



Novel Structured Metal Bipolar Plates for Low Cost Manufacturing

PI: Conghua “CH” Wang

TreadStone Technologies, Inc.

June 6, 2017

Project ID# : FC105

Project Overview

Timeline

- Project start date: Jan. 15, 2017
- Project end date: Jan. 14, 2018
- Percent complete: 15%

Budget

- Total Funding Spent:
as of 3/31/17: \$63,000
- Total Project Value: \$437,500
- Cost Share Percentage: 20%

Barriers

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

Supporting Partner

- Strategic Analysis, Inc.
- Hawaii Natural Energy Institute, University of Hawaii.

Objective of the Project

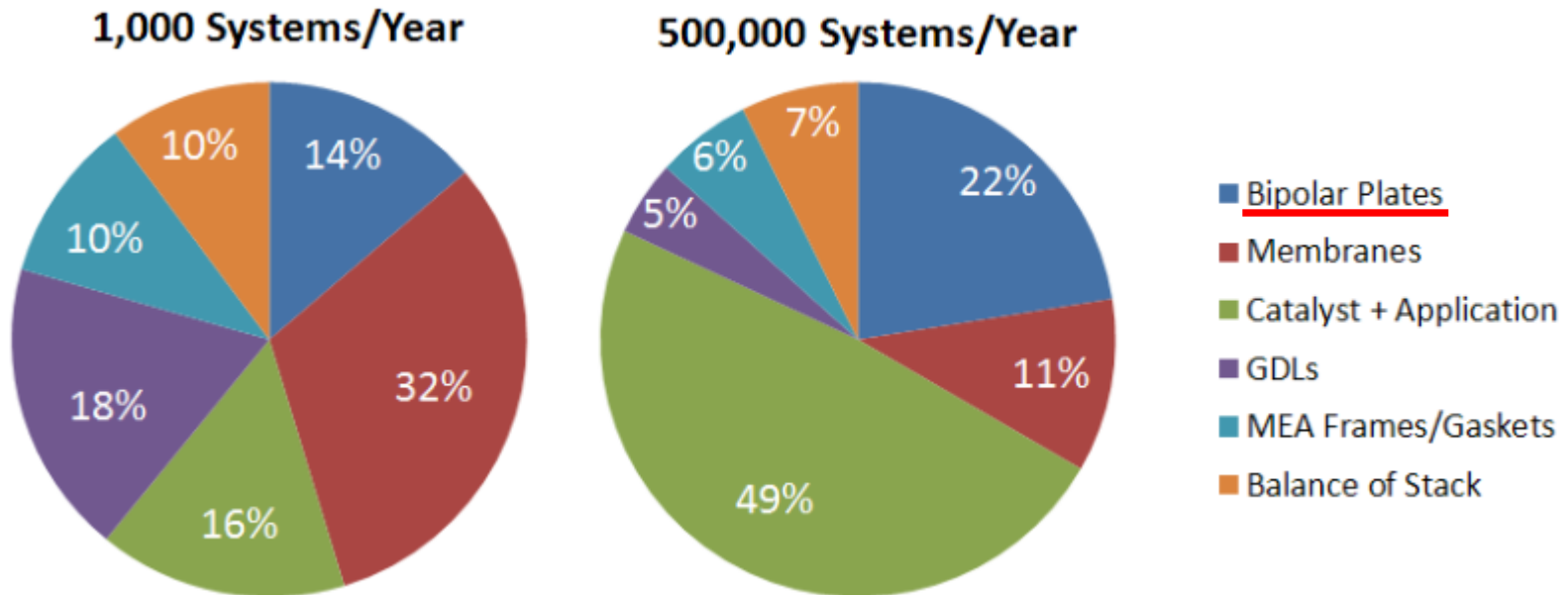
- Overall Objective: Develop lower cost metal bipolar plates to meet performance target and 2020 cost target (<\$3/kW)
 - Develop large scale manufacturing process for the doped titanium oxide coating technology demonstrated in previous SBIR project.
 - Investigate the relationship between processing conditions and doped titanium oxide properties for production quality control system development.

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 Cost

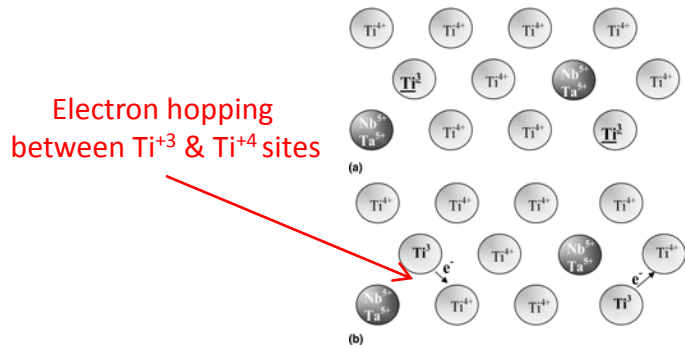


J. Spendelow, J. Marcinkoski, "Fuel Cell System Cost – 2013"
DOE Fuel Cell Technology Office Record # 13012

Approach: Coating Material for SS Plates

--- Semiconductive Doped TiO_x

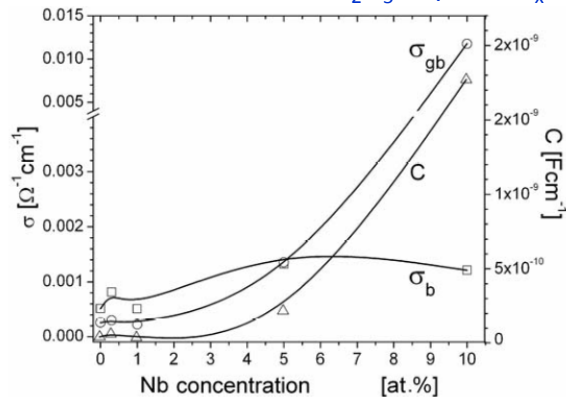
Doping TiO_2 with +5 valence elements enforce the formation of Ti^{+3} in TiO_2 lattice structure, and result in the higher electronic conductivities.



Challenges to use doped TiO_x coating:

1. Doped TiO_x is semi-conductive. The electrical conductivity is not high enough.
2. The weak bonding of doped TiO_x to 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

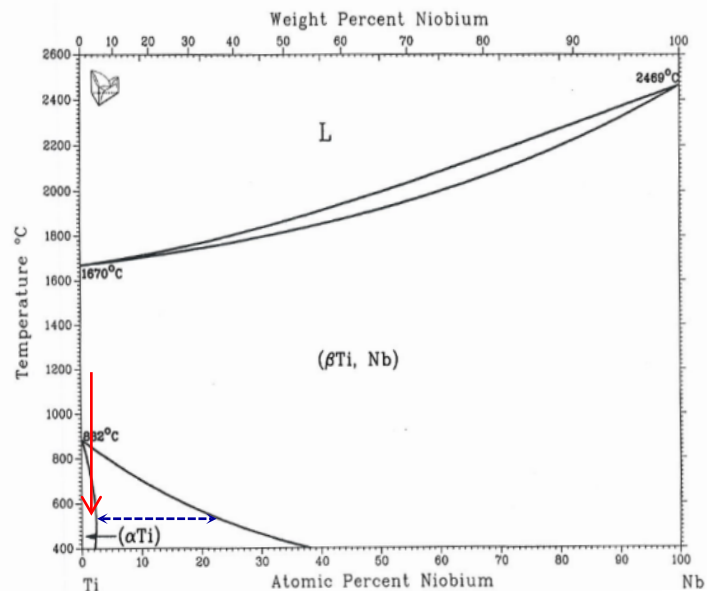
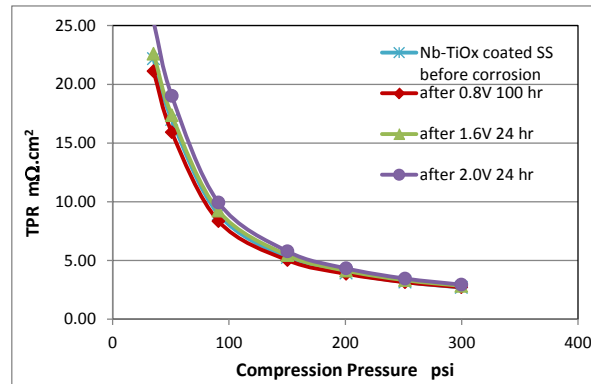
TreadStone's approach:

- 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).

Achievements and Challenges

--- from previous SBIR project

- Low contact resistance and superior corrosion resistance have been demonstrated using the doped TiO_x coating on SS substrate for PEM fuel cell applications.
- Challenges:
 - Processing technology has to be optimized to obtain the desired structure of surface oxide layer.
 - Segregation of Nb or Ta alloy element from the coating surface leads to the pure Ti surface oxide layer of the sputtering coated surface.
 - Need to determine the optimized coating composition.
 - Understand the relation of the processing condition with the surface oxide layer structure.



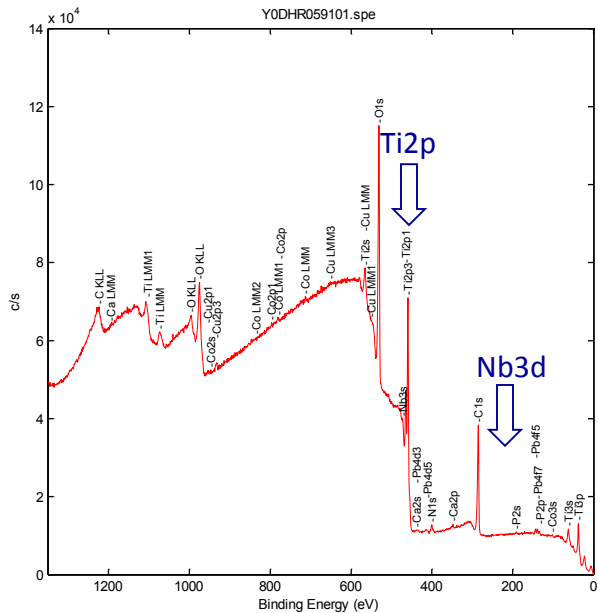
The objective of this project is to develop technical solutions to overcome these challenges..

PVD Process Condition Optimization

--- Minimizing Surface Layer Alloy Element Segregation

- Previous Processing Condition

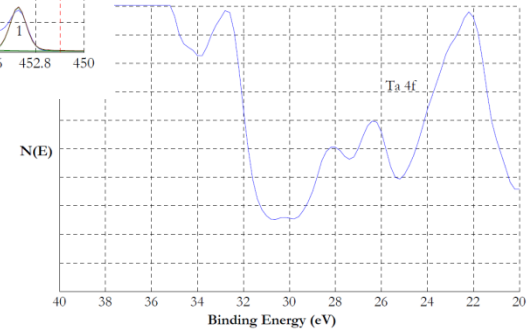
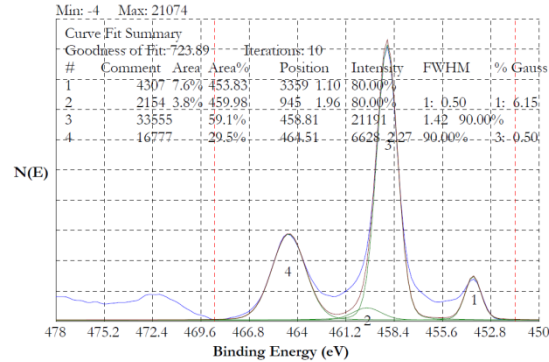
XPS survey spectrum of as-coated Ti3Nb on SS.



No Nb (or Ta using Ti-Ta alloy target) was detected on the coating surface

- Modified Processing Condition

XPS core level spectra of as-coated Ti2Ta on SS.

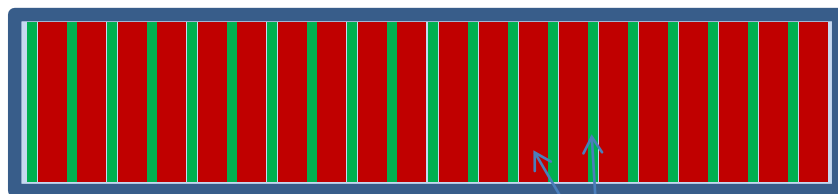


Surface layer Ta to Ti ratio is 0.4% : 99.6% (using Ti2Ta target)

The modified processing condition has brought Ta to the surface. Further optimization is needed to increase its concentration.

Coating Composition Optimization

- Need to prepare targets with various compositions.
 - Challenge: the cost and lead time for custom composition targets.
 - Typically \$2,000 – \$3,000, 6-8 weeks for one target.
- Conventional co-deposition: using several targets, alternately deposit from each target to control the bulk composition of the coating by the sputtering time of each target.
 - The coating is nano-scale layer by layer structure, not the atomic scale mixing. The surface oxide composition (several nm thick) could only contain the element of last layer of the coating.
- **“Mosaic” target approach for coating composition optimization.**



Ti tray to hold target strips

Target strips of alloy or metal

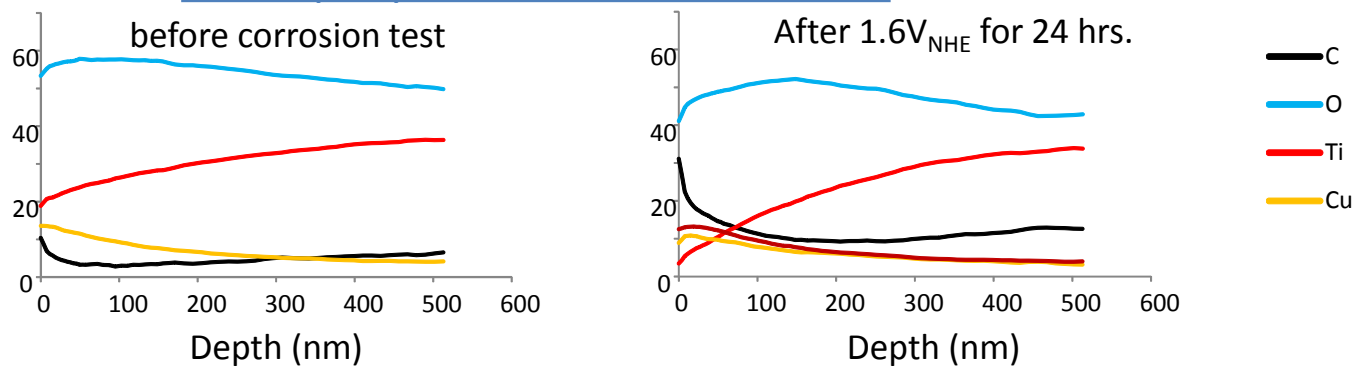
- The coating composition can be controlled by the width of each target strip.
- The distance from the target to the substrate has to be long enough for sufficient mixing of vapors.

Mosaic target approach makes it feasible for composition optimization in this project.

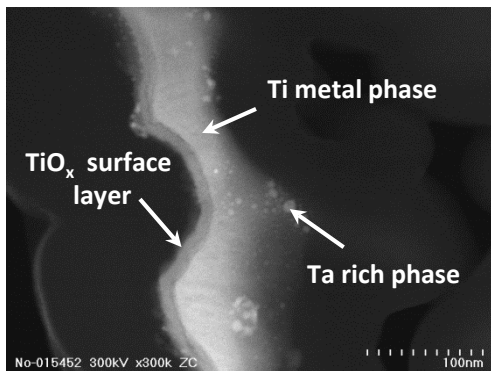
Surface Oxide Layer Characterization

- Previously, X-Ray Photoelectron Spectroscopy (XPS), Auger Electron Spectroscopy (AES) and Rutherford Backscattering Spectroscopy (RBS) have been used to characterize the surface oxide layer.
 - The rough surface topography makes the analysis difficult.
 - The thickness of the surface layer are relative (typically to SiO₂).

XPS depth profile of Ti₂Ta coated SS



- **FIB/STEM is more suitable method for the surface analysis.**



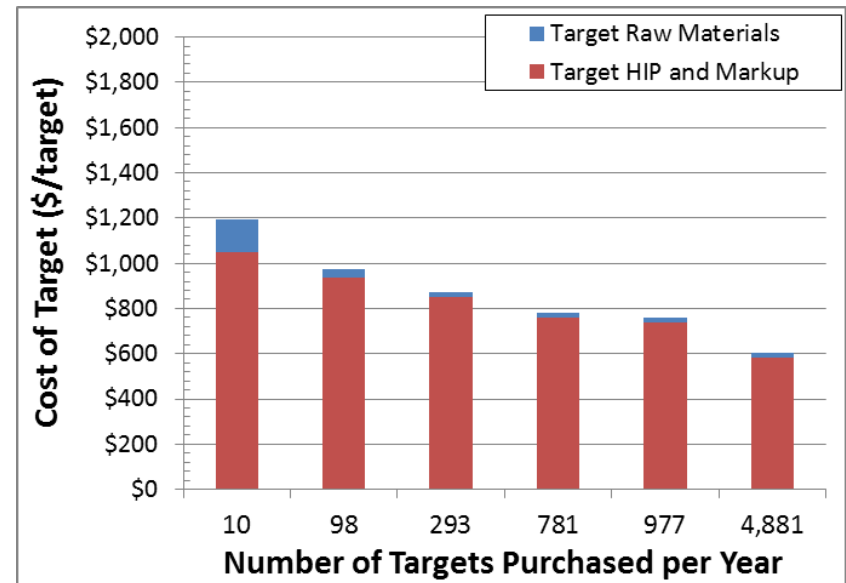
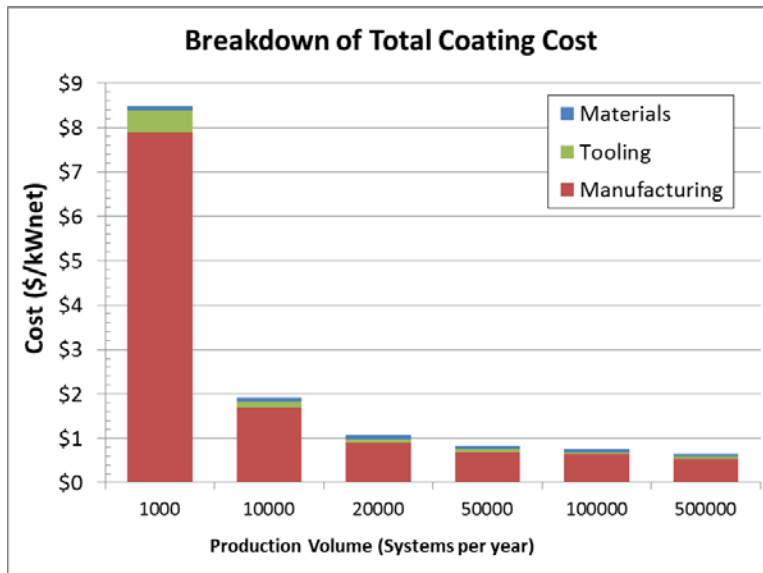
- The TiO_x surface oxide layer is 15-20 nm.
- There is Ta rich phase in the Ti₂Ta coating layer.
- The small amount of the β phase can not be detected by XRD.

The data was from a DOE SBV project, teaming with LANL and ORNL.

Fabrication Cost Analysis

--- Conducted by Strategic Analysis, Inc.

- 377 BPP assemblies per stack, 1095 mW/cm² power density,
- The target material cost is dominated by the manufacture cost. It could be reduced with lower cost manufacture method.
- Total cost of doped TiO_x coating is roughly \$0.14/welded pair of plates (\$0.85/kW) at 500k sys/year. The largest contributor is the PVD capital cost.



Planned Future Work

- **PVD Process Development**

- Using the methods developed in the project, conduct a detailed investigation of the process conditions and coating layer structure to enable a reliable manufacturing process for large scale production
 - Working with two PVD companies for the process development.

- **Doped TiO_x Surface Oxide Characterization**

- Using FIB/STEM, investigate the relation of the surface oxide thickness/composition to the processing condition and its stability under PEM fuel cell operation conditions.
 - Having user agreement with IAC, Princeton University for the access to their facility. Their new equipment, installed last summer, is capable for the task.

- **Durability Evaluation**

- *ex-situ* and *in-situ* tests of the doped TiO_x coating for PEM fuel cell applications will be conducted at TreadStone and HNEI, U. Hawaii

Milestones

- **Milestone 1.1:** Manufacture Cost Analysis. By the end of 3rd month, the manufacture cost analysis is finished.
- **Milestone 1.2:** PVD Process Development. By the end of 6th month, the target material is prepared and the initial metal plate coating process for has been demonstrated.
- **Milestone 1.3:** PVD Process Development. By the end of 9th month, the optimized metal plate coating process development will be finished.
- **Milestone 2.1:** Doped TiO_x surface layer properties determined. By the end of 9th month, the properties of the doped TiO_x surface will be determined.
- **Milestone 2.2:** Compatibility with bipolar plate stamping process characterized. By the end of 12th month, the feasibility of the doped TiO_x coating on low cost 304 [and 202] stainless steel substrates and as the pre-stamping coating is evaluated.

Summary

- **Objective:**
 - Develop low cost fabrication process of the doped TiO_x coating for PEM fuel cell applications.
 - Investigate the relation of the properties of doped the doped TiO_x surface layer with processing condition and its durability in PEM fuel cell operation environment.
- **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 processing factors for the coating layer surface composition control.
 - *Developed a low cost method for coating composition optimization.*
 - Manufacturing cost analysis indicates that the cost of this precious metal free coating technology is lower than other competing technologies.
- **Future Work:**
 - Detailed development of the PVD process for the optimized coating properties.
 - Investigation the relation of the surface oxide layer properties with processing conditions and its durability.
 - Conduct *ex-situ* and *in-situ* tests to demonstrate the performance of the coating processed with low cost fabrication process.

Acknowledgements

- DOE EERE Fuel Cell Team.
- DOE SBIR Program.
- Supporting Partners.
 - Strategic Analysis, Inc.
 - HNEI, U. Hawaii