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Membrane Electrode Assembly Manufacturing Automation Technology for the Electrochemical Compression of Hydrogen

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April 30, 2019

DOE Hydrogen and Fuel Cells Program
2018 Annual Merit Review and Peer Evaluation Meeting

Project ID: h2006

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Overview

Timeline and Budget

- Project start date: 05/01/18
- Project end date: 11/01/19
- Total project budget: $371,240
  - Total partner share: $150,000
  - Total federal share: $150,000
  - Total partner in-kind: $71,240
  - Total DOE funds spent*: $55,155

* As of 1/31/19

Barrier

- Reliability and Costs of Gaseous Hydrogen Compression (Delivery B)

Partners

- HyET
- National Renewable Energy Laboratory (NREL)
- Lawrence Berkeley National Laboratory (LBNL) (associated membrane project with HyET)

Relevance

- Relevance (from H_2@Scale CRADA call):
  - Develop materials, processing techniques, and/or innovative designs for components used in hydrogen equipment and compressors, to enhance durability
  - Design **novel manufacturing approaches** and technologies for compressors and pipeline fatigue life and durability
- Objectives:
  - Full rheological understanding of **optimum ink formulation** for roll-to-roll coating method for catalyst layers, followed by electrochemical hydrogen compression (EHC) relevant tests
  - Development of **real-time optical/areal quality inspection** techniques for membrane electrode assembly (MEA) materials, with a focus on the membrane
  - Compare **EHC performance** of roll-to-roll (R2R) and manually manufactured MEAs (HyET)
  - Design and specification of an **EHC MEA manufacturing line** (HyET with NREL)

Approach

- Project leverages NREL in-line MEA inspection and electrode scaling capabilities and expertise developed for fuel cell and electrolysis materials, and apply to EHC
- NREL is developing quality control (QC) techniques and inks/coating process understanding and transfering knowledge to HyET
- HyET is performing in situ testing of coated materials in their hardware/system
- Project outcomes will inform manufacturing line specification

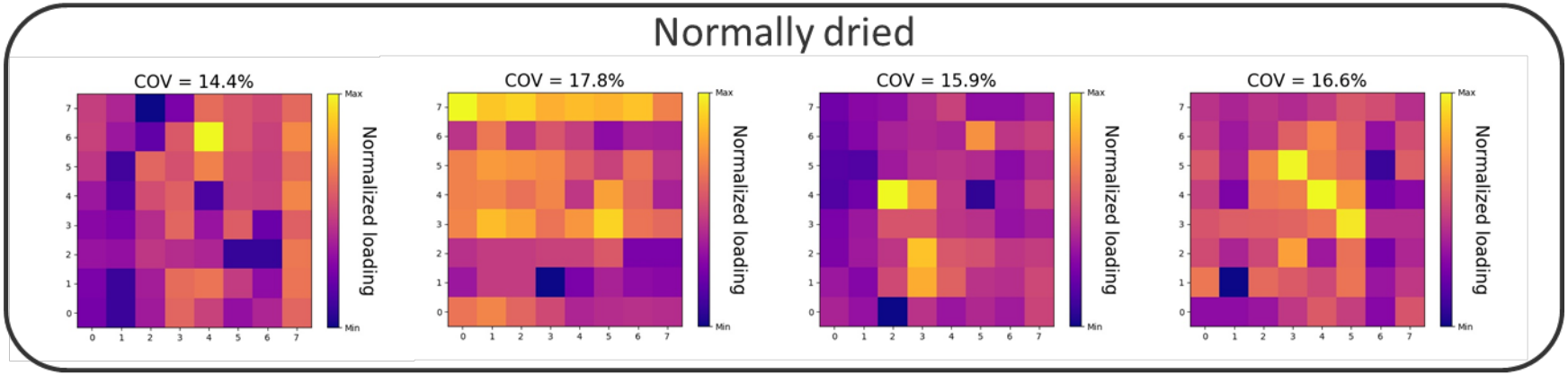
Approach: Milestones

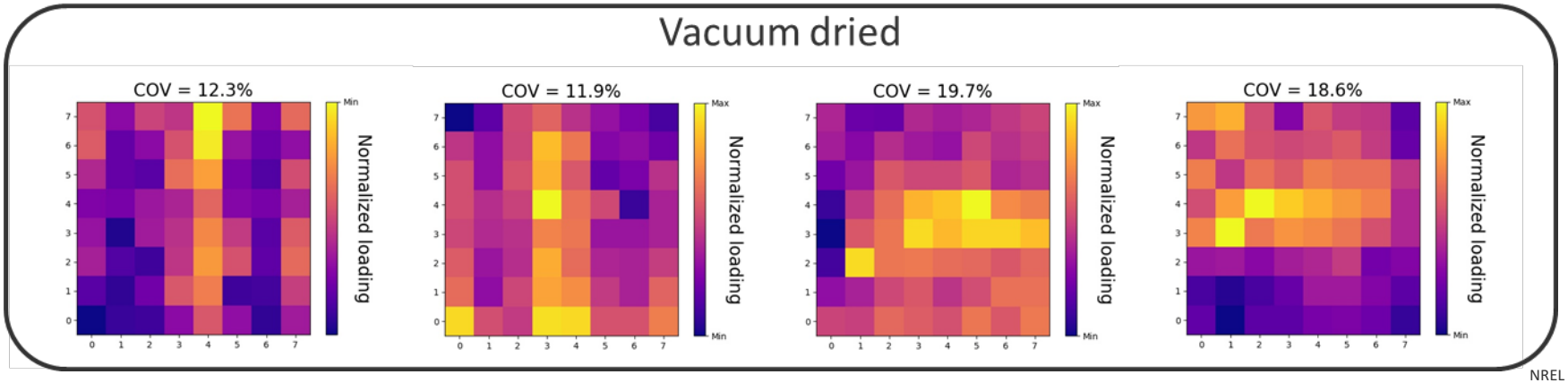
Milestone #	Project Milestones	Type	Completion	Progress Notes
1	Suitable ink formulation	Milestone	100%	Ink formulations were developed for initial R2R coating run, and provided acceptable quality and coatability.
2	Coated electrodes	Milestone	100%	Small-scale coating and spraying experiments were completed, informing the R2R coating run.
3	Selection report on in-line QC inspection methods	Milestone	75%	Current focus, as directed by HyET, is membrane defect characterization and database development.
4	EHC test report on manual vs automated MEA production quality	Milestone	30%	Ongoing.
5	Manufacturing line design spec	Go/No-Go	0%	Will initiate based on milestone 4 results.

Accomplishments and Progress

Performed x-ray fluorescence (XRF) mapping of HyET-produced electrodes to assist in their quality evaluation

- Assessed the impact of drying methodology
- *Results indicated that electrode coating homogeneity did not depend greatly on drying method*



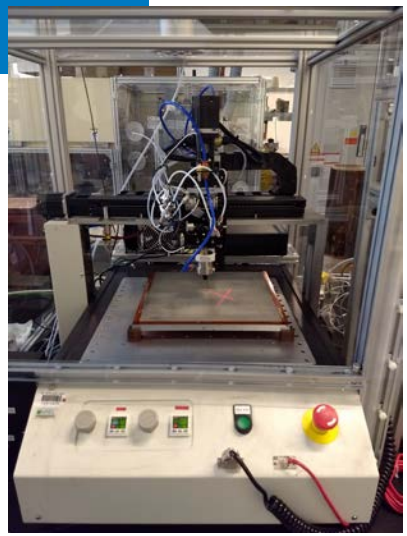


Accomplishments and Progress

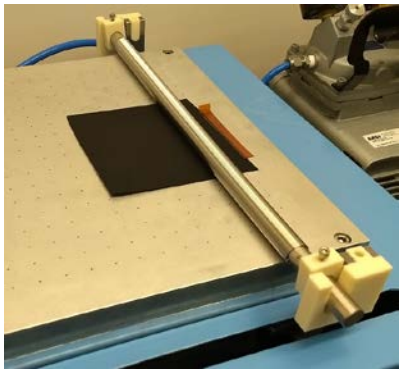
Fabricated baseline electrodes with NREL-style inks

- Coating processes
  - Ultrasonic spray
  - Rod
- Electrodes
  - Using two platinum/carbon (Pt/C) catalysts
  - Using two gas-diffusion layer (GDL) substrates
- Coated electrodes sized for HyET test cell
- Validated electrode loading and variability via XRF
- Provided to HyET for in situ testing

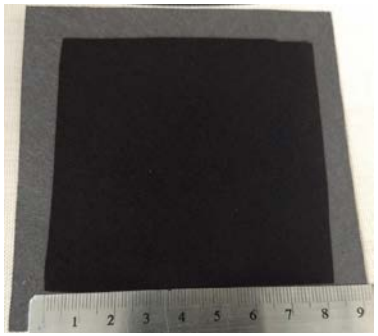
Ultrasonic spray coating



Automatic bar/rod coater



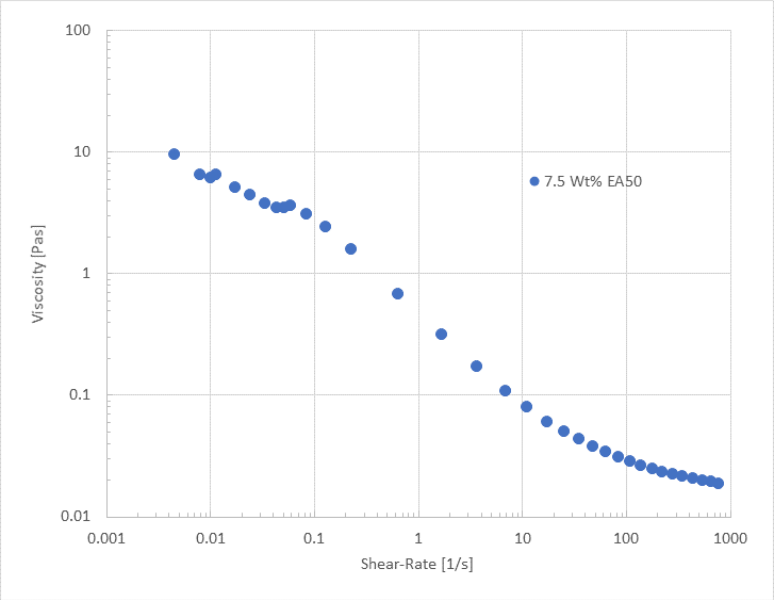
Spray-coated electrode



Accomplishments and Progress

Established ink formulation and coating conditions

- Ordered and slit 50 m rolls of both GDL types
- Trial run coating onto polyethylene terephthalate (PET) film to set conditions
  - Initial ink formulation (5 wt% Pt/C – similar to that used for spray and rod coating) was not high enough viscosity
  - Reformulated to 7.5 wt% to achieve desired viscosity
- *Target loadings were able to be achieved within the operating window*



Rheology of 7.5 wt% ink

Accomplishments and Progress

Coated electrodes using R2R slot-die

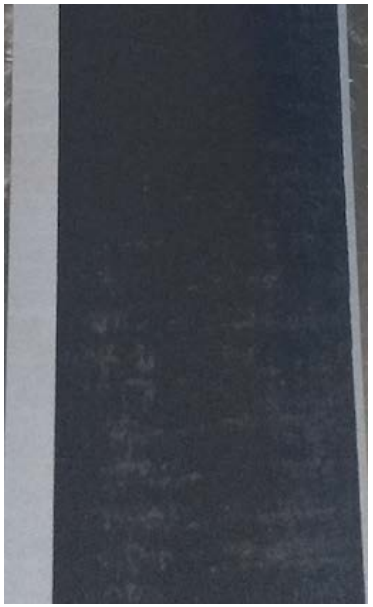
- Coating onto both GDLs
  - 18 cm wide, both substrates
  - Three target loadings
  - Turrax homogenizer, then stirred overnight to degas
  - Web-speed 1 m/min
  - Oven 1: 80 C, oven 2: 90 C
- Used cross-sectional scanning electron microscopy (SEM) to study electrode structure
- Validated electrode loading and variability via XRF
- Electrodes coated at all conditions and provided to HyET for in situ testing
- *Expanded our understanding of formulating inks for and coating onto porous substrates*

Accomplishments and Progress

Identified material issues with GDL A

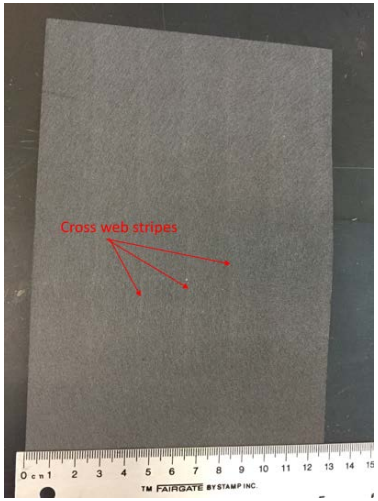
- Apparent cross-web variations in coating (no similar variations in coatings on GDL B)
- Spacing of coating variations seemed to match visible variations in the substrate
- Such substrate variations were not observed on previous material samples received from HyET
- Via XRF, the "stripes" in the coated electrodes were measured to have lower Pt loading than nominal sections
- Working with supplier and HyET to identify source of variation
- *Expanded our knowledge base of relevant manufacturing defects*

CL on GDL type A



CL on GDL type B

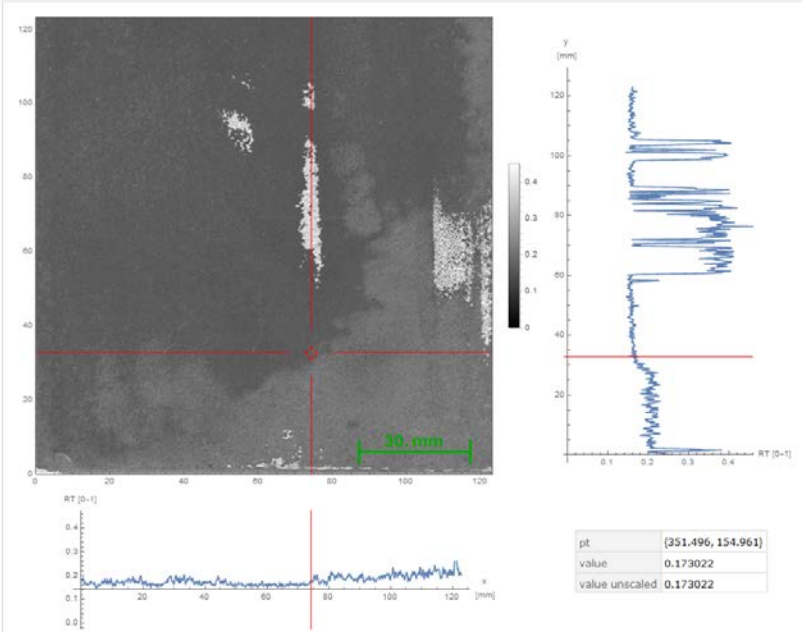
Non-uniformities on GDL A



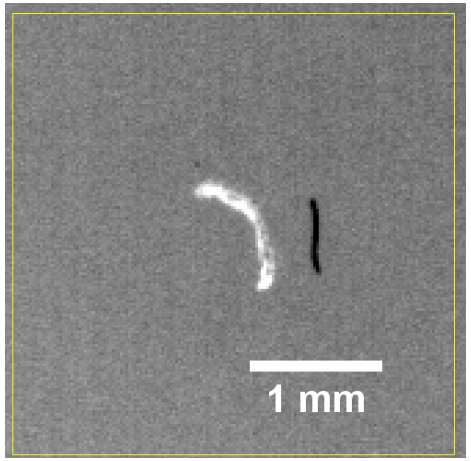
Accomplishments and Progress

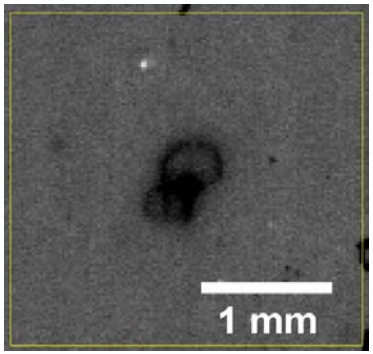
Optical imaging – electrode

- Optical scanning of HyET-fabricated electrodes with known defects
  - Defects easily imaged in reflectance mode
- Comparison of defects in reflectance and transmission modes
- *Demonstrated capability of optical techniques for electrode defect detection*

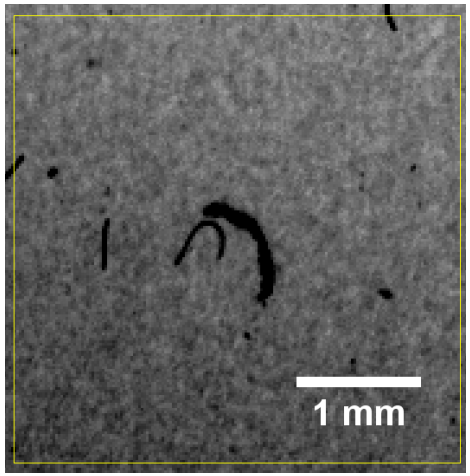


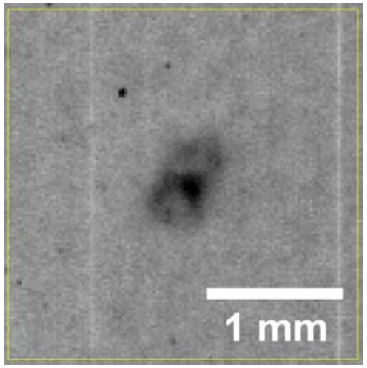
Reflectance mode scanning of electrode with coating defects





Reflectance mode



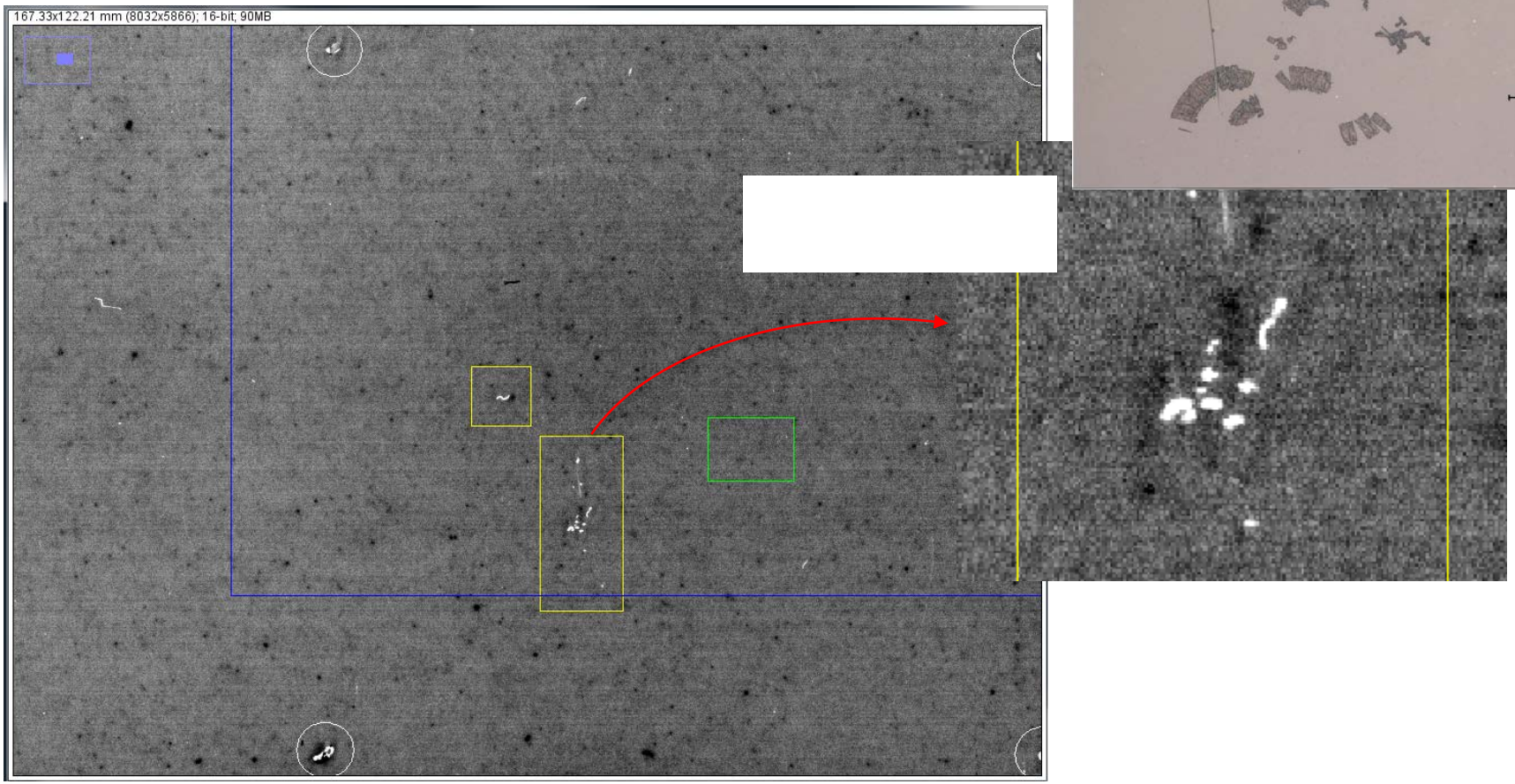


Transmission mode

Accomplishments and Progress

Optical imaging – membrane

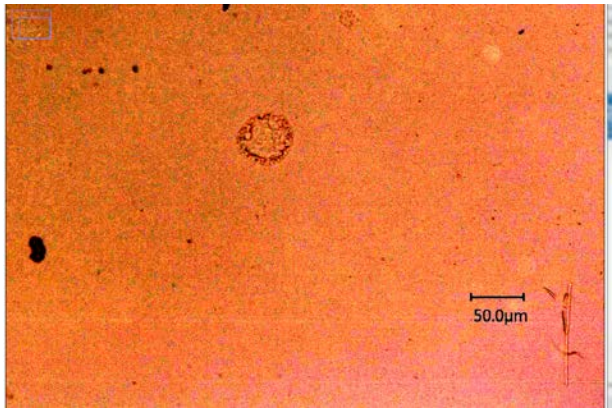
- Performed fast reflectance mode scanning of membrane
- *Reflectance scanning demonstrated for membrane defect detection*



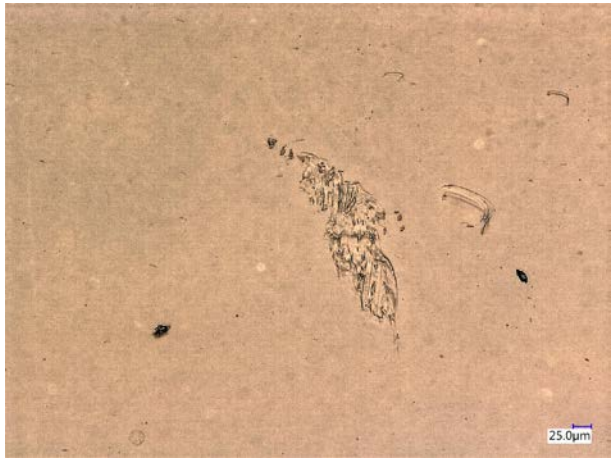
Accomplishments and Progress

Optical imaging – identification of samples for in situ defect testing

- Used algorithms to detect defects from fast scanning images
- Performed optical microscopy of membranes with defects to further characterize and confirm that the defect is "in" the membrane, not a piece of dust or other matter
- *Goal is to provide samples with confirmed defects to HyET to understand if these defects affect performance*

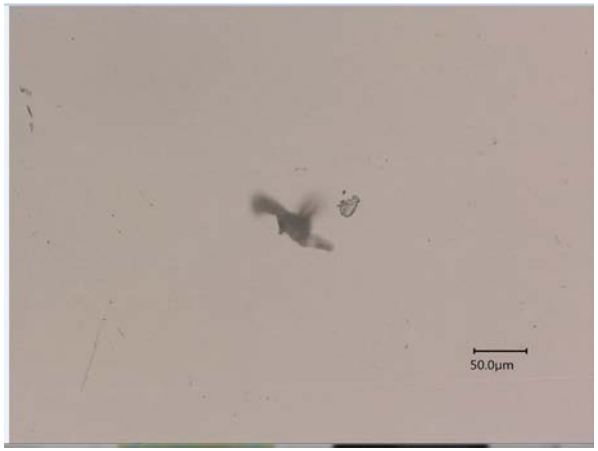


Deposited liquid or microbubble



Surface feature

Embedded object
(not typical)



Accomplishments and Progress

Optical imaging – identification of samples for in situ defect testing (cont'd)

- Focus on understanding object distributions and finding objects of specific size for in situ testing
  - Target objects of several 10s of µm
- Measured object distributions, but found that most identified objects are not attached to membrane – likely come from lab or handling
- Working on methodologies to minimize dust and other "external" objects during measurements
- *Leveraged FCTO investments in optical inspection methods and automated algorithm development*
- *Expanded our knowledge base of relevant manufacturing defects*

Accomplishments and Progress: Responses to Previous Year Reviewers' Comments

- This project was presented as a poster, but not reviewed last year

Collaboration and Coordination

- Industry partner: HyET
  - Defines objectives
  - Defines materials and structures
  - Provides information about applicable manufacturing processes and techniques
  - Performs in situ cell testing of NREL-produced cell materials
  - Defines major parameters for MEA manufacturing line
- National lab partner: NREL
  - Explores and develops relevant inspection techniques for MEA materials
  - Formulates and characterizes inks for scalable electrode processing
  - Explores applicable coating processes
  - Provides methods and coated materials to HyET
  - Assists in the specification of MEA manufacturing line
- Additional partner: LBNL
  - LBNL has membrane-focused project with HyET; NREL and LBNL will coordinate on research as applicable

Challenges and Barriers

- Identifying suitable in-line inspection techniques for HyET MEA materials
- Formulating inks for scalable processes
- Identifying scalable process methods and procedures for high-volume electrode production

Proposed Future Work

- Pending HyET in situ testing of spray-coated, rod-coated, and slot-die-coated electrodes, review ink formulation and perform follow-on R2R coating run for optimization
  - Obtain GDL roll material without striping defects
- Continue to work on optimizing optical inspection and microscopy methods to reduce or eliminate external contaminants
- Develop membrane defect database (data, samples) to assist HyET in understanding impacts of defects in the membrane
- Assist HyET in the development of the manufacturing line plan and estimate

Technology Transfer Activities

- Under the CRADA, applicable technology will be transferred to the industry partner
- NREL will assist HyET in the implementation of project outcomes

Summary

- Approach: leverage NREL manufacturing R&D capabilities developed for fuel cells to similar EHC materials and processes
- Relevance: develop materials, processes, techniques and manufacturing approaches for electrochemical hydrogen compression MEAs
- Accomplishments
  - XRF mapping
  - Small-scale and R2R coating
  - Electrode structure characterization
  - Optical scanning and microscopy for defect identification
  - *Advanced our general understanding of coating onto porous substrates and relevant manufacturing defects for MEA materials*
- Future Work
  - 2nd coating run
  - Improved optical scanning and microscopy methods
  - Assist HyET with manufacturing line design

Acknowledgements

HyET

- Martijn Mulder, Industry Partner PI
- Albert Bos, CTO

NREL

- Peter Rupnowski, QC Development sub-lead
  - Brian Green
- Scott Mauger, MEA Scaling sub-lead
  - Jason Pfeilsticker
  - Sunil Khandavalli

LBNL

- Gao Liu, Sister-project collaborator

Thank You

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Publication Number

NREL is a national laboratory of the U.S. Department of Energy, Office of Energy Efficiency and Renewable Energy, operated by the Alliance for Sustainable Energy, LLC.

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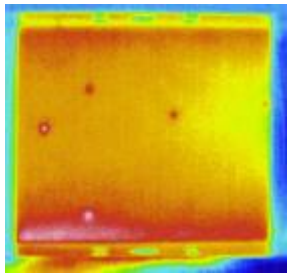
Technical Back-Up Slides

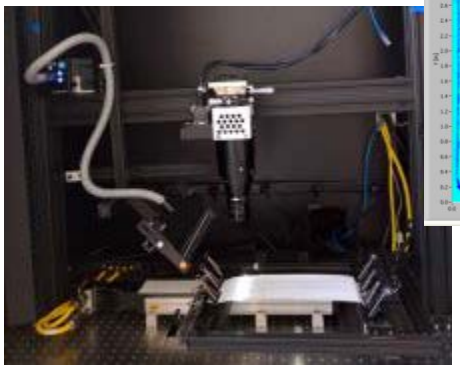
NREL Capabilities: In-line Inspection

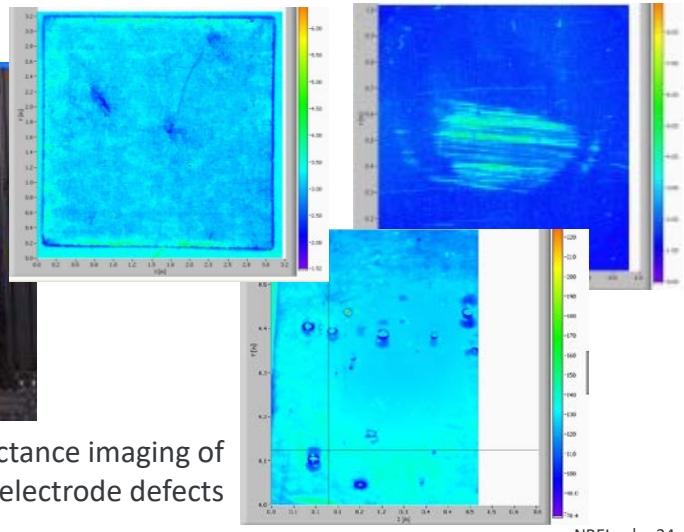
Development of real-time, in-line quality control diagnostics to support the continued scale-up of MEA components for low-cost production

- Material portfolio: Fuel cell, battery, and electrolyzer materials
- Membrane defect imaging and thickness mapping
- Electrode and GDL uniformity
- MEA shorting and gas crossover
- Property measurement, e.g. porosity
- Optical and IR diagnostic platforms
- Non-destructive, 100% inspection



Infrared imaging of electrode and MEA defects





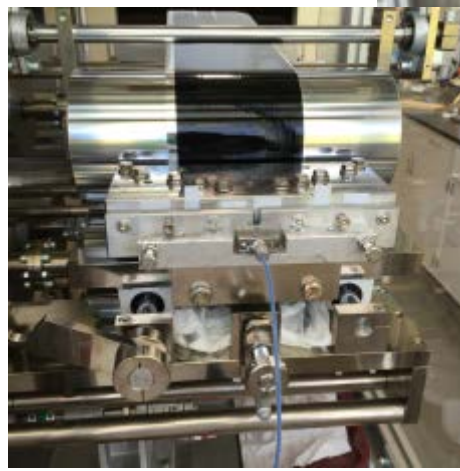
Optical reflectance imaging of membrane and electrode defects

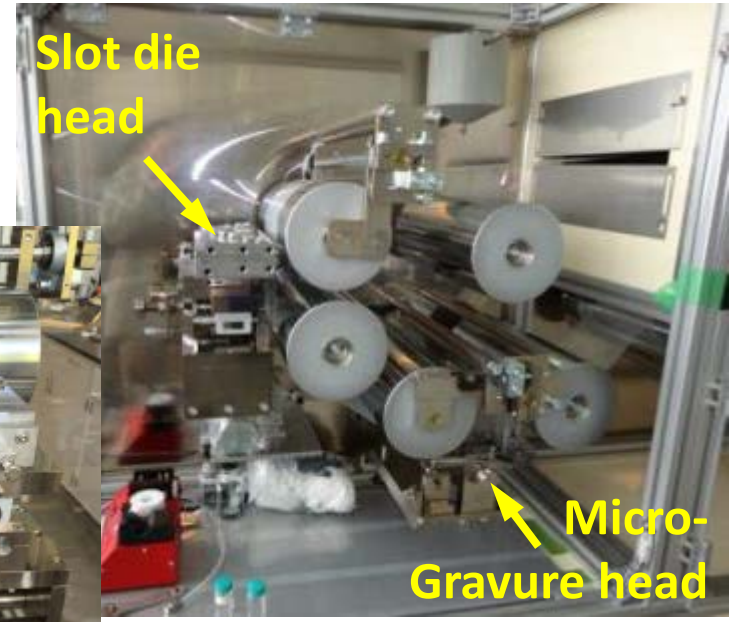
NREL Capabilities: MEA Scaling

Understanding how parameters of scalable, high-volume processes effect MEA morphology, uniformity, and performance

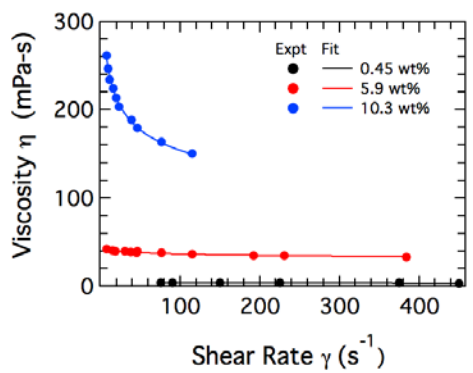
- Roll-to-roll coating station
  - Slot/knife coating head
  - Micro-gravure coating head
  - 50-300 mm coating width
  - 4 independently controllable drying sections
  - Coating speeds from 0.2-10 m/min
- Applicable to various thin-film technology applications
- Formulation, rheology, coating, drying studies

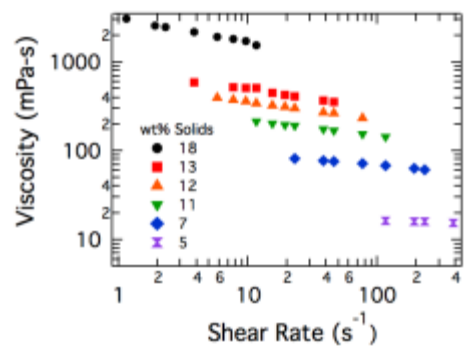
Slot-die coating





Multi-head coating enclosure





Viscometry of electrode inks

Project Task Details

- **Task 1: Inks and Coating Process Development (NREL)**

  NREL will bring to bear ink characterization tools, including rheology, dynamic light scattering, and zeta potential, and electrode fabrication equipment across several scales including a roll-to-roll coating line to evaluate and perform initial optimization of inks and coatings for EHC electrodes. NREL will perform parametric studies to understand the impact of formulation and process variables on the thickness and uniformity of electrode layers. NREL will provide electrode sheet materials to HyET for assembly into cells and in situ testing at HyET's facility.

- **Task 2: Quality Inspection Development (NREL)**

  NREL will bring to bear multiple test-beds for the development of real-time quality inspection techniques for electrodes, membranes, and MEA subassemblies for HyET's EHC MEA materials. NREL's activity focuses on areal inspection techniques, i.e. techniques that utilize optical or infrared imaging to provide the potential for 100% inspection of MEA material webs (rather than point measurements). These techniques enable detection of small discrete defects in MEA materials as well as determination of overall film or layer uniformity. If appropriate, NREL can utilize its industrial-style web-line to validate techniques with sheet or roll materials.

Project Task Details

- **Task 3: EHC testing of R2R vs manual MEA based EHC single cell & stack (HyET)**

  HyET will manufacture the MEAs and perform EHC cell testing which will include standard diagnostics (as used for PEMFC) like shorting tests, IV-curve recording, (Electrochemical Impedance Spectroscopy) EIS and cyclic voltammetry (CV analysis), hydrogen (H2) cross-over testing, H2 pumping at ambient pressure and finally compression testing to maximum pressure. Duration testing to evaluate the performance stability will conclude the membrane evaluation test program. Performance of the manual vs automated manufactured MEAs will be compared.

- **Task 4: Specification of a manufacturing Line (HyET+NREL)**

  Using its experience with R2R equipment and fabrication of MEA materials, and based on its work and results in Tasks 1 and 2, NREL will assist HyET in the design and specification of a manufacturing line for the high volume production of its MEAs. NREL will specifically provide inputs relative to electrode coating and quality inspection devices and integration. US suppliers of required production line equipment will be identified.