



PROTON

THE LEADER IN **ON SITE** GAS GENERATION.

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Single Step Manufacturing of Low Catalyst Loading Electrolyzer MEAs

Award #: DE-SC0009213

Project Team: Proton OnSite (Prime)

Partner: University of Connecticut (Subcontractor)

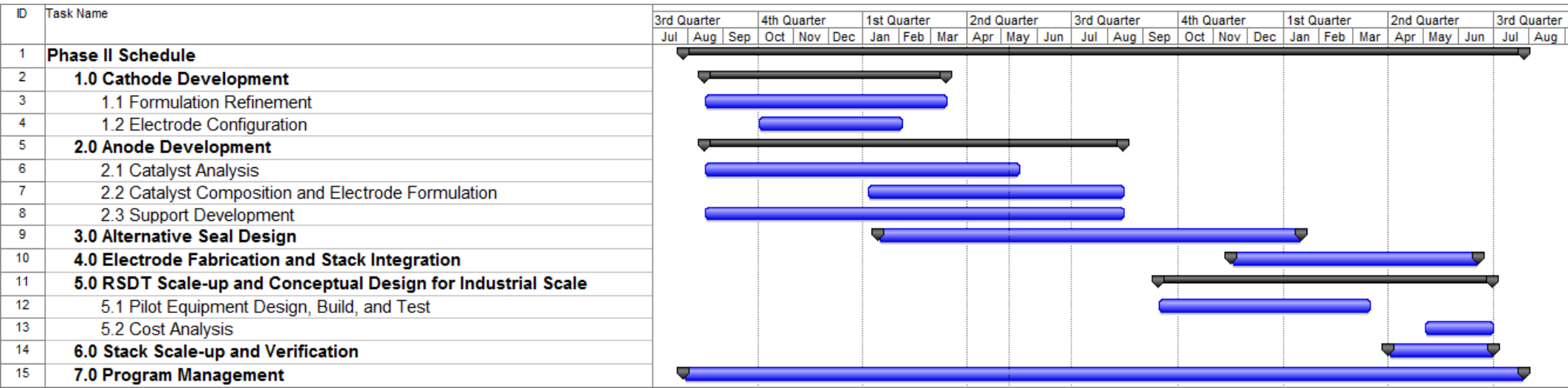
U.S. DOE Advanced Manufacturing Office Program Review Meeting
Washington, D.C.
May 28-29, 2015

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Project Overview

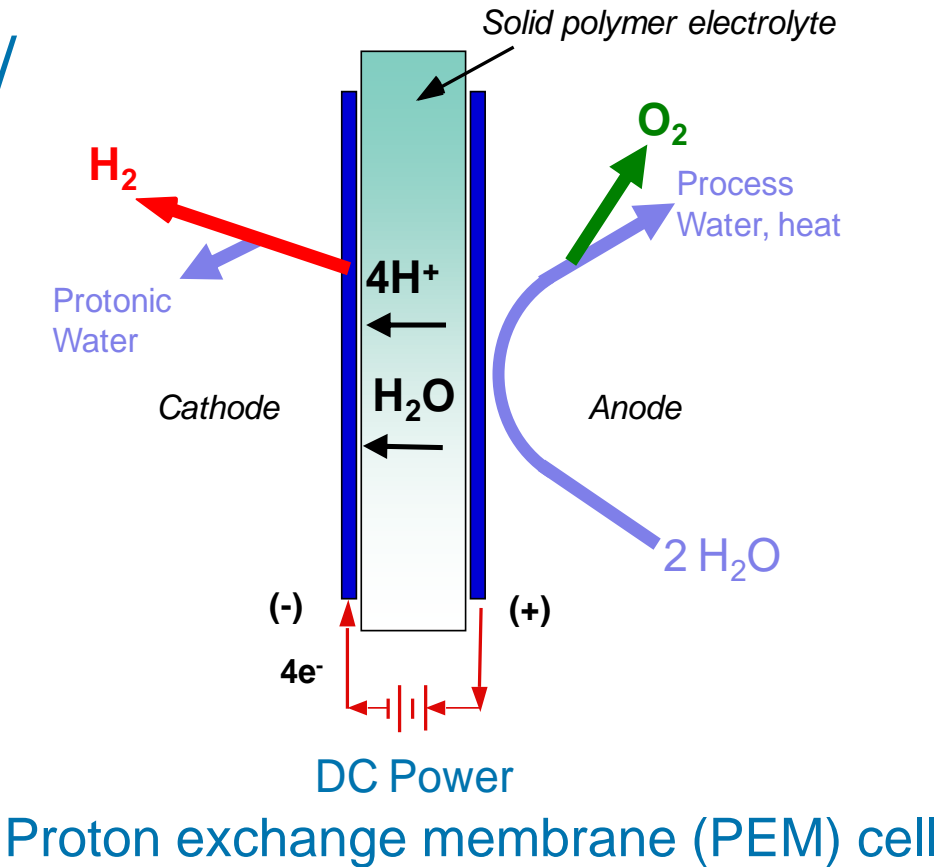
Project Management		Total Project Budget	
P. I. Name	Dr. Katherine Ayers	DOE Investment	\$1,150,000
Presenter		Cost Share	\$0
Organization	Proton OnSite	Project Total	\$1,150,000

Project Start: 15 Nov 2012 (PH I)
 Project End: 27 July 2016 (PH II)



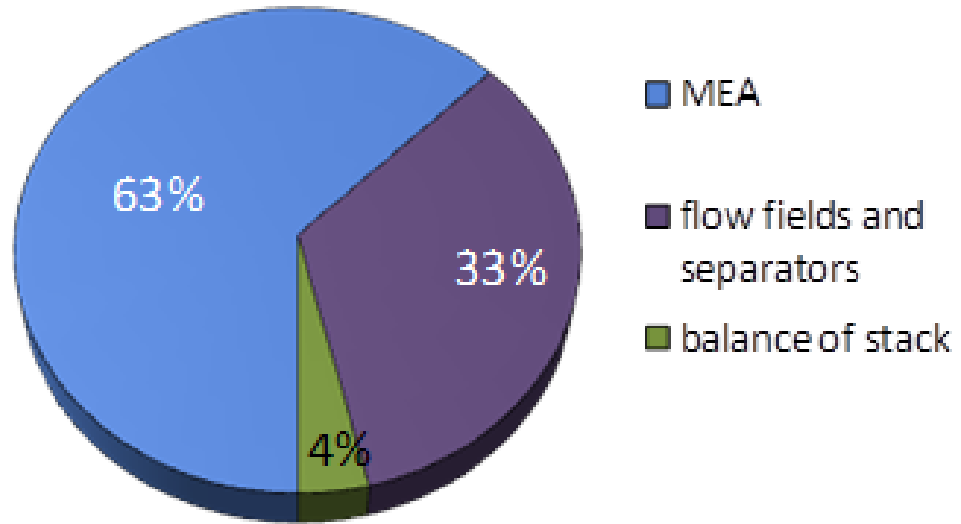
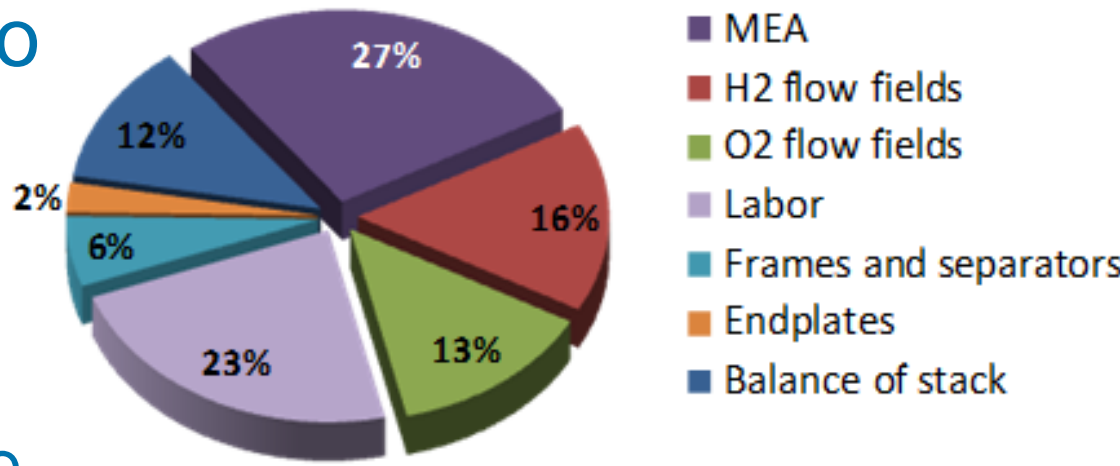
Project Rationale

- Water electrolysis is the only viable near term solution for renewable hydrogen generation
- PEM electrolysis is the technically preferred option
- Need to address high cost/
high energy electrode manufacturing processes
 - High catalyst loadings:
high energy cost in mining



Current Practice

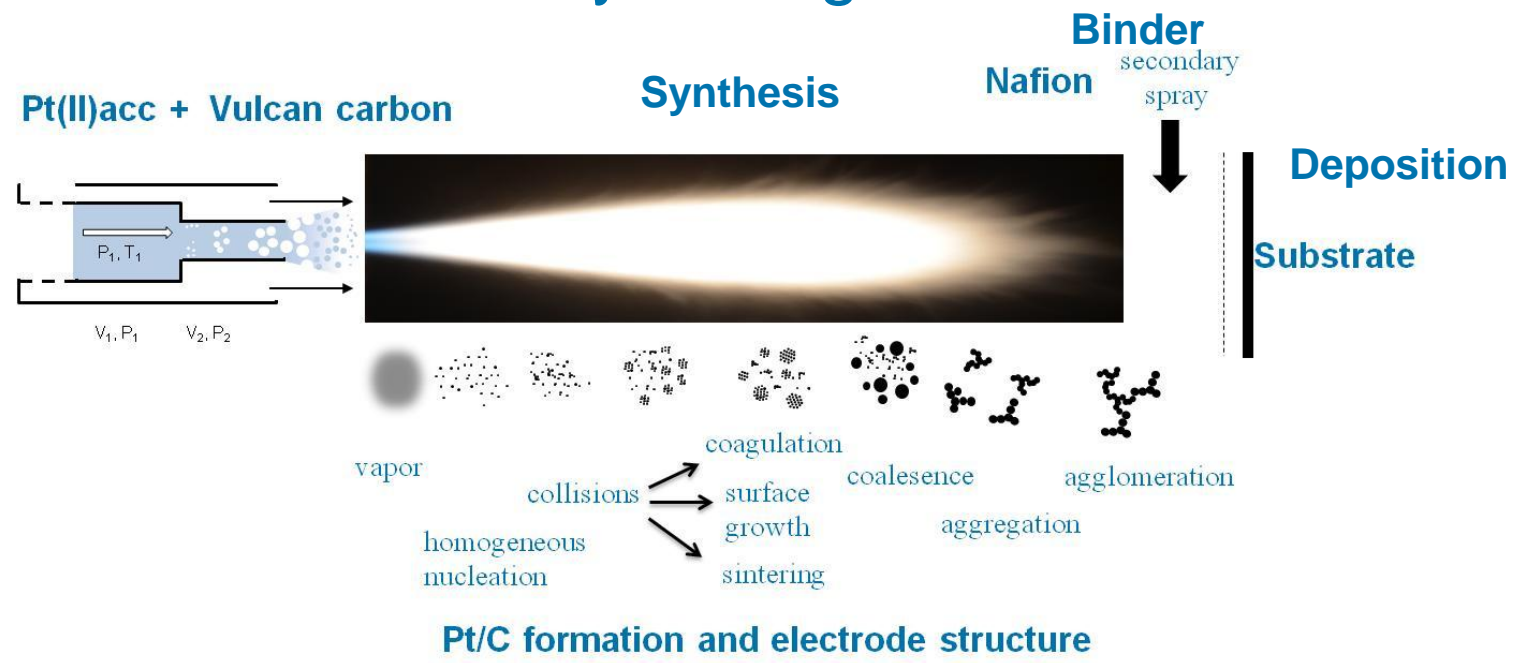
- Legacy process dating to Apollo space program
- Manual operation uses high loadings
 - Overdesigned to manage process inconsistencies.
 - Multiple process steps and significant amount of “art”
- Energy intensive steps throughout process.



Cost breakdown (top) and labor breakdown (bottom) for major stack components

Solution

- Reactive spray deposition technique (RSDDT)
 - Addresses major cost and energy contributor in PEM electrolyzer through reduced PGM usage
 - Flexible to catalyst type (metal, oxide) and substrate (polymer, carbon, metal)
 - Enables reduced catalyst usage and roll to roll fabrication

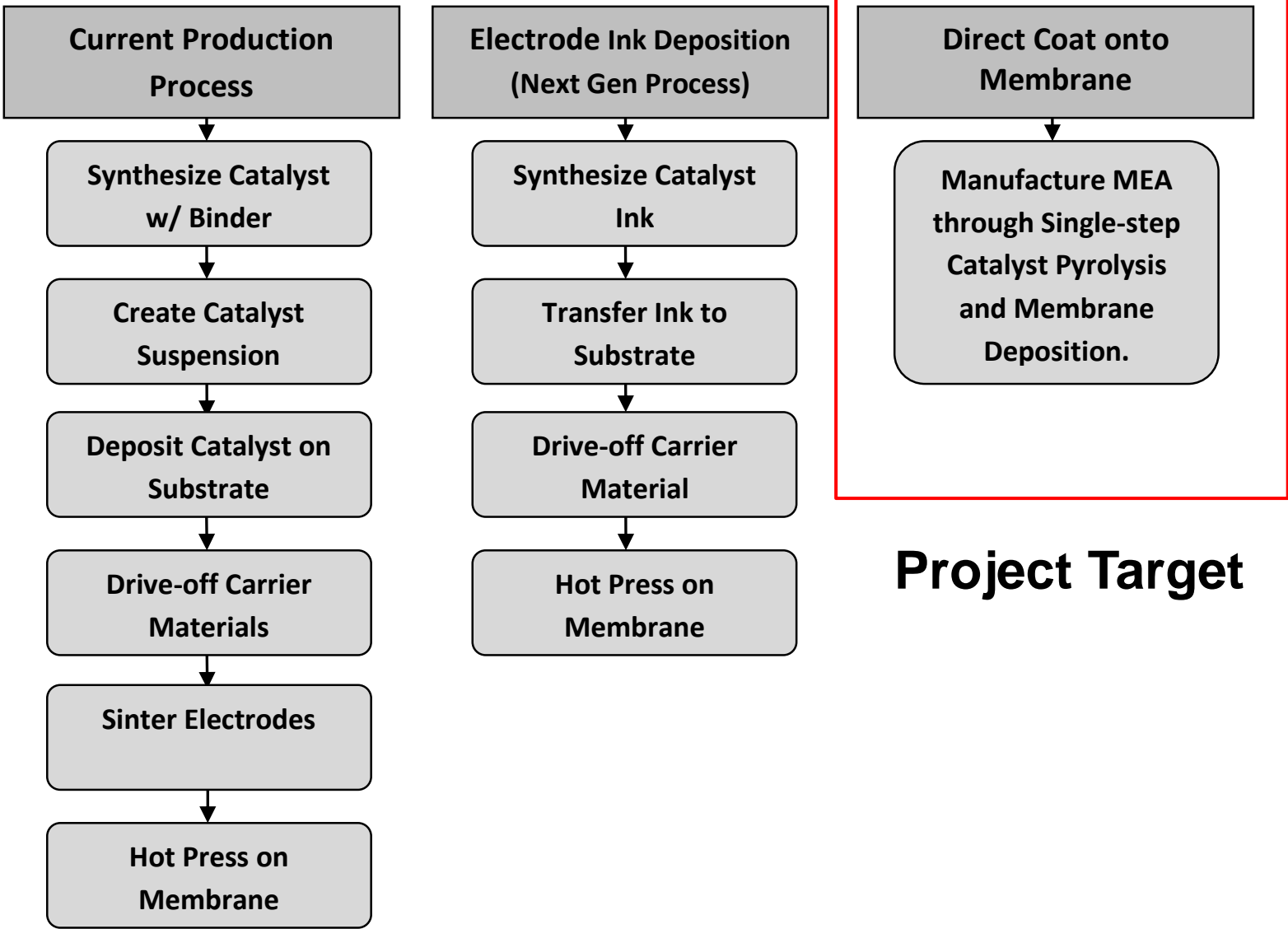


Project Objectives

- Develop low metal-content electrodes
 - Optimize catalyst composition
 - Down-select electrode configuration
 - Develop optimal formulation for RSDT
 - Show feasibility of low membrane usage MEAs
- Scale-up to high volume, low-energy deposition of catalyst with improved utilization
 - Develop pilot scale RSDT apparatus
 - Scale up MEAs & demonstrate stable performance

Project Objective: Scope

MEA Manufacturing Process Development Pathway



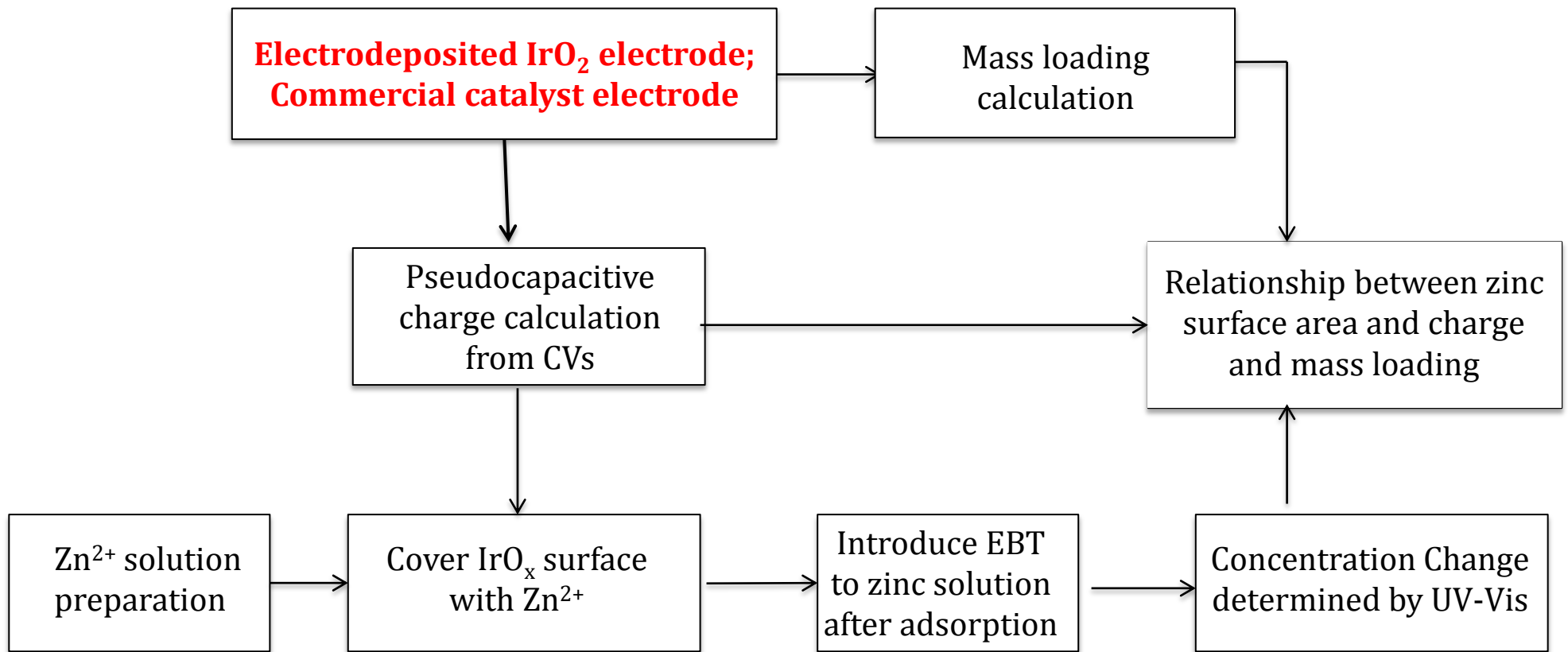
Project Target

Technical Barriers

- Determination of anode catalyst utilization
 - Traditional electrochemically active surface area techniques not viable
- Catalyst optimization by RSDT
 - Tune parameters for ideal structure, thickness, support/ionomer ratio, and porosity
- Development of stable electrocatalyst supports
 - Anode material compatibility presents challenge
- Scale-up and process demonstration
 - Translate practice to apparatus capable of high volume, low energy manufacturing

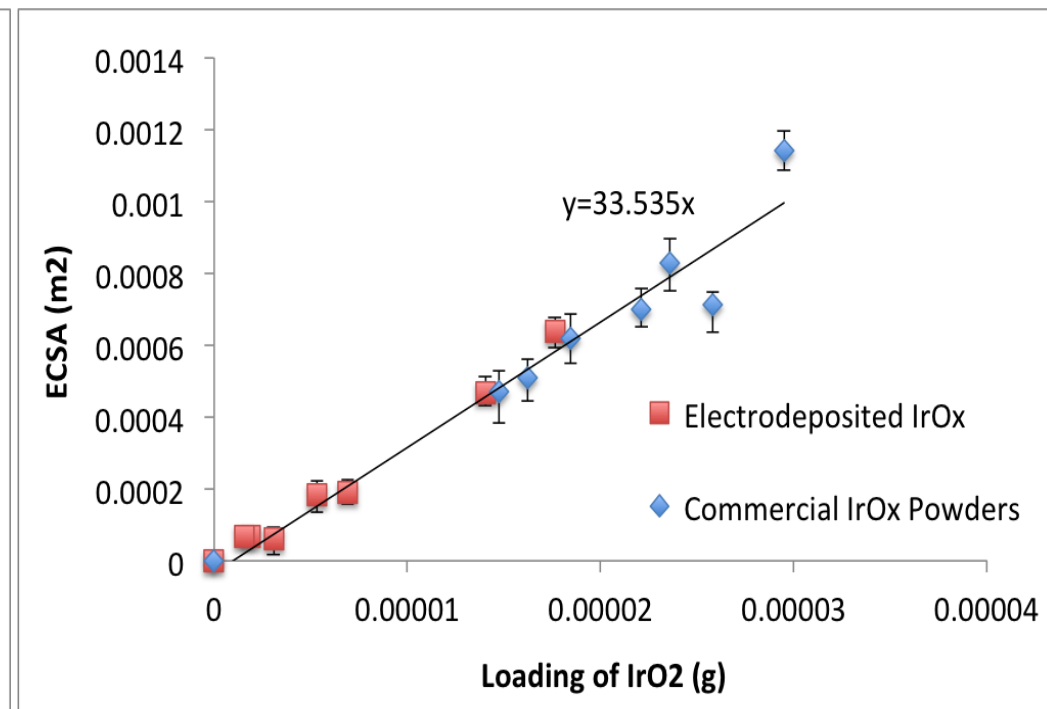
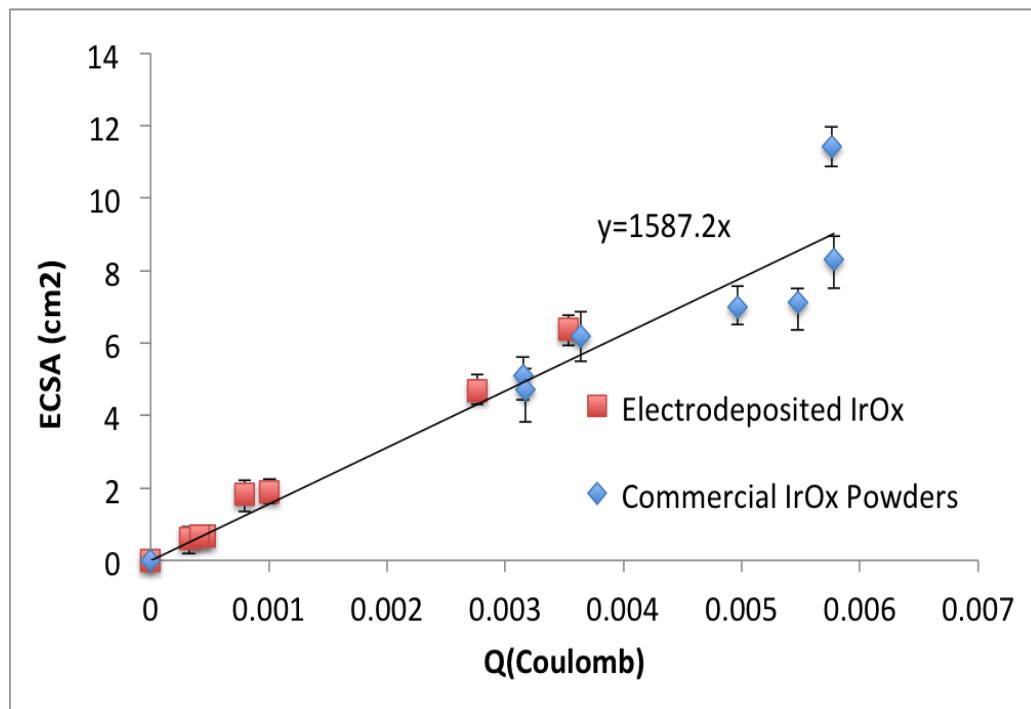
Technical Approach: IrO_x ECSA

- Zinc cation adsorption method



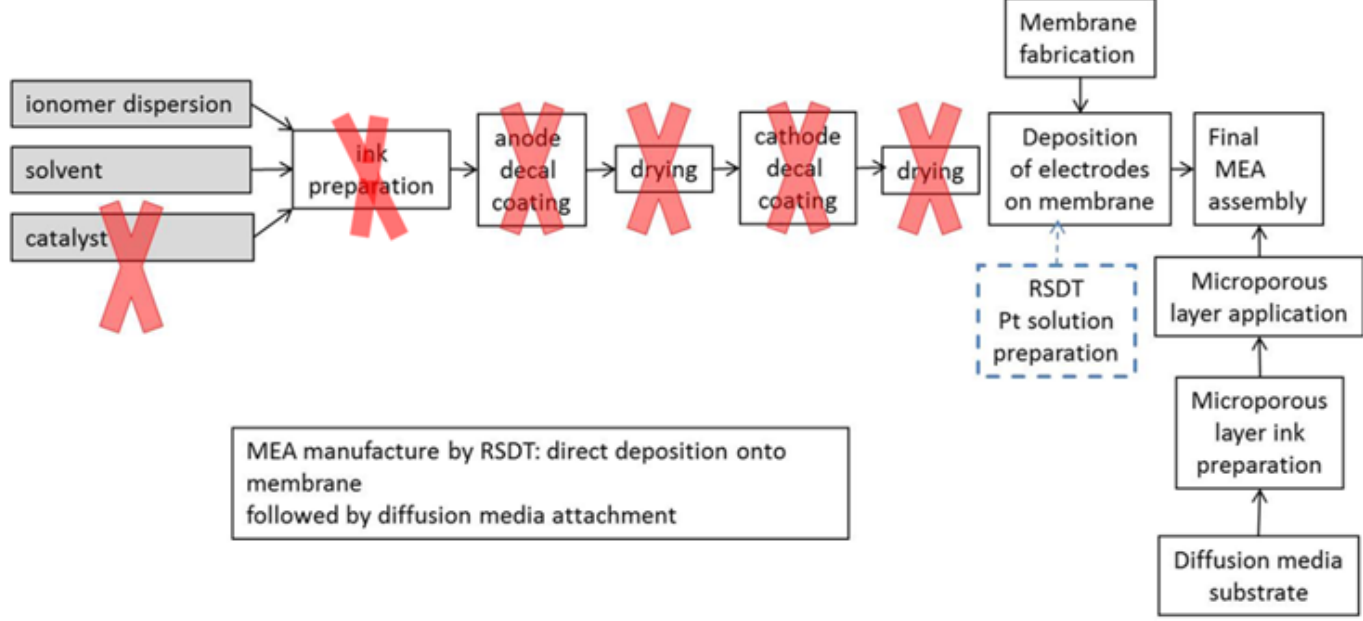
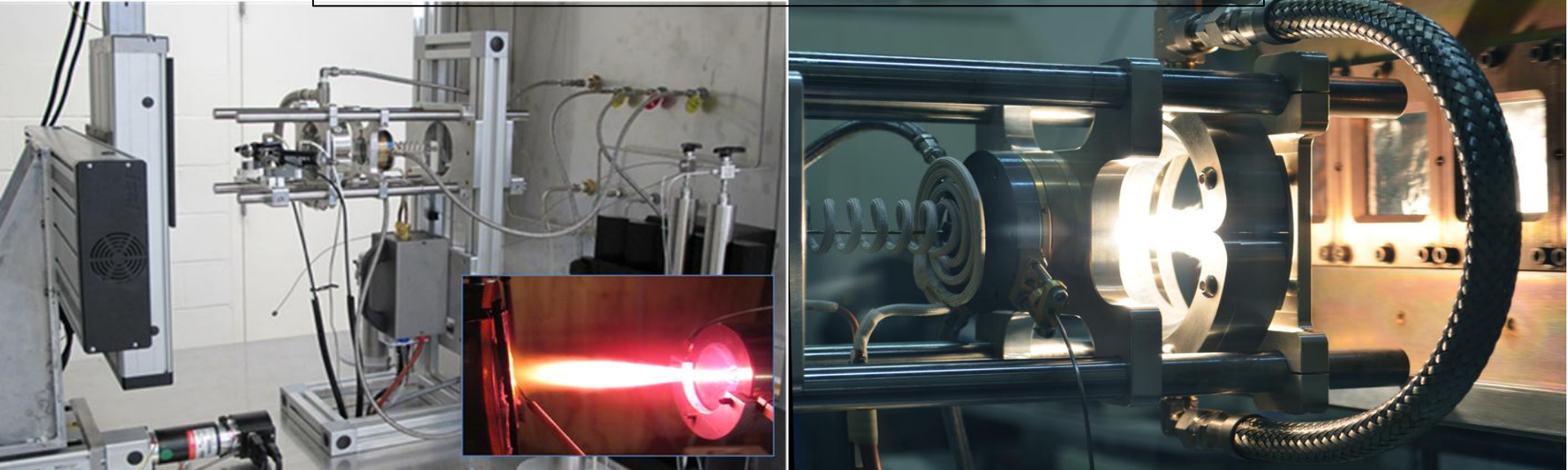
Technical Approach: IrO_x ECSA

- Two electrochemical systems merged together to give out the final correlation values
- Method of quality control for RSDT



Technical Approach: RSDT

Co-PI Maric's RSDT Manufacturing Setup at UConn



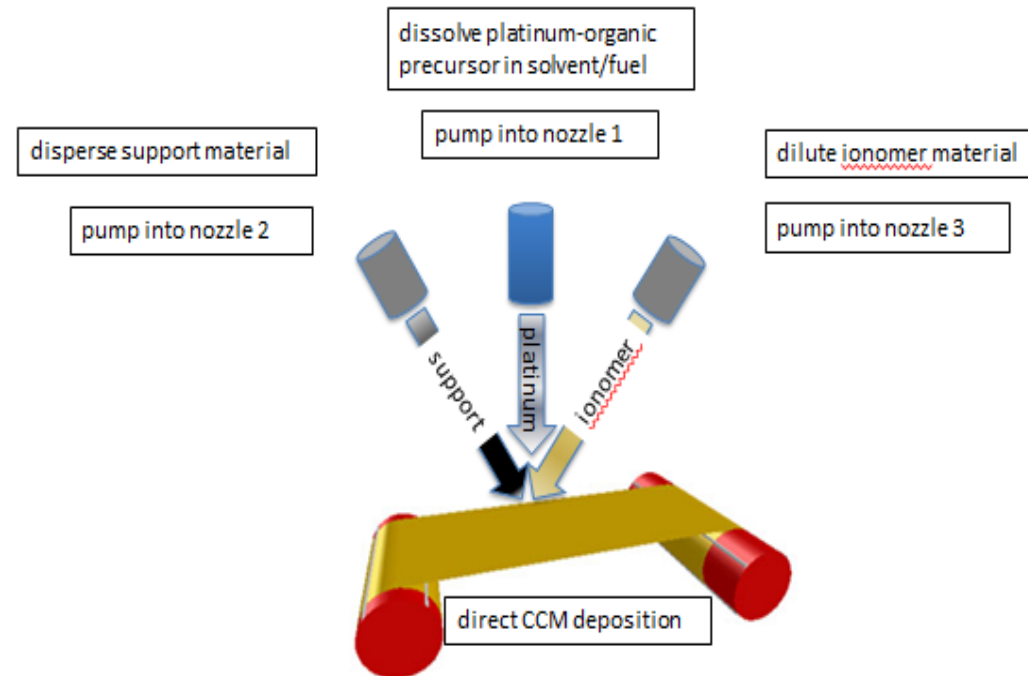
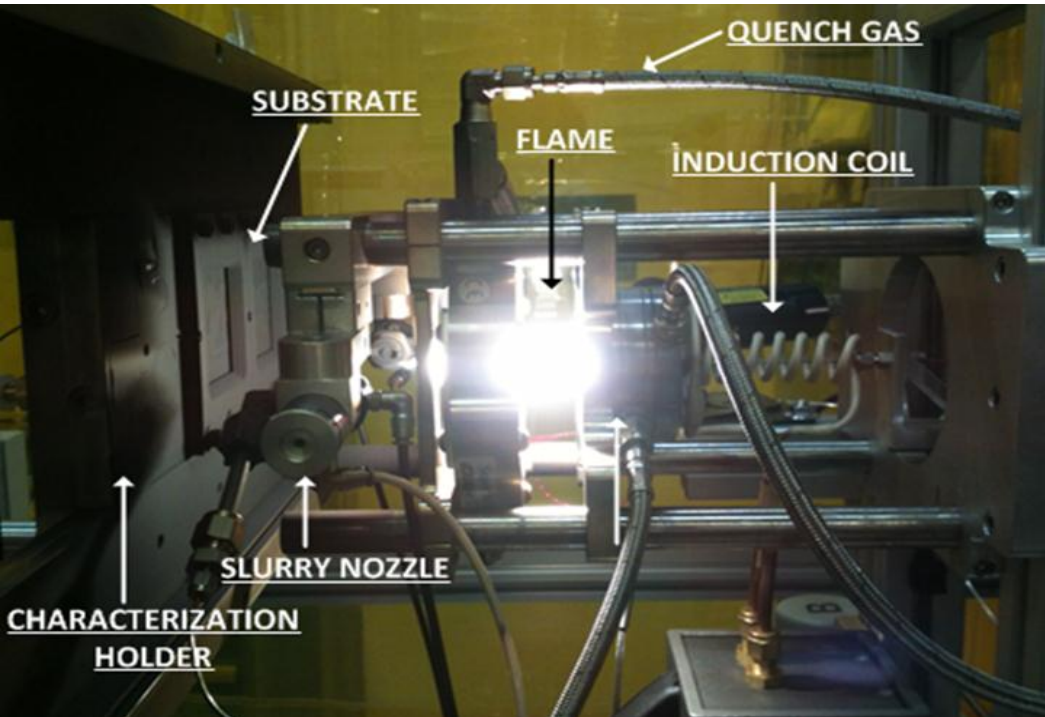
Technical Approach: Optimization

- Independent parameter control during deposition process

Process Parameter	unit	area of interaction
precursor concentration	molarity	metal deposition rate, platinum morphology, droplet size and back pressure
flow rate	mL/min	flame length, atomization, particle formation and residence time, temperature profile
nozzle temperature	°C	droplet size, dryness of droplets, back pressure and spray stability
collimating gas	SLPM	spray angle, flame velocity, laminar or turbulent flame, oxidizing potential, and substrate temperature
pilot oxygen and methane	SLPM	maintain combustion of spray
equivalence ratio	unitless	flame temperature, shape, droplet residence time
quench air flow rate	SLPM	cools reaction zone, Pt deposition pattern
quench angle	θ from centerline	deposition rate and pattern, substrate temperature, causes eddys

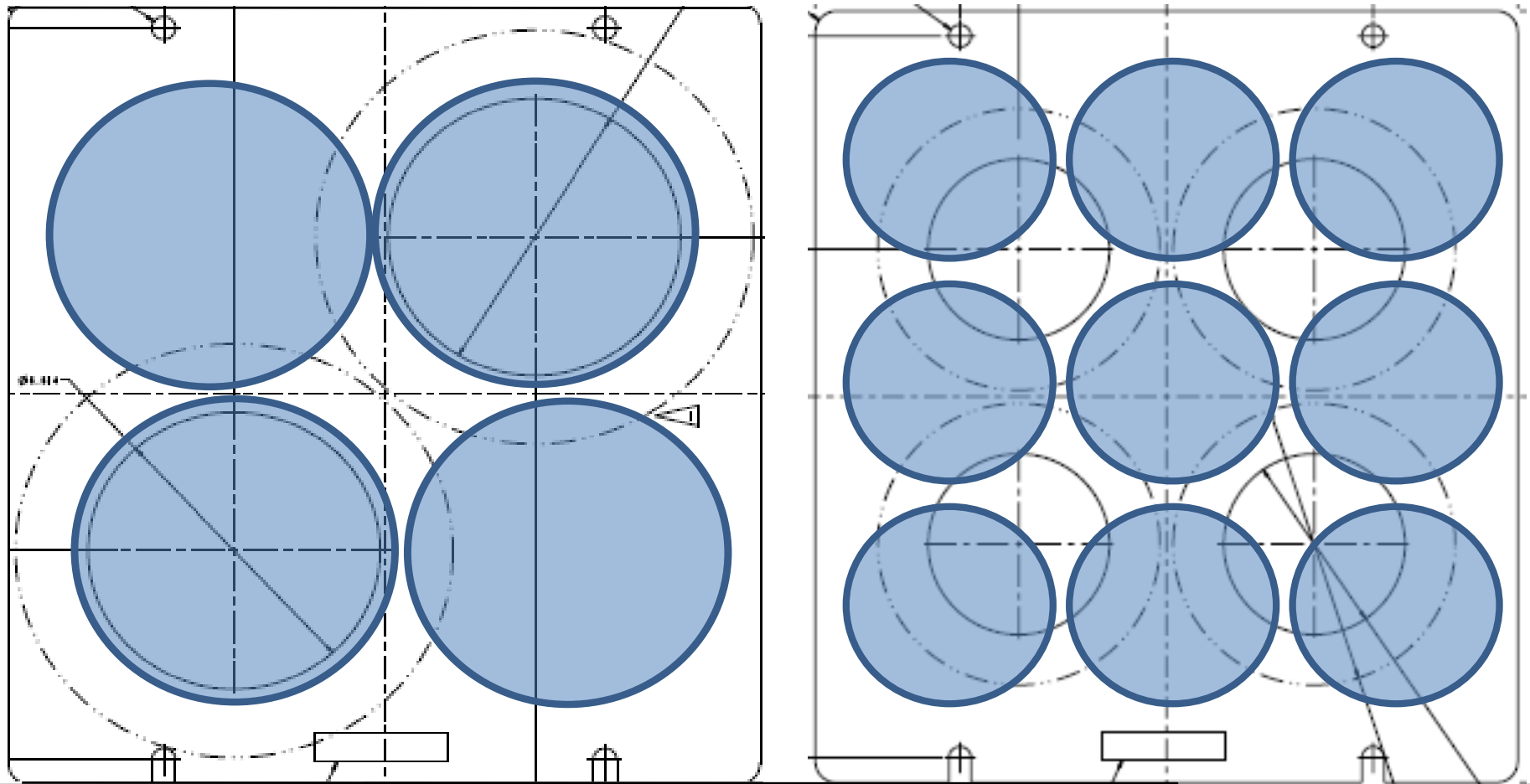
Technical Approach: Scale Up

Current RSDT equipment at UCONN (left) and a schematic of proposed roll-to-roll process (right).



Technical Approach: Alternative Sealing

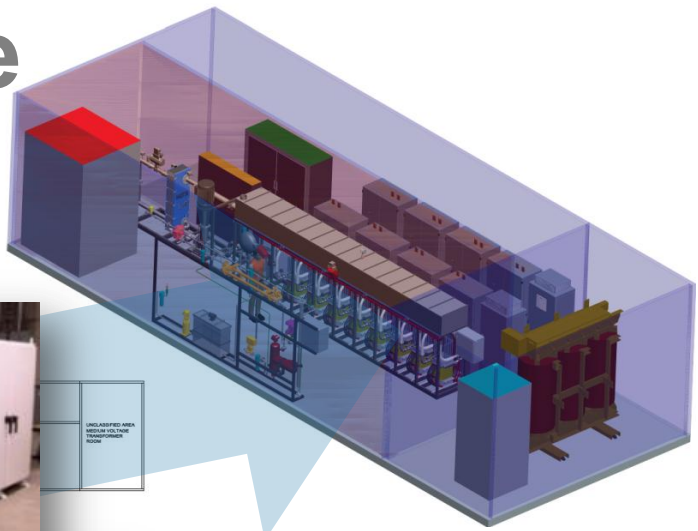
- Development of low membrane usage MEA design enables throughput up to 2X.
- Allows for easier integration with web process



210 cm² MEA (left) and 86 cm² (right).

Commercialization Experience

From Single to Multi-Stack Systems



M Series



HOGEN® C Series



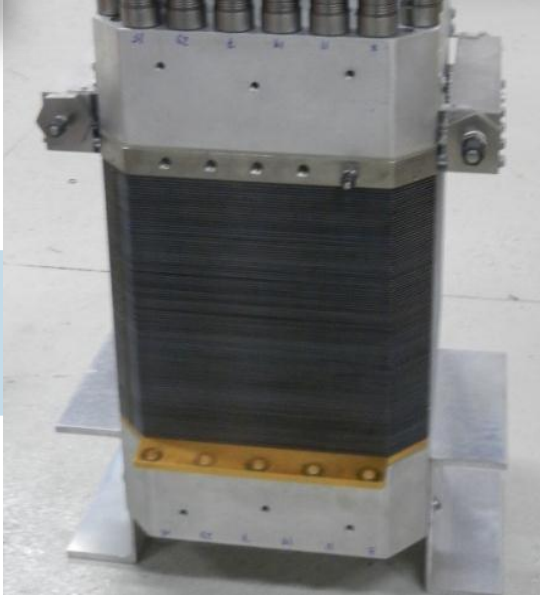
HOGEN® H Series



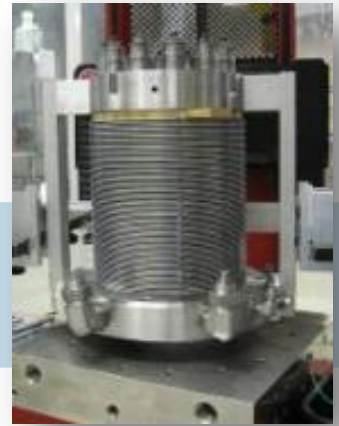
HOGEN® S Series



HOGEN® GC



680 cm²
50 Nm³/hr
100 kg/day



210 cm²
10 Nm³/hr
21.6 kg/day



86 cm²
2 Nm³/hr
4.3 kg/day



28 cm²
0.05 Nm³/hr
0.01 kg/day

Transition and Deployment

Today:

- Power plants
- Industrial (reducing env.)
- Lab applications
- Lifting gas (Weather balloons/Aerostats)



Power Plants



Heat Treating



Laboratories



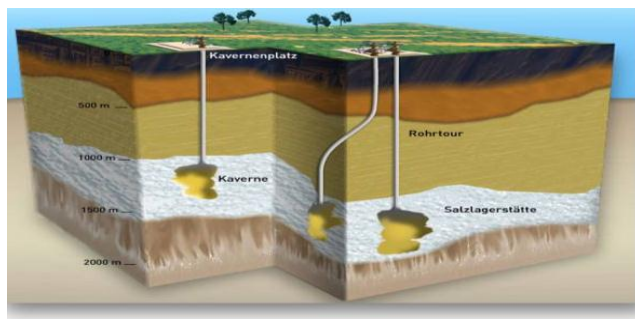
Semiconductors



Government

Emerging:

- Storing renewable energy
- Making methane (biogas)
- Fueling vehicles

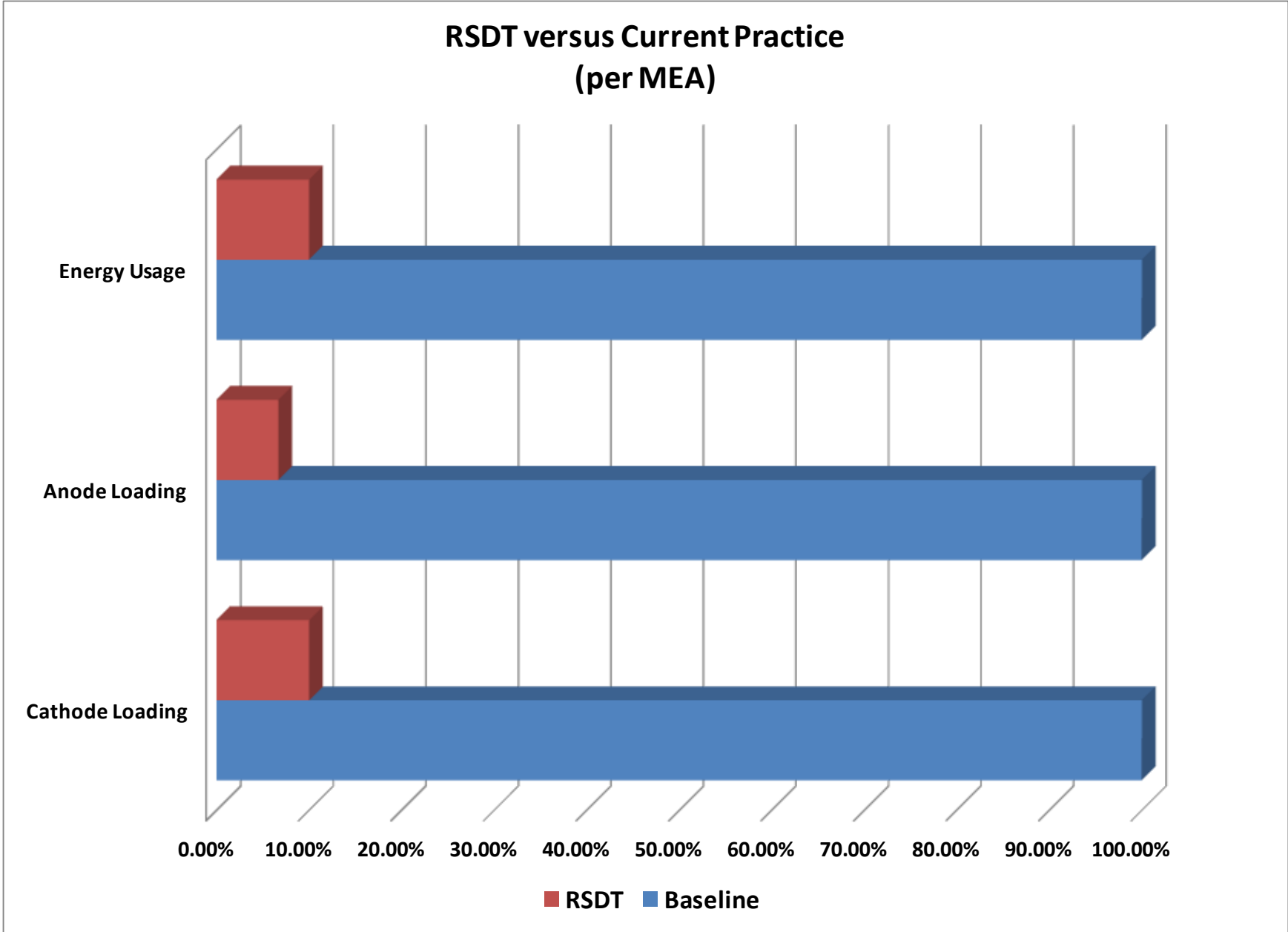


Transition and Deployment

- Megawatt scale PEM launched
 - Would further benefit from RSDT.
- Addresses demand for:
 - Wind capture (100GW)
 - Biogas (>3000 plants)



Expected Improvements: RSDT



Config. Score Sheet for Downselect

	All CCM		CCM Anode GDE Cathode		CCM Cathode GDE Anode		All GDE	
	Technique	Score	Technique	Score	Technique	Score	Technique	Score
Build type	Dry	+3	Dry	+3	Dry	+3	Wet & Dry	+1
Next Gen Cell Design (Reduced Seal Area)	Gasket & O-ring	+1	Gasket & O-ring	+1	Gasket & O-ring	+1	O-ring	+3
Substrate changes	<u>1-Step</u> Anode Cathode X-Over	+1	<u>2-Step</u> a) X-Over/Anode b) Cathode	+3	<u>2-Step</u> a) X-Over/Anode b) Cathode	+3	<u>3-Step</u> a) X-Over b) Anode c) Cathode	+5
Readiness of deposition technologies	a) X-Over b) Anode c) Cathode	+1 +3 +1	a) X-Over b) Anode c) Cathode	+1 +3 0	a) X-Over b) Anode c) Cathode	+1 +3 +1	a) X-Over b) Anode c) Cathode	0 +3 +1
Impact to current cell design	Drop-in	+1	GDE integration required	+3	GDE integration required	+3	GDE integration required	+3
Total		11		14		15		16

Progress: Project Milestones

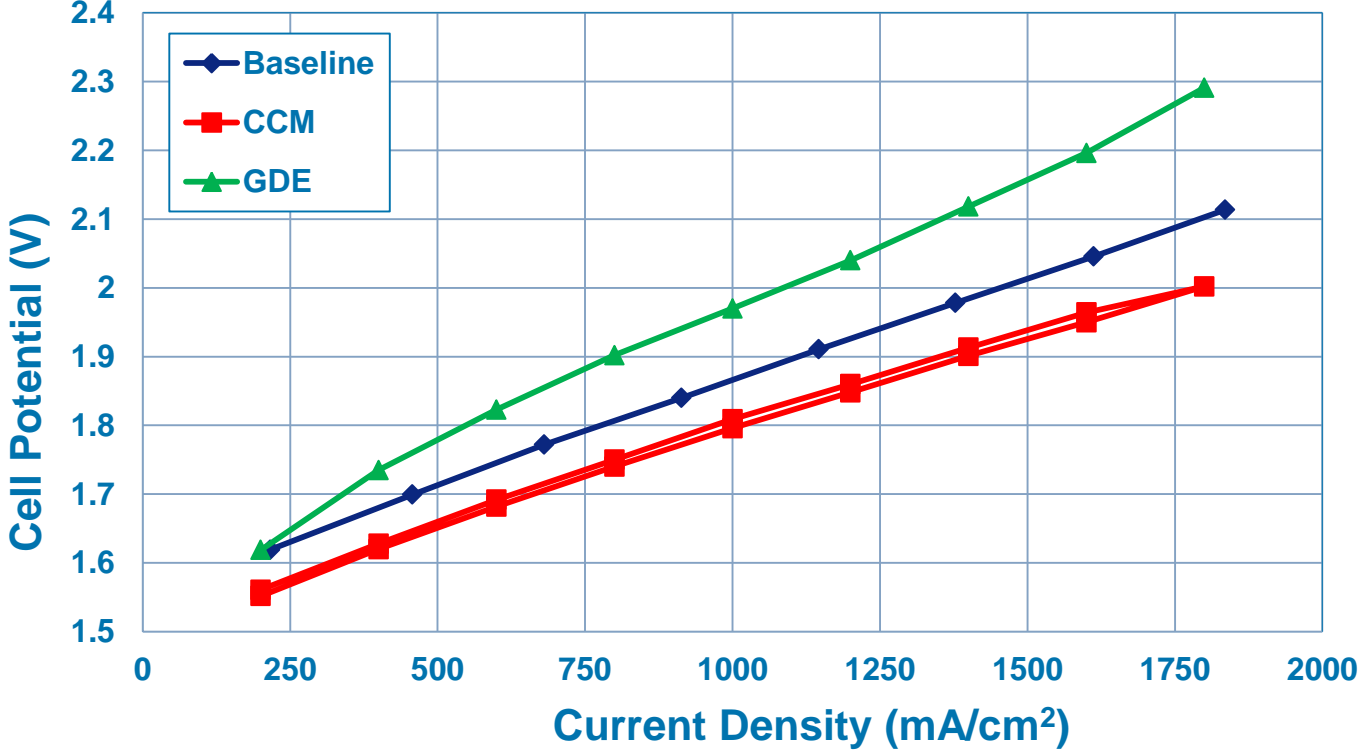
Milestone Description	Responsible	Due Date
Project Kick-off	Proton, UCONN	08/19/2014
Baseline ITO stability	UCONN	10/15/2014
Demonstration of ECA measurement of model catalyst in RDE	UCONN	12/1/2014
Down-select cathode electrode configuration (CCM versus GDE) and formulation	Proton, UCONN	3/1/2014
Complete alternate frame load and stress calculations	Proton	3/1/2014
Conduct 200 hour durability test of down-selected cathode config. at 28 cm ² level	Proton, UCONN	4/15/2014
Extension of ECA measurement to in situ catalyst measurements	UCONN	5/1/2015
Finalize design and CAD drawings for alternate frame concept	Proton	5/15/2015
Demonstrate performance and stability of first IrO ₂ /ITO catalysts	Proton, UCONN	6/15/2015
Demonstrate RSDT anode performance equivalency to Proton baseline	Proton, UCONN	8/15/2015
Procure and verify prototype parts through hydrostatic sealing tests	Proton	9/15/2015
Conduct 200 hour durability test of down-selected config. at 28 cm ² level	Proton, UCONN	10/15/2015
Scale-up and conduct operational tests of alternative frame design	Proton	12/15/2015
Demonstration of 89 cm ² MEAs using optimal RSDT Scale-up	Proton, UCONN	1/1/2016
Conceptual Design for Industrial Scale RSDT	UCONN	5/1/2016
Achieve 500 hours of operation at 89 cm ² cell level	Proton, UCONN	6/1/2016
Scale-up Cost Analysis	Proton	7/1/2016
Complete Final Reporting	Proton, UCONN	7/27/2016

Technical Accomplishments

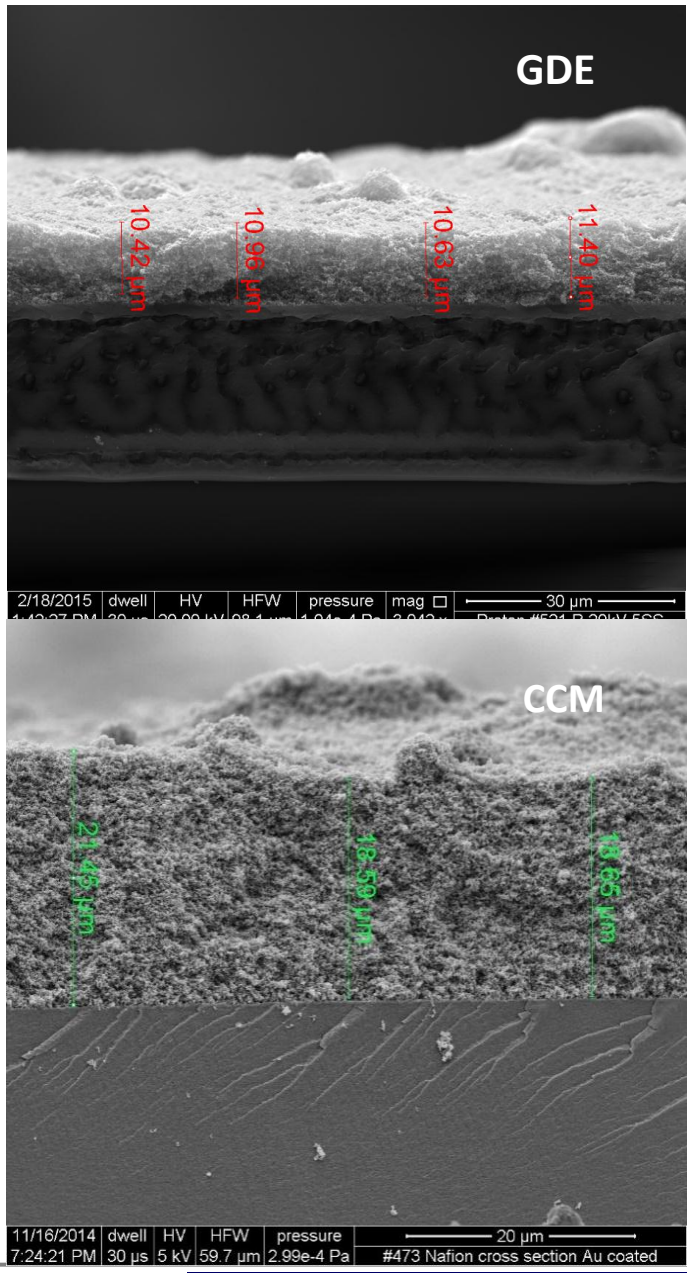
- Cathode Development
 - Demonstrated single step manufacture of electrode.
 - Down-selected the catalyst coated membrane (CCM) pathway
- Anode Development
 - Initial evaluation of single-step fabrication
- Alternate Seal Design
 - Concepts have shown reduction in membrane usage by ~40%
 - Computational modeling has verified cell concepts to work within acceptable mechanical loading and stress values
- Stack Scale-up and Verification
 - Initiated scale-up activities by transitioning cathode test from bench hardware to commercial platform

Technical Accomplishments: Cathode

**RSDT Cathode Comparison
CCM vs. GDE**

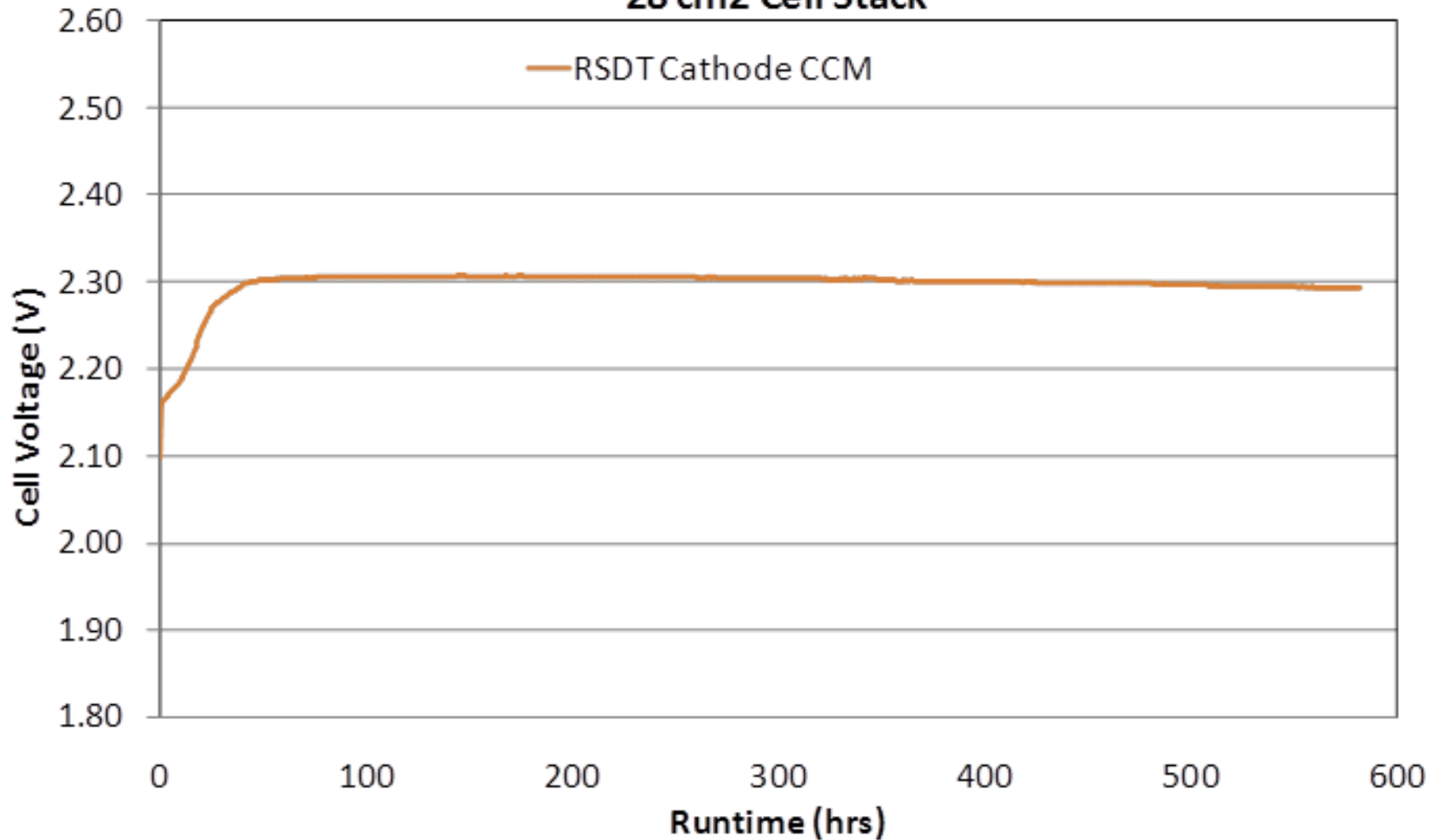


- Demonstrated improved performance compared to baseline at 1/10 loading



Technical Accomplishments: Stability Testing

Steady-State Durability Test of UCONN Fabricated Cathode CCM
28 cm² Cell Stack



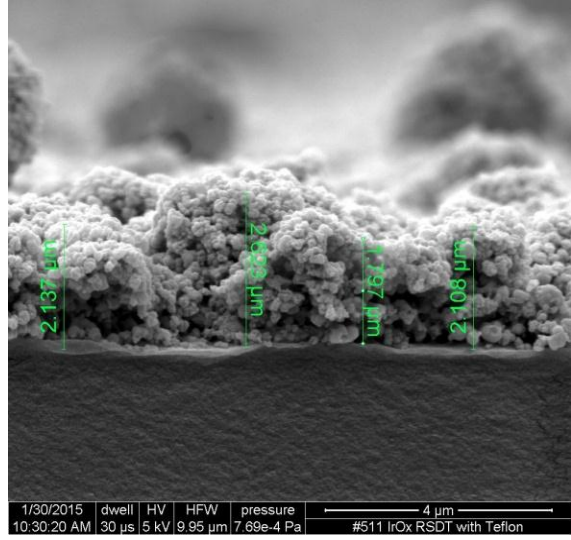
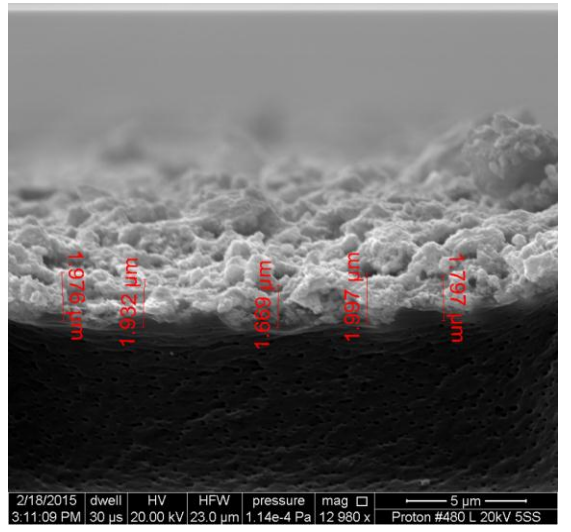
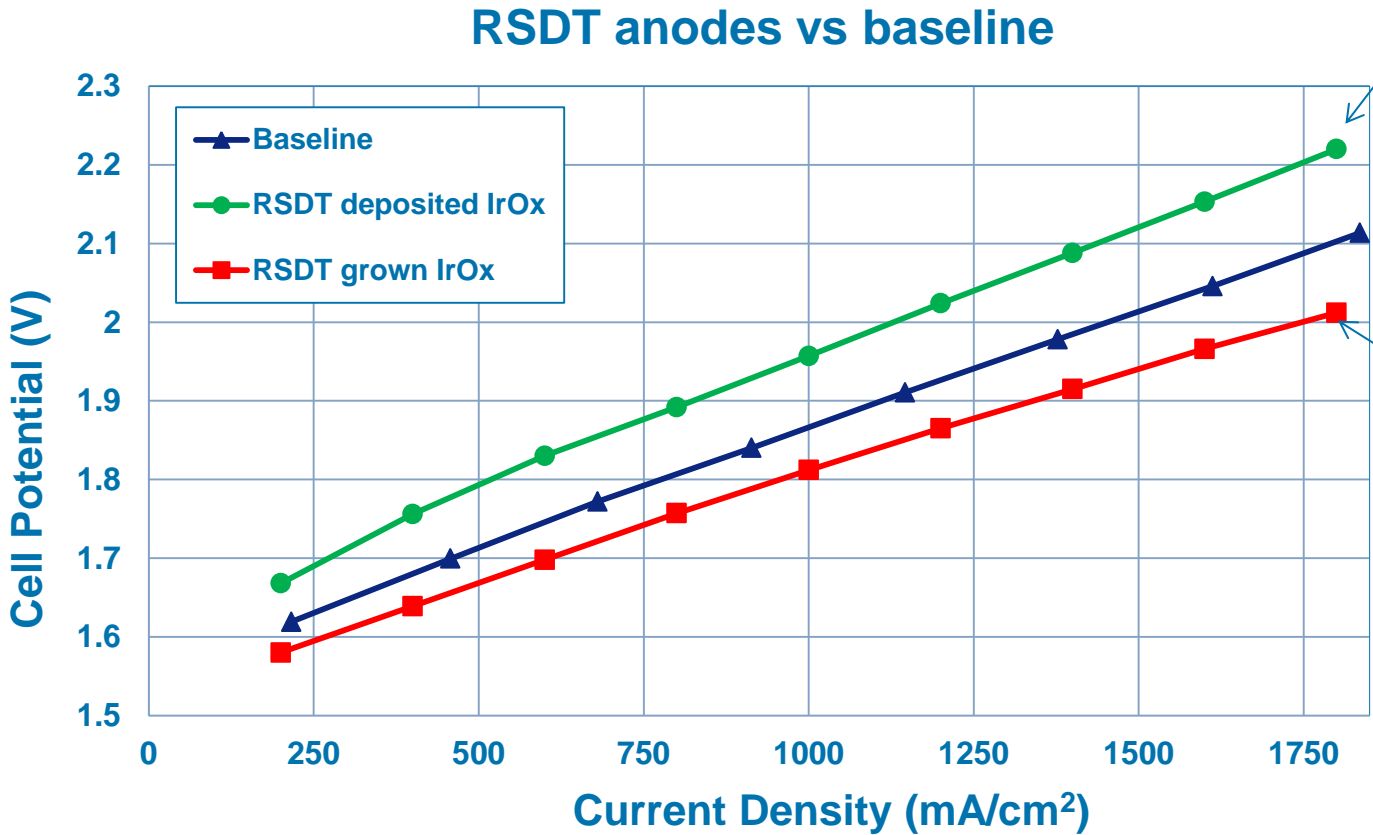
Demonstration of stability achieved with scale-up cathode CCM

Cathode vs. Anode Comparison

Input	Cathode	Anode
Composition	Metal – Polymer Composite	Oxide – Polymer Composite
Particle Size	~5 – 10 nm	> 10 nm
Porosity	Gas Transport	Gas/Water Transport
Loading	0.15 mg/cm ²	>0.5 mg/cm ²
Precursor	Known	Known
Carrier Gas/Flame	Known	Known
Support	Carbon	Oxygen/Potential Compatible

Completed
In-Process
Needs Development

RSDT Grown IrOx Anode vs RSDT Deposited Anode



- Performance in favor of in-situ grown electrode, refinement in stability is on-going

Conclusions

- **Cathode Development**
 - Cathode CCM down-selected over GDE
 - Comparable durability; enables single-step process
 - Scale-up demonstrated
- **Alternative Sealing Design**
 - Concepts evaluated and drawings created.
 - Feasibility confirmed through modeling
 - Stress modeling of parts completed.
 - Prototype hardware has been designed for sealing tests

Future Work

- **Anode Development**
 - Robust OER catalyst at reduced loading
 - Apply in-situ ECSA measurements
 - Translate best-practice from cathode
 - Down-select GDE vs CCM
 - Verify through operational testing
- **RSDT Scale-up and Concept Design**
 - Complete design and build apparatus
 - Demonstrate in scale-up parts
- **Stack Scale-up and Verification**
 - Operate multi-cell stack w/RSDT electrodes

Collaborators

- University of Connecticut
 - Designed and developed RSdT apparatus
 - Conduct and optimize electrode depositions
 - Build and verify scaled-up prototype unit
 - Develop and apply ECSA QC technique
- CAE Associates
 - Design verification through computer modeling/simulation