2012 DOE Vehicle Technologies Program Review

AMD 704 Development of Steel Fastener Nano-Ceramic Coatings for Corrosion Protection of Magnesium Parts

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Automotive Metals Division

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Overview

Timeline

- Start - October, 2007
- Finish - September 30, 2011
- 100% Complete

Budget

• Total project funding -	$884,000
– DOE share -	$442,000
– Contractor share -	$442,000
• Funding received in FY11 -	$129,000
• Funding for FY12 -	$ 30,000

Barriers

Barriers

A. Performance

B. Cost

C. Joining and Assembly

Targets

- Less than -1.2 mA/cm^2 galvanic current between nano-ceramic coated steel and magnesium
- Coated steel fasteners generate less magnesium mass loss than aluminum fasteners in cyclic salt spray testing

Partners

- USAMP (Chrysler, Ford and General Motors)
- Alfred University , Visteon, General Fasteners, KAMAX, NIST

Relevance

Goal

- To facilitate the use of light weight magnesium in automotive applications by developing and validating nano-ceramic coatings for steel fasteners that will inhibit galvanic corrosion between the fasteners (cathode) and magnesium (Mg) components (anode).
  - This project is considered an enabling project to facilitate vehicle lightweighting through increased use of magnesium by addressing galvanic corrosion in dissimilar metal joints, one of the fundamental barriers to the use of magnesium as cited in the "Magnesium Vision 2020" USCAR publication.

Objectives

- Develop a nano-ceramic coating for steel fasteners with equivalent or better performance than aluminum fasteners with respect to galvanic corrosion.
- Determine if the Galvanic Current Measurement Test is a good predictor of galvanic corrosion performance.

Expected Result

- Successful development of nano-ceramic coatings will enable improved fuel economy through vehicle lightweighting by providing a low cost solution for joining magnesium with steel fasteners.

Transfer case exhibiting perforation and oil leak without effective isolation

Milestones

Month-Year	Milestone		Deliverable
Jan. ’11	1	Design of Experiments to determine the nano-ceramic coating materials and parameters that best isolate steel from magnesium.	DOE Study
Mar. ’11	2	Nano-ceramic coated steel substrates have a galvanic current equal to or less than aluminum when coupled with magnesium in salt water (Galvanic Current Measurement Test).	An effective Nano-ceramic coating (materials/thickness) that reduces or eliminates galvanic current between steel and magnesium
Sept ’11	3	When placed in contact with magnesium, nano-ceramic coated fasteners induce less magnesium mass loss than aluminum fasteners during cyclic salt spray testing	Effective Nano-ceramic coated fasteners that eliminates or significantly reduces galvanic corrosion with magnesium

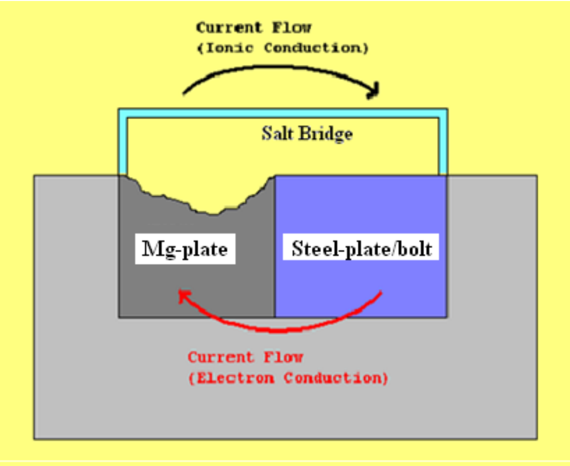
Approach / Strategy

The most common means of attaching magnesium components to automotive steel structures is to isolate the steel from magnesium through use of aluminum washers, spacers and/or fasteners.

- The difference in electrochemical potential is less between aluminum and magnesium than it is for steel and magnesium, thereby generating less driving force (voltage) for galvanic corrosion.
- Nano-ceramic coatings may be considered a viable alternative to aluminum if the coatings can better isolate steel from magnesium and generate less corrosion than aluminum.

Note: There is no industry accepted test for evaluating galvanic corrosion. This project selected two tests and used uncoated steel as the base line and aluminum as the target.

- The galvanic current measurement test: This test would assess the ability of the nano-ceramic coatings to isolate steel from magnesium.
- Cyclic salt spray testing (VDA): The VDA test has been developed in Europe and shows promise in evaluating galvanic corrosion in dissimilar metal joints.

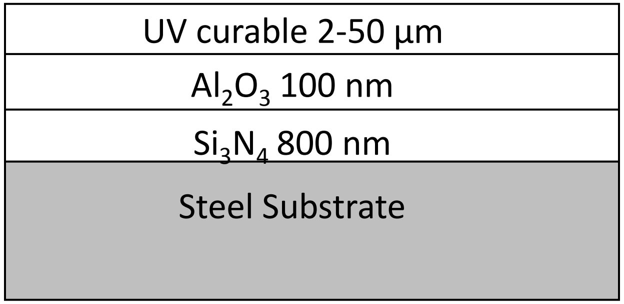


Approach / Strategy

The project established a two phase approach.

- Task 1: Apply nano-ceramic coatings to steel coupons, evaluate the galvanic current between the coated steel coupon and magnesium and compare results against aluminum coupled with magnesium.
  - Decision Gate: If the nano-ceramic coated steel coupons exhibited less galvanic corrosion than aluminum then Task 2 can begin.

- Task 2: Apply nano-ceramic coatings to steel fasteners, evaluate the galvanic and general corrosion of the coated steel fasteners against magnesium using cyclic salt spray testing, and compare results against aluminum fasteners mounted against magnesium.
  - Decision Gate: The program is successful if the nano-ceramic coated fasteners induces less corrosion than aluminum fasteners.



Multi-layer Coating Thickness

Technical Accomplishments and Progress

Task 1.1: Design of Experiments

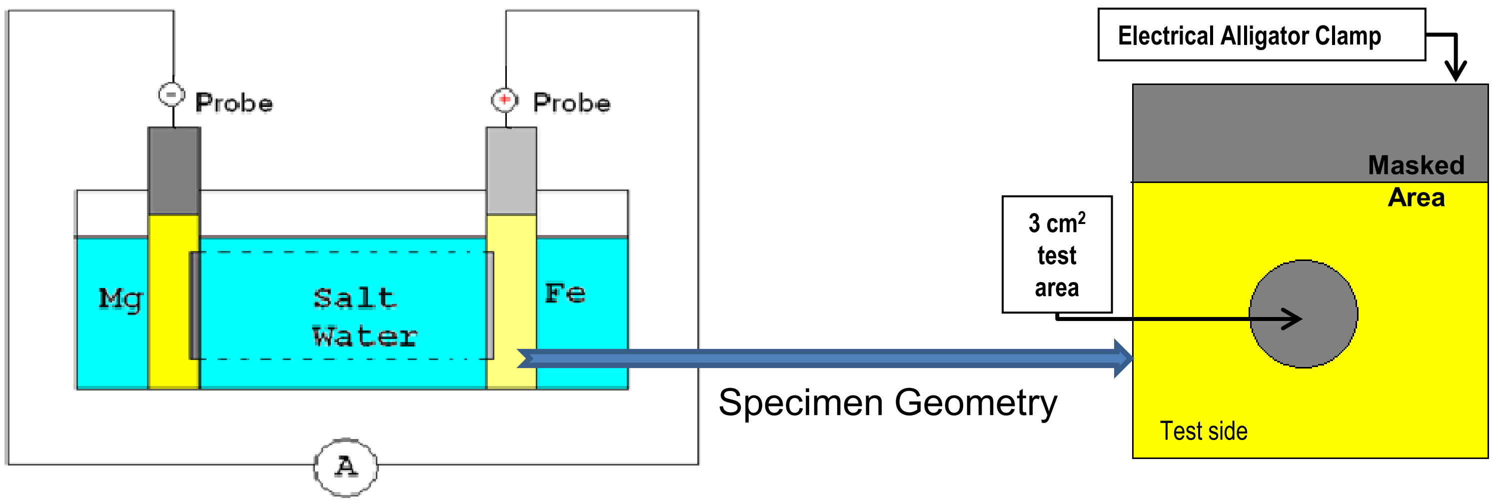
- A DOE was performed using galvanic current measurements for single layer and multilayers of SiN, AlO and UV curable AlO in different combinations (A, B, C, AB, AC, BC, ABC)
  - The DOE revealed that the SiN nano-ceramic coating had the most impact on galvanic corrosion.
- Multilayer coatings could have an additional benefit on general corrosion.
- The team concluded that multilayer coatings using SiN as the base coat showed the most promise.

Milestone 1 Achieved: DOE completed.

Technical Accomplishments and Progress

Tasks 1.2 – 1.5: Galvanic Current Measurement Testing

- The galvanic current measurement test measures the current between dissimilar metals placed into a fixture and selectively exposed to a 3.5% sodium chloride solution.
  - The samples are masked to expose an area of 3 cm^2 on each side of the galvanic couple.
  - A saturated calomel electrode (SCE) is utilized as the standard reference electrode (+0.241 V vs. standard hydrogen electrode (SHE).
  - The fixture holds the two samples at opposite ends of a 6.7 cm long PVC tube containing the salt solution.
  - Electrical contacts are connected to the samples and to a zero resistance ammeter.
  - Two measurements with nominally identical samples are conducted. Each measurement lasts for 30 minutes (1800 seconds).



Technical Accomplishments and Progress

Task 1.2 – 1.5: Galvanic Current Measurement Testing

- Average Galvanic Corrosion Current
  - 1050 Steel (control) = **8.83 mA/cm²**
  - 6061 Aluminum (Target) = **1.26 mA/cm²**
  - Single layer SiN nano-ceramic coating = **0.79 mA/cm²**
  - Multilayer nano-ceramic coating of SiN-AlO-UVAlO = **0.55 mA/cm²**

- Single layer SiN exceeded aluminum target corrosion current

- Multi-layer nano-ceramic coating improved upon single layer SiN coating.

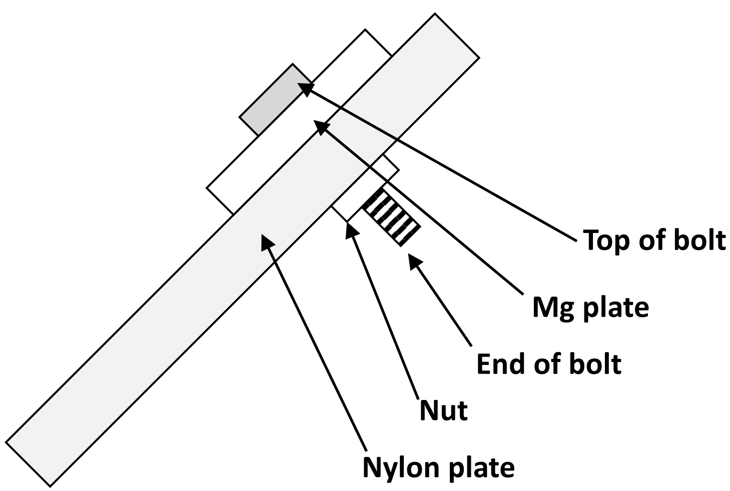
- **Milestone 2 Achieved: Nano-ceramic coated steel induced less galvanic current than aluminum.**

Sample	Average Corrosion Current (mA/cm^2)
Control-1 (1050 steel)	8.83
SiN 1	0.79
AlO-1	8.12
UVAlO-1	8.05
AlO-UVAlO-1	6.95
SiN-UVAlO-1	1.10
SiN-AlO-UVAlO-2	0.55
Al-Mg-1 (Target)	1.26

Technical Accomplishments and Progress

Tasks 2.1 -2.2: VDA Testing

- The VDA is essentially salt spray testing using a fixturing as shown below.
- Fasteners are coated and assembled into fixtures.
- The sample is inclined during testing
- Samples are removed daily, photographed and measured for mass loss/gain.



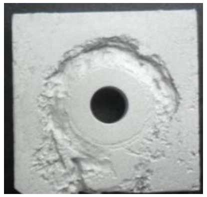
Side of substrate holder (inclined 45 degrees)

Salt Spray Chamber

Technical Accomplishments and Progress

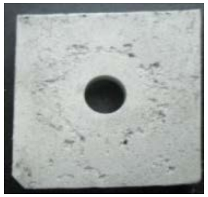
Task 2.1 – 2.2: Modified VDA Test Results

Steel fastener uncoated (Control): Day 0



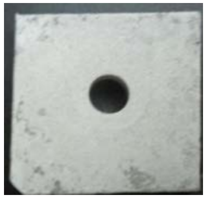
Steel fastener uncoated (Control): Day 3 **3.32 % mass loss**

Fastener removed and Mg plate sandblasted



6086 Aluminum fastener (Target): Day 3 **0.65 % mass loss**

Fastener removed and Mg plate sandblasted



Multilayer nano-ceramic coated fastener: Day 3 **0.08 % mass loss**

Fastener removed and Mg plate sandblasted

Milestone 3 Achieved: Nano-ceramic coated fasteners induced less corrosion in the magnesium than the aluminum fasteners.

Collaboration

- Project Team:
  - USAMP (Chrysler, Ford, GM)
  - USCAR Fastening Task Team - Provided guidance on testing and qualifying automotive fastener finishes
  - Alfred University – Prime contractor which performed mechanical properties and corrosion testing
  - General Fasteners: Technical advisor KAMAX provided fasteners for coating and testing
  - Materials Technologies – Project management
  - McCune and Associates – Technical Advisor
  - NIST - Coated steel samples and fasteners
  - Visteon: Technical advisor

Proposed Future Work

- This project demonstrated the concept feasibility of nano-ceramic coatings for use on steel fasteners as a means of mitigating galvanic corrosion between magnesium and steel.

- Future work would be to demonstrate the technical feasibility of using nano-ceramic coated steel fasteners on magnesium substructures.
  - Establish the ability of nano-ceramic coatings to achieve reliable torque/tension requirements.
  - Evaluate residual torque of assembled joints with nano-ceramic coated fasteners
  - Determine if nano-ceramic coatings have sufficient durability to resist damage induced by handling and assembly as well as assembly stress
  - Evaluate the general corrosion performance of nano-ceramic coated fasteners

- Alfred University submitted a DOE proposal on December 7, 2011 in response to FOA 560 to evaluate the technical feasibility of using nano-ceramic coatings for fasteners.

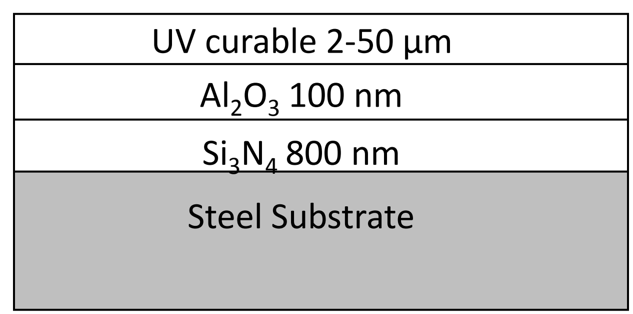
Summary

Project Summary:

- The Design of Experiments showed that the SiN nano-ceramic coating has the most significant impact on galvanic current and was not overly sensitive to coating layer thickness. Tested multilayer coating thickness is shown below.
- Multi-layer nano-ceramic coated steel exhibited less average galvanic current (0.55 mA/cm^2) than 6061 aluminum (1.26 mA/cm^2) when coupled with magnesium in salt water.
- Multi-layer nano-ceramic coated steel fasteners induced less galvanic corrosion in magnesium than aluminum, 0.08 % versus 0.65% respectively, after three days of cyclic salt spray testing.

Next Steps

- Issue Final Report
- Technology Transfer

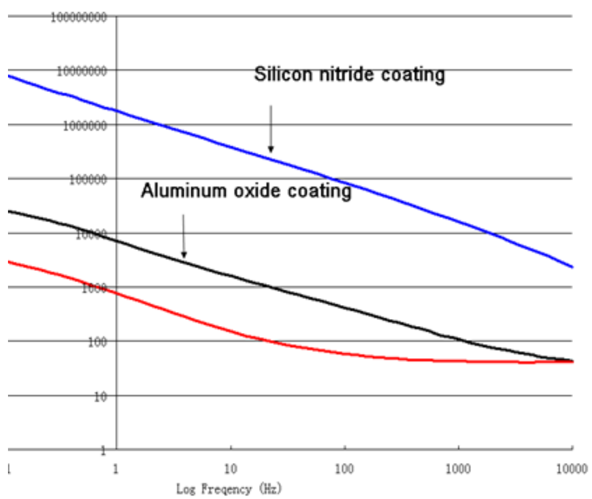


Multi-layer Coating Thickness

Technical Back-Up Slides

Experiments - Thin film coatings

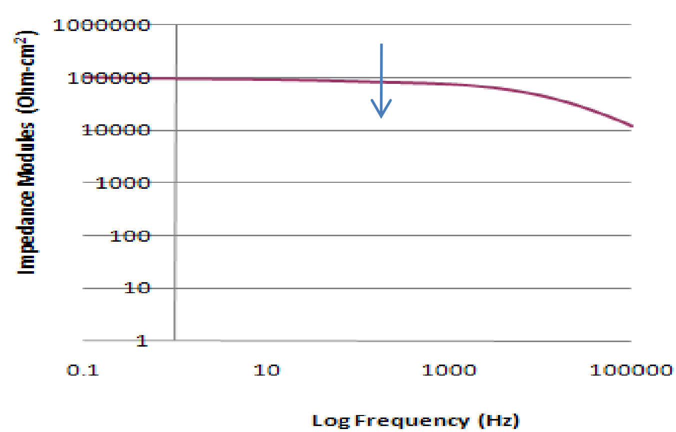
- Silicon Nitride
  - Techniques: PECVD / ALD
  - Thickness: 300 - 850 nm /20-60 nm
- Aluminum Oxide
  - Techniques: E-beam
  - Thickness: 100 - 700 nm
- UV-curable materials
  - Techniques: UV-light
  - Thickness: depending on the method of curing



- Back ground:
  Thin film coating materials on flat surface
  - Previous study (on a flat steel plate):

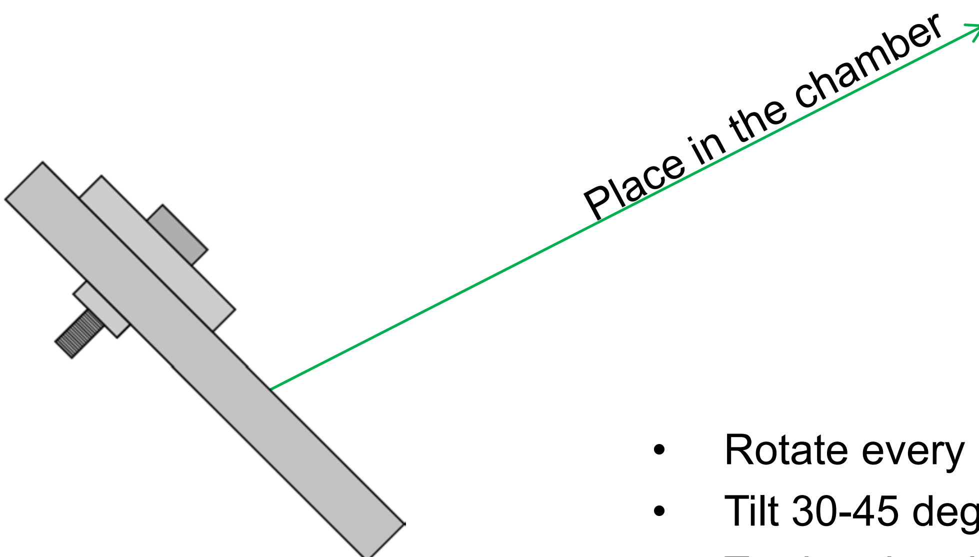
  Impedance module:
  SiN>UVCeO>AlO>control
  - Thin film coating applied on bolts (This work)

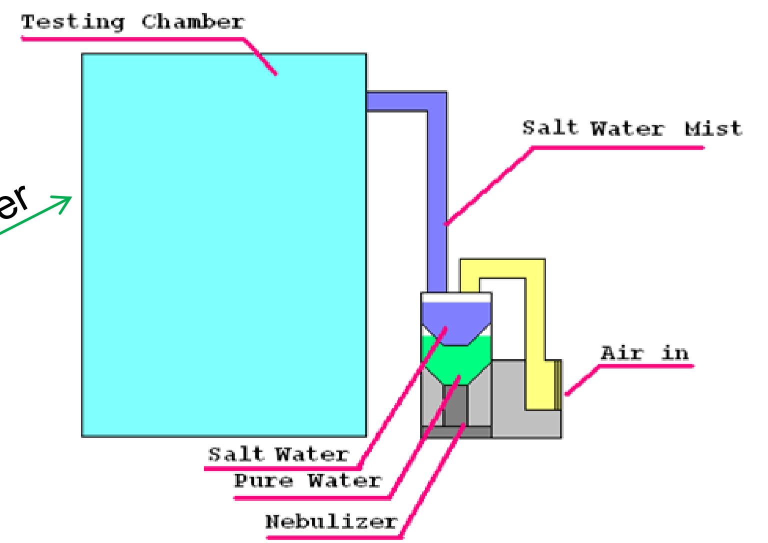
UV-curable Cerium Oxide



Corrosion test on bolt

- Weather chamber
  - Salt Water (50 g NaCl, 1000 g distilled water)
  - Feed salt mist 15 minutes every one hour





Sample

- Rotate every 24 hours
- Tilt 30-45 degree
- Testing duration:
  - 11~12 days
  - Corrosion product is built to its “maximum” stage for one of the samples
- Photo record everyday

Example Modified VDA Test Result:

- Under bolt head: area A
- Galvanic corrosion: area B
- General corrosion: area C
  - Estimated depth of corrosion ring: 2mm (area B)
  - Estimated width of corrosion ring: 2.5mm (area B)
  - 0.334g (Estimated from the galvanic corrosion ring)
  - 0.318g (from measurement)