Point-Of-Manufacturing Microwave Plasma Jet Material Coatings

Control Number: 1465-1578, Emerging Research Award DE-EE0008319

“Atmospheric Cold Plasma Jet Coating and Surface Treatment for Improved Adhesive Bonding Performance of Dissimilar Material Joints Subject to Harsh Environmental Exposure”

Starfire Industries LLC, General Motors LLC, University of Illinois
Project period: Phase 1, October 2018 – September 2019

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Presenter: Ivan Shchelkanov PhD, Starfire Industries LLC

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Overview

Project Title:
Atmospheric Cold Plasma Jet Coating and Surface Treatment for Improved Adhesive Bonding Performance of Dissimilar Material Joints Subject to Harsh Environmental Exposure

Timeline
Project Start Date: 01/10/2018
Budget Period End Date: 30/09/2019
Project End Date: 30/09/2020

Barriers and Challenges
- Obtain extended plasma jet for 3D printed parts processing
- Eliminate bulky waveguides and Magnetrons
- Obtain good process repeatability and scalability

AMO MYPP Connection:
Technical Area 4: Materials for Harsh Service Conditions
Technical Target 4.3: Achieve performance-based cost parity for the manufacture of alternate materials and parts for use in harsh service conditions.

Project Budget and Costs:

<table>
<thead>
<tr>
<th></th>
<th>DOE Share</th>
<th>Cost Share</th>
<th>Total</th>
<th>Cost share %</th>
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</thead>
<tbody>
<tr>
<td>Overall budget</td>
<td>$0.8M</td>
<td>$0.2M</td>
<td>$1M</td>
<td>20%</td>
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<tr>
<td>Approved Budget (BP – 1&amp;2)</td>
<td>$0.8M</td>
<td>$0.2M</td>
<td>$1M</td>
<td>20%</td>
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<tr>
<td>Costs as of 3/31/19</td>
<td>$181 614</td>
<td>(~20%)</td>
<td>$181 614</td>
<td>(~20%)</td>
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</tbody>
</table>

Project Team and Roles

TPOC: Dr. Robert Stubbers
APOC: Dr Brian Jurczyk

Dr. Blair Carlson - Lead
R&D group, Dissimilar Material Joining, Coatings, Requirements, Costs, Energy saving
- J. Wells - Automatization and Immigration

Dr. Eitan Barlaz – Lead
Prof. Dr. David N. Ruzic – sabbatical
- Director of CPMI

Dr. Daniel Krogstad - Lead
- Environmental Testing lab Manager
- Accelerated Aging Materials Research
Project Objectives

Point of Manufacturing **Anti-Corrosion Conversion Coating**

| Coat Only Bonding/Joining Surfaces Instead Of Entire Part | Treat Complex 3D Parts/Surfaces With Extended Plasma Jet |

Achieve High-Strength, **Long-Life Joints**

| Maintain Minimum 80% Lap-Shear Strength For Epoxy Joining | Resist Corrosion Under Harsh Environment Accelerated Life Conditions |

Eliminate Wet Chemistry Steps

| Eliminate Off-Site Transport, Energy Use & Waste Water Effluent | Reduce Cost By $100-300/Vehicle For Wet Chemistry |

Enable Vehicle Lightweighting

| Enable Dissimilar Material Joining (e.g. Al/Mg/CFRP) & New Combinations | Spillover Energy Reduction Effects For Lighter Weight Vehicles |
The conventional approach is to use wet chemical treatments (surface clean, etching, conversion coatings) to achieve a material transformation/deposition to resist corrosion on vehicle parts

- Parts are totally treated by wet chemistry even though only a few % of the surface area is used for joining
- For epoxy-jointed surfaces of dissimilar materials, the joining surface quality and reliability is paramount

The near-term state of the art is to use laser ablation for aluminum to generate a native, deep surface oxide that resists corrosion and provides excellent surface adhesion for bonding

- Performed immediately prior to joining to limit surface contamination to build on the opposing mating surfaces
- Laser oxide formation is only good for aluminum and is suitable for flat, planar or easily accessible surface parts (not 3D printed, lightweight shapes)- Can not be used on Carbon, and low temperature ignition point materials

Impact is significant

- Eliminates transport, fuel consumption, bulk chemical use, waste water treatment, and lowers energy/emissions
- 33% reduction in cost compared to near-term laser ablation methods
- Single treatment process for Al, Mg, CFRP @ point of manufacturing; inhibitor for carbon fiber galvanic corrosion
Plasma-based techniques use non-equilibrium chemistry for surface cleaning (e.g. the DC gliding arc & dielectric barrier discharge) prior to adhesive bonding

- The DC gliding arc is limited to downstream chemical precursor injection with a mm-scale plasma zone due to very high gas temperature & turbulent flow
- Dielectric barrier discharges are power density limited and require bulky support hardware
- Industrial scale systems need multiple source heads and treat planar surfaces only

Starfire’s Innovation:

- Miniature solid-state power amplifiers using latest high-electron mobility transistors generate microwave energy directly at the coaxial plasma applicator allowing efficiency and small size
- Zonal shield gas, process gas and centerline material delivery enables novel applications using chemical precursors and direct physical sputtering of an electrode at atmospheric pressure
- Simultaneous surface cleaning, radical/etch, material deposition and reactive plasma chemistry with shielded contaminant protection

Atmosphere Plasma Processing

1. Turn the applicator ON
2. Process only required area
3. Turn the applicator OFF

Wet Chemistry Processing

1. Prepare the bath where to dip the part
2. Get Chemicals
3. Dip the whole part into Chemicals
4. Clean the processed part
5. Deal with WASTE

To replace
Develop industry compatible plasma applicator

- Demonstrate compact microwave plasma generator with integrated solid-state power amplifier in small form and flexible power delivery to plasma applicator
- Demonstrate streamline flow for extended plasma jet and extended reach for material coating for complex parts and 3D surfaces

Demonstrate plasma coating recipe stable against minor process variations

- Use chemical precursor delivered silane/siloxane chemistries for surface cleaning, coating and sealing
  - Evaluate potential for alumina/zirconia thin-film chemistries using hybrid sputtering or chemical precursor delivery

Experiments on Material Coatings are set in motion

- Perform surface analysis screening tests for initial recipes, upgrade to tactical wet exposure lap-shear strength evaluations and down-selected strategic corrosion testing analysis on ideal material coating

Advanced Implementation Readiness

- Demonstrate implementation readiness with field demonstration at General Motors in 2nd Phase
- Leverage University-Industry Collaboration
Results & Accomplishments

Project Status

- Small plasma applicator and power supply development
  ◦ Completed

- Champion process parameters for required tactical tests
  ◦ Found and tested

- Coating process variability
  ◦ Under investigation.

- Integration requirements for GM factory
  ◦ Initial evaluation in progress

Required Future Work

- De-risking, engineering design, R&D evaluation

- Evaluation of native oxide effect, environment conditions, handling requirements

<table>
<thead>
<tr>
<th>Schedule</th>
<th>Q1-2 (Completed)</th>
<th>Q3-4 (Undergoing)</th>
<th>Q5-6 (Planned)</th>
<th>Q7-8 (Planed)</th>
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<tbody>
<tr>
<td>Microwave Plasma Applicator</td>
<td>Requirements Scoping Study EM Design (Completed)</td>
<td>Compact Plasma Applicator Design (Completed)</td>
<td>Compact Plasma Applicator</td>
<td>Demo @ GM</td>
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<td>Materials DoE</td>
<td>SiOx Coatings (Completed)</td>
<td>Al/Zr Coatings (In progress)</td>
<td>Selected Recipe optimization and validation</td>
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<td>Material Eval</td>
<td>Basic Material Screening (Completed)</td>
<td>Tactical Corrosion Testing (In progress)</td>
<td>Strategic Corrosion Testing</td>
<td>Demo @ GM</td>
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<tr>
<td>Go/No-Go</td>
<td>&gt;80%Lap Shear 2L, 5kg, 200W</td>
<td></td>
<td></td>
<td>Transition</td>
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</tbody>
</table>

After High Temperature Water Soak
Even a small variation in process affects final performance more than it is allowed

- Not treated _ Accelerated exposure
- Champion recipe _ Plasma treated _ Accelerated exposure
- Plasma treated _ No environmental exposure
- No treatment

Lap Shear strength [MPa]
Immediate transition plan is for in-line vehicle manufacturing with Partner General Motors
  ◦ Interface with robotic arm delivery system

Secondary market opportunities for transition through NSF member companies in I/UCRC Lasers and Plasmas For Advanced Manufacturing
  ◦ Trinity Industries (Rail Car, Barge, Wind Towers)
  ◦ National Oilwell Varco (Oil & Gas Infrastructure)
  ◦ Lockheed Martin (Aerospace Components)

The discussion to transition away from “wet chemistry” to “dry chemistry” is underway in several markets