FY23 SBIR I: Solution Based Nanostructured Carbon Coatings for Reusable, Corrosion Resistant, Stamped Metallic Bipolar Plates

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Project Goal

- Renewables based green Hydrogen is a clean energy vector and a basis for an emerging industry that can help meet global decarbonization goals.
- Electrolyzers and Hydrogen fuel cells are the two major technologies required for the generation and effective use of green hydrogen in a cyclical fashion.
- High cost of the constituent stacks is the major impediment for their mass market adoption of Hydrogen fuel cell stacks.
- The bipolar plate is a critical component (see section II) that constitutes a significant portion of the hydrogen fuel cell stack cost.
- **Westwood, MA based Nano-C and its focused spin-off Clenersys**, are aiming at the cost-effective manufacturing of **Nanostructured Carbon Materials (NCM)** and key components based on those respectively, thus enabling US supply chains for a green Hydrogen adoption
- The goal of this SBIR Phase I project is to develop cost-effective, corrosion resistant, stamped metallic bipolar plates.

Overview

Combining the elegance of low-cost stamped steel with the electrochemistry of an ink based NCM coating, Clenersys' patented innovation CORE™, provides the most viable path among the competing technologies for the large-scale deployment of cost effective, metallic bipolar plates. During the SBIR Phase I our team has demonstrated the technical viability and pathways for commercialization.

Timeline : 9 Months (July 2023- Apr 2024)

Budget : $ 199,765.00

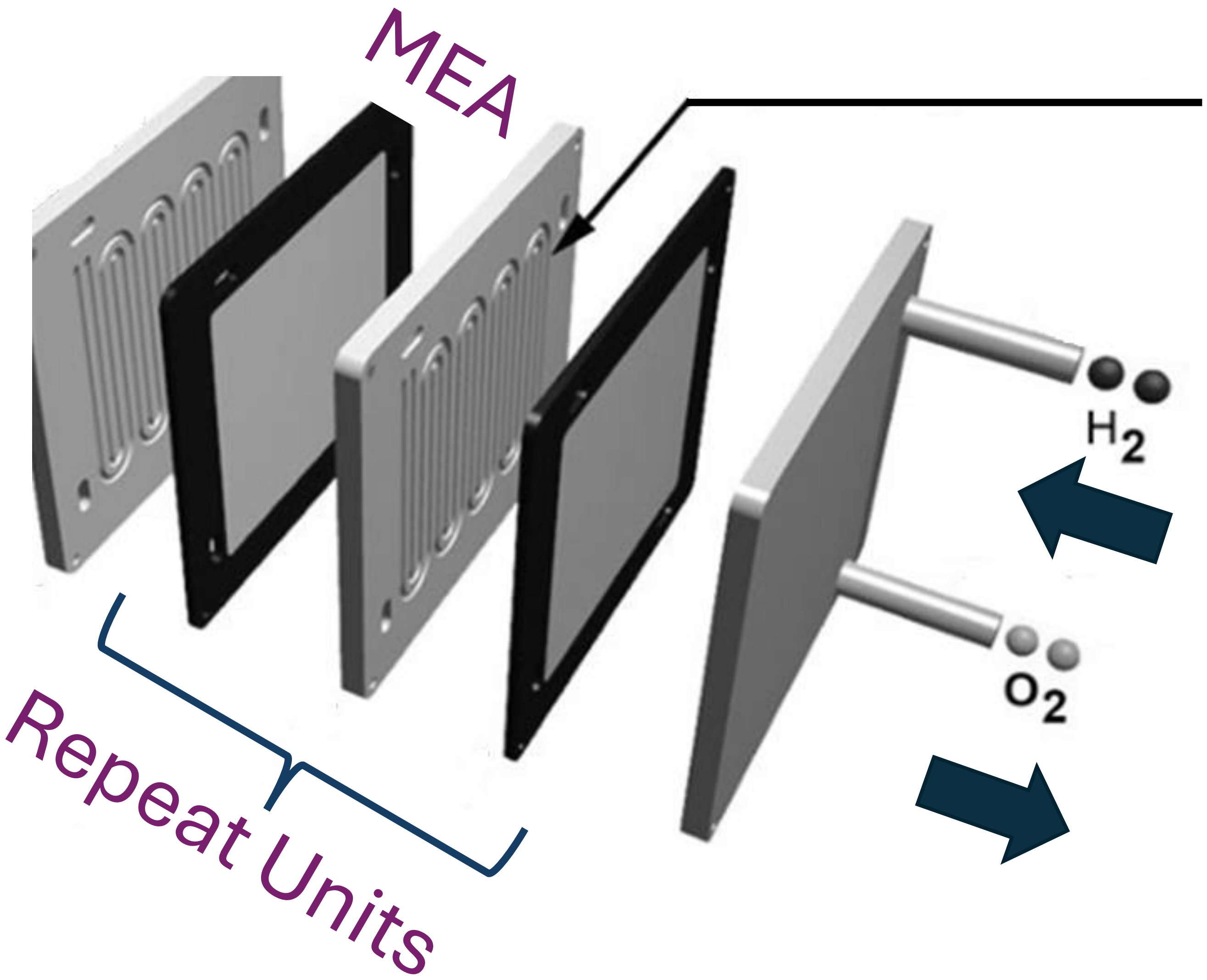
Barriers : See slide # 5

Partners :

Northeastern University

Center for Renewable Energy Technology

Basic Technology



A bipolar plate (BPP) in a PEM fuel cell serves multiple critical functions!

- Structural support for stack
- Electrical connectivity
- Distribution of gases
- Support for Membrane Electrode Assembly (MEA)
- Separation of cells
- Housing for coolant flow

Barriers and Market Needs

Competing Options	Value / Challenges
Machined Graphite	• Proven electrochemical performance. • Not suitable for vehicle use due to volume, cost of machining and fragility. Sheet process limits scale.
Stamped Titanium	• Cost is a concern for mass deployment. • Corrosion without additional protective coatings.
Gold/Nitride on Stainless Steel	• High-cost materials. • PVD process adds to costs and limits throughput. • Post deposition stamping is a challenge.
PVD Coated Carbon	• Carbon is less expensive than gold. • Coating provides good corrosion protection. • Expensive PVD process and limited throughput.
Clenersys ink Based Nanostructured Carbon Coatings on Metallic Substrates	• **Elegance of steel and electrochemistry of carbon.** • **Ink-based coatings and scalable post-deposition stamping for cost-effective, R2R manufacturing.** • **Utility and methods patents allowed worldwide.**

Potential Impact

- When renewables are used to generate green Hydrogen, it can be an effective clean energy vector for meeting the daunting decarbonization goals.
- Stamped metallic bipolar plates offer a cost-effective manufacturing pathway for the mass market adaptation of hydrogen fuel cells but face a steep technical challenge due to corrosion.
- The innovation presented in Phase I SBIR project aims to address the corrosion challenges, employing an ink based, corrosion resistant coating of nanostructured carbon materials.
- If the approach is successful, millions of square meters of stamped, coated metal plates will be manufactured in the United States, thus linking our highly skilled workforce from traditional steel manufacturing and automotive industries with the new generation jobs in a green hydrogen economy.

Collaboration and Coordination

- During the Phase I effort Nano-C, Clenersys and NUCRET collaborated
- Development of carbon materials, inks and coatings were carried out by Nano-C and Clenersys at their Westwood, MA facility
- Electrochemical characterization of the coatings were carried out at the Northeastern University Center for Renewable Energy Technologies (NUCRET) led by Professor Sanjeev Mukerjee.
- An undergraduate student, a graduate student and a post-doctoral were involved in the research work and closely collaborated with the senior scientists and staff members from Nano-C and Clenersys

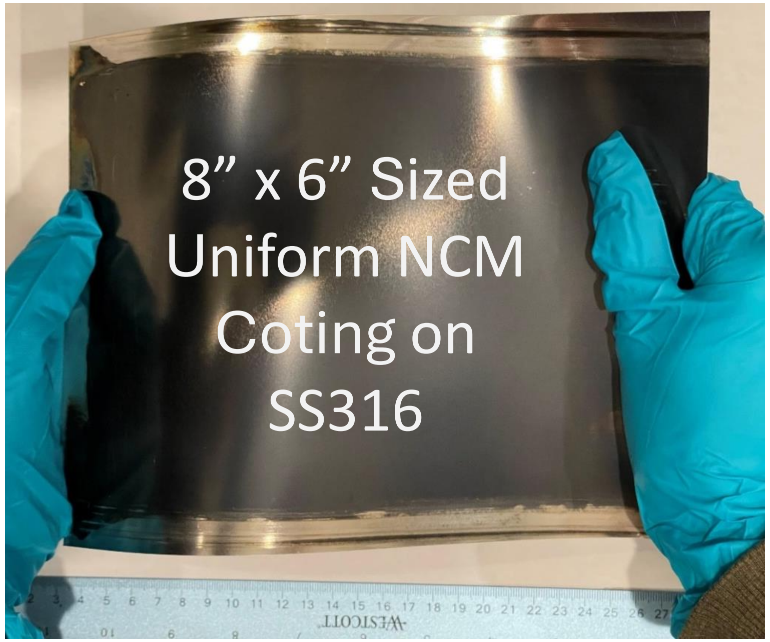
Phase I Accomplishments and Progress

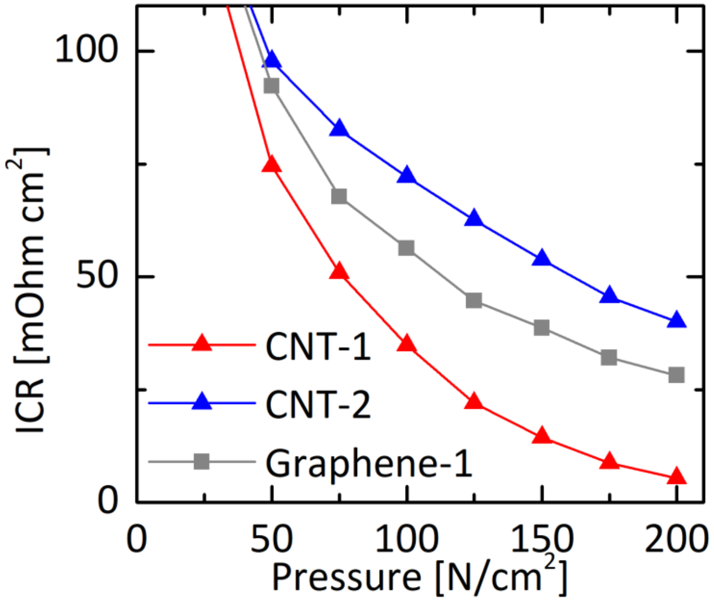
- Lab scale validations and proof of concept results related to CORE™ were obtained prior to the formal launching of Clenersys in 2021.
- Phase I efforts were aimed at further validation tests relevant to industry needs.
- Key Phase I objectives, approach for validation and accomplishments are summarized in the table

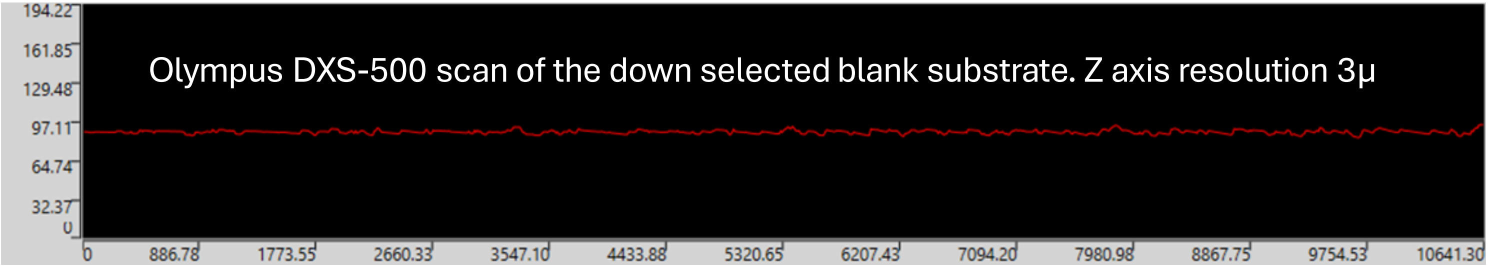
Key Objectives	Approach for Validation	Status
Interfacial Contact Resistance (ICR)	Screen multiple, commercial S316 steel substates. Optimize ink system and coatings to achieve an ICR of 10mΩ.cm^2 or closer.	✓
Corrosion Protection	Verify corrosion current density below 10^{-6} A/cm^2 sing three electrode corrosion cell .	✓
Adhesion and Structural Durability	Verify adhesion employing ASTM peel tests and abrasion using a Taber mechanical abrader	✓
NCM Electrochemical Stability Under Cathodic Conditions	Potentiostatic runs of coated steel at steady state cathodic conditions. Exposed coatings tested for adhesion and by Raman spectroscopy.	✓
Major Parameters for Scaled R2R Production	Coating in a lab-scale R2R system. Stamping of NCM coatings post deposition and surface scans.	✓
Meeting Cost Target for Deployment of 20,000 Stacks by 2027	Initiate third party cost analysis of the Clenersys supply chain, costs and cost sensitivity. Scoping for US supply chains to support production.	✓
Validation at Operating Conditions Emulating a PEM fuel cell	Test corrosion as a function of time within an emulated test stack at 80°C and > 90% RH when in direct contact with an acidic membrane.	(in progress)
CORE™ Validation Within Functioning Cell	Verify employing 25 cm^2 active area single test cells with solid graphite plates as reference control.	(in progress)

Inks and Coatings and Development

- SS316 stainless steel substrates from about half a dozen commercial sources and with varying thickness and surface roughness examined.
- Proprietary inks and dispersions of nanostructured carbons like different carbon nanotube types, graphene or their combinations were formulated and tested using multiple coating methods.
- Rod or bar coating method used for all ink-depositions presented here.

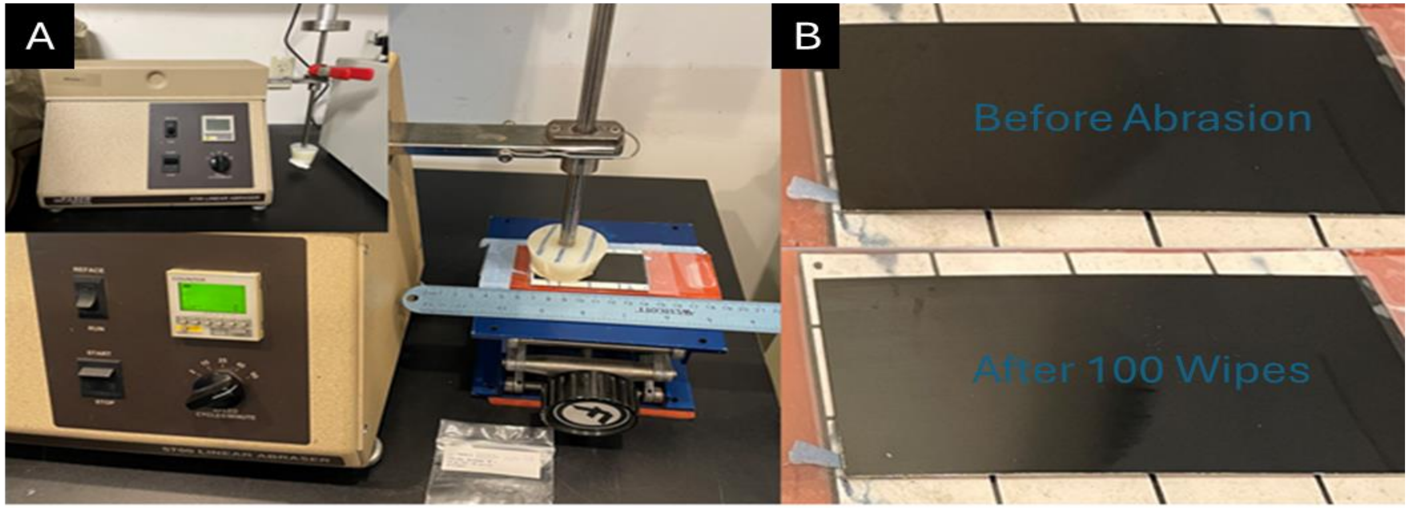


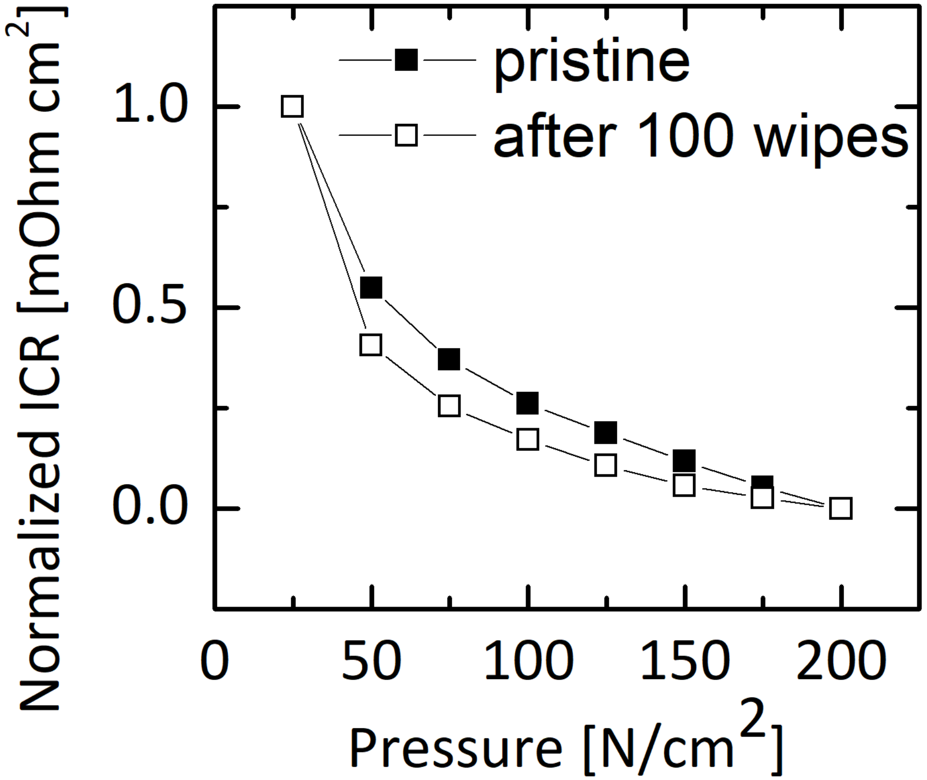




Coating Adhesion and Mechanical Durability

- Various NCM coatings were screened for adhesion (ASTM peel tests).
- Those passing test were further subject to abrasion tests using a Taber abrader
- Optical inspection and ICR measurements were carried out before and after the abrasion wiping to evaluate mechanical durability of the coatings.

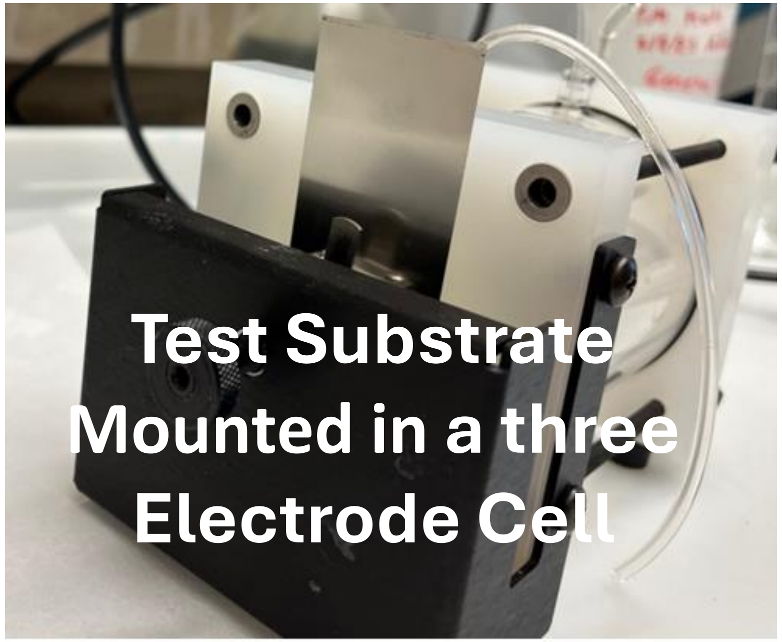


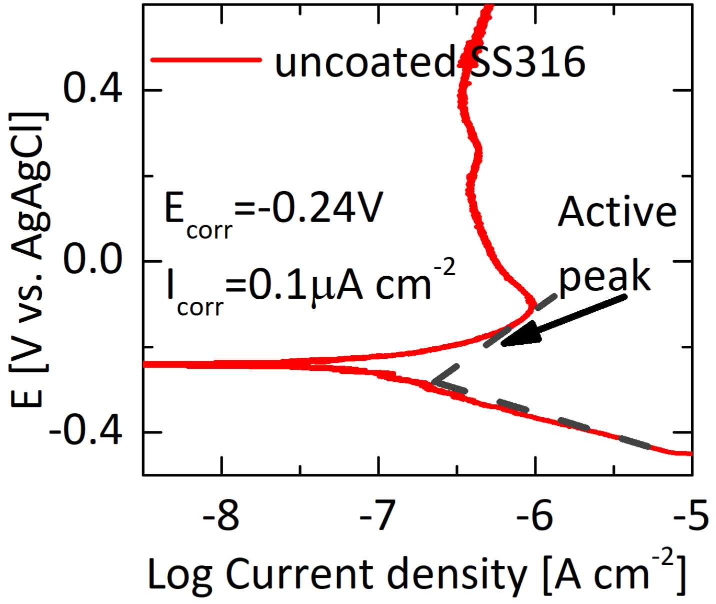


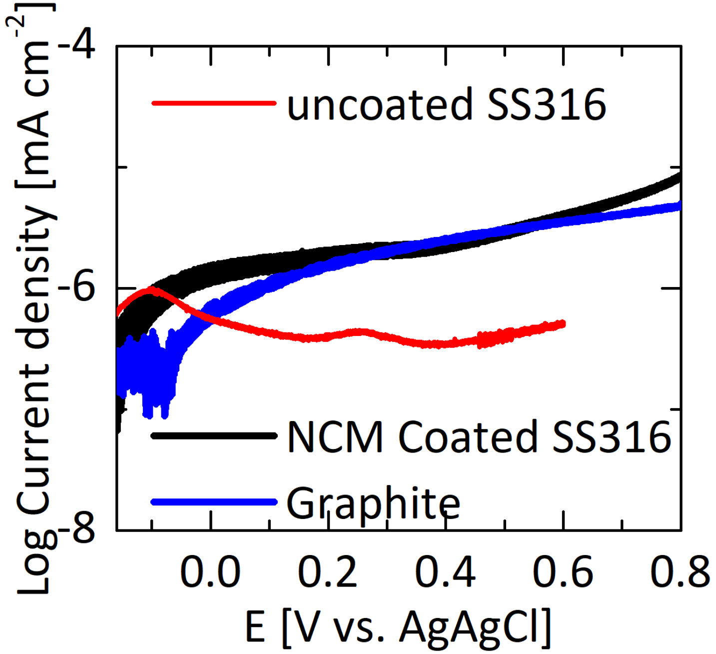
Corrosion Behavior of Coating Similar to Graphite!

- Polarization measurements* in 1mM H_2SO_4 + 1ppm F^- under Ar. Active peak marked in the case of uncoated SS316. **No active peaks seen with NCM coating**.
- Polarization behavior of NCM coated SS316 similar to Graphite than to steel.

** Conditions same as reported by Kopasz, J., and T. Benjamin., Argonne National Laboratory. Bipolar Plate Workshop Report., ANL 85 (2017):* 654-668).

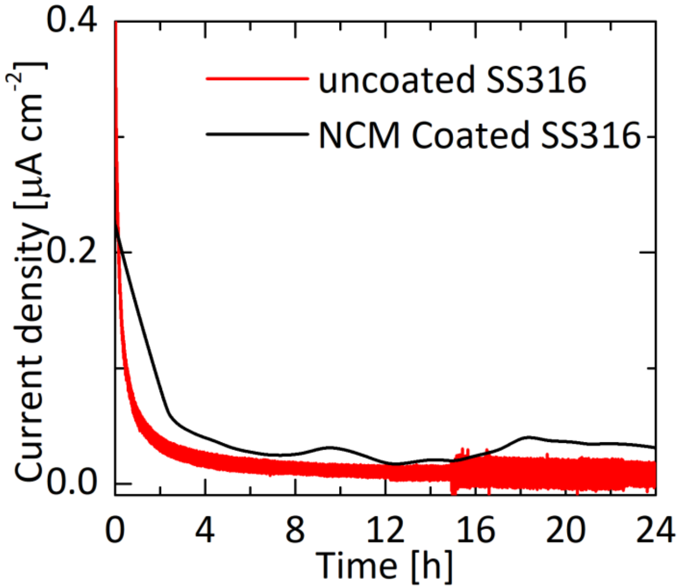


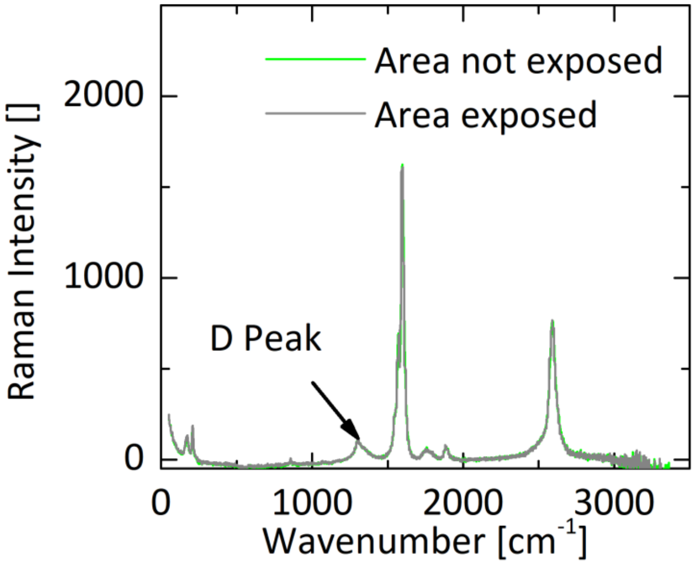


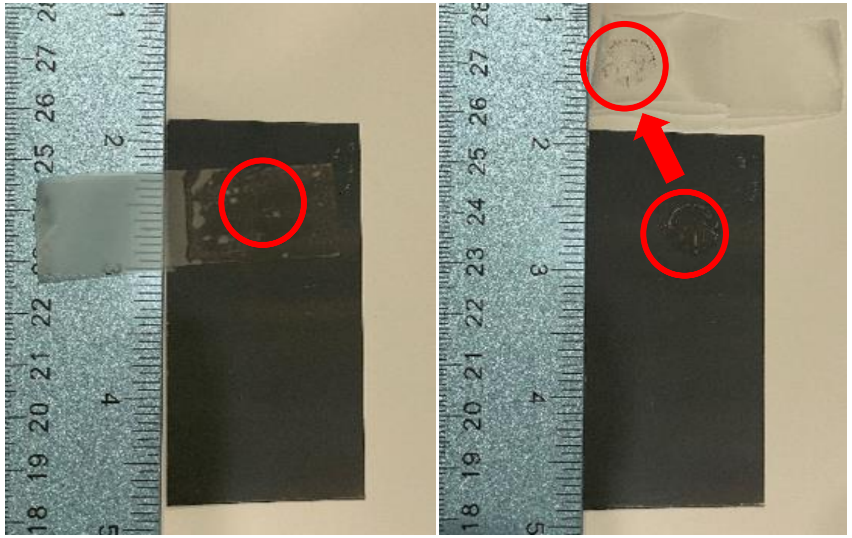


Electrochemical Stability

- Potentiostatic tests were conducted at steady state conditions for 24 hours. Corrosion currents below DOE specified limits.
- Recovered samples were evaluated *ex-situ*. **Raman spectra showed no Eklund shift or change in D-peak intensity, indicating high electrochemical stability.**
- Peel tests showed that exposed areas remained adhered to the steel substrate

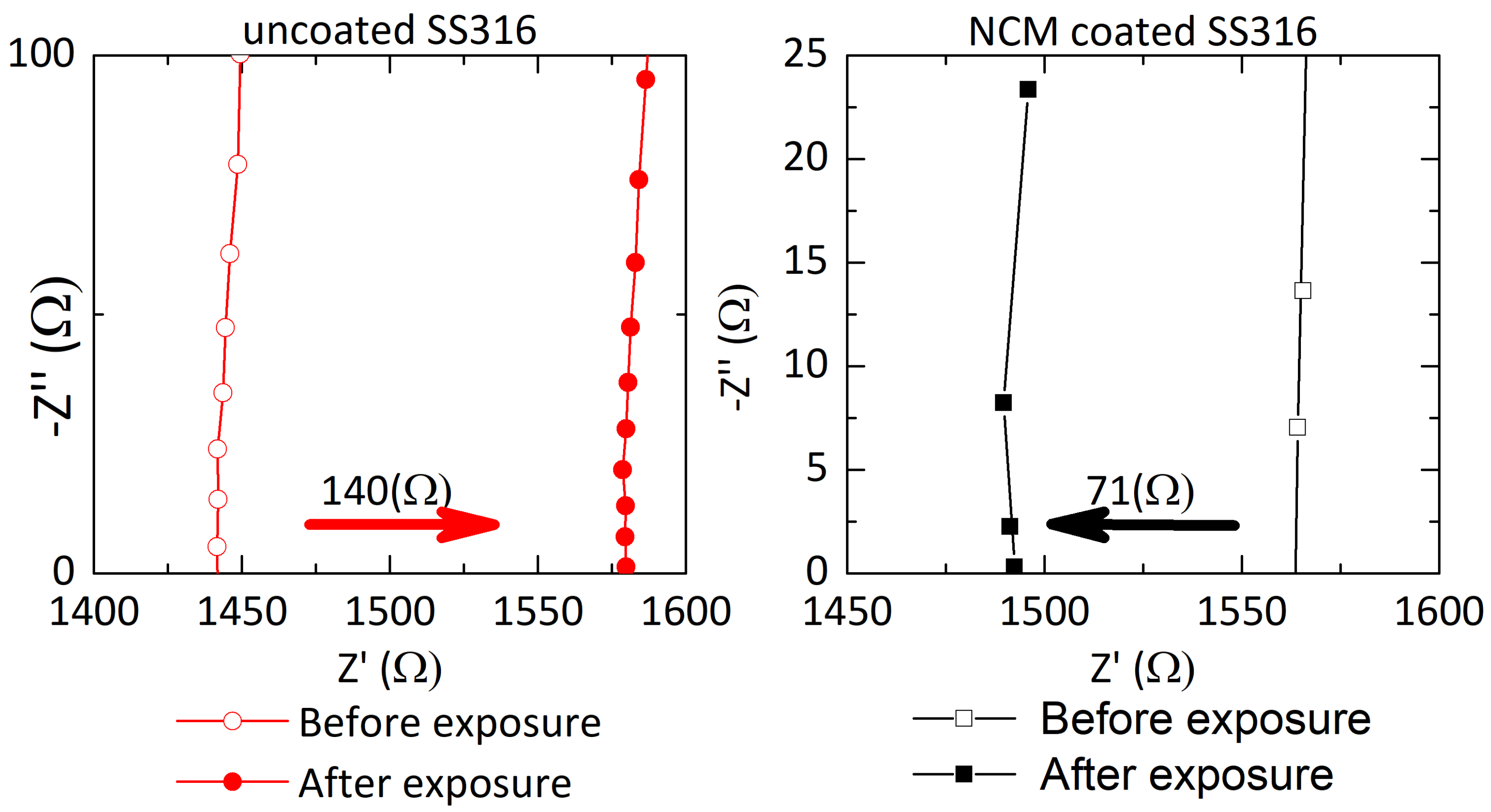






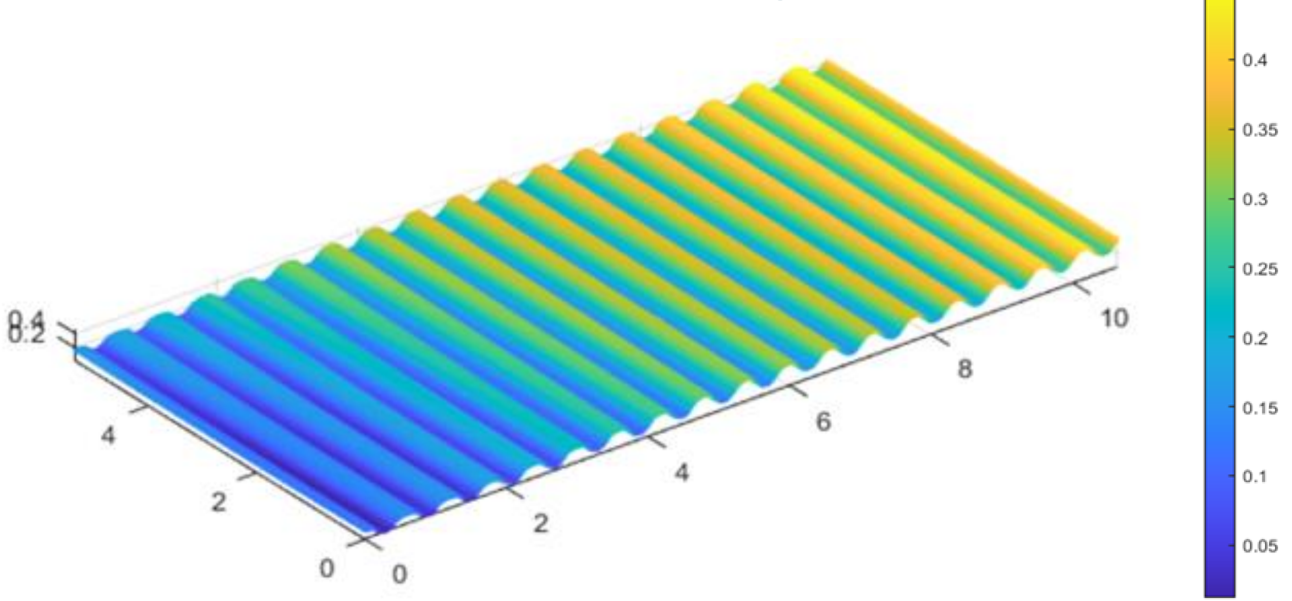
Corrosion and Electrochemical Impedance

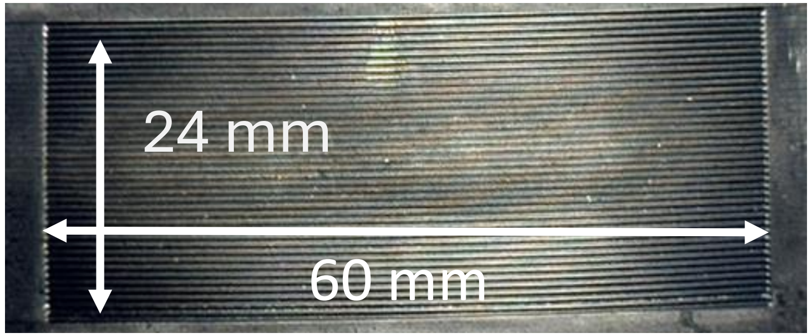
- Electrochemical impedance measurements in the (1Hz-100 kHz) range were recorded *in-situ* at the beginning and end of the 24 hours cathodic exposure.
- Uncoated SS316 recorded an increased Z' compared to the NCM coated sample.

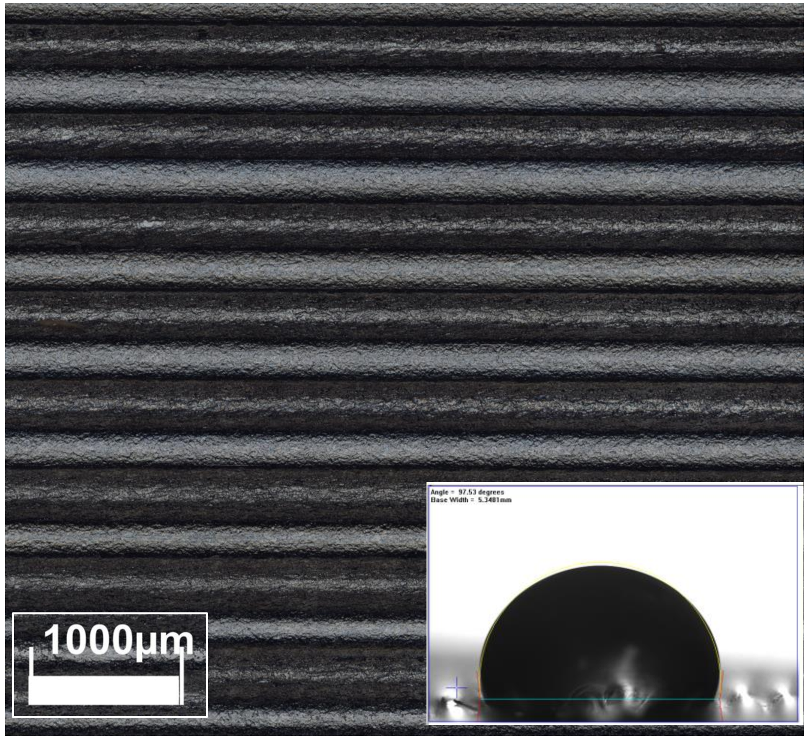


Validation of 'Stamping After Coating'

- NCM coatings on SS316 can be formed post deposition by high-velocity stamping.
- High resolution optical profilometric scans reveal conformal flow of the NCM coating during the strenuous stamping process.
- Water contact angle measurements post stamping show that the formed NCM surface is highly hydrophobic.

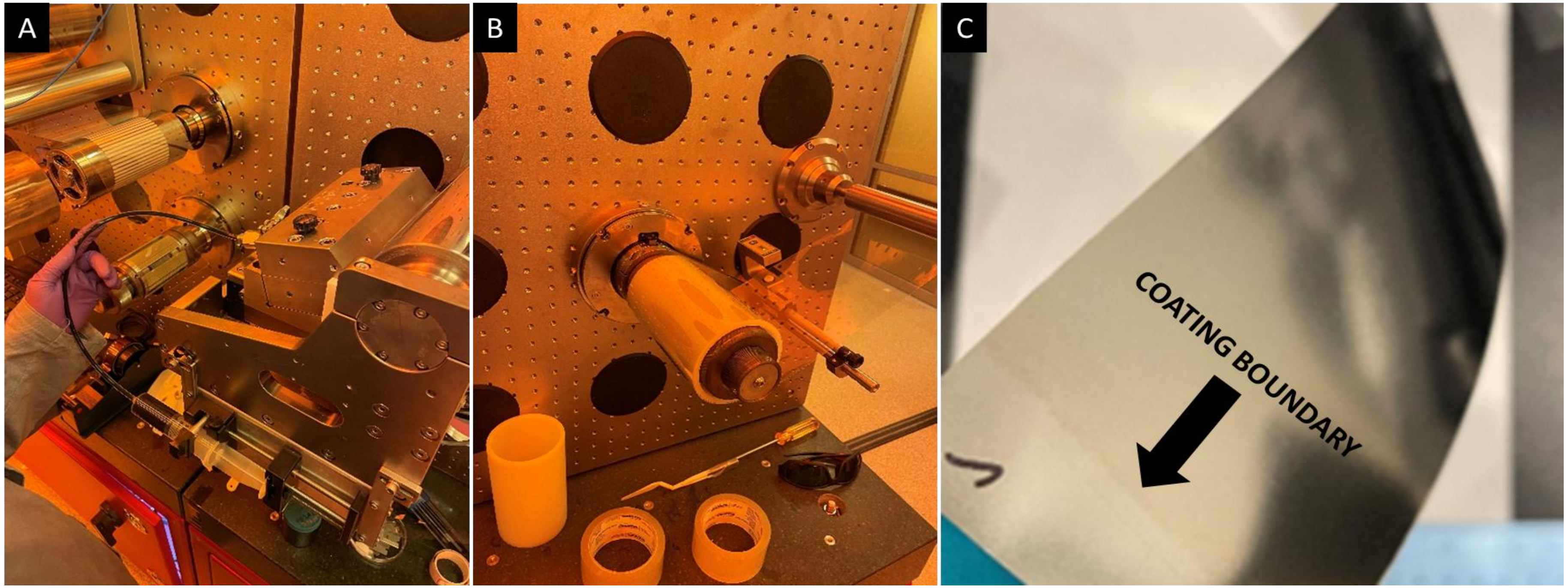






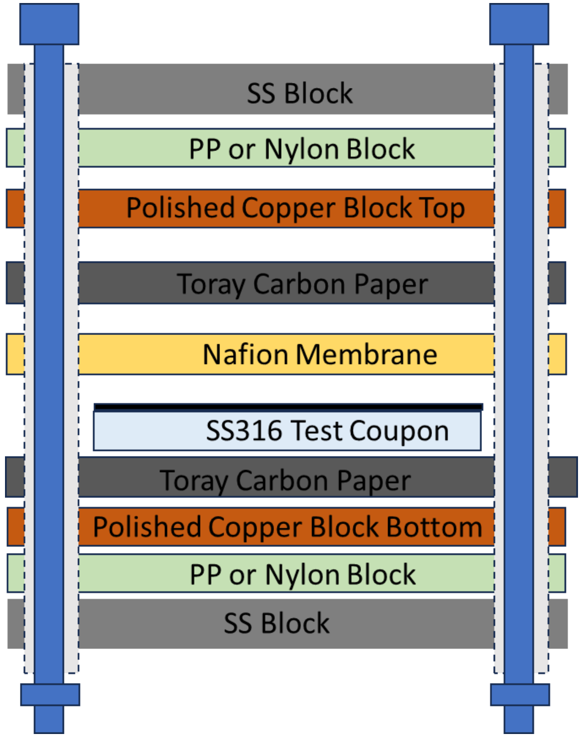
Compatibility with Roll-to-Roll Manufacturability

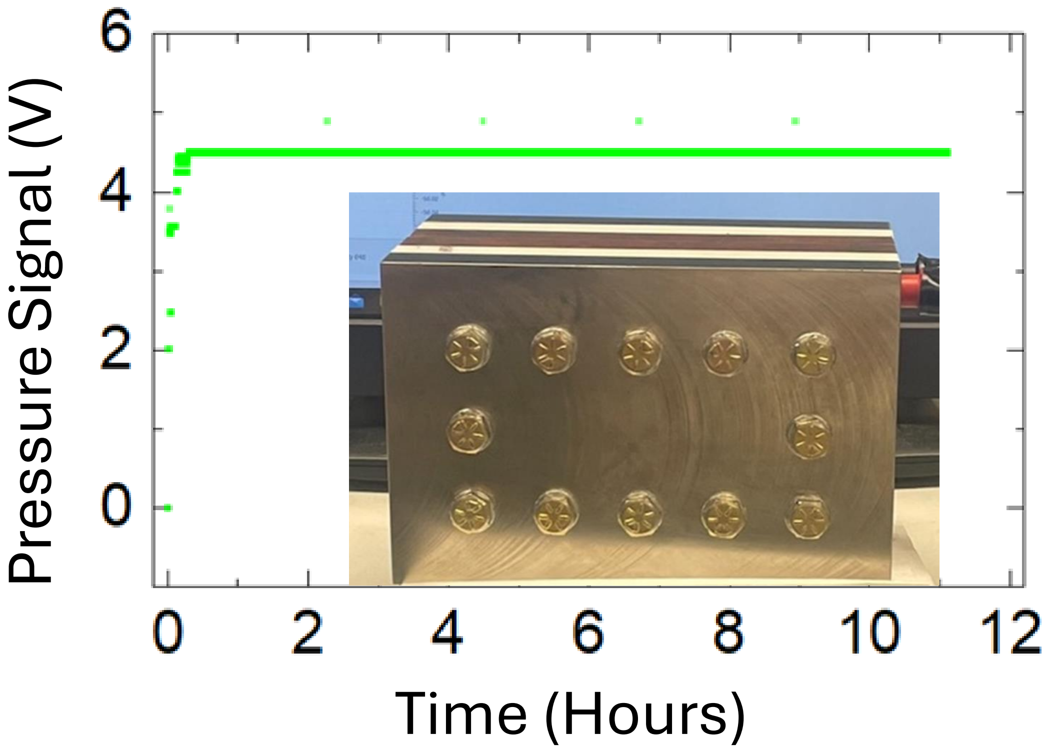
- Sections of the R2R coating system used to verify roll-to-roll coating compatibility
- Thinner ink used for test run
- SS316 substrate of size 12”x3” was tethered to a moving plastic web
- Uniform coating and boundary seen



Stack Emulating PEM FC Operation

- Emulation test stack built and verified to sustain 1.5 MPa pressure.
- Stack can be operated inside a controlled environment chamber.
- Real time data acquisition at 80 °C and > 90% relative humidity.
- Performance data presented as part of Phase I final report





Proposed Future Work

- Apply for an SBIR initial Phase II award as a follow-up to the Phase I project presented here
- If a Phase II award is received, further elevate the Technology Readiness Level (TRL) and commercialization progress by addressing the following tasks in collaboration with an expanded team of experts from industry and academia

  - Coating performance and coating yield or large area coatings in line with cost target.
  - Stamping of NCM coatings on steel compatible with R2R and throughput needs
  - Performance of the NCM coatings within a PEM stacks of power exceeding 1kW or more
  - Beachhead customer/ potential customer involvement

- Any proposed future work is subject to change based on funding levels.

Summary

- Inks and their coatings based on Nanostructured Carbon Materials (NCM) such as single walled carbon nanotubes and graphene were developed.
- Uniform, large area NCM ink coatings on SS316 substrates demonstrated.
- Electrochemical response of NCM ink coatings on steel verified to be similar to Graphite than to steel.
- NCM ink Coatings on SS316 verified to pass adhesion and abrasion tests.
- Corrosion current for NCM ink coated SS316 seem below the DOE set target levels.
- Electrochemical stability of NCM ink coatings verified by Raman spectroscopy.
- NCM Ink coatings on steel can be stamped after deposition.
- Early trials showed compatibility of the NCM inks with an R2R coating process.
- Stack emulating real-life PEM-FC operating conditions designed and constructed
- Performance of the NCM ink coatings within the emulated stack and a functional PEM cell with 25cm^2 MEA are ongoing. Proprietary performance data to be presented as part of the Phase I final report.