Development and Manufacturing for Precious Metal Free Metal Bipolar Plate Coatings for PEM Fuel Cells

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TreadStone Technologies, Inc.

DOE project award DE-EE0009613

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DOE Hydrogen Program

2023 Annual Merit Review and Peer Evaluation Meeting

Project ID# : FC345

Project Goal

- Reduce the manufacturing cost of PEM fuel cell metal bipolar plate in order to meet DOE’s cost target of < $5kW.
  - Using low grade stainless steel and aluminum as the substrate material.
  - Develop precious metal-free coating technology.
  - Develop the coating process that is suitable for roll to roll (R2R) manufacturing process.
- Meet the performance and durability requirements for HDV application.
  - Develop the accelerated stress test (AST) protocols for rapid evaluation of PEM fuel cell bipolar plates.
  - Investigate the coating electrical conductance and degradation mechanism.
- Focused low grade stainless steel material

Chemical formulation of low grade stainless steel (typical value %)									
#	Cr	Ni	Ti	Cu	Mo	C	Si	Mn	Fe
430	17.0	<0.5	-	<0.5	<0.5	<0.12	<1	<1	Bal
JFE443CT	21.0	0	<0.3	<0.4	-	-	-	-	Bal
JFE409L	11.0	<0.5	-	-	-	<0.08	<1	<1	Bal

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Project Overview

Timeline

- Project Start Date: Sept. 17, 2021
- Project End Date: March 31, 2014
- Percent Complete: 50%

Budget

- Total Project Funding: $1,469,112
  - DOE Shares: $1,115,162
    - BP1 $562,725
  - Contractor Shares: $353,950
    - BP1 $180,975
- Spending (to Feb. 28, 2023)
  - DOE Share: $377,325
  - Contractor Shares: $145,818

Barriers

- Barriers Addressed : Bipolar Plate Durability and cost
  - Cost: < $5/kW (2025)
  - resistivity < 10 $m\Omega.cm^2$
  - corrosion < 1 $x10^{-6} A/cm^2$

Partners

- Las Alamos National Lab.
- Pacific North Wester National Lab.
- Univ. Tenn, Knoxville
- Austin Power.

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Relevance/Potential Impact

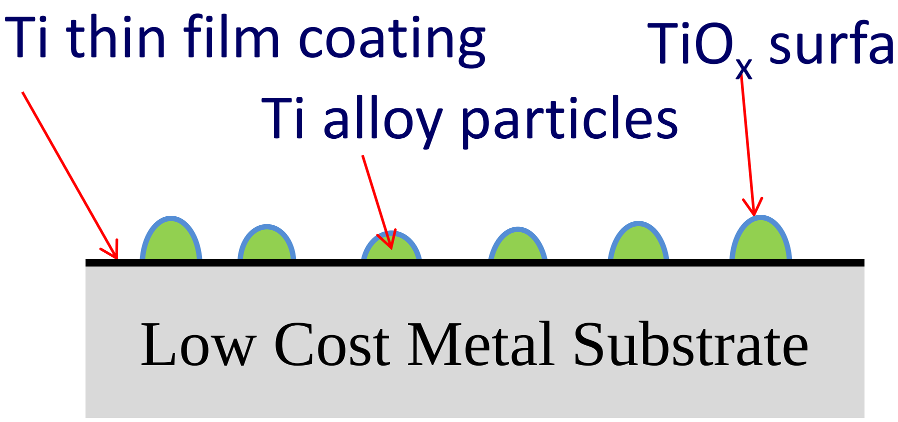
- Bipolar plates are the second most expensive component in PEMFC stacks.
- Low-cost metal bipolar plates and fabrication techniques are critical to meet HFTO 2030 cost and performance targets for heavy-duty applications.

Barriers	DOE Targets	Project Impacts
Cost	<$5/kW	Develop precious metal free coating technology to enable low grade stainless steel and aluminum based metal plate to meet the cost target.
Durability	>25,000 hrs	Develop rapid AST protocol for rapid evaluation.
Performance	Corrosion Current: $< 1\ \mu A/cm^2$ Contact Resistance: $<10\ m\Omega.cm^2$	Investigate the coating degradation mechanism to achieve the superior performance.

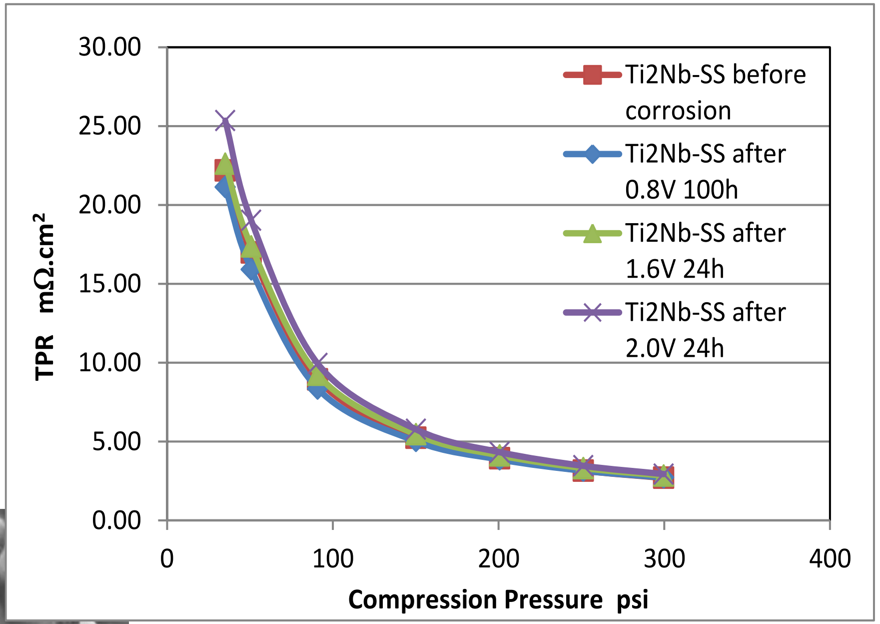
Project objectives and impacts directly contribute to meet HFTO 2030 Targets

Technical Approaches

Schematic drawing of TiOx coating Structure



TiOx coating has demonstrated the superior durability.

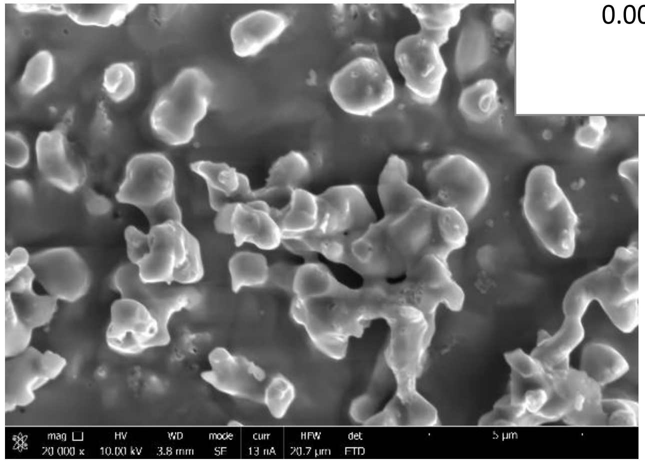


<u>Key parameters of the coating:</u>

1. Surface topography
2. Surface oxide layer composition

<u>Manufacturing Technology:</u>

- ➢ Bond Ti alloy particles on metal substrate surface to obtain the desired topographic structure & composition. (DPB process)



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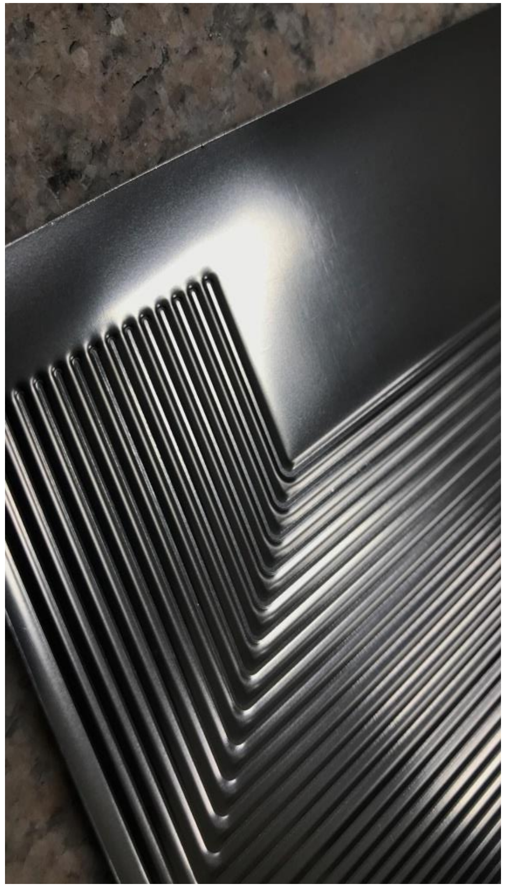
PNNL Aluminum Based BP Background

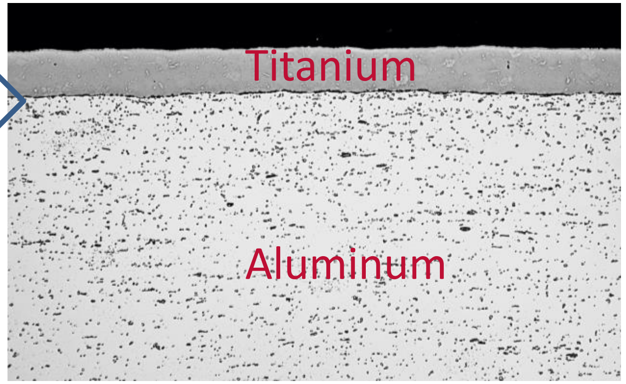
- Initial projects demonstrated that diffusion bonded titanium to aluminum was viable
  - Demonstrated that all 2020 DOE bipolar plate technical targets for light duty vehicles could be met with this solution
    - Cost targets were shown to be capable of being attained, but would require significant process improvement to enable cost reduction of the diffusion bonding process
    - Required the addition of coating to reduce the interface contact resistance (ICR) of the plate. Demonstrated with TreadStone's DOT technology.
- Focus of this project is to demonstrate the feasibility of using precious-metal-free TiOx coating to reduce the ICR.

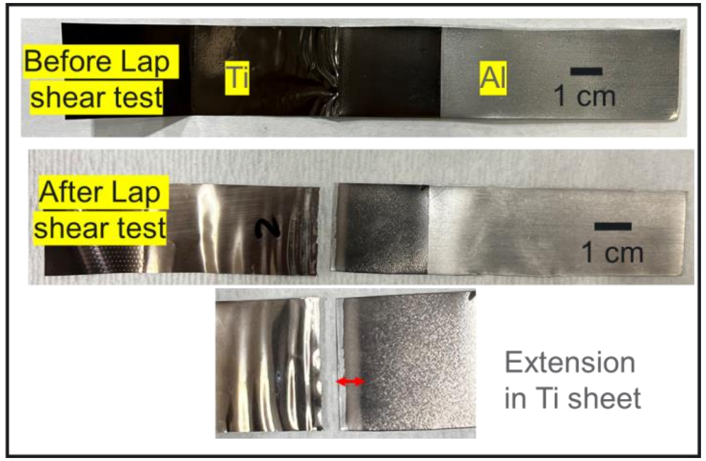

Simultaneously formed and diffusion bonded titanium foil to aluminum bipolar plate demonstration

Diffusion Bonding Cycle Time Improvement

- In Phase 1, numerous opportunities to improve diffusion bonding cycle time identified
  - Multiple parts per cycle (readily implementable)
    - ✓ Stacking
    - ✓ Large sheets
- Focus on Process improvements
  - **Temperature/Time:** 510 – 640˚C / 10 to 30 min
  - **Pressure:** 1000 – 3750 psi (7-26 MPa)
  - **Atmosphere:** Inert (Argon)
  - **Surface preparation:** Acetone cleaned or 400 grit polished
- Results
  - Diffusion bonding at higher pressures and temperatures feasible
  - Diffusion bonding times reduction of at least 50% possible
  - Further reduction in cycle time looks highly probable

Diffusion Bonded Ti to Al Cross Section





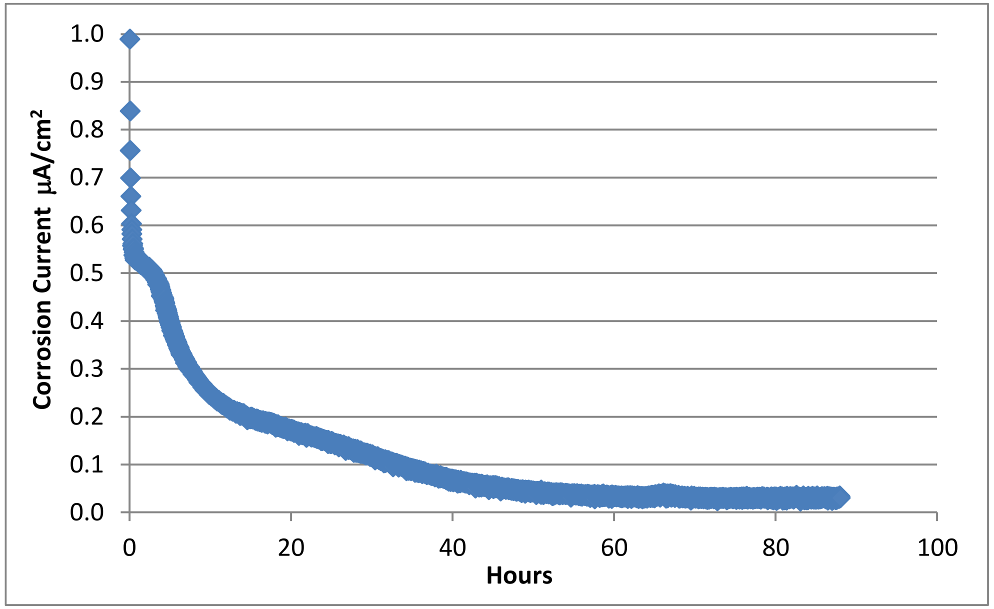
Lap Shear Test: All Failures in Ti base Material

Accomplishments and Progress

Performance of Nb-TiOx coated Aluminum Plate

Corrosion current of Nb-TiOx coated Aluminum

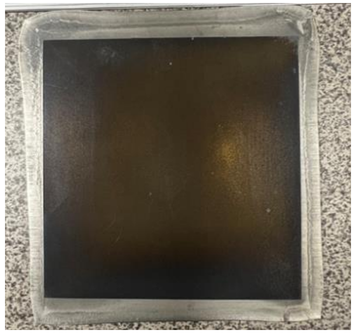
at $0.8V_{SHE}$ in pH 3 H_2SO_4 + 0.1ppm HF solution at 80^oC



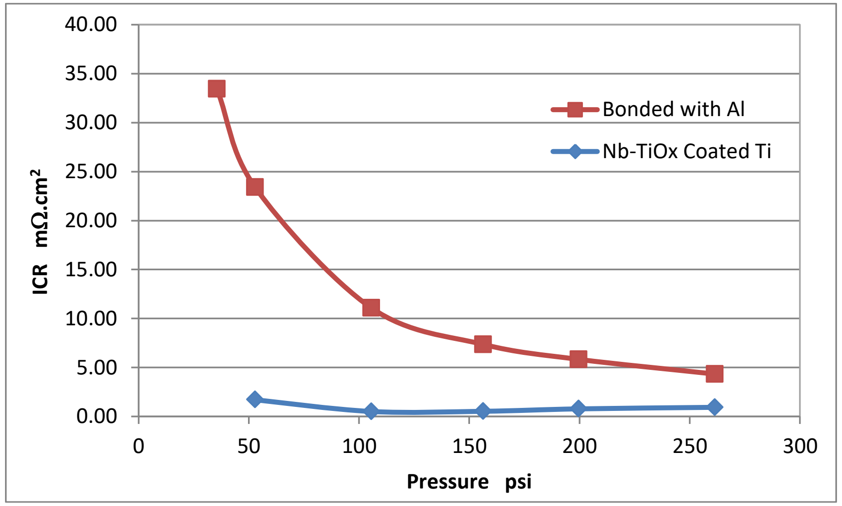
Nb-TiOx coated Al plate is ~ $0.03\mu A/cm^2$ meet Performance target ($< 1\mu A/cm^2$)

Nb-TiOx coated Ti foil

Nb-TiOx coated Al plate



ICR of Nb-TiOx coated Ti foil and bonded with Al plate



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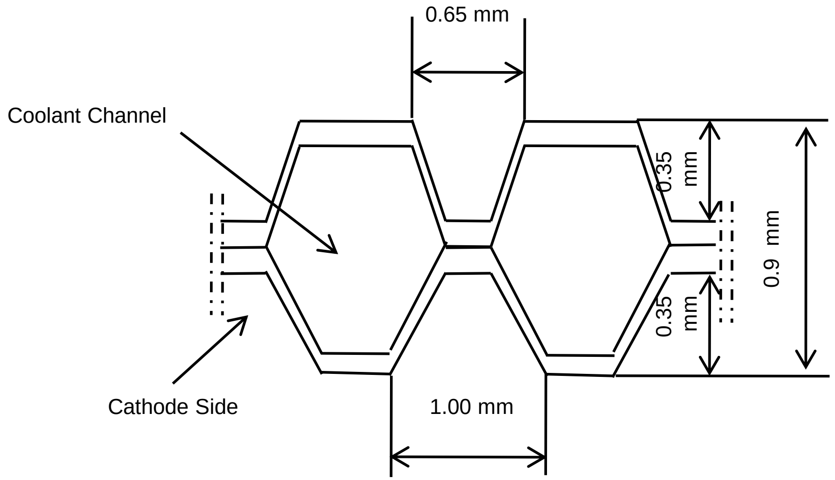
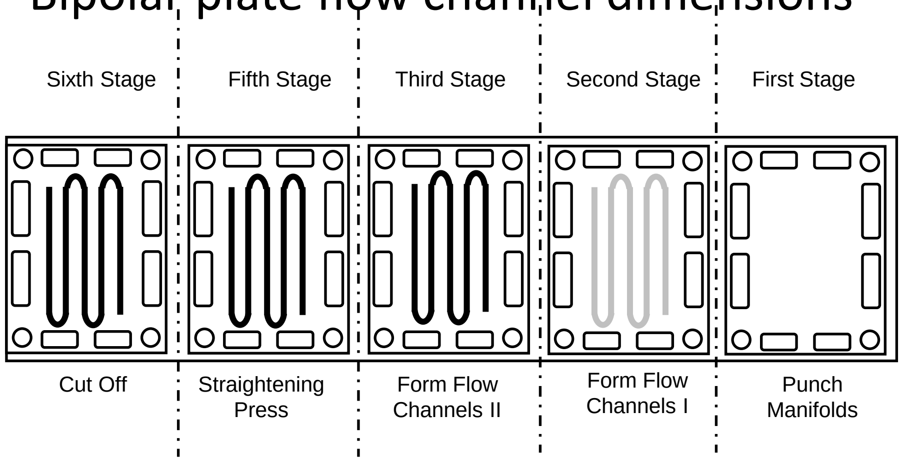
Cost Analysis Assumptions

Assuming pre-stamping coating and two stamped plates jointed by laser welding.

- 80 kWnet fuel cell system
- Power density: 1,150 mW/cm^2
- Cell voltage: 0.663 V
- Bipolar plate area: 328 cm^2 (L 20 cm x W 16.4 cm)
- 378 cells in one stack
- A five-stage progressive die stamping process (hydro-forming could be an alternative)
- Laser welding to join the two plates (Adhesive bonding could be alternative, lack of long-term durability)
- 75 µm thick SS409 foil substrate price: $4.0/kg
- Niobium powder price: $250/kg
- Ti powder price: $183/kg
- Ti sputtering target price: $199/kg



Bipolar plate flow channel dimensions

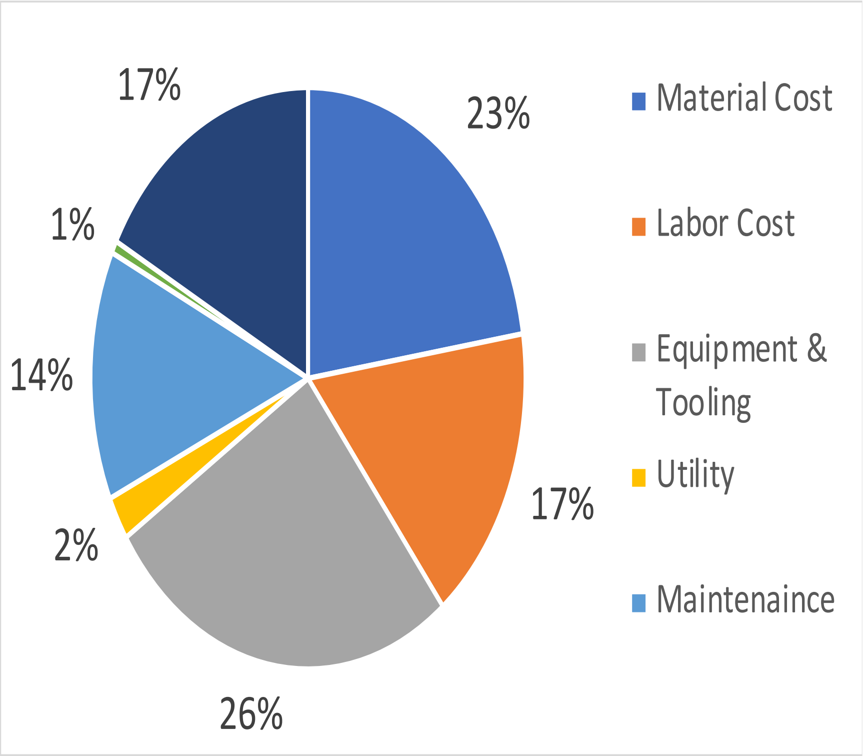


Five-stage progressive die stamping

TiOx Coated Metal BP Total Cost

The SS409 foil with TiOx pre-coating bipolar plate costs about $4.76 /kW at the production volume of 500,000-80kW stacks.

Cost	$/cell	$/kW
Material Cost	$0.23	$1.07
Labor Cost	$0.17	$0.80
Equipment & Tooling	$0.27	$1.26
Utility	$0.02	$0.12
Maintenaince	$0.14	$0.66
Building	$0.01	$0.03
Capital Costs	$0.17	$0.82
Total	$1.01	$4.76



409 SS foil coating cost: $1.93/kW
Plate manufacturing cost: $2.83/kW

- Annual production volume: 500,000 80kW stacks – 190 million BPPs
- 80 kW Stack
- 378 cells
- Cell area 327 cm^2
- Power density: 1,150 mW/cm^2

Accomplishments and Progress

AST Protocol

Proposed BPP AST Initial Tests	
AST	Start/Stop
Cycle	Triangular sweep cycle: 1 V/s between 1 V_{SHE} and 1.5 V_{SHE}
Number	10,000 cycles
Cycle time	1 seconds
Temperature	80°C
Solution	1 mM H_2SO_4
Metric	**Frequency**
Anodic Corrosion Current < 1uA corrosion current	After 0, 10, 100, 200, 500, 1k, 2k, 5k and 10k cycles*
Effluent ICP/MS**	After 0, 2k, 5k and 10k cycles

*Solution was replaced after 2k cycles due to evaporation

Samples collected **(in process)

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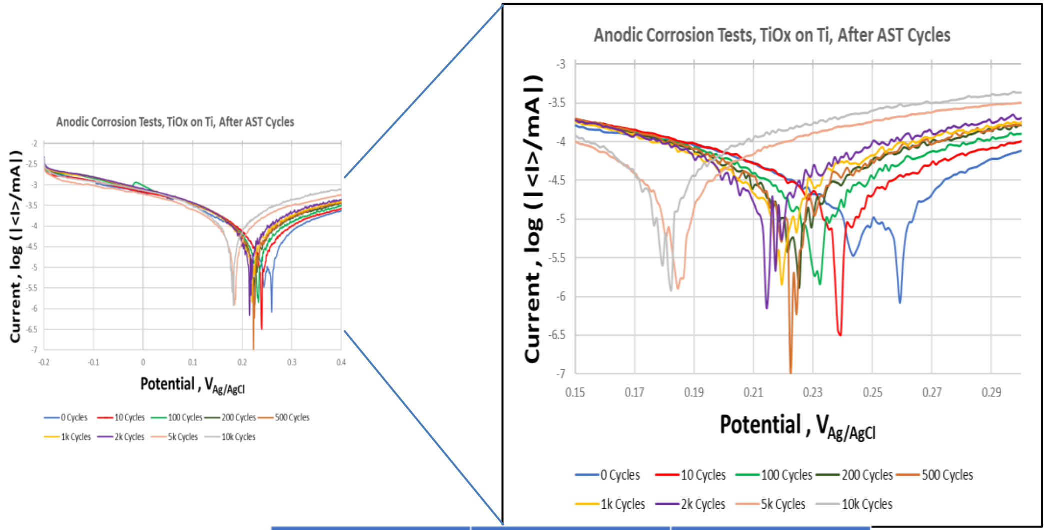
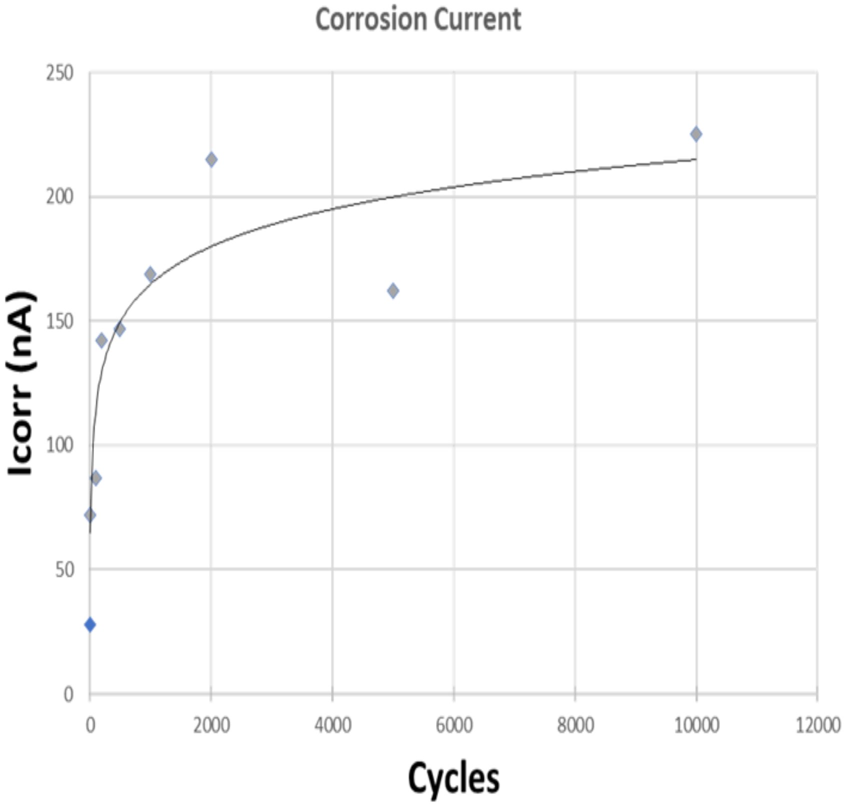
Accomplishments and Progress

AST Protocol



# of Cycles	Ecor	Icor
0	0.260 V	0.028 uA
10	0.238 V	0.072 uA
100	0.231 V	0.087 uA
200	0.225 V	0.142 uA
500	0.222 V	0.147 uA
1,000	0.219 V	0.169 uA
2,000	0.214 V	0.215 uA
5,000	0.185 V	0.162 uA
10,000	0.180 V	0.225 uA



- Clear trend observed with E_{corr}
- Passivation layer decays as cycles increase
- Providing less protection thus increasing corrosion current
- E_{corr} decreases with increasing cycles

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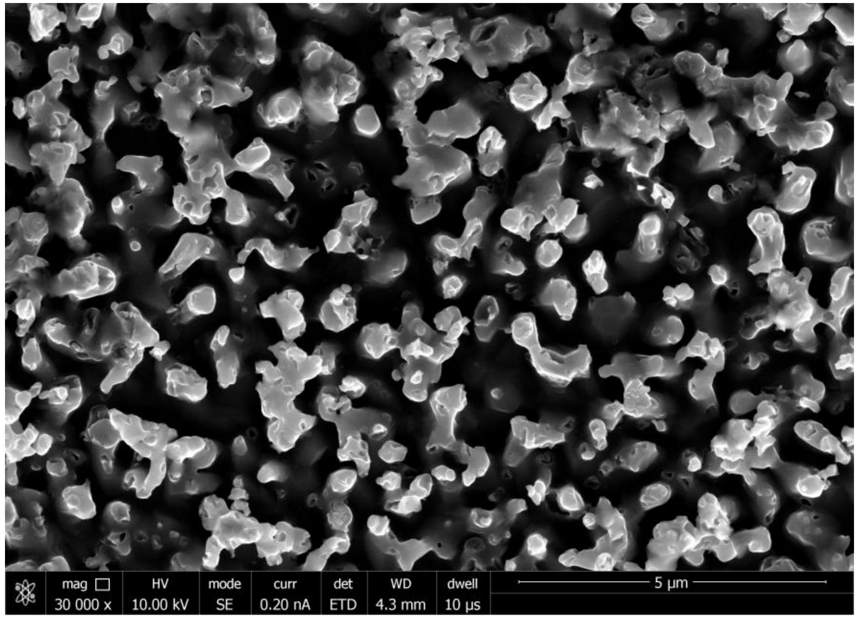
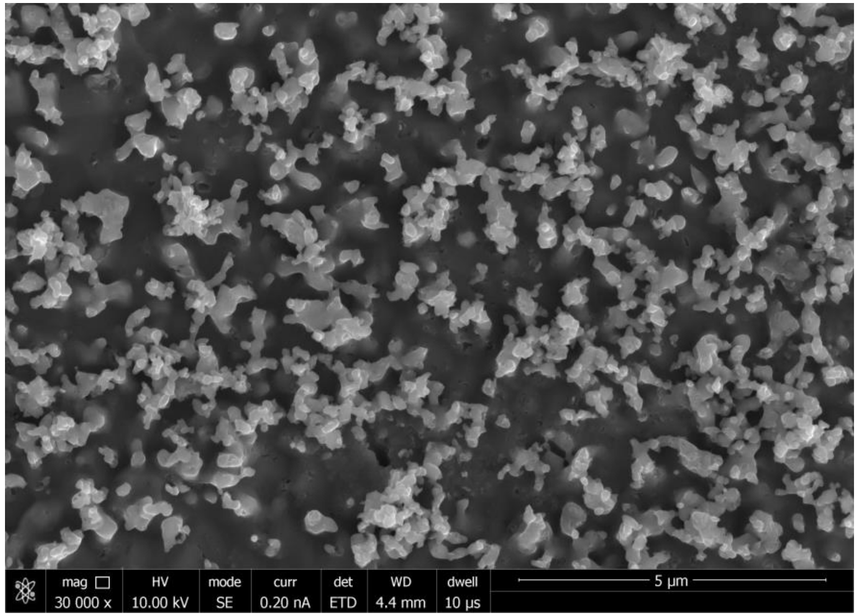
Accomplishments and Progress

Microscopic structure of the Nb-TiOx coated SS.





- The Microstructure of the coating topographic structure can be tailored by processing condition

Surface oxide layer composition.

- Using XPS for surface layer composition analysis was not reliable, because it is difficult to avoid substrate material, even at small angle.
- It is better to do the analysis by TEM.

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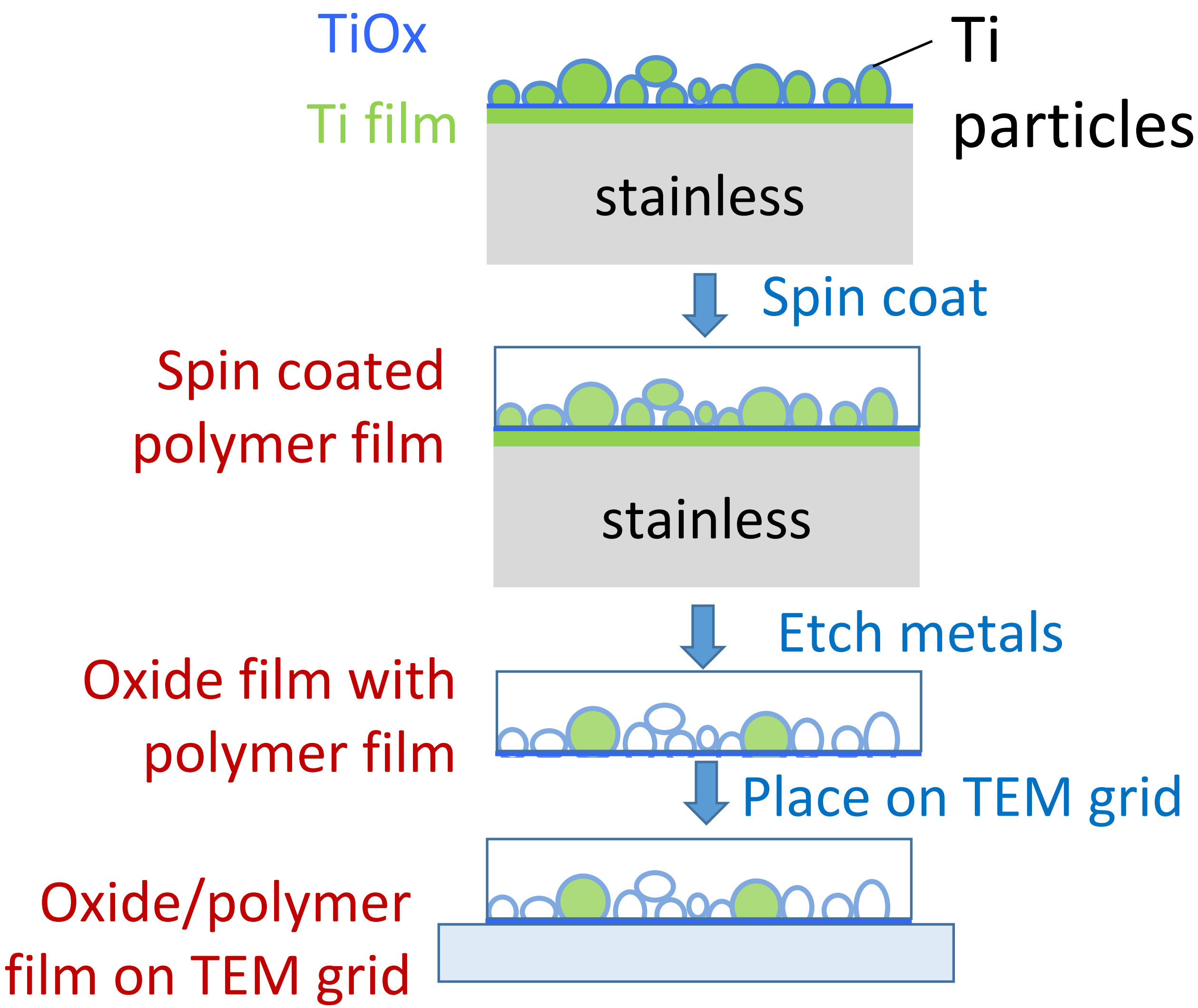
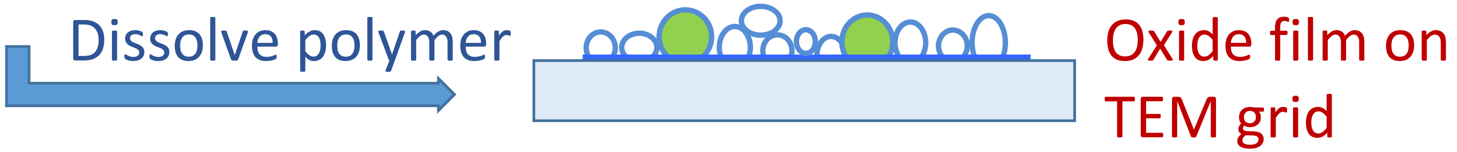
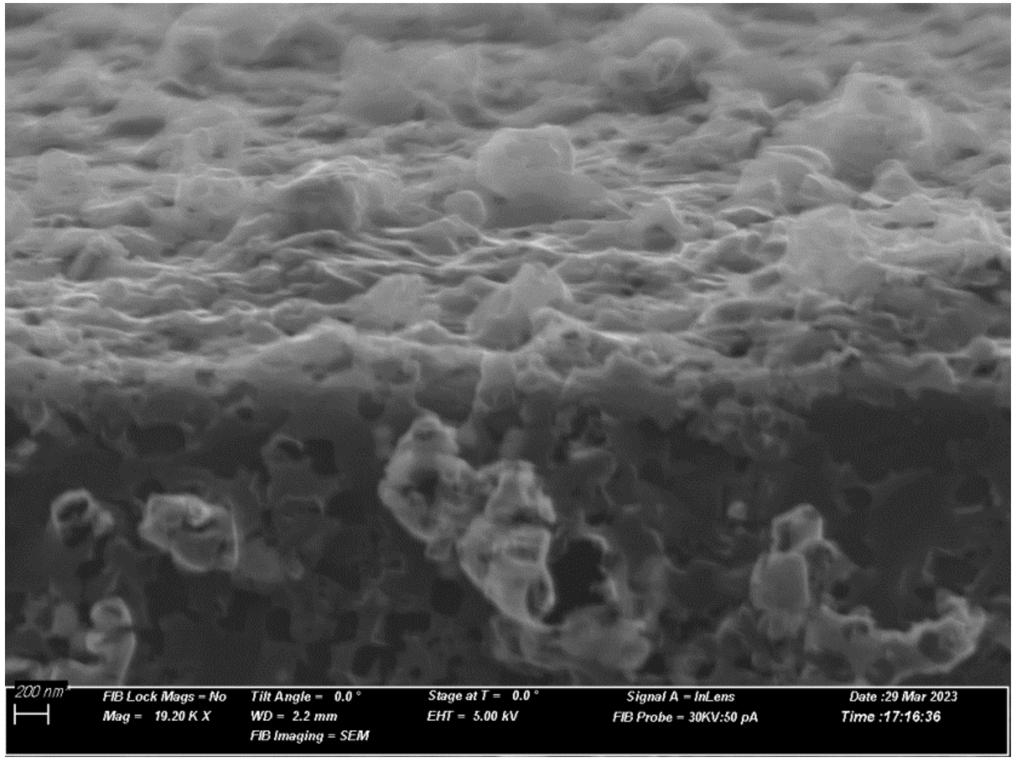
Accomplishments and Progress

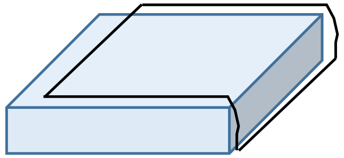
New method for surface oxide analysis – surface lift-off







Lifted TiOx coating sheet can be draped over a substrate edge, so that surface morphology can be viewed in TEM from different angles.



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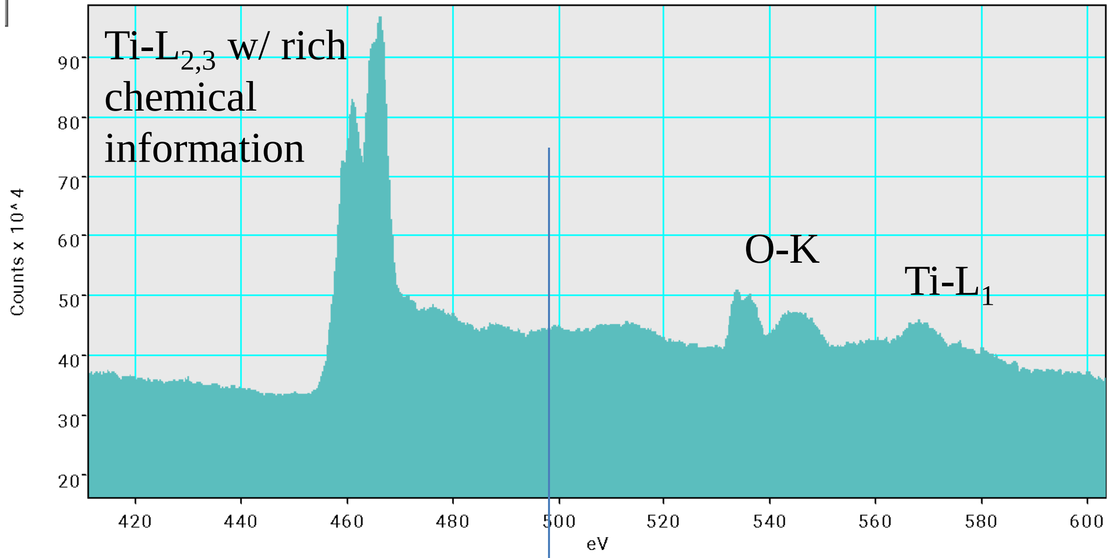
Accomplishments and Progress

TiOx Coating Structure and Electrical Conductance Mechanism

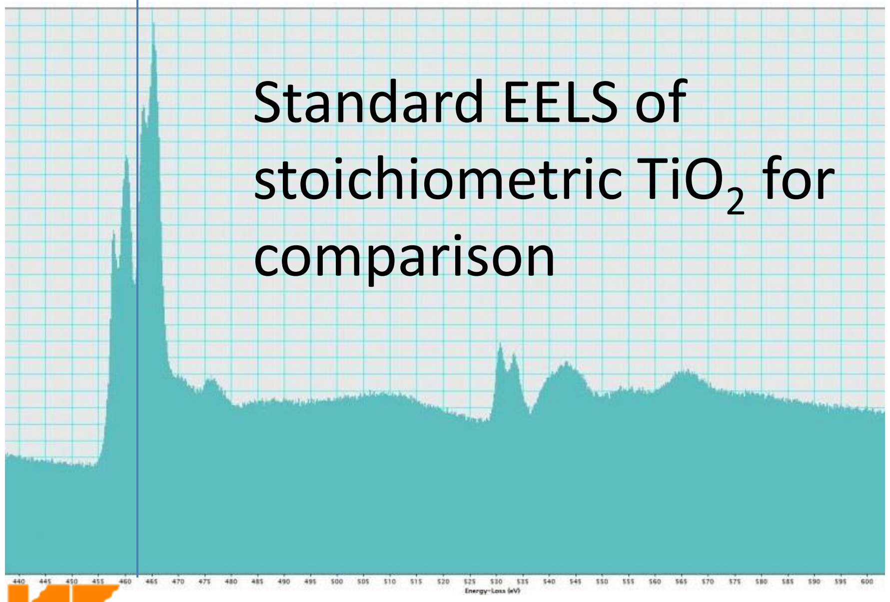


Electron energy loss spectrum (EELS) Our sample. This particular specimen went through longest etching (for substrate removal), thus suboxide at bottom removed.

Relative quantification (in 70 nm dia. Circle)

Ti (33.18 ± 4.1) at.%

O (66.82 ± 8.3) at.%



Standard EELS of stoichiometric TiO_2 for comparison

- Sampling size can go to atomic level (< 1nm).
- Suitable for large size sample with micron textured surface analysis.

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Collaborations

TreadStone
Project Management
Coating Process Development & Corrosion Tests
PI: Conghua Wang 20%
PM: Gerald DeCuollo 5%

LANL
Tommy Rockward
BP AST Protocol Development & Test

Austin Power
Yong Yang
Manufacturing Cost Analysis

PNNL
Christopher Smith
Aluminum Based BP Development

UT. Knoxville
Gong Gu
Degradation Mechanism Investigation

Future Work

- Optimization of the TiOx formation process on Ti-Nb particle surface and complete the manufacturing cost analysis.
- Demonstration of the TiOx coated BPs in PEMFC single cell evaluation, including operation under AST conditions.
- Investigation of performance degradation mechanisms of the TiOx coating under PEMFC application conditions.

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

Summary

- ***Objective:***
  - Develop low cost fabrication process of the doped TiO_x coating on low cost metal substrate for PEM fuel cel HDV applications.
  - Develop AST protocol for rapid evaluation of BPs for HDV applications.
  - The coating conductance mechanism investigation
- ***Relevance:*** Reducing the metal bipolar plate cost to DOE's performance and cost target.
- ***Approach:*** Using doped TiO_x coating on low nickel content SS and aluminum surface for HDC fuel cell applications.
- ***Accomplishment:***
  - Demonstrated the coating process for low cost metal substrates.
  - *The AST and conductance mechanism investigation are underway.*
- ***Future Work:***
  - Finish the AST protocol development and use it as the rapid evaluation method.
  - Investigation the TiOx coating conductance mechanism.
  - Further development of the coating process to reduce the process cost.

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