



Novel Structured Metal Bipolar Plates for Low Cost Manufacturing (SBIR Project)

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SBIR Phase II Project Overview

Timeline

- Project start date: May 15, 2014
- Project end date: May 14, 2016
- Percent complete: 50%

Budget

- Total Funding Spent in FY 2015:
as of 3/31/14: \$230,615.10
- Total DOE Project Value: \$988,784
- Cost Share Percentage: 0%

Barriers

- Barriers Addressed : Bipolar Plate Durability and cost
 - Cost: < \$3/kW (2020)
 - resistivity < 10 mΩ·cm²
 - corrosion < 1 x10⁻⁶A/cm²

Partners

- Hawaii Natural Energy Institute, University of Hawaii.
- Ford Motor Company

Objective of the Project

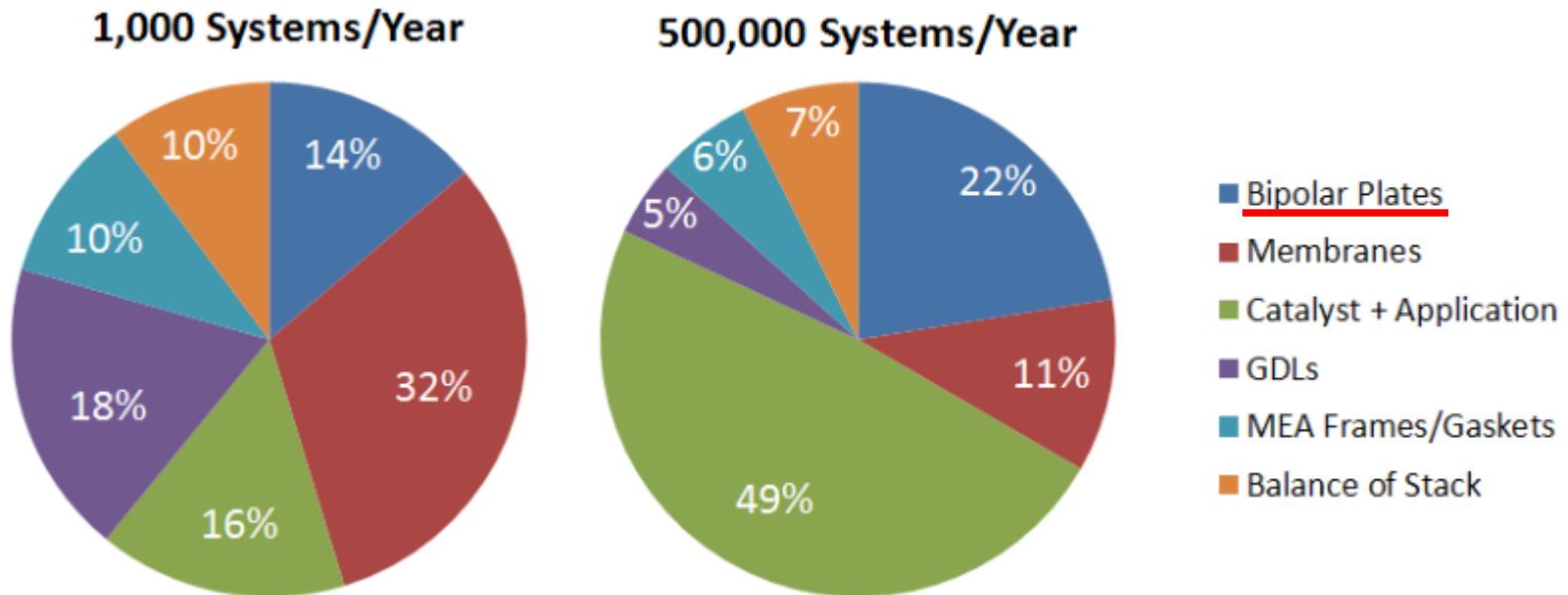
- Overall Objective: Develop lower cost metal bipolar plates to meet performance target and 2020 cost target (<\$3/kW)
 - Scale up and optimize doped titanium oxide coating technology demonstrated in Phase I project
 - Full size short stack demonstration under automobile dynamic testing conditions.

Key Technical Targets

Characteristic	Unit	2011 Status	2017 Targets	2020 Targets
Cost	\$ /kW	5-10	3	3
Corrosion	$\mu\text{A}/\text{cm}^2$	<1	<1	<1
Resistivity	$\Omega.\text{cm}^2$	<0.03	<0.02	<0.01

Relevance

Bipolar Plate Cost is a Major Portion of Stack

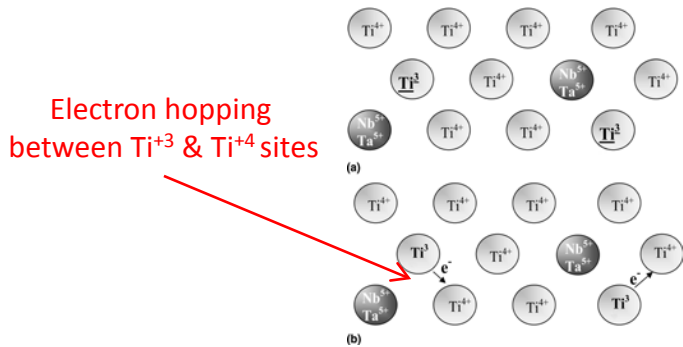


J. Spendelow, J. Marcinkoski, "Fuel Cell System Cost – 2013"
DOE Fuel Cell Technology Office Record # 13012

Approach: Coating Material for SS Plates

--- Doped TiO_x

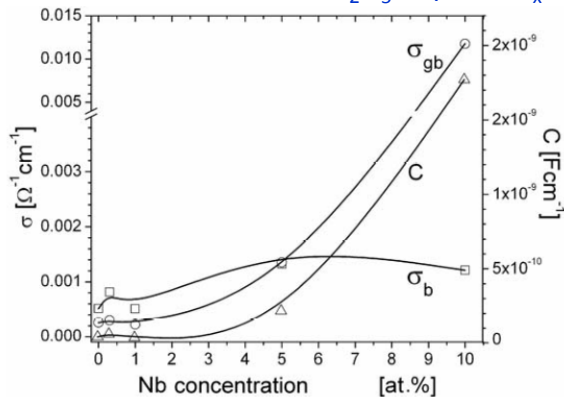
Doping TiO_2 with +5 valence elements will enforce the formation of Ti^{+3} in TiO_2 lattice structure, and result in the higher electronic conductivities.



Challenges to use doped TiO_x coating:

1. Doped TiO_x is semi-conductive. The electrical conductivity is not high enough.
2. How to obtain reliable bonding of doped TiO_x on metal substrate surface.

Electrical conductance of Nb_2O_5 doped TiO_x



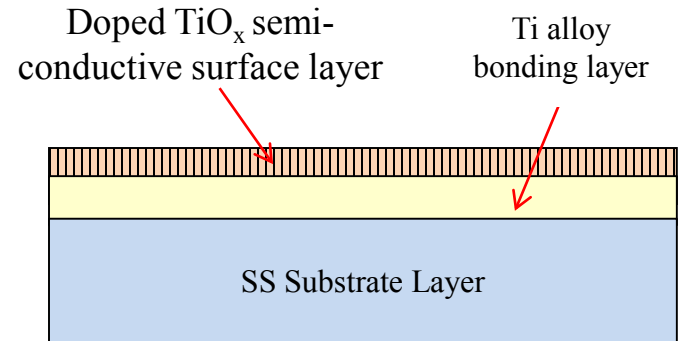
A. Trenczek-Zajac, M. Rekas, Materials Science-Poland, Vol. 24, No. 1, 2006

TreadStone's approach:

- To coat stainless steel substrate with Ti-Nb or Ti-Ta alloy. Then, grow the doped TiO_x surface layer on the Ti alloy coating layer.
1. The doped TiO_x on Ti alloy surface is thin and reliable.
 2. Ti alloy coating has excellent adhesion on metal substrate (stainless steel or aluminum).

Approaches: Fabrication Process

- Based on industrial available Physical Vapor Deposition (PVD) technology for the coating materials deposition.
 - Ready for high volume production
- Focused on the electrical conductive and corrosion resistive doped titanium oxide as the coating materials.
 - Low cost materials.
- Focused on the deposition and post deposition treatment conditions to obtained the desired structure of the surface coating.
 - Superior adhesion of coating layer with substrate.
 - Post deposition treatment for the desired phase structure of the coating layer.



Leybold Optical's DynaLine
Inline Sputtering System

Approach: Tasks and Milestones

Coating Target Material
Optimization

May '14 – Jan. '15

- Determine the composition of the target material. (Finished)
- Coat SS foil for ex-situ test. (Finished)

PVD Process
Development

Nov. '14 – Feb. '16

- Demonstrate the coating in single cell test. (Finished)
- PVD process development. (on-going)

- Components preparation. (start in June '15)
- Long-term durability test in short stack. (start in July '15)

Demonstrate in Auto.
Fuel Cell Stack

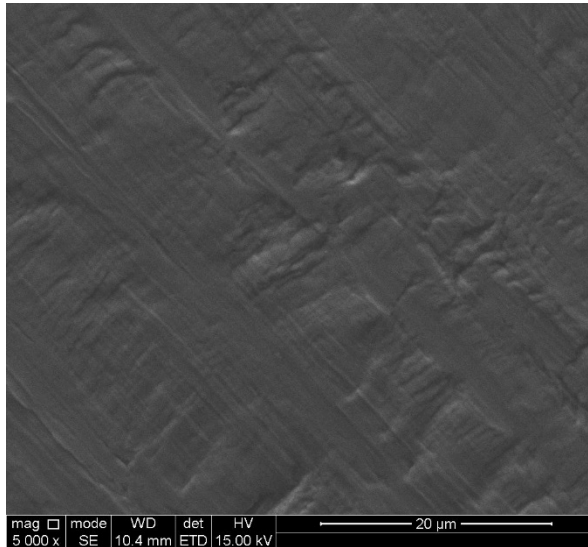
Jun. '15 – May '16

Project Duration: 24 months

Accomplishments: PVD Process Development

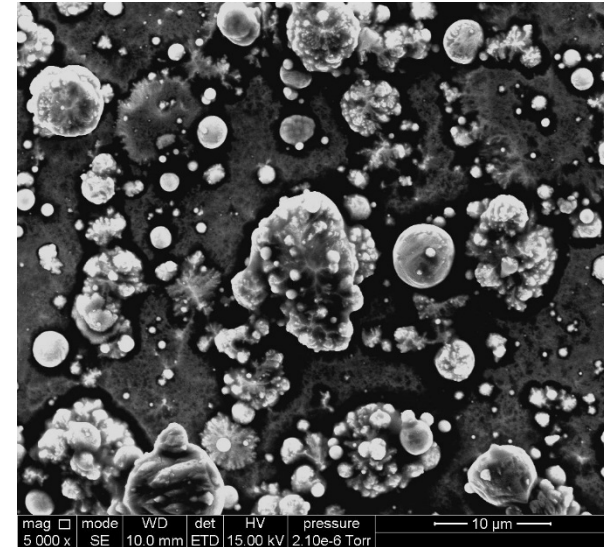
Nb-TiO_x coating on SS foil surface

DC Magnetron Sputtering



- Relative smooth coating surface.
- Alloy element segregation on the very surface layer with current sputtering process.
 - No Nb in the surface layer (5-10nm), and very high contact resistance.
- Need to etch off the surface layer to obtain the desired Nb doped TiO_x (Nb-TiO_x) surface layer for low electrical contact resistance.

Cathodic Arc Deposition



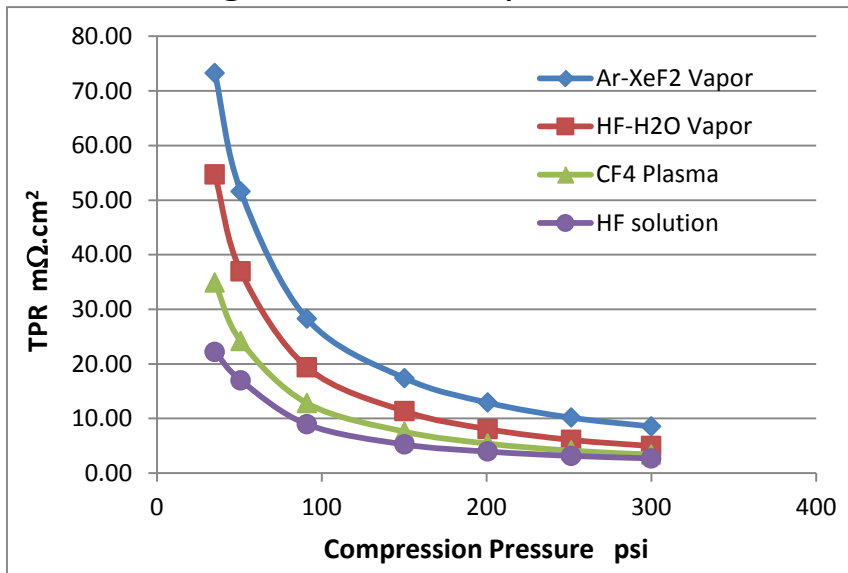
- Rough surface with micron particles.
- Uniform composition of the coating surface.
- The electrical contact resistance is not as low as the etched sputtering coated SS.
- Target material utilization of Cathodic Arc is low.
- Difficult to obtain thin (<0.5 μm) coating.

Accomplishments: Coating Materials

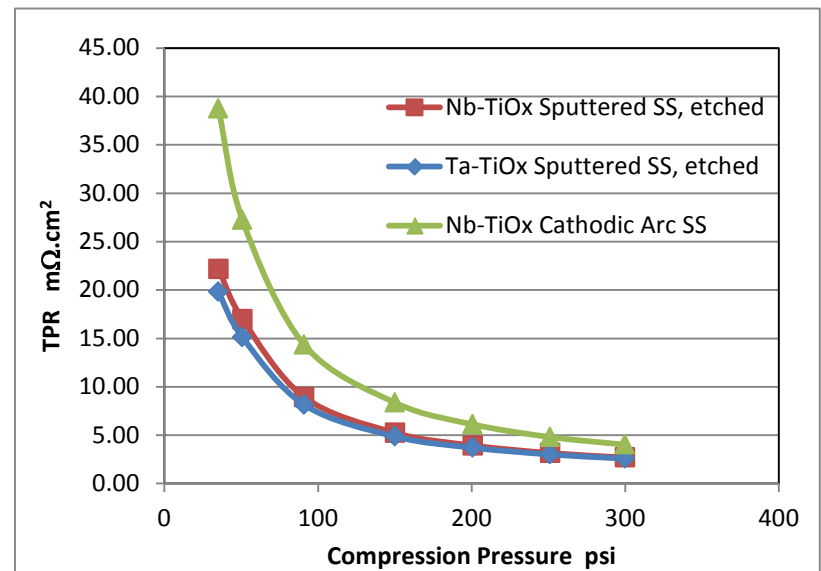
Sputtering coated Nb-TiO_x on SS

SS with doped TiO_x coating

Etching method comparison



Composition and deposition methods



- Hydrofluoric acid etching is more repeatable and has lowest surface contact resistance.
- Vapor etching is easier to be integrated with PVD process. More developments are needed.

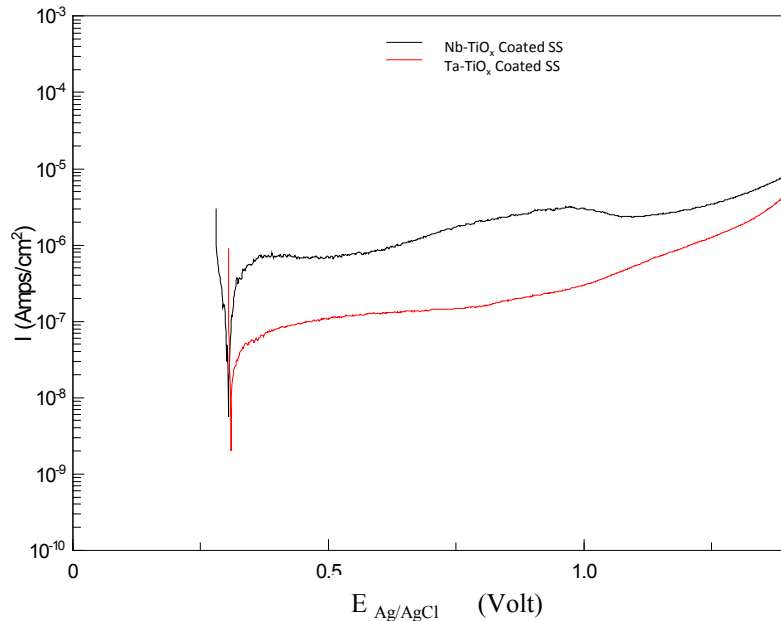
Note: TPR, through plate resistance measured of the plate in contact with TGP-H-060 Toray Paper.

Accomplishments

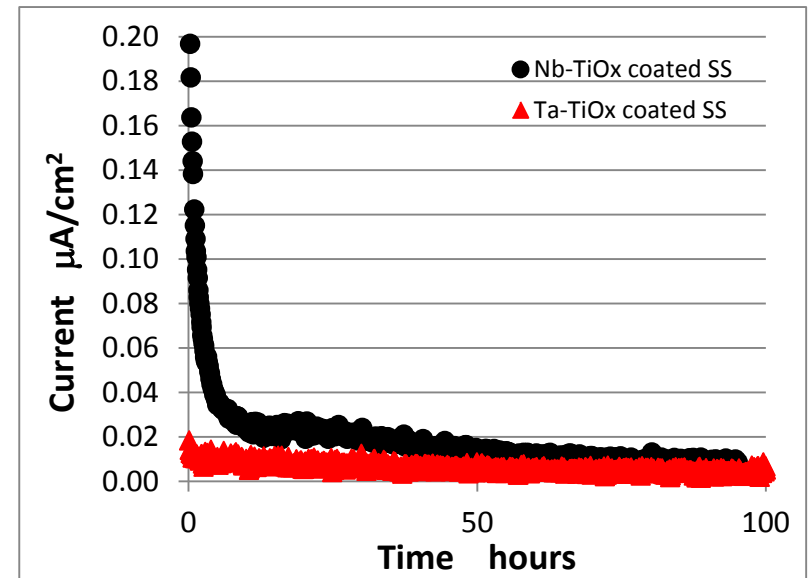
ex-situ Tests of Doped TiO_x coated SS

in pH 3 H₂SO₄ + 0.1 ppm HF at 80°C

Potentiodynamic Test (@10 mV/min)



Potentiostatic Test (@0.8V_{NHE})



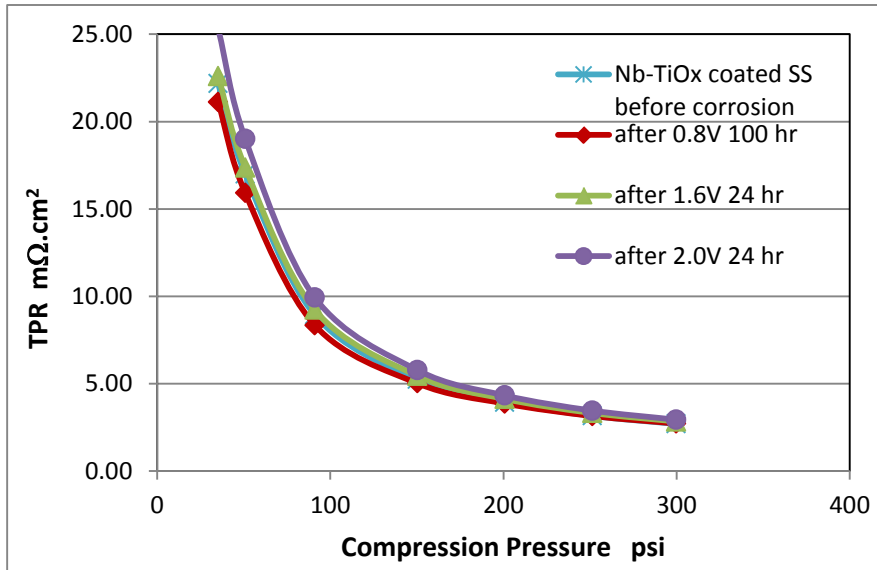
- Both Nb and Ta doped TiO_x coated SS can meet the corrosion current target (<1 μA/cm²)
- Ta-TiO_x coated SS has lower corrosion current than that of Nb-TiO_x

Accomplishments:

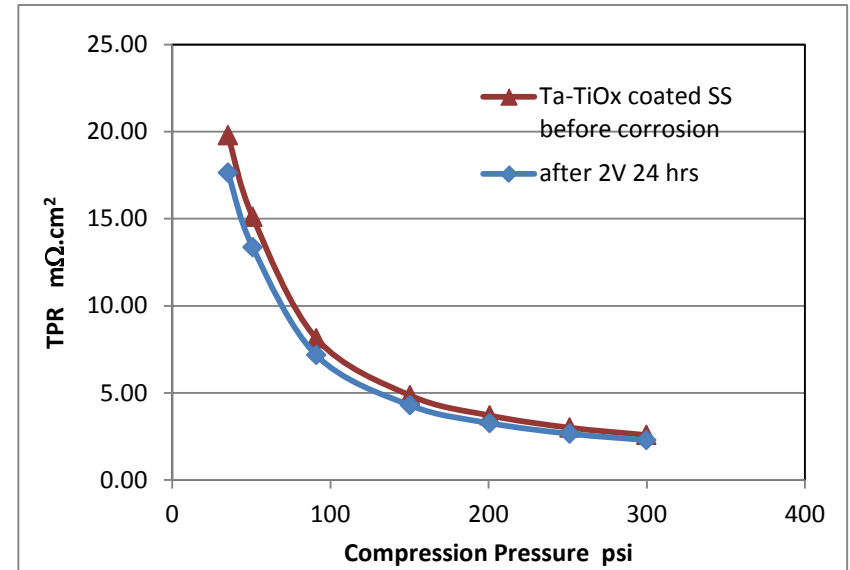
Coating Stability Test in Extreme Conditions

in pH 3 H_2SO_4 + 0.1 ppm HF at 80°C

316L SS with Nb-TiO_x coating
before and after corrosion tests



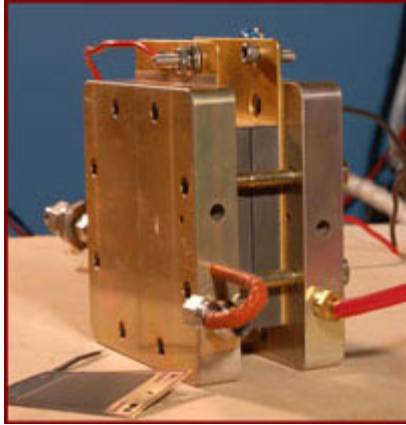
316L SS with Ta-TiO_x coating
before and after corrosion tests



- Doped TiO_x coated SS has low surface electrical contact resistance.
- The coated SS has superior corrosion resistance for PEM fuel cell applications.
- The extreme corrosion condition (@ 1.6V_{NHE} or 2 V_{NHE}) *ex-situ* tests are not included in regular standard, but it is very attractive to OEMs.

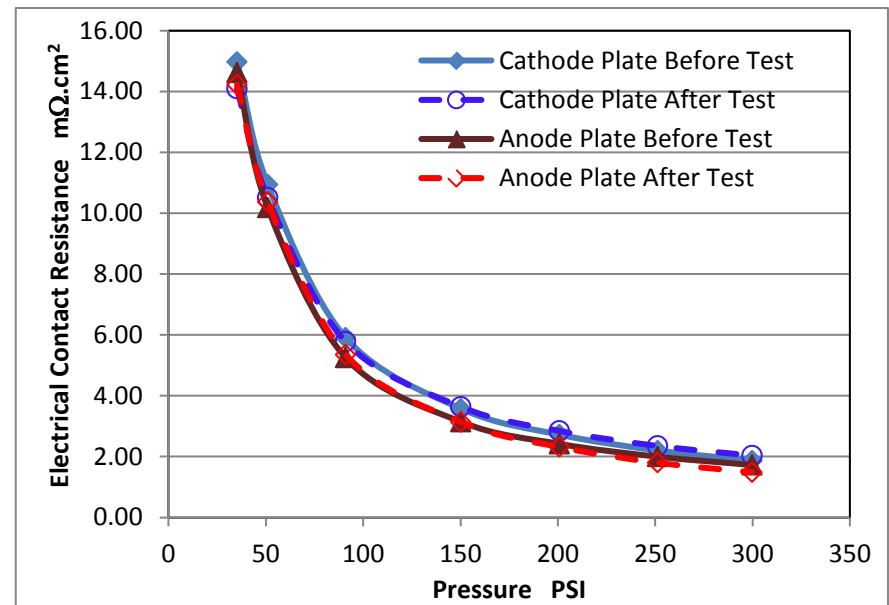
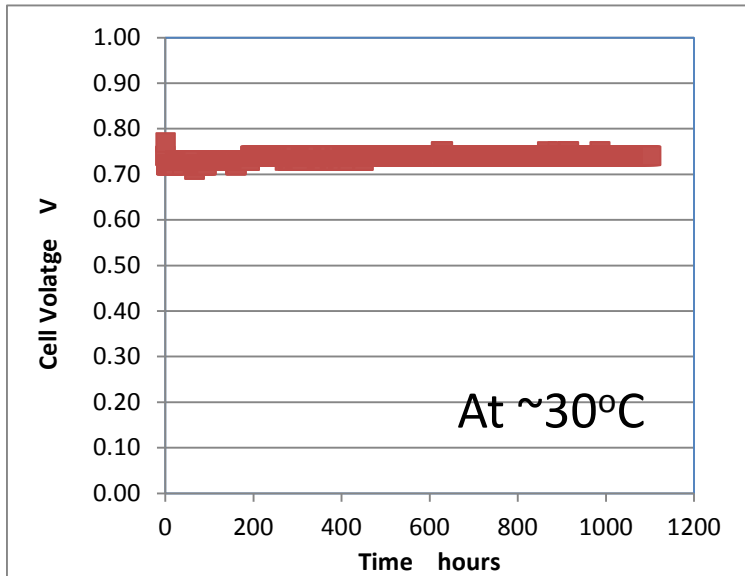
Accomplishments

Single Cell Test with Nb-TiOx Coated SS Plates



16 cm² active area cell using Fuel Cell Technology hardware

Contact Resistance with GDL before and after 1,100 hrs. single cell test



Responses to Previous Year Reviewer's Comments

This Project was not reviewed last year.

Collaborations

Team Partner:

HNEI, Univ. Hawaii

5 kW stack testing under automobile
dynamic operation conditions.

Dr. Jean St-Pierre

Industrial Supporter:

Ford Motor Company

Independent *ex-situ* test evaluation
Provide automobile stack for durability test

Mr. Shinichi Hirano

Mr. Mark Ricketts

Remaining Challenges and Barriers

- **Large scale fabrication process.**
 - Current PVD + hydrofluoride acid etching process may not be suitable for large scale fabrication.
 - The second year of the project will develop a simpler process, in addition to the planned stack durability demonstration.
- **Fundamental understanding of the coating material.**
 - The performance of the coating material is much better than expected. What is the scientific principles behind it?
 - **The fundamental study is not included in this SBIR project.**

Proposed Future Work

- **Task 2. PVD Process Development**
 - Simplify current coating process, evaluate the coating properties by *ex-situ* corrosion tests and single cell tests.
- **Task 3. Demonstration in Automobile Stack Durability Tests**
 - Ford will contribute a short stack (no cost to the project) for the durability test.
 - The stack tests will be conducted at U. Hawaii with the technical supports from Ford.

Summary

- **Objective:** Develop a low cost metal bipolar plate coating that does not need to use precious metals.
- **Relevance:** Reducing the metal bipolar plate cost to meet FY20 requirements.
- **Approach:** Using doped TiO_x coating on metal plates surface for fuel cell applications..
- **Accomplishment:**
 - Identified the high performance, stable coating material.
 - DC Magnetron Sputtering and cathodic arc processes have been used for the coating material deposition.
 - *Ex-situ* tests indicate that the coated stainless steel has superior stability.
 - Corrosion resistance easily meet the targets.
 - Electrical contact resistance is low and stable after aggressive *ex-situ* corrosion tests.
 - The superhydrophilic surface properties has additional benefits to plate flow field design.
 - 1100 hours single cell evaluation demonstrate its durability in PEM fuel cells.
- **Collaborations:**
 - Teaming with HNEI, Univ. Hawaii for stack long term durability test.
 - Ford will contribute a full size, short stack for the demonstration.

Acknowledgements

- DOE EERE Fuel Cell Team.
- Team Members. HNEI, U. Hawaii
- Industrial Partners. Ford