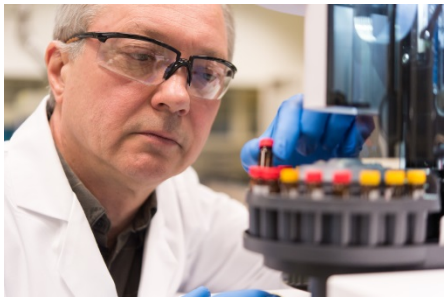




ANL: Materials Engineering Research Facility (MERF)

- Develop scalable manufacturing processes
- Evaluate emerging manufacturing technologies
- Develop analytical methods and quality control procedures – establishing materials specifications
- Make kilogram quantities of the material available for industrial evaluation and further research





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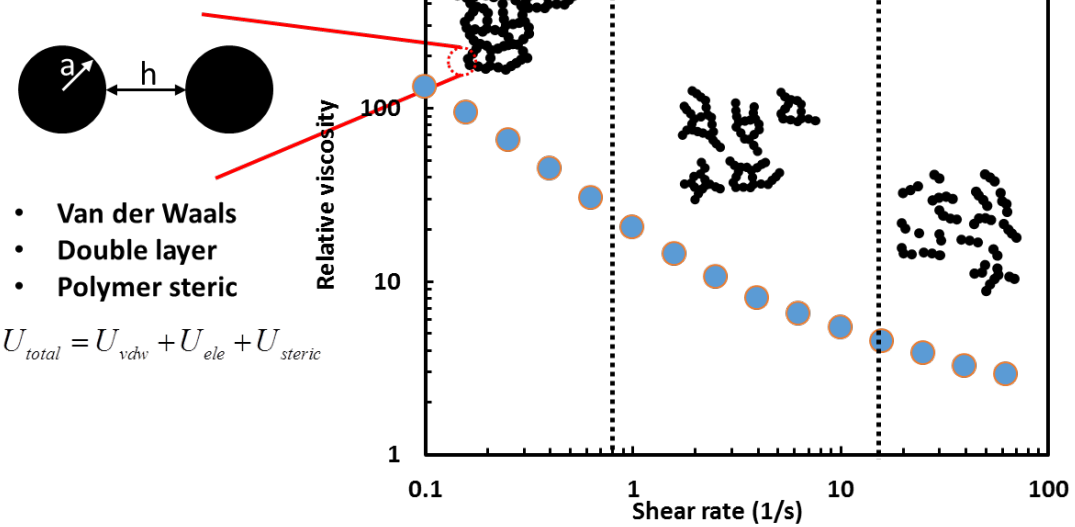
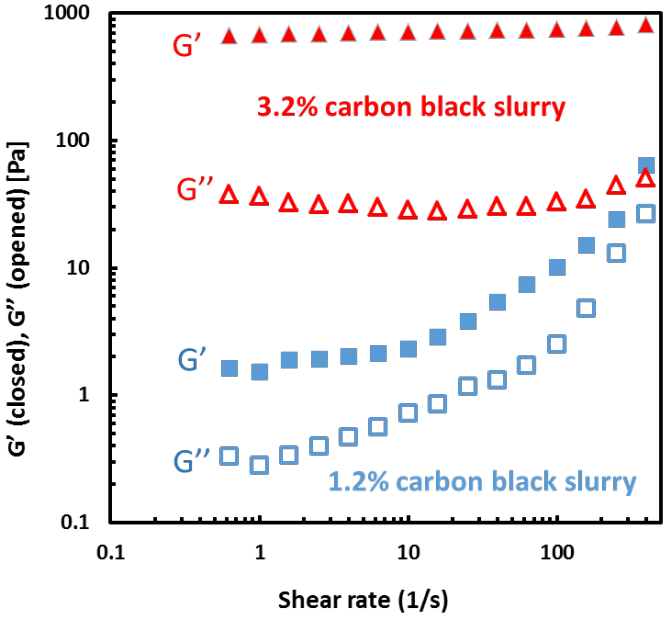
LBNL: Fundamental ink modeling and characterization

Colloidal interactions → Microstructure → Rheology → Drying → Mechanical properties





- Study: Role of colloidal interactions in Li-ion battery fabrication

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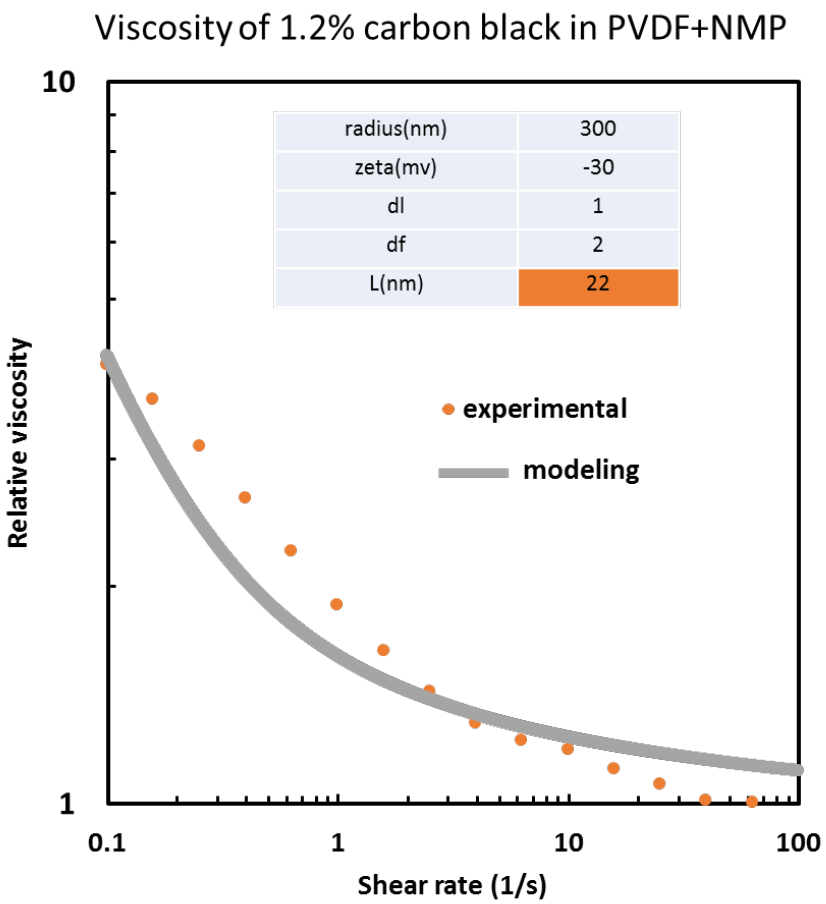
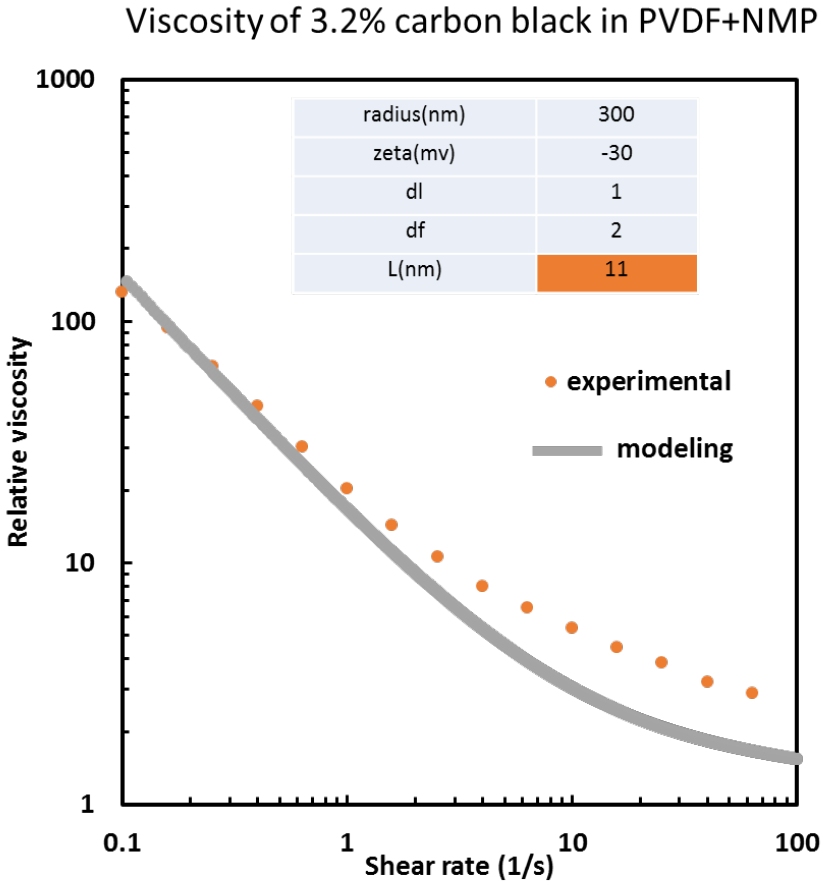
LBNL: Fundamental ink modeling and characterization





- Study: Role of colloidal interactions in Li-ion battery fabrication

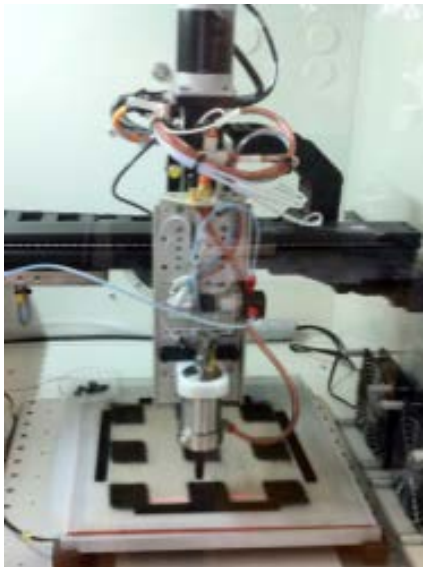
ANL: Polymer extruder R2R pilot plant

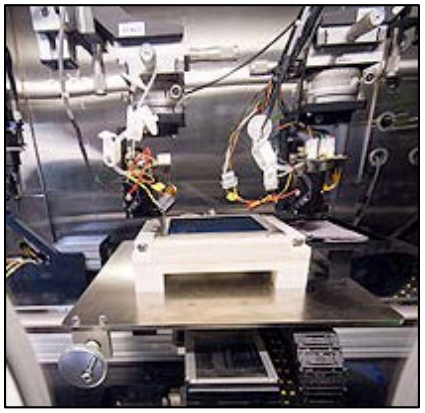
- Can produce experimental polymer rod or sheet with specified solids loading

NREL: Understanding scaling science and process-performance relationships

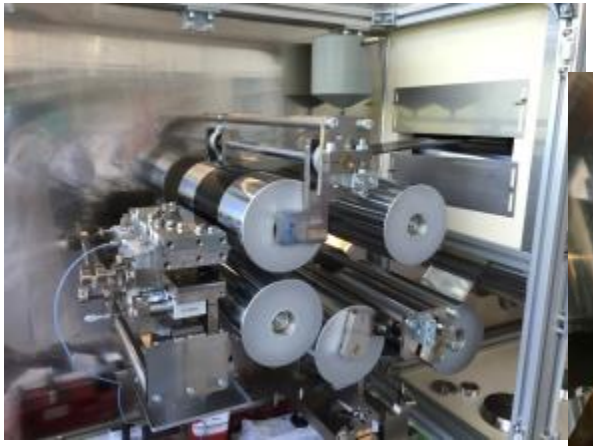
Understanding Process Science

- Synthesis of active materials
- Ink and solution formulation and properties
- Substrate-coating interactions
- Material-process-performance relationships
- Gradient structures
- Scalability of new multi-layer, multi-functional structures
  - Explore pathways to low cost using industrially relevant processes





Ultrasonic and jet spraying



Fuel cell electrode

OPV BHJ layer

Processing Capabilities

- Small-scale solution processing
  - Formulation, mixing, viscosity, rheometry
- Small-scale coating
  - Spin, knife, rod
- Spray coating
  - Ultrasonic, aerosol jet, ink jet, electro-spin/spray
- R2R coating
  - Slot die, gravure

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ANL: Cell Analysis, Modeling and Prototyping (CAMP)

- Coat and hot press electrodes
- Fabricate xx3450 pouch and 18650 cells
- Electrochemical evaluation for electrode matching

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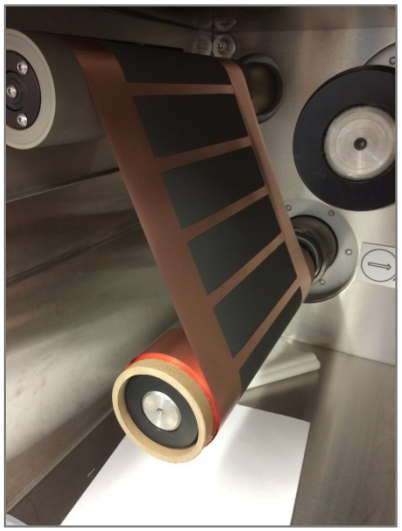
ORNL: Pilot-scale active layer processing

Planetary Mixer (≤2 L)

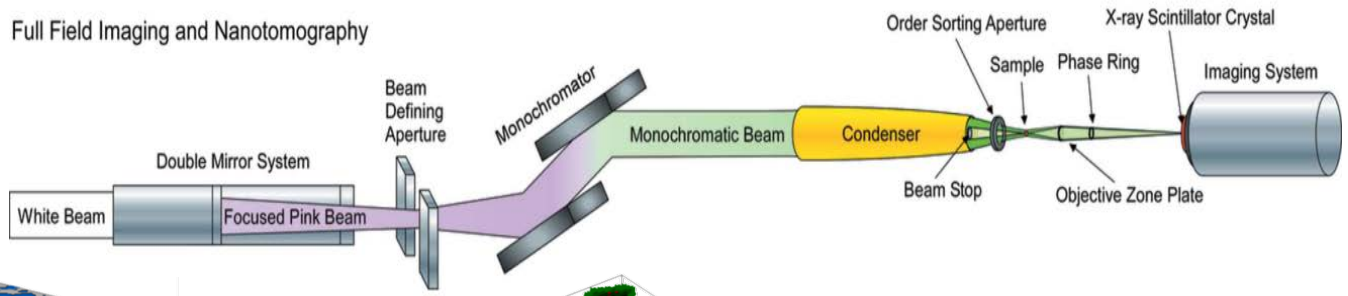
Corona Plasma Treater

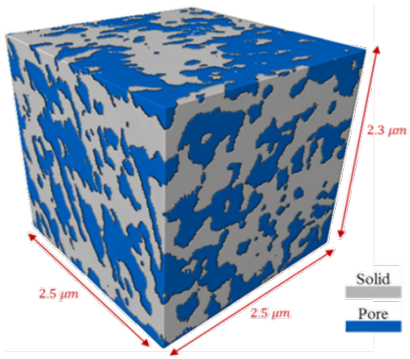
Heated Calender (80,000 lb_f)

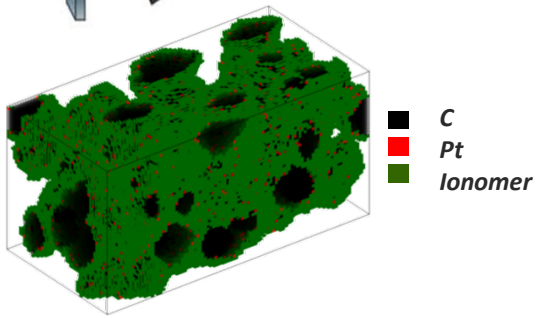


Patch Coating

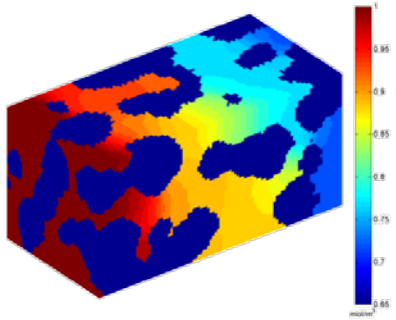
ANL: X-ray tomographic structural characterization of active layers



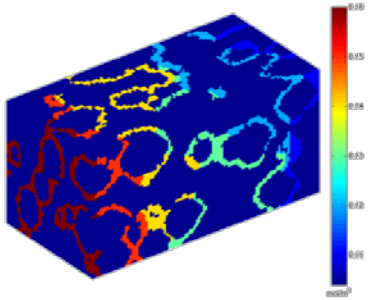




Advanced Photon Source nanoprobe beamlines 26-ID and 32-ID



O_2 Concentration in Pores



O_2 Concentration in Electrolyte

- Hard X-rays can penetrate and image the full thickness of active layers
- Reconstructed images can be used to determine structural properties limiting performance

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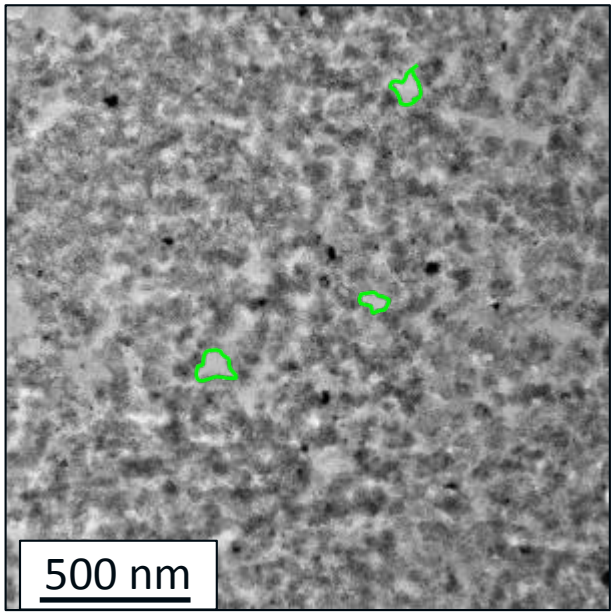
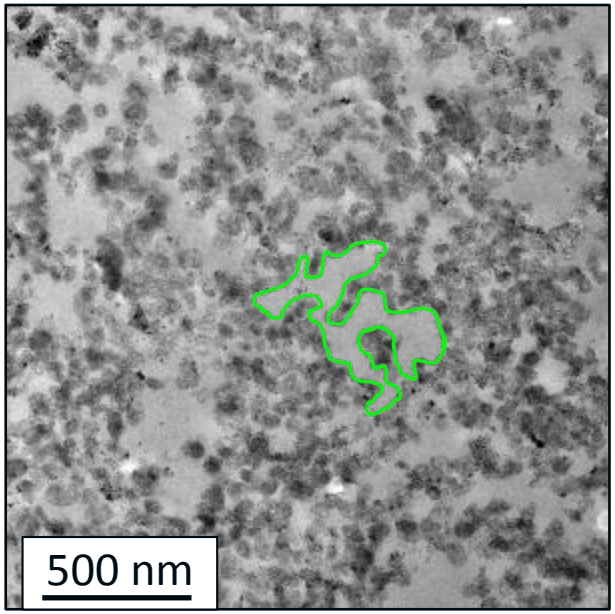
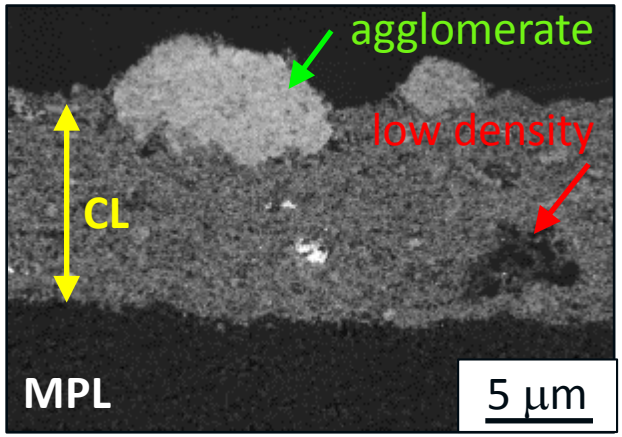
ORNL: High resolution electron microscopy

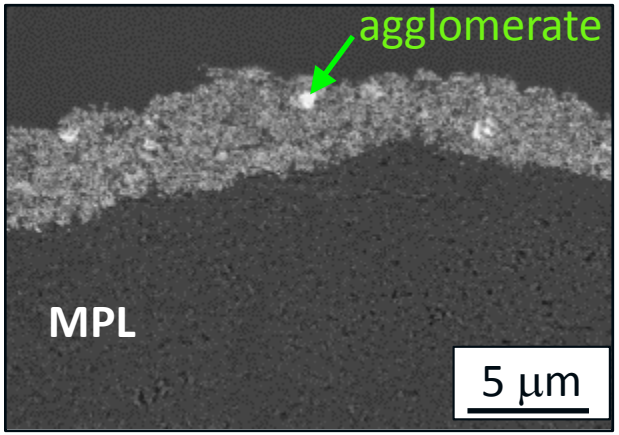
Coating #1



Coating #2







- TEM imaging of nano-scale morphology of coated active layers

NREL: Application-specific device testing

- Understand materials-interfaces-process-performance relationships
- E.g., fuel cells, electrolysis, solar, window films, HVAC membranes
- Performance, durability, lifetime, reliability, efficiency
- Accelerated aging

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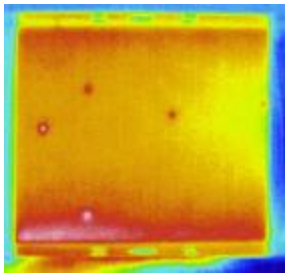
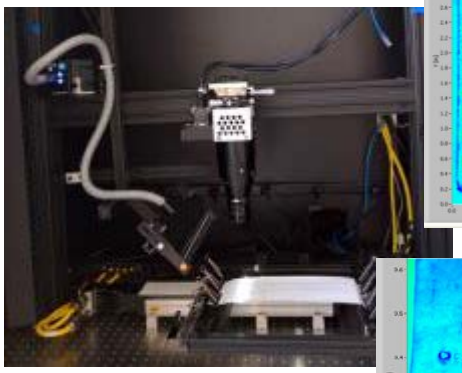
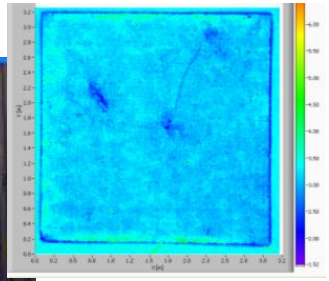
NREL: In-line quality diagnostic development

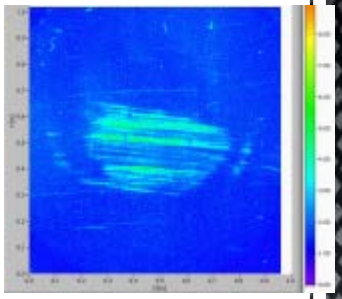
- Large clean energy material portfolio, e.g. fuel cell, battery, and electrolyzer materials
- Membrane defect imaging and thickness mapping
- Active layer uniformity
- Thru-plane defects, e.g. shorting and gas crossover
- Property measurement, e.g. porosity
- Optical and IR diagnostic platforms
- Non-destructive, 100% inspection

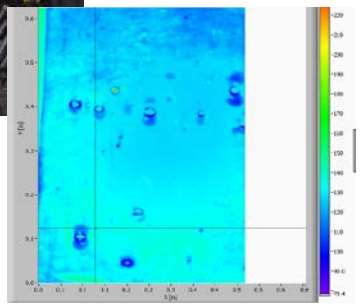


Infrared imaging of electrode and cell defects









Optical reflectance imaging of membrane and electrode defects

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Development assistance opportunity

- Roll-to-Roll (R2R) Advanced Energy Materials Manufacturing – FedBizOpps.gov, Solicitation Number: ORNL-R2RAMM-2017-02-02

CRADA solicitation

- Industry-proposed work
- Work done at the labs, funded by DOE
- 50% cost share requirement
- Standard (no negotiation) cooperative research and development agreement (CRADA)
  - Protects IP and proprietary information
- Selection criteria
  - Potential change in MRL due to proposed barrier being removed
  - Technology alignment with EERE mission and consortium goals/capabilities
  - Impact of primary metrics of success: throughput, energy, yield
- 10-18 month project duration

CRADA solicitation: examples for collaboration with the consortium

- Development of processes
  - Synthesis
  - Coating/deposition
  - Drying/curing
  - Thermal/treatment
- Application of new materials to R2R
  - Use of lab facilities for small-scale testing
- Development of material-specific inspection techniques
- Application of advanced and high-energy characterization tools
  - Inks and slurries
  - Membranes and active layers
- Modeling and simulation
- Testing R2R equipment/processes in new applications
- Application-specific device fabrication and testing

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Acknowledgements

David Hardy, DOE AMO

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Peter Faguy, DOE VTO

Many collaborating researchers at ORNL, NREL, ANL, and LBNL

OAK RIDGE National Laboratory

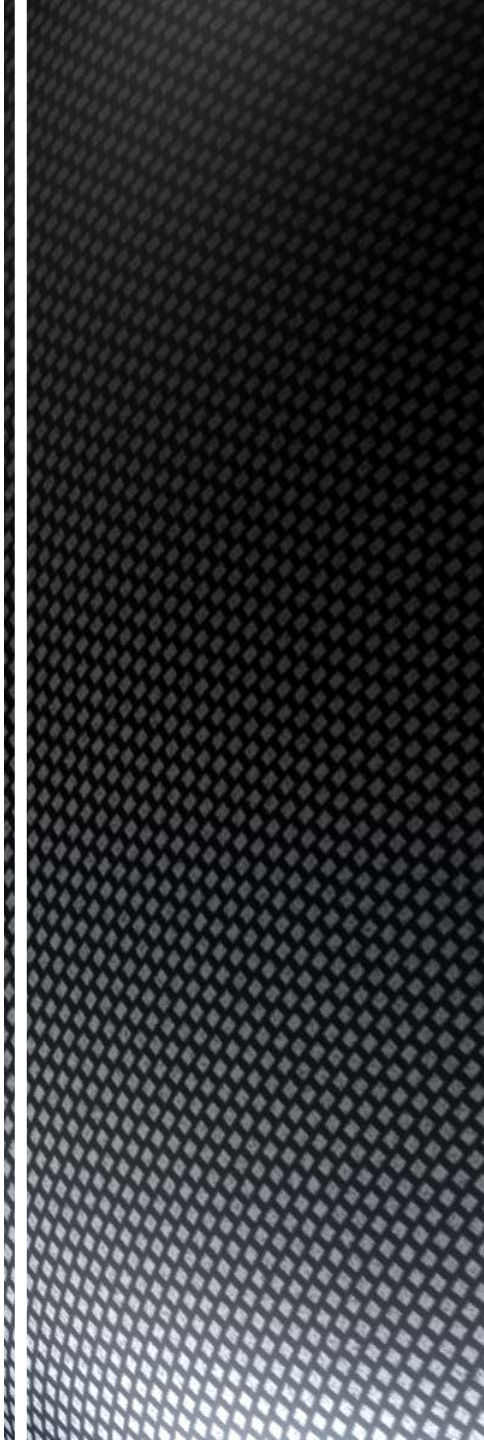
Argonne NATIONAL LABORATORY

R2R

BERKELEY LAB

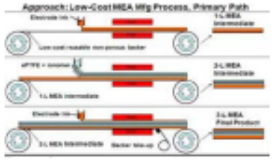
NREL NATIONAL RENEWABLE ENERGY LABORATORY

Backup Slides



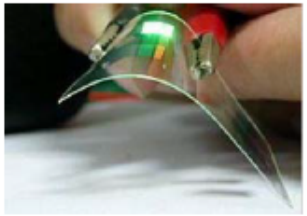
AMO R2R Workshop: Cross-agency linkages

- **DOE**
  - FCTO – FCEVs, PEM fuel cells, fuel cell backup power, Hydrogen gas separation
  - SETO – CIGS PVs, flexible PVs
  - BTO – Airflow panel membranes, electrochromic window coatings, sensors
  - AMO – CdTe solar cells, solar reactive coatings, battery/super-capacitor/superconducting cable/sensor technologies
  - OFE - Polymeric and ceramic/metallic membranes for CO2 separation
  - NREL – Defect diagnostics, quality control for scale-up of fuel cells on weblines
- **DOD**
  - Micro-electronics for flat panel displays, thin film transistor arrays for flexible displays, digital x-ray detectors, flexible reflective displays, self-aligned imprint lithography, zinc-polymer battery chemistries, R2R processed OLED, flexible Si CMOS chips on paper
- **NSF**
  - Nanomanufacturing research
- **Others -**
  - Organic-based TFTs for displays and RFID, flexible electronic OLED displays, anodes and cathodes in a continuous process, planarization, imprint embossing and patterning, alternative materials and membranes, functional hybrids, viscoelastic fluids, thermoelectrics, micro-electronics lithography printing



PEM Membranes

Flex Electronics



OLEDS

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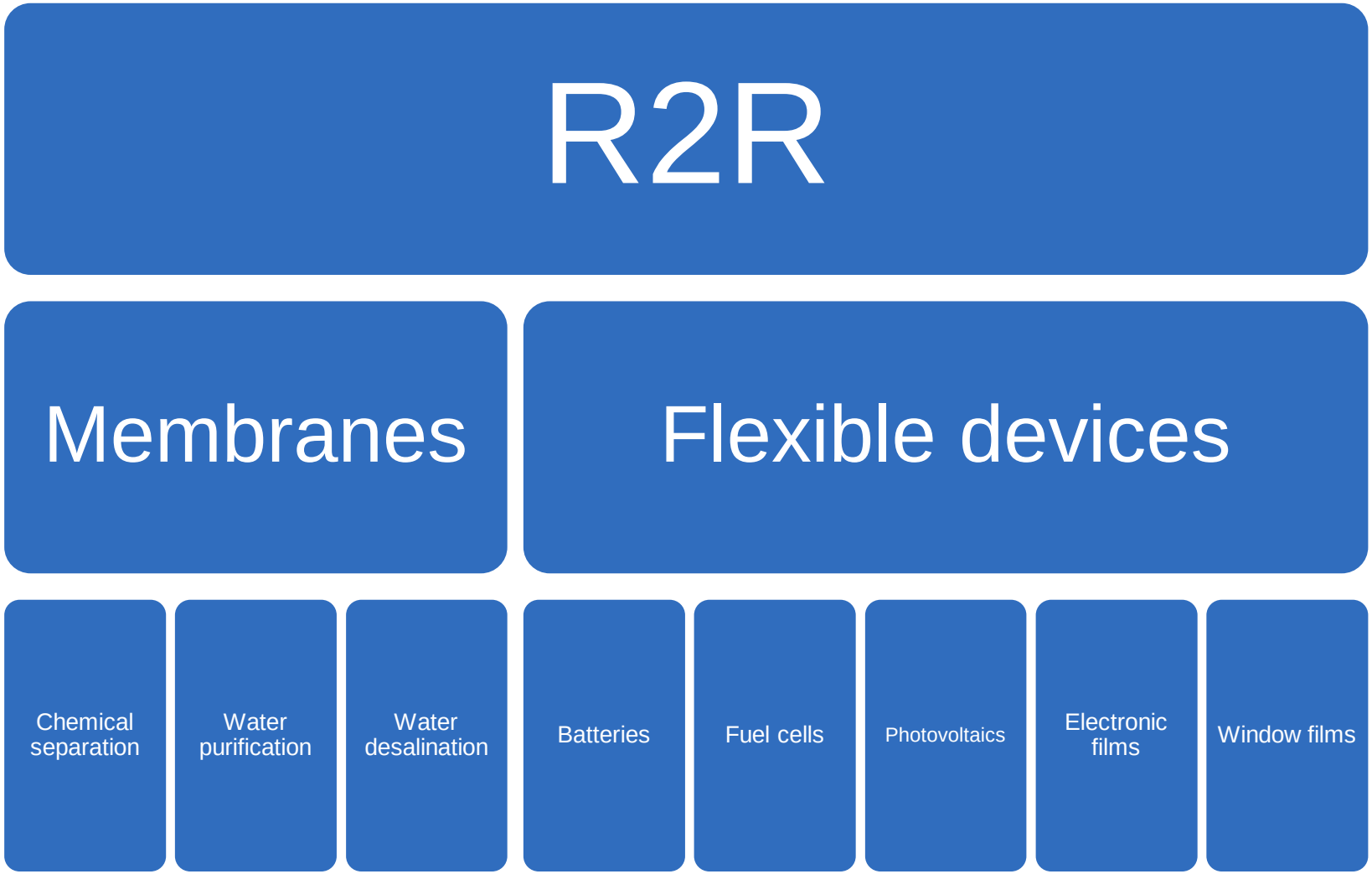
Key R2R applications for investment by DOE



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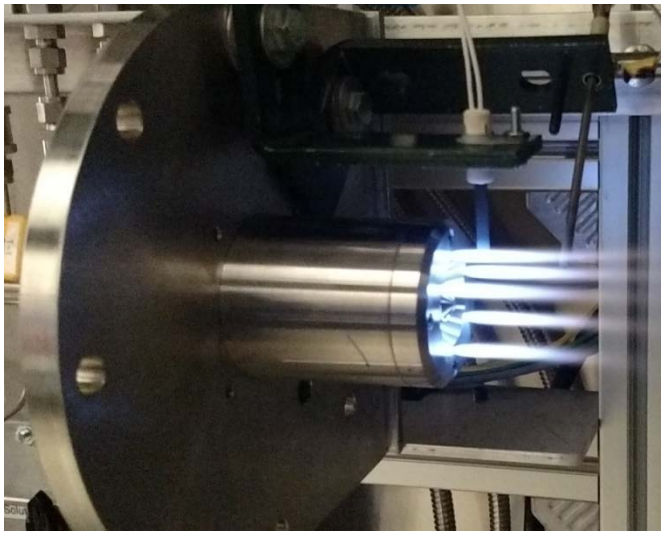
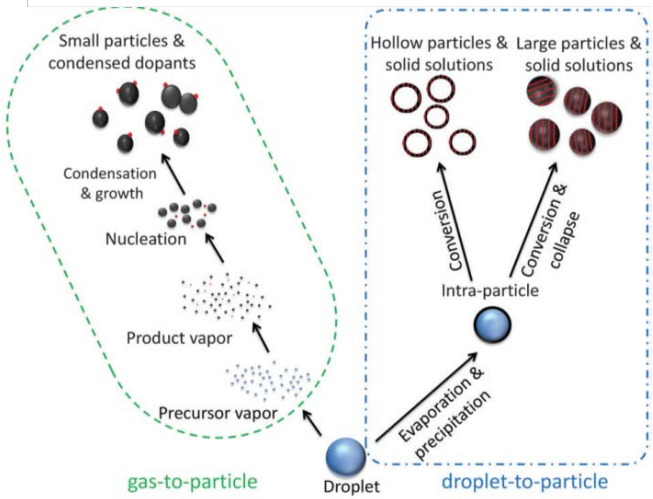
ANL: Flame Spray Pyrolysis Facility

- 20 g/hour production
- Daily operation turnover (new material day after day)
- Industry standard safety systems
- In-situ annealing section
- Extensive front-end controls (combustion flows and liquid feed)





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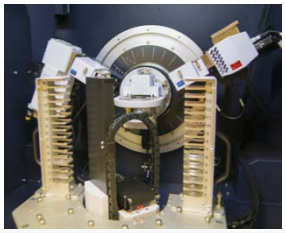
ANL: High-Throughput Research Laboratory

Synthesis/Fabrication

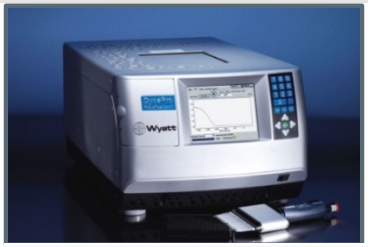
Robotic System – Air Sensitive Synthesis

Robotic System – non-Air Sensitive Synthesis

Characterization



X-ray Diffractometer

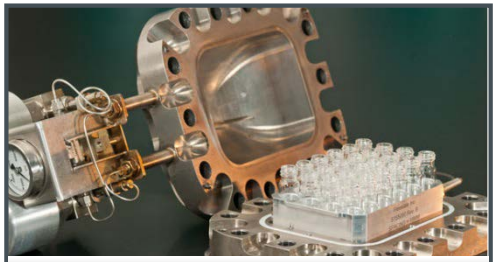


Particle Size Analyzer

25-electrode Fuel Cell

Treatment/Evaluation

Parallel Plug-Flow Reactor



Screening Pressure Reactor

- Two robotic systems for exploring a wide range of compositional phase space
- X-ray diffractometer designed to integrate seamlessly with HT equipment
- Nuvant 25-electrode array fuel cell hardware for electrocatalytic activity and electrode performance evaluation
- Reactors with variety of analytic capabilities (e.g., GC-MS, liquid chromatography)

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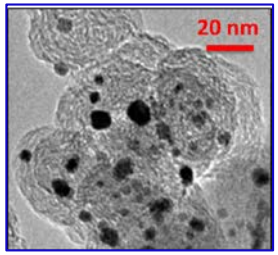
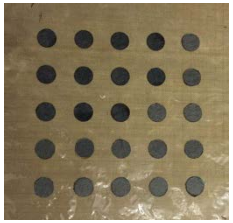
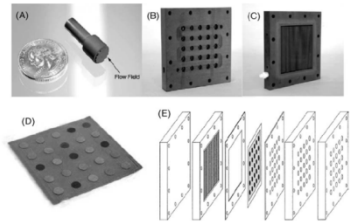
ANL: High-Throughput/Combinatorial Synthesis and Characterization of Inks and Electrodes



Pt/C Catalyst

FreeSlate High Throughput Catalyst Ink Synthesis and Deposition





Combinatorial Fuel Cell Test Fixture

NuVant Systems, Inc.
Nuvant.com

- High throughput/robotic synthesis of catalyst-ionomer-solvent inks of various ionomer to carbon ratio and solvent type, as defined by NREL and ORNL effort
- Analysis of microstructure of inks and dried inks via SEM and TEM for microstructure
- High throughput/robotic deposition of inks on substrates (blanks or gas diffusion layers)
- Combinatorial performance testing and characterization of twenty-five electrodes in a membrane-electrode assembly

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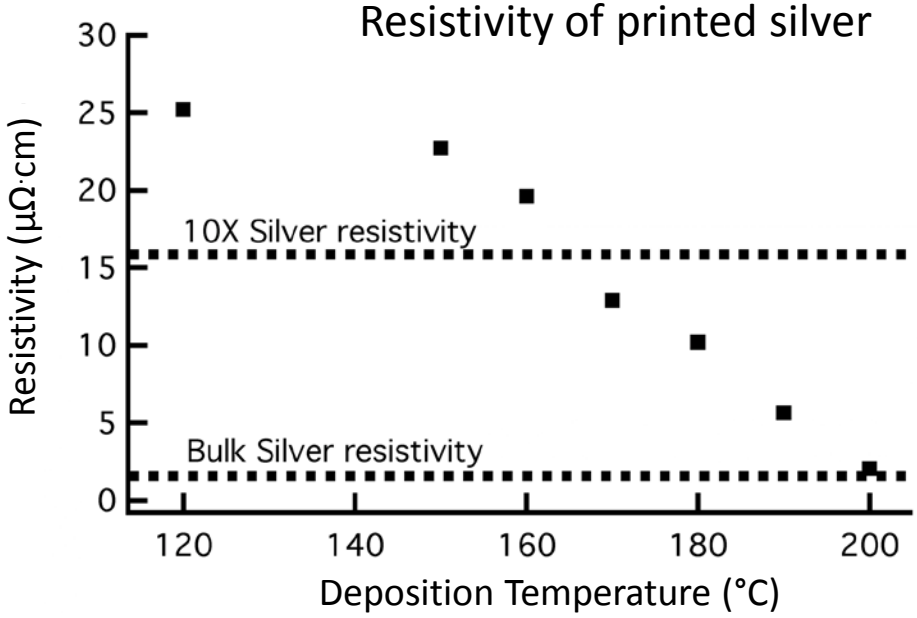
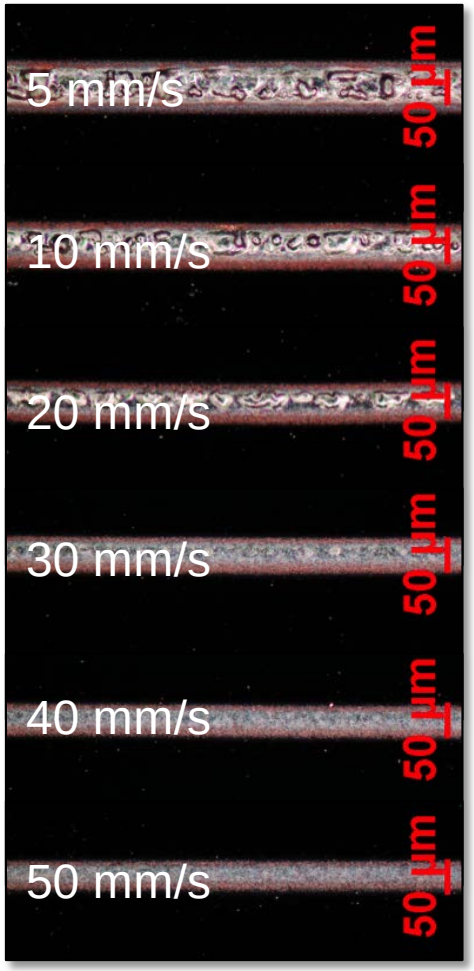
NREL: Printing of metal contacts

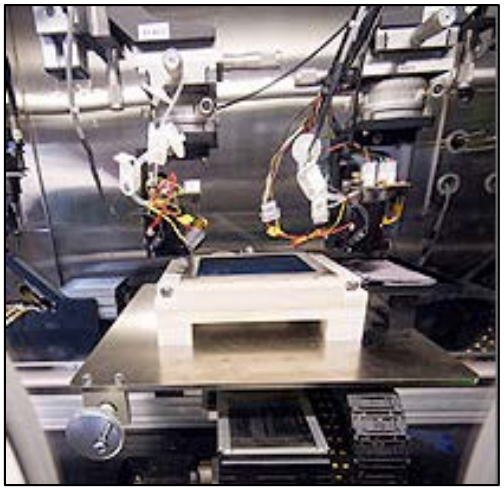
Resistivity of printed silver



Copper lines

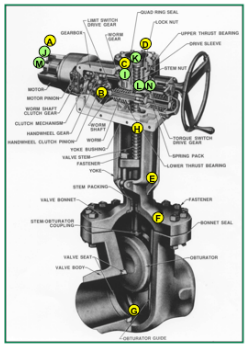


- Inkjet printing and Aerosol Jetting in inert atmosphere
- Metals: Ag, Cu, Ni, Al with resistivity close to bulk
- Patterns with line widths < 50 μm
- Organic and inorganic insulators can also be printed

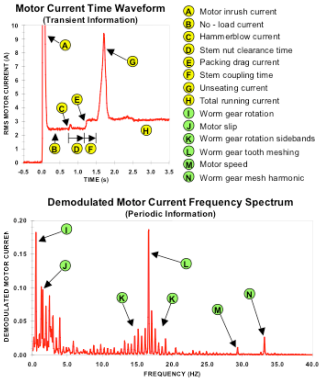


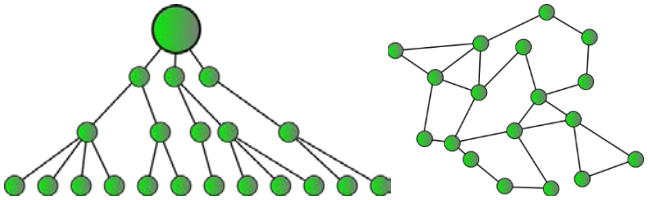
ORNL: Data and controls

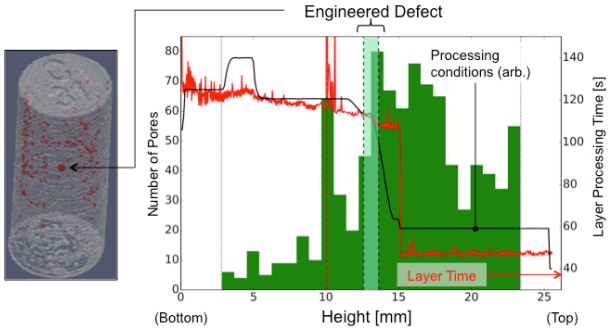
- Advanced sensor technologies
- Electrical signature analysis
- Large scale structured and unstructured data management and utilization
- Multi-dimensional data visualization
- In-situ process monitoring of additive manufacturing
- Distributed, stochastic model predictive controls development



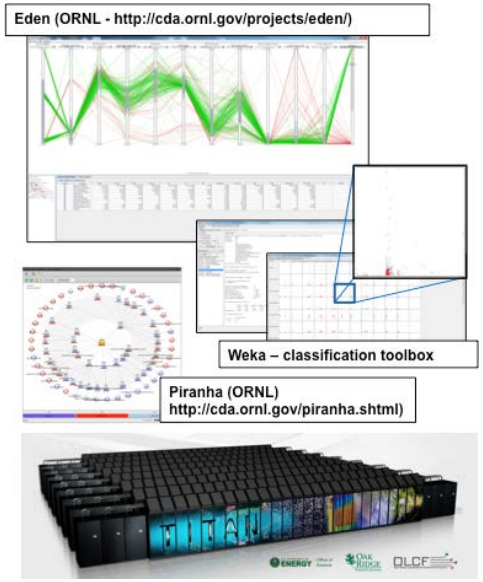
Motor-Operated Valve







Anomaly/interruption: Execution ↔ Outcome correlation
Late stage porosity: Planning ↔ Outcome



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