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

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Project ID# : FC105

SBIR Phase II Project Overview

Timeline

- Project start date: May, 2014
- Project end date: May, 2016
- Percent complete: 90%

Barriers

- Barriers Addressed : Bipolar Plate Durability and cost
  - Cost: < \$3/kW (2020)
  - resistivity $< 10\ m\Omega \cdot cm^2$
  - corrosion $< 1\ x10^{-6} A/cm^2$

Budget

- Total Funding Spent:
  as of 3/31/16: \$835,450
- Total DOE Project Value: \$988,784
- Cost Share Percentage: 0%

Partners

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

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Objective of the Project

- <u>Overall Objective:</u> 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.

<u>Key Technical Targets</u>

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

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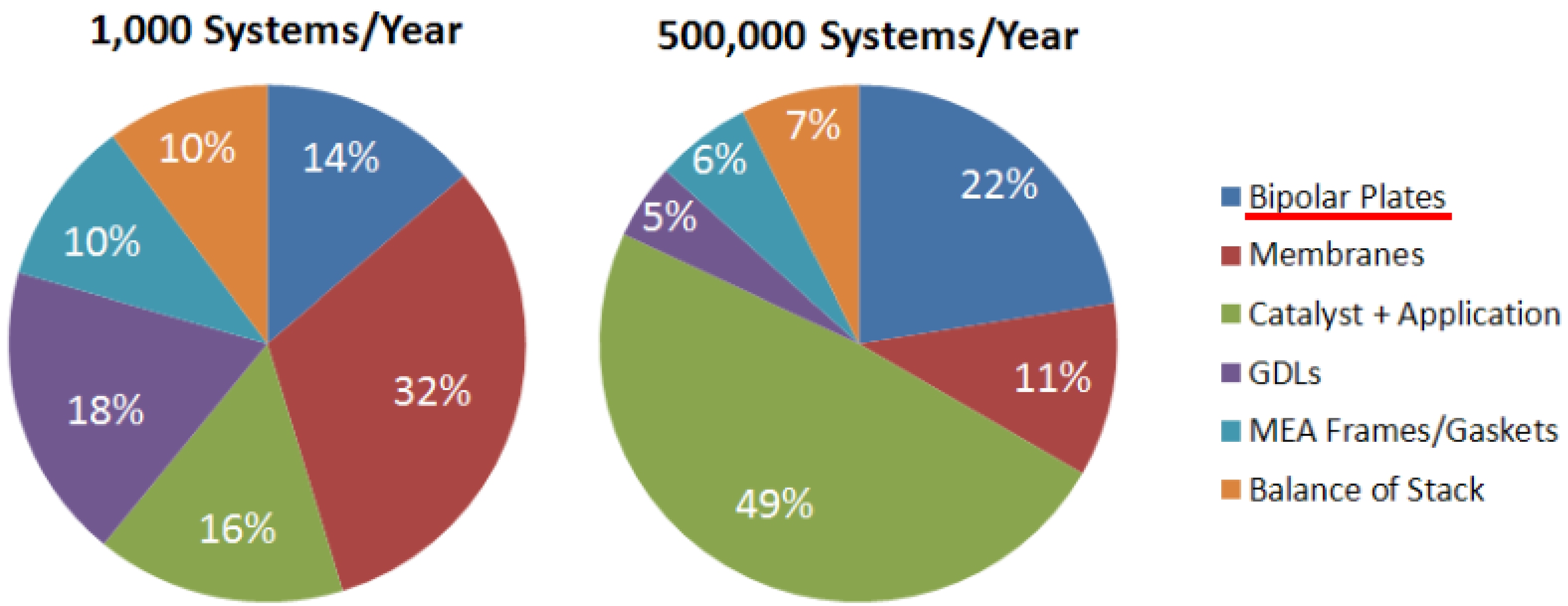
Relevance

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

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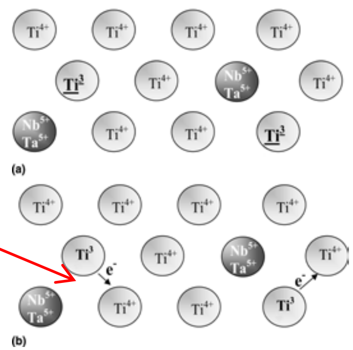
Approach: Coating Material for SS Plates

--- Semiconductive 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.

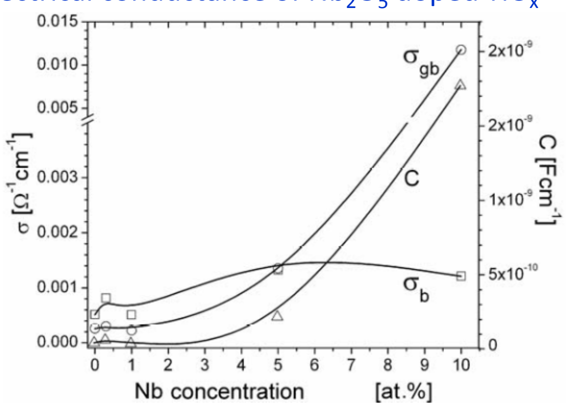
Electron hopping between Ti^{+3} & Ti^{+4} sites



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

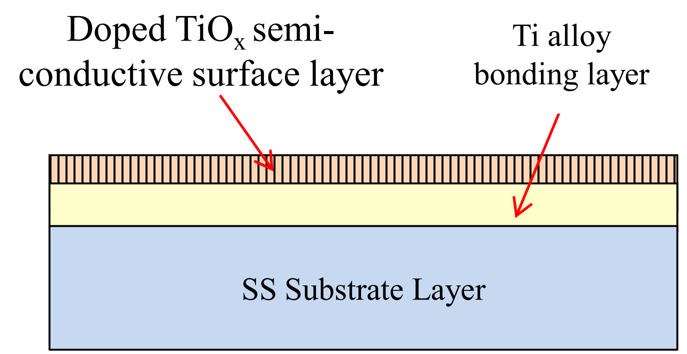
TreadStone's approach:

➢ 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

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Approach: Tasks and Milestones

Coating Target Material Optimization

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

May ‘14 – Jan. ‘15

PVD Process Development

- Demonstrate the coating in single cell test.
- PVD process development.

Nov. ‘14 –Feb. ‘16

- Components preparation.
- Long-term durability test in short stack.

Demonstrate in Auto. Fuel Cell Stack

Jun. ‘15 – May ‘16

<u>Project Duration: 24 months</u>

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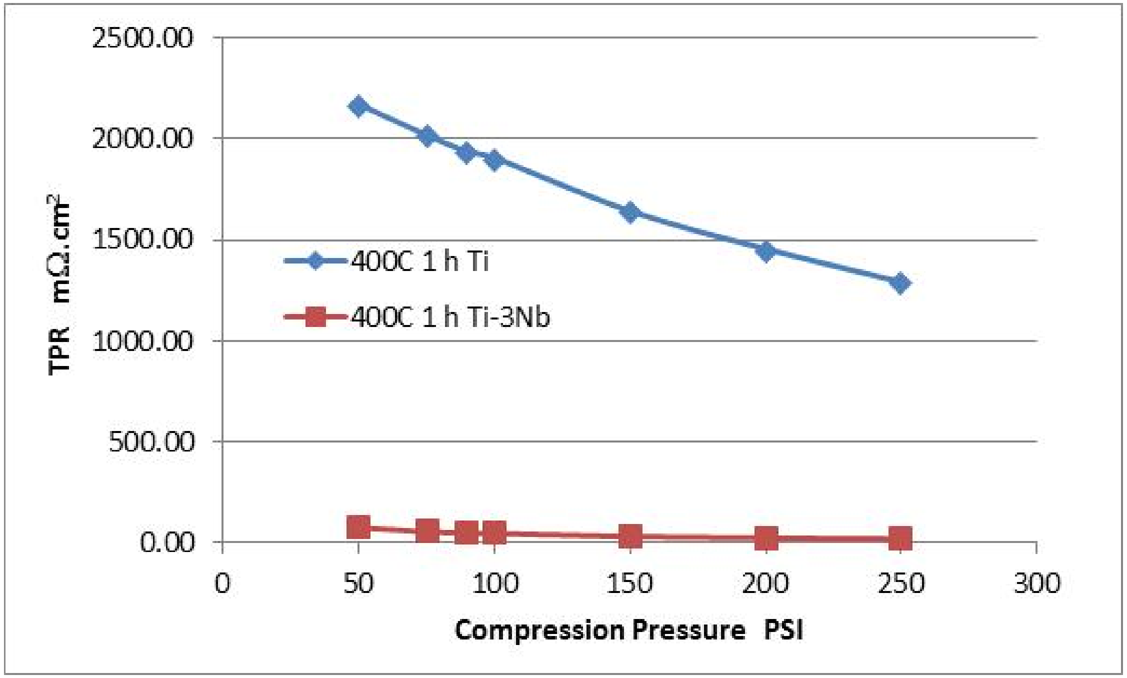
Proof of Concept Experiment

Through Plate Resistance (TPR) comparison of pure Ti and Ti-3Nb alloy plate having thick oxide layer by thermal oxidization in air (400^{o}C).



The Nb doped Ti oxide layer on Ti-Nb alloy surface has much lower resistance than that of pure Ti oxide layer on pure Ti plate surface.

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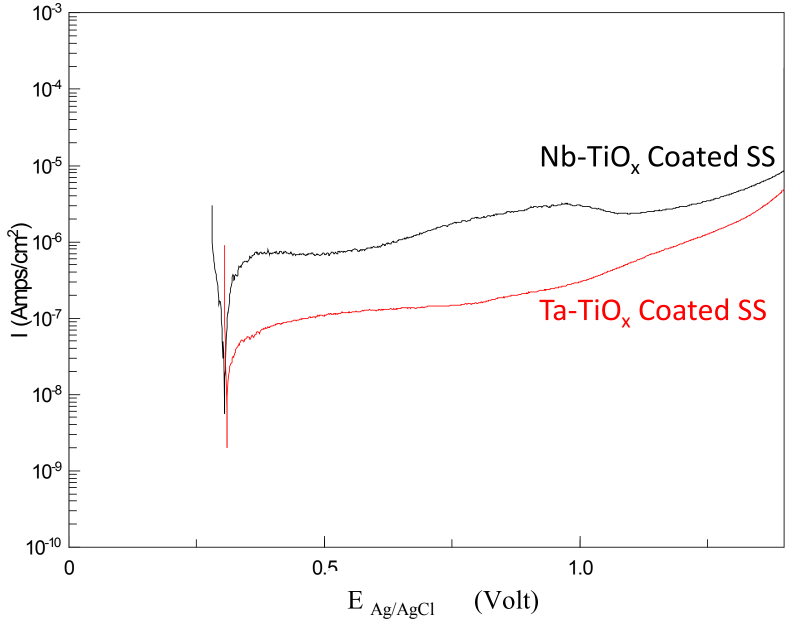
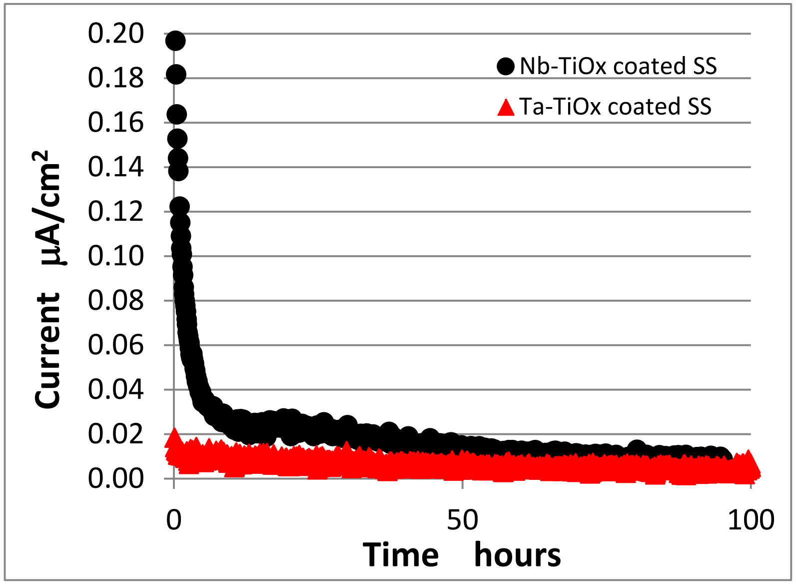
ex-situ Corrosion Tests of Doped TiOx coated SS

in pH 3 H_2SO_4 + 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
- Titanium oxide surface layer, as the corrosion product, can further reduce the corrosion current of the coated plates.

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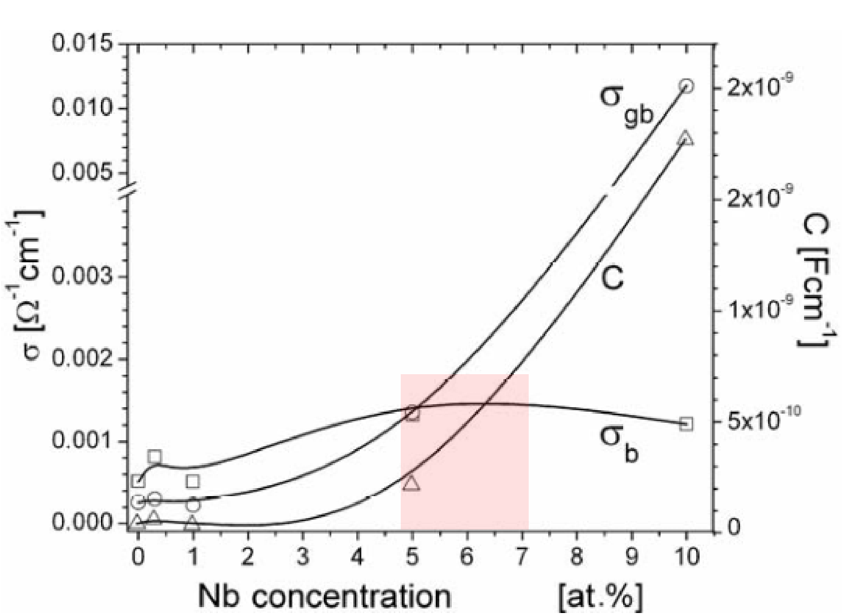
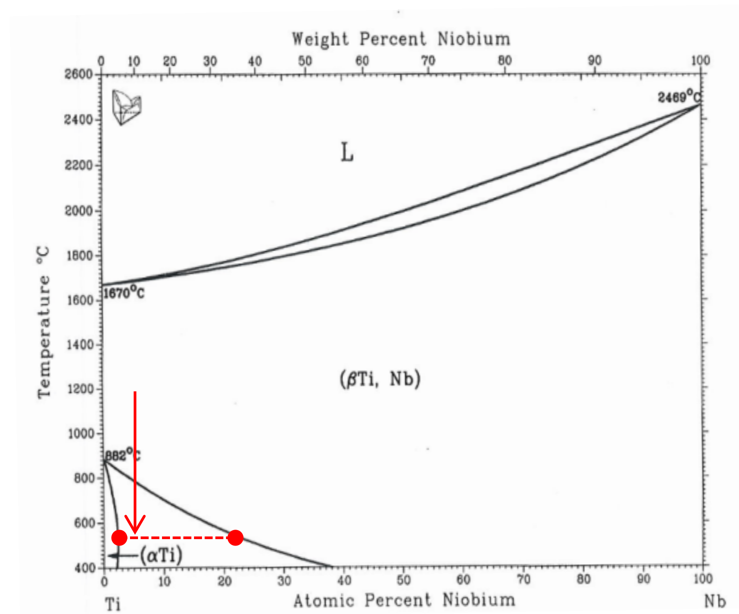
Ti Alloy Composition Selection

1. Nb doped TiOx
   - The solubility of Nb in Rutile TiO_2 structure is ~10 at%. $TiNb_2O_7$ phase was found at high Nb concentration.
   - TiO_x with ~5 at% Nb has the highest electrical conductance. $TiNb_2O_7$ is not conductive.
2. Ti-Nb alloy
   - Ti with < 2 at% Nb is in single α phase.
   - Small amount of high Nb concentration β phase was identified by XRD in Ti with 5-7 at% Nb. β phase was not detected in Ti 3 at% Nb alloy by XRD.





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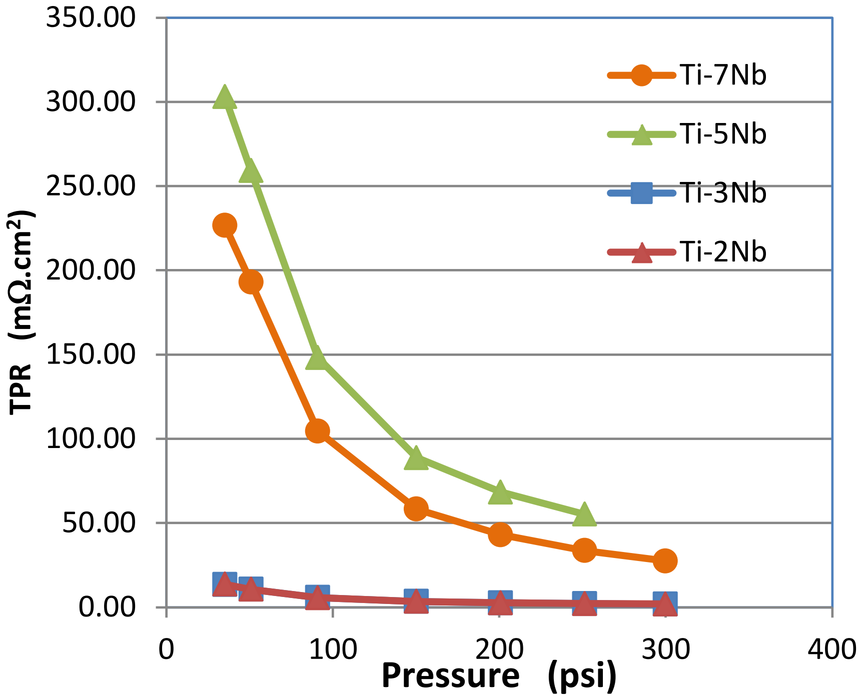
Nb-TiO_x Coating Formed on Different Ti-Nb Alloy Bonding Layer

GDL free TPR (with Toray H-060) of thermally oxidized Ti-Nb coated Stainless Steel



Note: Thermal oxidization is used to simulate the oxide surface layer growth of the plate in long term operation in fuel cells.

- Nb-TiO_x formed on Ti2Nb and Ti3Nb coated SS have similar low resistance.
- Nb-TiO_x formed onTi-5Nb and Ti-7Nb coated SS has much higher resistance.

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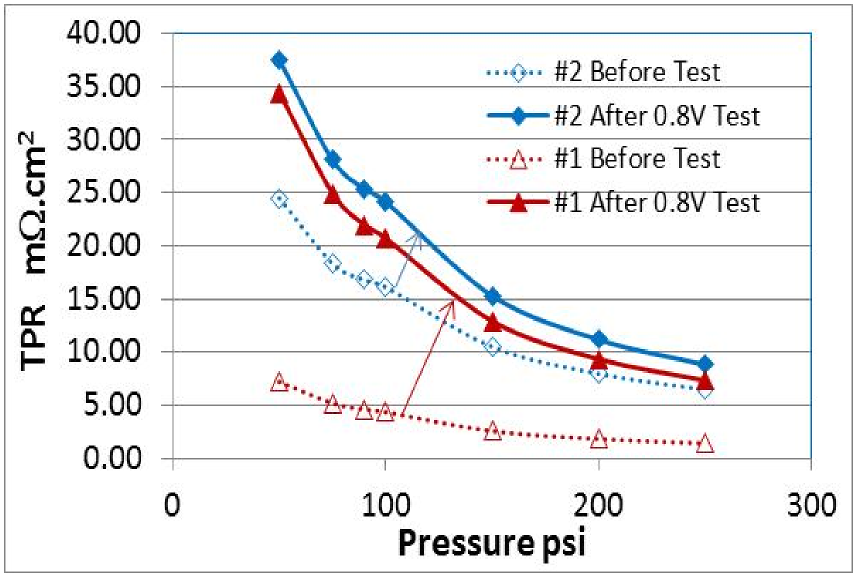
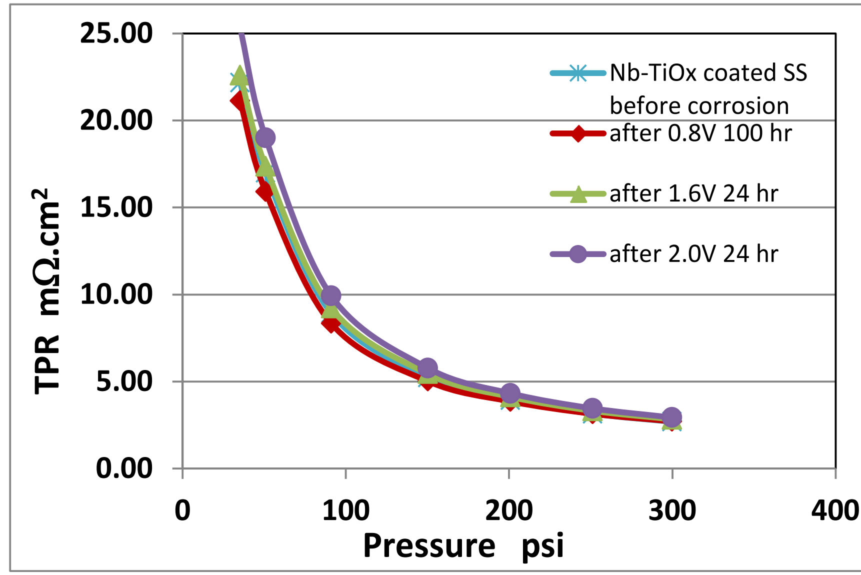
Corrosion Resistance of Nb-TiO$_x$ Coated SS

TPR of Nb-TiO$_x$ coated 316L SS with in pH 3 H_2SO_4 + 0.1 ppm HF at 80^{o}C before and after corrosion tests

Nb-TiO$_x$ coating formed on Ti3Nb bonding layer



Nb-TiO$_x$ coating formed on Ti2Nb bonding layer



- Doped TiO$_x$ coated SS has low surface electrical contact resistance.
- The coating with Ti-2Nb on SS has better corrosion resistance than that of Ti-3Nb.
- 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.

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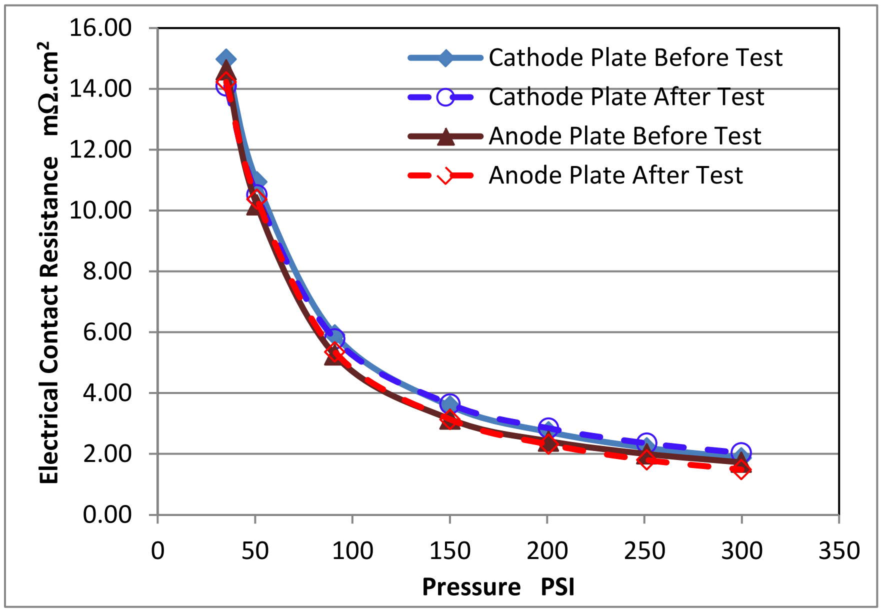
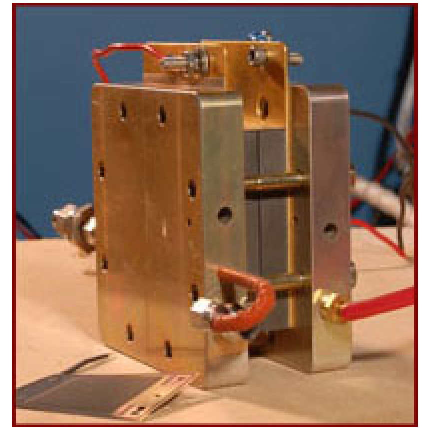
Single Cell Tests with Nb-TiO_x Coated SS Plates

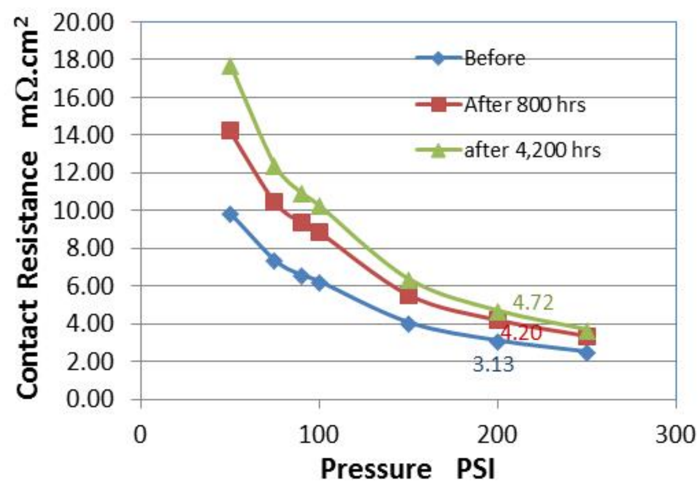
Contact Resistance of Ti2Nb coated SS plates with GDL before and after 1,100 hrs. single cell test



There is no resistance increase of the Ti2Nb coated SS plate after the single cell test.



Contact Resistance of Ti3Nb coated SS cathode plate with GDL before and after 800hrs. and 4,200 hrs. single cell tests



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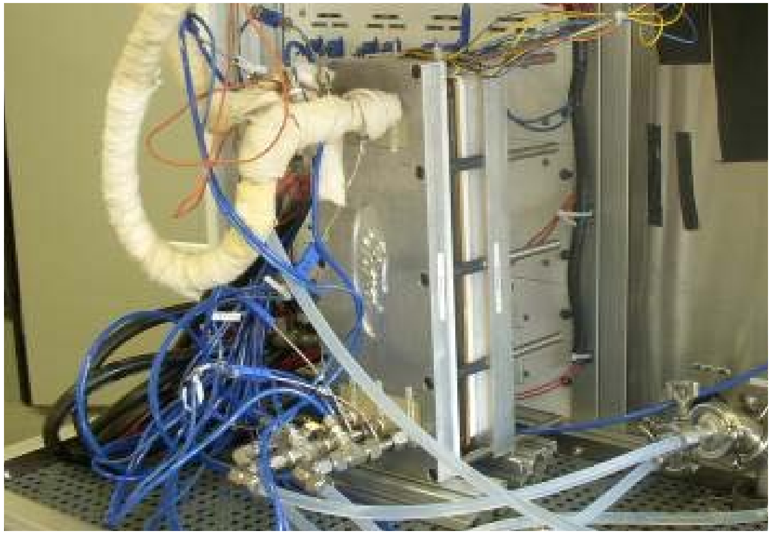
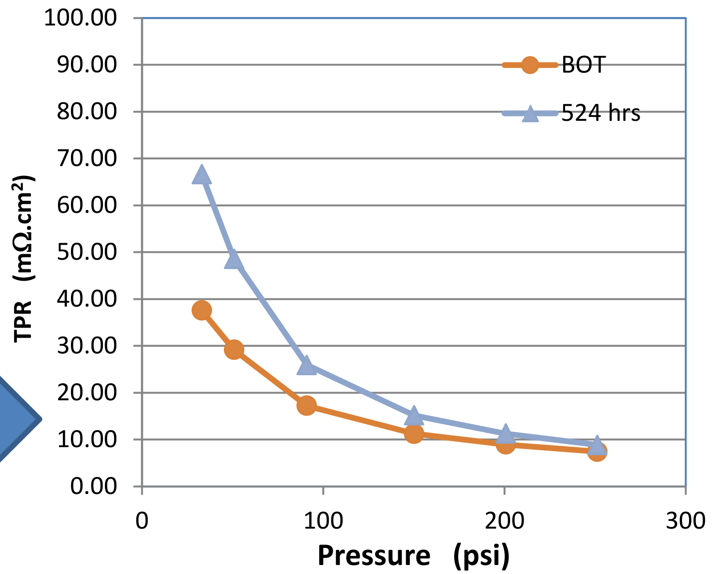
Durability Test in Short Stack under Dynamic Driving Conditions

- *TreadStone SS plates with Nb-TiO_x coating were tested in-situ for durability demonstration.*
- *Ford provided the stack and conducted the initial performance tests.*
  - *10-cell, 2.5 kW , 300 cm^2 active area, with TreadStone's coating*
- *The stack long term durability test is conducted at HNEI, Univ. Hawaii*
  - *The stack is tested for durability utilizing durability cycle (which includes FTP cycle along with others) mimicking real world driving conditions.*



- No corrosion marks on the tested plate.
- Plate TPR has very small increase after 524 hours test in the stack.



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Collaborations

Team Partner:

HNEI, Univ. Hawaii

2.5 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

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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.
  - Identified processing factors that determine the plate performance.
  - *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.
  - 1100 hours single cell evaluation demonstrated its durability in PEM fuel cells.
  - Long term durability test in short stack is on going at HNEI, U. Hawaii.
- ***Future Work:***
  - Complete the R&D to fully understand the working mechanism of the coating.
  - Develop the fabrication process and quality control systems to ensure the low cost production.
  - Conduct the fabrication cost analysis for large volume production.

Acknowledgements

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

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