TAPA NFE-16-06190

Novel Coating for Fuel Cell Metal Bipolar Plates (SBV with TreadStone Technologies, Inc.)

Karren L. More

August 10, 2017

TAPA (CRADA) FINAL REPORT
NFE-16-06190

OAK RIDGE NATIONAL LABORATORY
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TAPA/NFE-16-06190

Center for Nanophase Materials Sciences
&
Fuel Cell Technologies Office

Final CRADA Report for Small Business Voucher (SBV) Pilot Project with TreadStone Technologies, Inc. – Novel Coating for Fuel Cell Metal Bipolar Plates

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Date Published:
August 10, 2017

Prepared by
OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee 37831-6283
managed by
UT-BATTELLE, LLC
for the
US DEPARTMENT OF ENERGY
under contract DE-AC05-00OR22725

Approved For Public Release

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ACKNOWLEDGEMENTS

This TAPA NFE-16-06106 was conducted as a DOE Small Business Voucher Pilot Project within the Oak Ridge National Laboratory (ORNL) sponsored by the US Department of Energy Fuel Cell Technologies Office. Opportunities for Small Business Voucher Pilot project collaborations are posted at https://www.sbv.org/about.html. The goal of the SBV is to put the resources of the national laboratories at the fingertips of U.S. small businesses. Research sponsored by the U.S. Department of Energy, Office of Energy Efficiency and Renewable Energy, Fuel Cell Technologies Office, under contract DE-AC05-00OR22725 with UT-Battelle, LLC.

ABSTRACT

Light-weight metal is the preferred material for bipolar plate in the automotive proton exchange membrane (PEM) fuel cell stack. The challenge using metal bipolar plates is the high surface electrical contact resistance and the poor corrosion resistance in PEM fuel cell operation conditions. This SBV project involved specialized capabilities and expertise at Los Alamos National Laboratory (LANL) and Oak Ridge National Laboratory (ORNL) supporting TreadStone Technologies, Inc. and included *ex-situ* and *in-situ* drive-cycle tests that incorporated coated metallic bipolar plates (LANL tasks) and analysis of the surface chemistry and microstructure of coatings on stainless steel substrates (ORNL tasks).

1. NOVEL COATING FOR FUEL CELL METAL BIPOLAR PLATES

This Small Business Voucher Pilot (SBV) project was begun on March 28, 2016 and was completed on June 30, 2017. The collaboration partner TreadStone Technologies, Inc. is a small business. The coating technology developed by TreadStone was characterized and found to be stable during fuel cell testing and protected the underlying metal bipolar plate.

1.1 BACKGROUND

Light-weight metal is the preferred material as the bipolar plate in the automotive proton exchange membrane (PEM) fuel cell stack. The challenge for using metal bipolar plates is the high surface electrical contact resistance and the poor corrosion resistance under PEM fuel cell operation conditions. When using metal bipolar plates, a low-cost coating technology is required that will protect the metal. Coating development companies have invested significant resources in this area.

TreadStone Technologies has developed two (2) generations of its corrosion-resistant coating technology. The first-generation technology uses a small amount of precious metal, which has been accepted by the market and is in the process for manufacture scale-up. The second-generation is a new precious metal free coating technology, which has been developed with the support of the DOE SBIR program. While the coating using precious metal has the advantage of better performance in more aggressive corrosion environments, the precious metal free technology has the potential for further lowering cost and immunizing against the price volatility of precious metals. The challenge for precious metal free coatings is the long-term durability of the coating material under fuel cell operation conditions, especially under transient, high potential conditions.

1.2 TECHNICAL RESULTS

For the second-generation precious metal free coating technology, TreadStone's approach is to use semi-conductive, doped TiO_x as the coating material on the metal substrate. The doped TiO_x layer is grown on a Ti alloy interface layer that is deposited on metal substrate by physical vapor deposition (PVD). The Ti alloy layer is the precursor of the doped TiO_x coating layer and the bonding layer between the doped TiO_x surface coating and metal plate surface.

The focus of this SBV project was to investigate coating degradation during harsh accelerated stress tests (ASTs), which were performed at LANL. The scope of the work at ORNL was focused on assessing the surface chemistry and microstructure analysis of a metal (TM)-doped TiO_x coating on 316L stainless steel substrate using X-ray photoelectron spectroscopy (XPS), scanning electron microscopy (SEM), and transmission electron microscopy (TEM). Pristine TiO_x-coated metal plates, TiO_x-coated plates after *ex situ* corrosion tests in pH=3 H_2SO_4 + 0.1 ppm HF solution at 80°C and at high polarization potentials (up to 2.0VNHE), and post-AST (*in situ* tests) plates (50cm^2 hardware) were characterized. The breakdown of tasks between LANL and ORNL was:

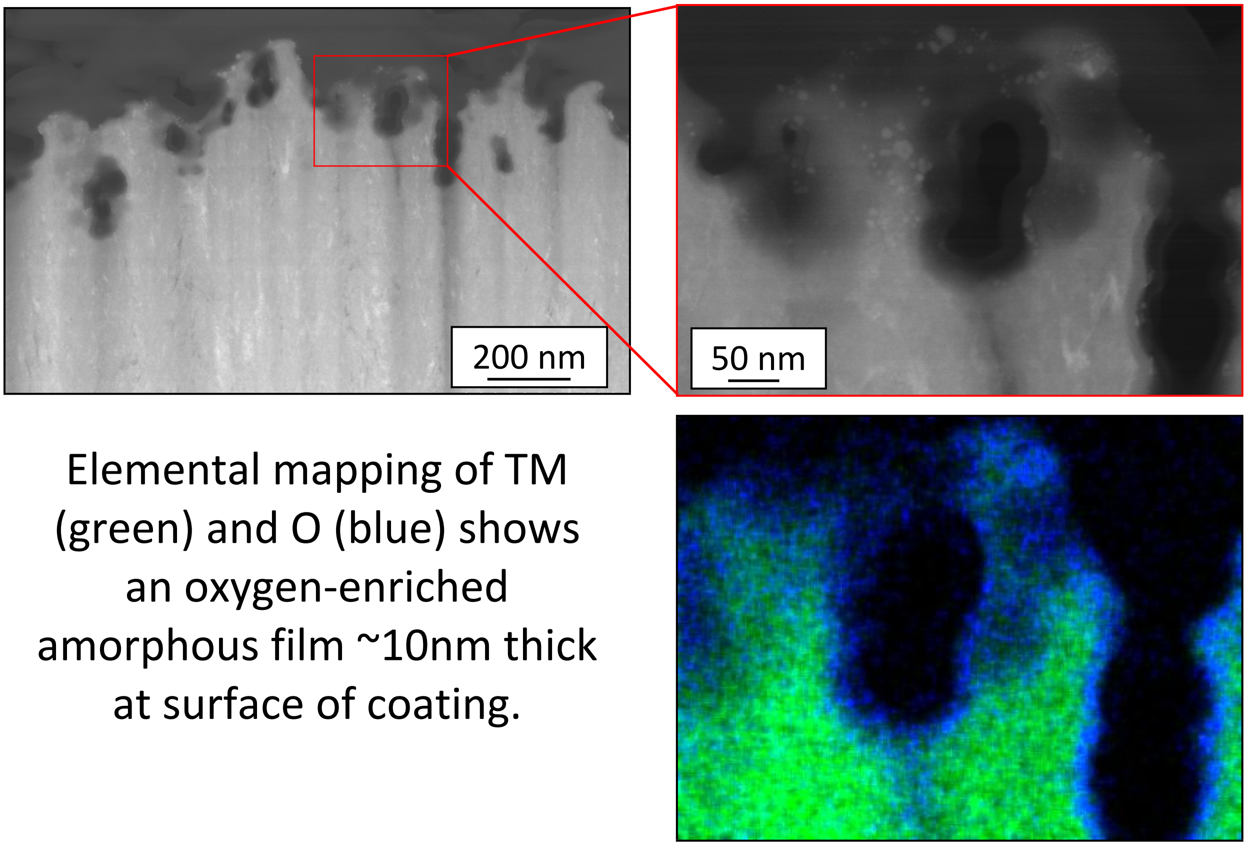
<u>Subtask 1: LANL</u>

Subtask 1a: *in-situ* fuel cell testing and accelerated stress tests of metal bipolar plates provided by Treadstone.
Subtask 1b: Chemical composition and elemental mapping (XRF)
Subtask 1c: Surface structure by 3D-tomography (X-Ray Computed Tomography)

<u>Subtask 2: ORNL</u>

Subtask 2a: Chemical composition and elemental mapping (XPS, SEM)
Subtask 2b: Oxide layer structure and changes in oxidation state (XPS, SEM, TEM)

The microstructure and composition of the pristine TiO_x coating is shown in the images and corresponding elemental map (Ti=green and O=blue) in Figure 1 show an ~10 μm thick TiO_x coating on the 316L substrate with a columnar morphology and a relatively porous surface. It is noted that the uppermost 10-50 nm surface layer of the TiO_x is oxygen-rich, which is consistent with XPS results from the same samples. No changes were observed in either the surface composition, bulk composition, or microstructure of the TiO_x coatings after *ex situ* corrosion testing (interfacial contact resistance), demonstrating that the TiO_x coating is stable in H_2SO_4 at high polarization potentials and protected the stainless steel substrate from corrosion.



Elemental mapping of TM (green) and O (blue) shows an oxygen-enriched amorphous film ~10nm thick at surface of coating.

Fig. 1. Series of electron microscopy images showing (top) columnar morphology of TiO_x coating and surface porosity and (bottom) titanium (green) and oxygen (blue) overlaid elemental map showing thin O-enriched surface layer on coating.

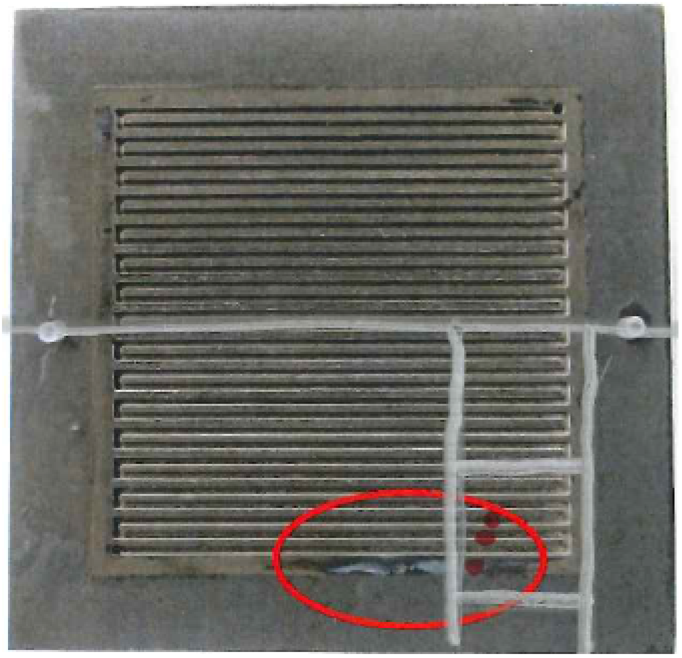


Fig. 2. TiO_x coating on metal bipolar plate after fuel cell testing at LANL.

TiO_x coated metallic bipolar plates after *in situ* drive-cycle tests (LANL) are shown in Figure 2 (note that areas marked were analyzed). Post-AST analyses of the surface chemistry and morphology of the TiO_x coatings by XPS and SEM (results not shown here) showed little change in the surface of the coatings after relatively harsh fuel cell testing.

1.3 IMPACTS

The fundamental investigations conducted as part of this SBV project into assessing the durability of coatings on metallic bipolar plates (chemistry and microstructure) will benefit the low-cost metal bipolar plate manufacture process and quality control system development. It will also enable the application of this technology in other fields, such as electrolyzers and batteries.

1.4 CONCLUSIONS

Work at ORNL was focused on microstructural and microchemical analyses of TiO_x-coated metal bipolar plates produced by TreadStone Technologies. ORNL partnered with Los Alamos National Laboratory and TreadStone to evaluate the stability and durability of the novel coating technology (doped TiO_x) after exposure to the harsh environments in an operating fuel cell. Post-test analyses showed that the composition and morphology of the TiO_x coating were not changed and provided excellent protection for the metal plates with no evidence for increased interfacial contact resistance. These preliminary analyses (before and after testing at LANL) provided some necessary data regarding stability of the coated plates, but additional work will be required to fully assess any degradation mechanisms and to further optimize the coatings.

2. PARTNER BACKGROUND

TreadStone Technologies is a small business corporation located in Princeton, NJ. TreadStone has developed an innovative, low-cost coating technology and processing methods that protects metal substrates used in electrochemical energy systems from corrosion while remaining electrically conductive. TreadStone owns the core proprietary technology (18 patents and patent applications) that is used on a range of electrochemical systems for energy generation and storage. Our technology is commercially proven and in use in electrochemical systems critical to the energy economy, including fuel cells and electrolyzers. Additional applications are being tested including flow batteries, Li-batteries, electrochemical compressors, and other systems.