In-Line Monitoring System for Membrane and Electrode Assembly Manufacturing

PI: Michael Kimble

Presenter: Daniel Carr

SkyVision Sciences, LLC

Phase I DOE SBIR

Contract # DE-SC0023840

May 8, 2024

DOE Hydrogen Program

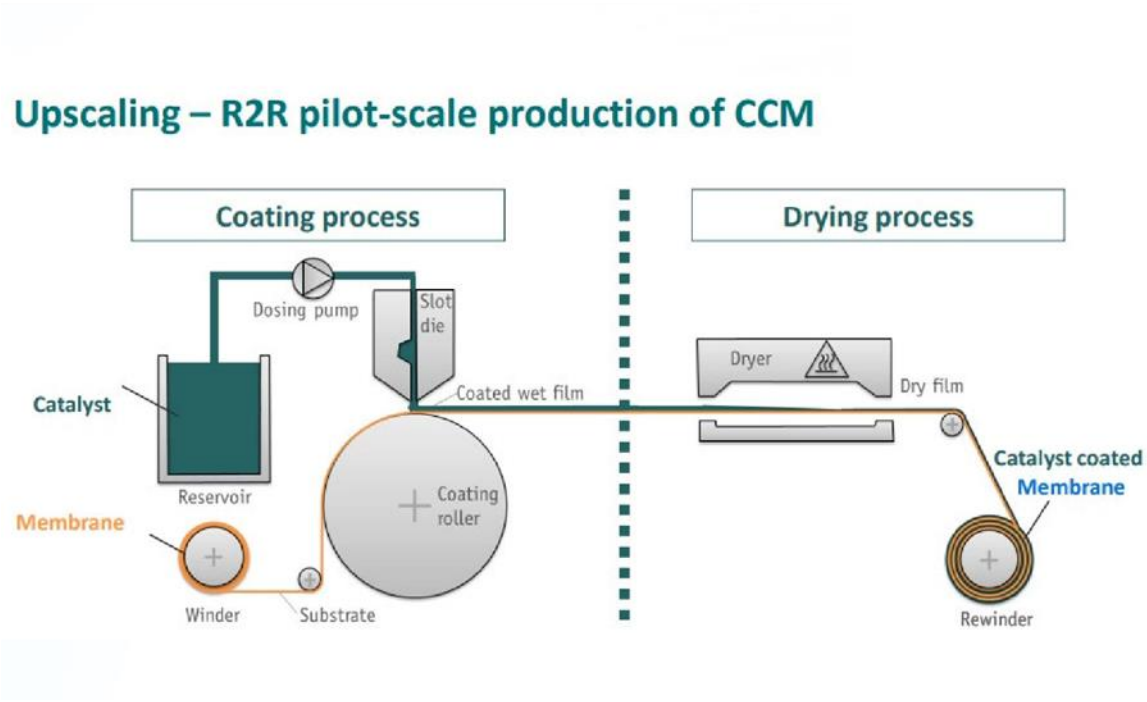
2024 Annual Merit Review and Peer Evaluation Meeting

AMR Project ID: MNF-BIL011

This presentation does not contain any proprietary, confidential, or otherwise restricted information

Project Goal

- Develop a real-time quality control sensor for assessing the electrochemical viability of membrane and electrode assemblies (MEAs) produced in a roll-to-roll process



- 10 m/min (faster speed requires more expensive stable materials)
- Continuous or patch coating
- Apply directly to membrane or onto carrier sheet (for subsequent decal-transfer to membrane)
- Other catalyst application options:
  - Ultra-sonic spray
  - Other

- **RD&D Issues/Desires:**
  - Higher speed
  - Lower capital cost
  - Ink formulations for high speed
  - Wider widths
  - Modularity
  - Design flexibility
  - In-line QC
  - Registration of defects

Project Overview

Timeline

- Project Start Date: July 10, 2023
- Project End Date: April 9, 2024
- Percent Complete: 100%

Budget

- Total Project Budget: $206,500
  - Contractor Share: $0
  - Federal Share: $206,500
- Funds Spent: $206,500

Barriers Addressed

- Design of 3 electrode electrochemical test system inside drum sensor platform
- Roll to roll interrogation

Partners

- US DOE: Project Sponsor and Funding
- SkyVision Sciences: Technical R&D

Relevance/Potential Impact

- Projections in the near-term call for 20,000 fuel cell stacks/year and 1 GW/year for proton exchange membrane electrolyzers
- Central to these stacks are catalyst coated membranes (CCM) or MEAs
- To meet these manufacturing quantities as well as to lower costs, an in-line metrology system enabling reel-to-reel MEA quality assessments to make rapid real-time methods fundamentally addressing the electrochemical viability of the MEA before it is incorporated into higher value electrochemical stacks is needed

80 kW Light Duty Vehicle	275 kW Heavy Duty Vehicle	1 MW Electrolyzer
1 stack per system	4 stacks per system	100 stacks per 100 MW
310 cells/stack	400 cells/stack	150 cells/stack
250-400 cm^2 active area	400-800 cm^2 active area	1,800 cm^2 active area
400-600 cm^2 total area	600-1300 cm^2 total area	3,000 cm^2 total area
3.4M CCMs/year	3.6M CCMs/year	150K CCMs/year

Relevance/Potential Impact: SkyVision Advantages Over Current State of the Art

Current Interrogation Methods

- NREL has developed a number of MEA inspection tools
  - Membrane thickness using hyperspectral imaging
  - Thermal scanning
  - Optical and Infrared imaging
  - Spectroscopy-based methods
  - Optical transmission imaging
  - These appear to be laboratory methods rather than industrial compatible methods

Current Method Disadvantages

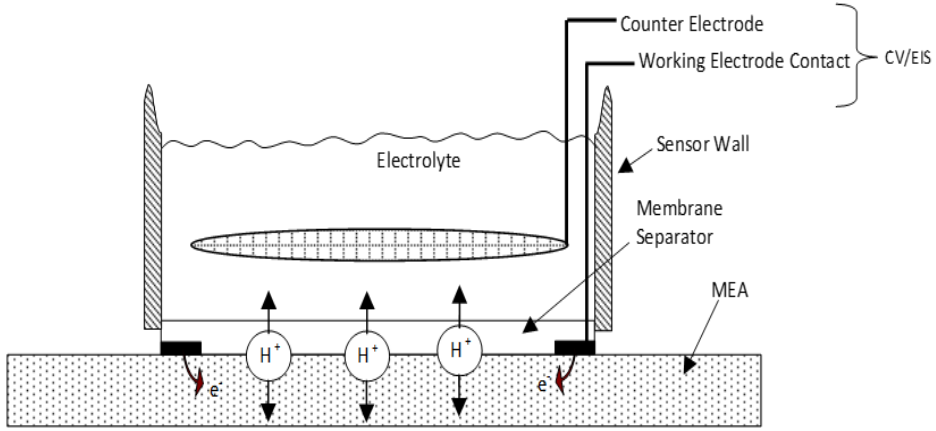
- Tests are destructive since the MEA cannot be reapplied to the final product
- These tests are also time consuming since either a fuel cell test or a separate electroanalytical test has to be conducted

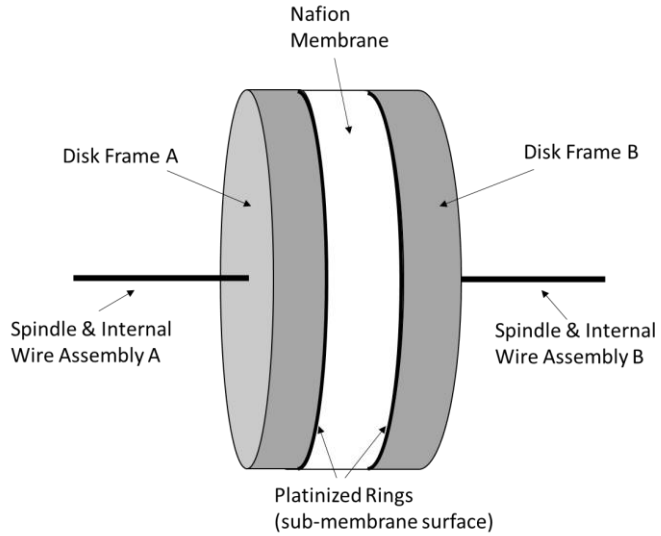
SkyVision Interrogation Method Advantages

- Is assessed for its electrochemical activity in a non-destructive manner
- Capable of having more regions analyzed for statistical comparison
- Capable of being assessed in a continuous manufacturing line, and
- Usable in its final product application

Technical Approach

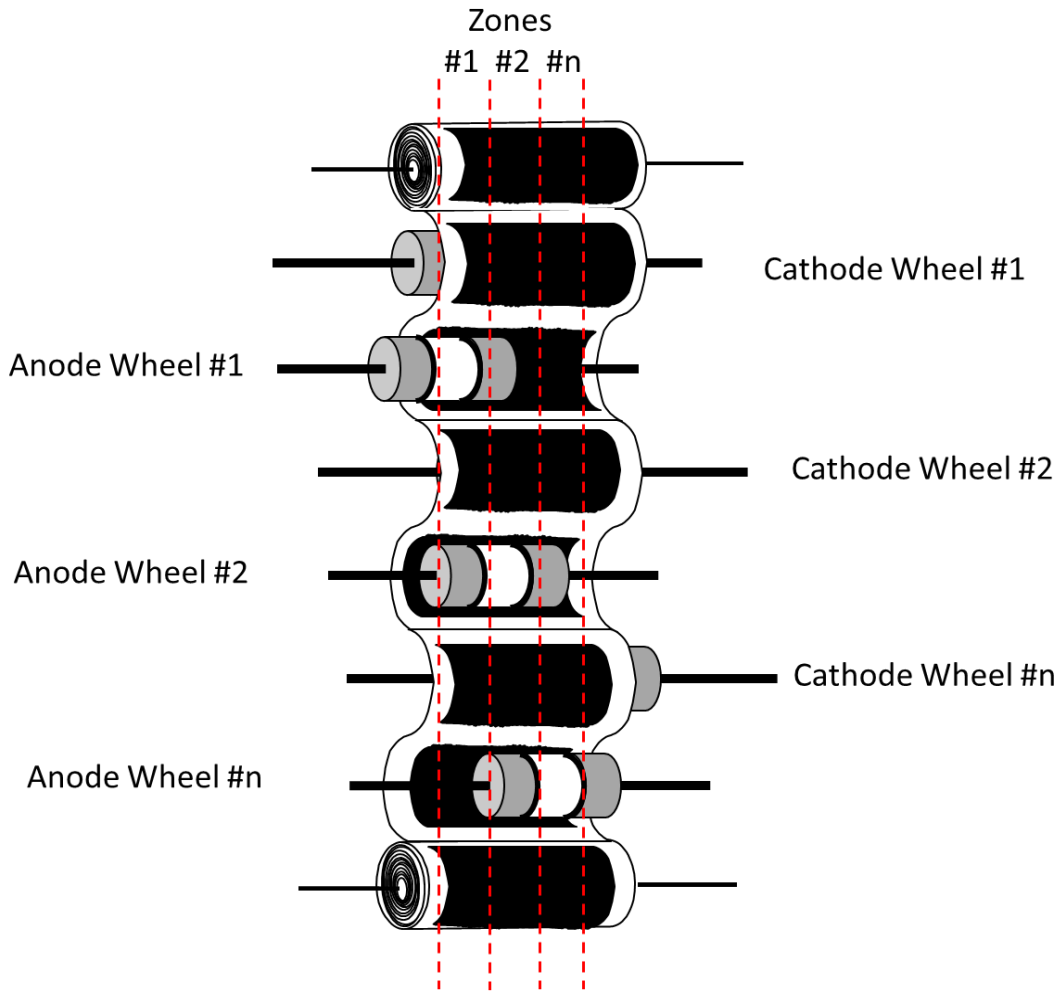
- Modify SkyVision's patented (US #8,221,603) hand-held electrochemical interrogation probe to a continuous sensor platform to support roll-to-roll MEA manufacturing
- Sensor packages the classic elements of a three-electrode wet-electrochemistry system (working electrode, counter electrode, reference electrode) into a drum to assess the electrochemical viability of MEAs by obtaining redox behavior of the working electrode surface (MEA)





Phase I Technical Challenges

- Design
  - Roll to roll interrogation of anode and cathode surfaces
  - Detecting defects across entire MEA surface
  - Sizing sensor drums for various sizes of MEAs and processing speeds
- Interrogation/analysis
  - Identifying electrochemical interrogation method and control parameters to be conducted
  - Need to be on the order of less than 1-2 seconds to give a go- to no-go QC acceptance criteria
  - Individual sensor QC over time

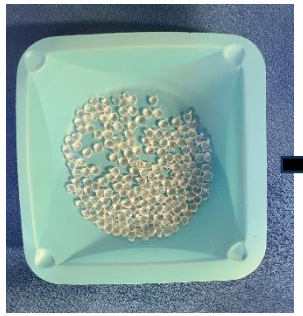


Approach: Safety Planning and Culture

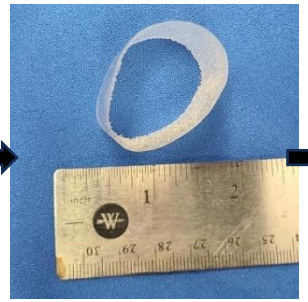
- Project was not required to submit a safety plan to the Hydrogen Safety Panel (HSP)

Technical Accomplishments: Drum Sensor Design

- Goal:
  - Package 3 electrode wet electrochemistry system into a drum sensor
  - Develop processes to produce membrane, working electrode, counter electrode, and drum housing



- Nafion precursor beads

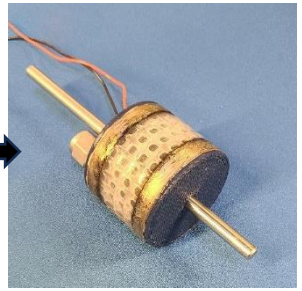


- Form tubular membrane

- Platinize membrane with working electrode current collector

- Pt coated Ti counter electrode

- 3D printed drum sensor housing components

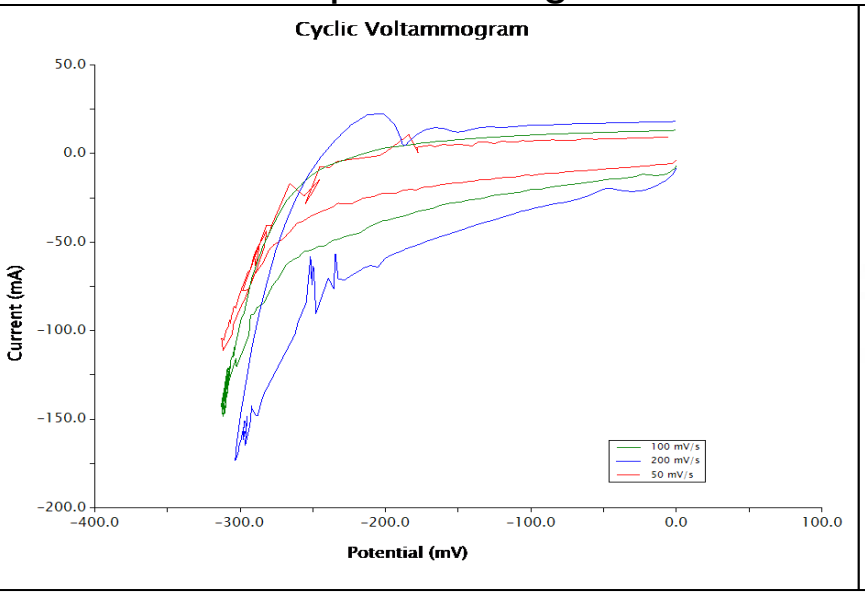


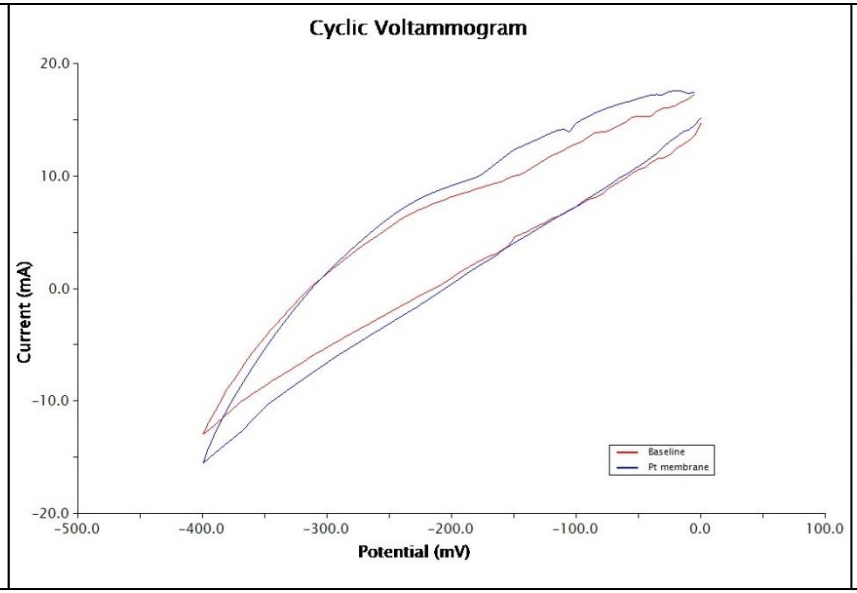
- Drum sensor

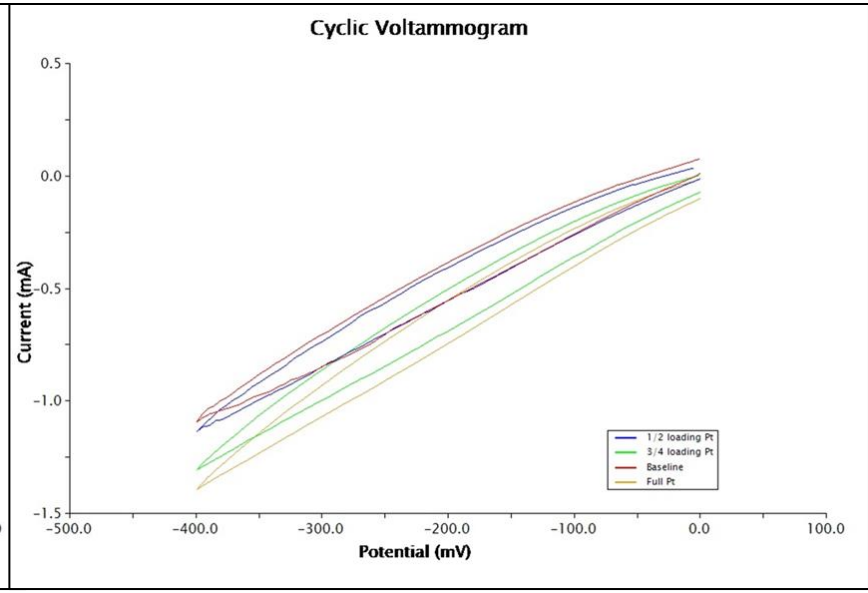
- Accomplishments:
  - Produced ionically conductive tubular membranes up to 2” ID with a 0.010” wall thickness
  - Developed procedures for plating working electrode current collector
  - Developed process to produce counter electrode
  - Designed and 3D printed drum sensor housing components
  - Packaged working, counter and reference electrodes into a drum sensor

Technical Accomplishments:
Drum Sensor Shows Electrochemical Assessment

- Goal:
  - Show drum sensor measures electrochemical signal
- Accomplishments:
  - Demonstrated drum sensor can operate at scan rates of 50 to 200 mV/s
  - Showed sensor can detect electrochemical activity of a catalyzed membrane surface
  - Rapid interrogation of less than 2 seconds



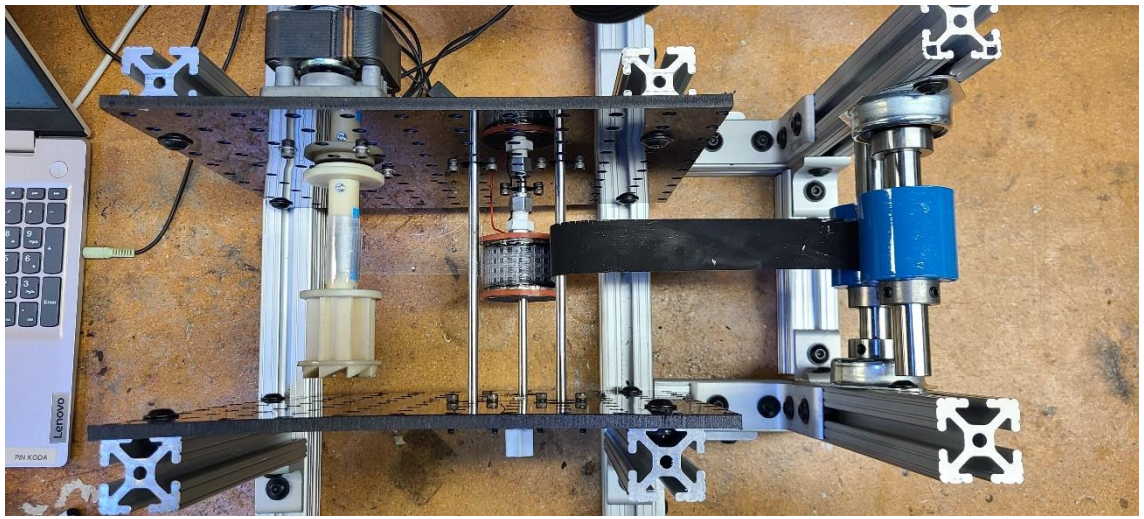


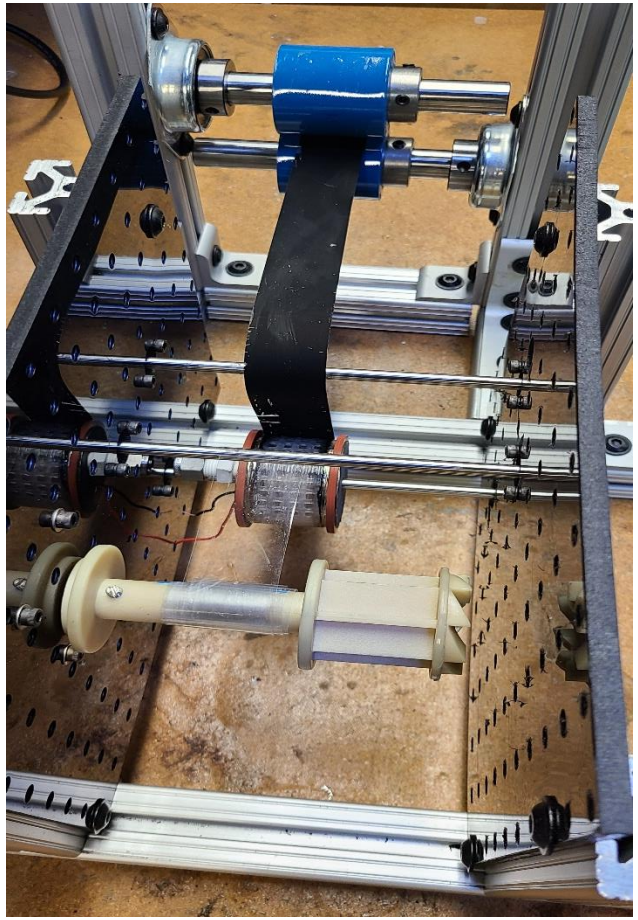


Technical Accomplishments: Reel to Reel Test System

- Goal:
  - Design reel to reel system for MEA assessment

- Accomplishments:
  - Designed automated reel to reel test system to interrogate MEAs with rotating single drum sensor



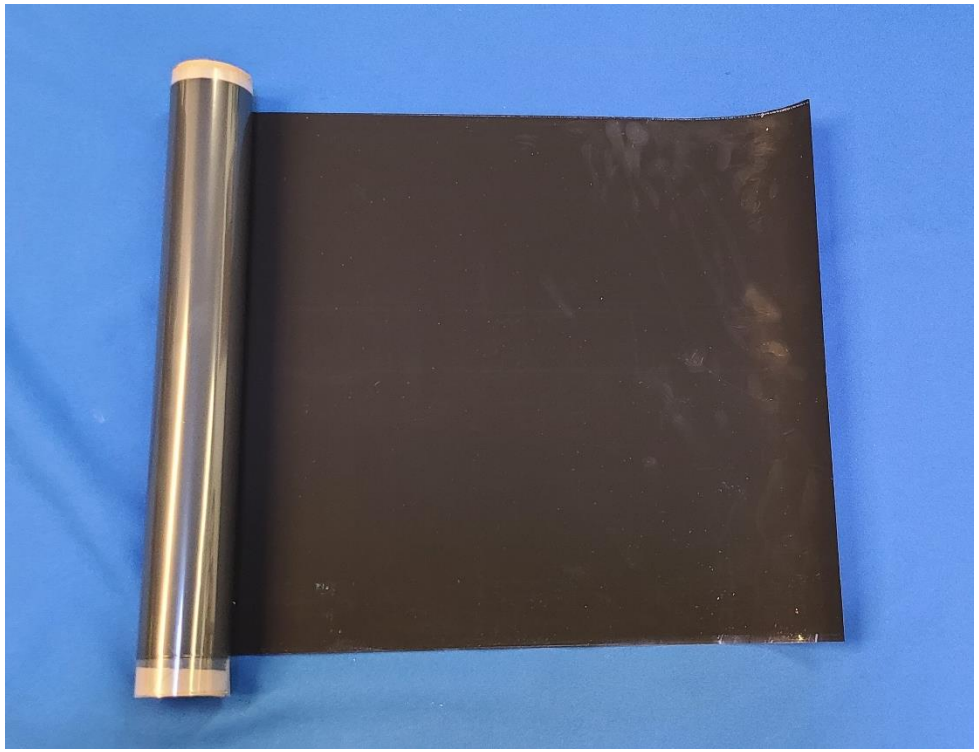


Accomplishments and Progress: Responses to Previous Year Reviewers' Comments

- This program was not reviewed last year

Collaboration and Coordination

- Working with a membrane manufacturer
  - They have catalytically coated decals in 100’ x 1’ rolls
    - Received a short run length to assess the catalytic variability with our QC sensor in a reel-to-reel mock up unit
    - Also looking into assessing decal-transferred coated membranes to assess delamination defects
  - They have catalytic coated GDLs in roll form
    - Attaining a short run length to assess catalytical and ionomer defects



DEIA/Community Benefits Plans and Activities

- Toward meeting compliance requirements of the Department of Energy's Office of Science's Promoting Inclusive and Equitable Research Plan, SkyVision Sciences has developed strategies and activities to promote diversity, equity, and inclusion into its research and development projects.
- SkyVision hires staff on the basis of technical excellence. Race, gender and sexual orientation do not play a role in our hiring practices. However, since 2010, SkyVision has hired more female scientists/engineers than male. SkyVision encourages a culture of inclusiveness and acceptance, where employees are comfortable communicating preferred pronouns, for example.
- The firm uses cross-matrix management where female and minority Principal Investigators or Program Managers are in charge of their projects, and team members assigned to those projects report up to the PI/PM; conversely, these female and minority employees that are team members on other projects report to those PI/PMs directing those other projects. In this manner, all employees work in different capacities as team members and project leaders promoting equity and inclusion.
- SkyVision will continue to use these strategies and present actions for meeting diversity, equity, and inclusion objectives for this proposed project.

Remaining Challenges and Barriers

- Rotational interrogation of MEAs
  - Interrogation of both sides (anode and cathode) of MEA
  - Interrogation of entire MEA surface using multiple sensors
- Interpreting data to detect defects in MEA
- Drum sensor fabrication improvements
  - Optimize individual component fabrication processes to improve quality control
    - Uniform membrane thickness
    - Conductive working electrode surface vs reactive surface
    - Optimize counter electrode

Proposed Future Work

- DOE SBIR Phase II Program will be used to focus on further development of technology including:
  - Optimization of individual sensor components including the sensor membrane, working electrode, counter electrode and reference electrode
  - Manufacturing scale up of drum sensor components
  - Assessment scale up
    - Assess larger membrane films with multiple drum sensors

Project Summary

- The Phase I DOE SBIR project has shown the technical feasibility of the in-line monitoring system for MEA manufacturing
  - Developed procedures to fabricate sensor membrane, working electrode, counter electrode
  - Designed and packaged the traditional three electrode wet electrochemistry test system into a drum sensor to interrogate MEA films
  - Sensor drums have shown the ability to measure catalytic activity of Pt coated films
  - Showed rapid response times of under 2 seconds using scan rates of 200 mV/s
  - Designed and fabricated reel to reel test system to rapidly evaluate MEAs