Large Scale Testing, Demonstration, and Commercialization of Fuel Cell Coolant

(SBIR Phase III)

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FC077

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Overview

SBIR Phase III: Large Scale Testing, Demonstration, & Commercialization of Fuel Cell Coolant

Total Project Funding	DOE Share: $1.016 M Planned FY13: 216,311
Timeline	Project start date : Oct 1st, 2010 Project end date: Sep 30st, 2013 80% Complete
Barriers Addressed	System Thermal Management Durability, Cost, & Performance
Collaborations	Protonex Technology Corporation University of Tennessee, Knoxville More than 15 Fuel Cell Companies

Relevance: Coolant Durability

DOE SBIR Phase III Project

DOE Barriers	Dynalene Inc.
A. Durability	• Understand the coolant durability requirements by working with different fuel cell types developed by various fuel cell manufacturers • **Coolant Durability:** Maintains low electrical conductivity and other thermo-physical properties for over 5,000 operating hours • **Coolant Compatibility:** Acceptable to be used with typical fuel cell components

Relevance: Coolant Cost

DOE SBIR Phase III Project

DOE Barriers	Dynalene Inc.
B. Cost	• Understand what the cost of coolant should be relative to current automotive coolants

Projected Selling Price of Coolant as Production Scaled Up	
Small Scale (55 gal batches)	$45/gal
Medium Scale (1,000 gal batches)	$30/gal
Pilot Scale (5,000 gal batches)	$15-25/gal
Full Scale (>100,000 gal/year)	$10-15/gal

Relevance: Coolant Performance

DOE SBIR Phase III Project

DOE Barriers | **Dynalene Inc.**

C. Performance

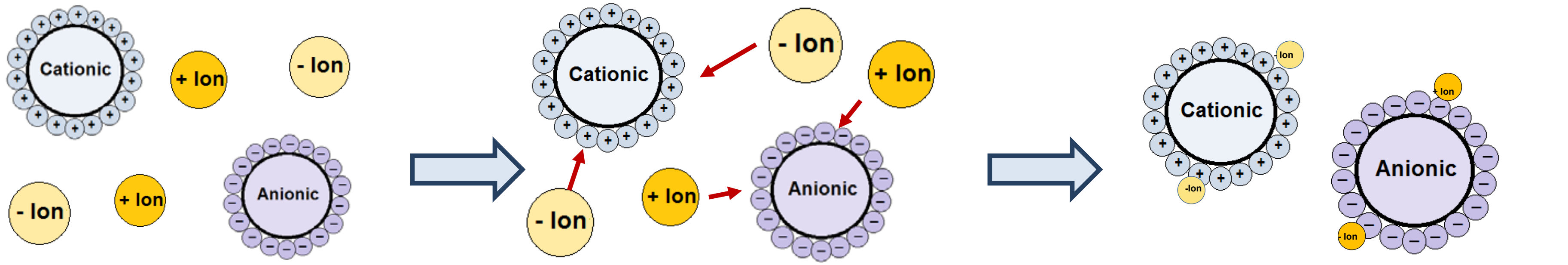
Coolant Criterion	Description	Value
Electrical Conductivity	Electrically conductive coolants reduce the performance of a fuel cell	< 2 µS/cm
Boiling Point	Similar to current automotive coolants	> 100°C
Freezing Point	Must be freeze-tolerant under extremely cold conditions	< -40°C
Thermal Conductivity	Higher is better for heat transfer	> 0.4 W/mK
Viscosity	Lower is better for heat transfer and pumping	< 1 cP at 80°C
Specific Heat	Higher is better for heat transfer	> 3 kJ/kgK
Cost/Toxicity/Flammability	Current automotive coolant	Similar

Approach: Coolant Characteristics

	Dynalene FC	Dynalene LC
Composition	• Glycol-water base fluid • Nanoparticle & non-ionic corrosion inhibitor additive package	• Glycol-water base fluid • Non-ionic corrosion inhibitor package (no nanoparticles)
Performance	• Both fluids are non-flammable and non-toxic • Physical and thermo-physical properties similar to typical automotive coolants • Superior heat transfer performance compared to non-aqueous fluids	
Low Electrical Conductivity	Low electrical conductivity maintained by two mechanisms: 1. **Nanoparticles:** Ion-scavenging in the working coolant 2. **Non-Ionic Corrosion Inhibitors:** Ion-suppression at the wall of the coolant channel	Low electrical conductivity maintained by one mechanism: 1. **Non-Ionic Corrosion Inhibitors:** Ion-suppression at the wall of the coolant channel

How do Dynalene FC & LC Work?

The Nanoparticles (for Dynalene FC)

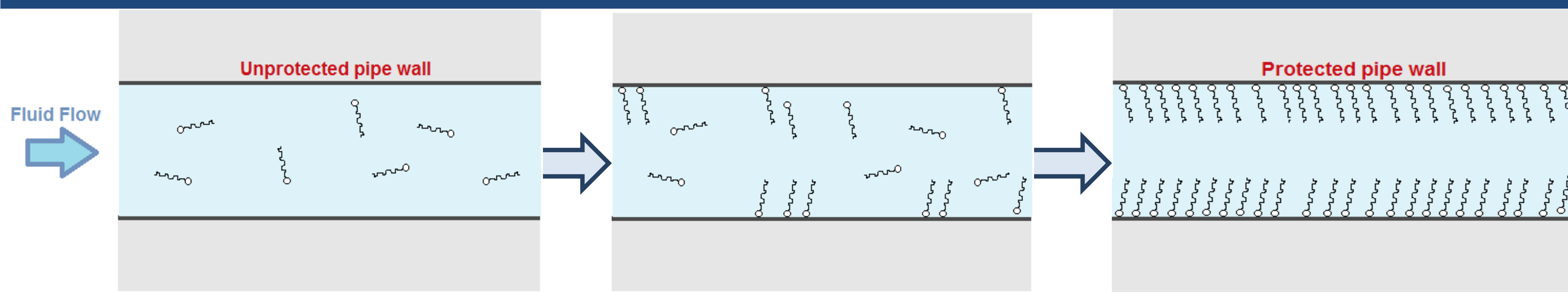


Free ions and nanoparticles in solution

Free ions attracted to + and - charges of the cationic and anionic nanoparticles

Nanoparticles finally scavenge and neutralize free ions

The Inhibitor (for Dynalene FC & LC)

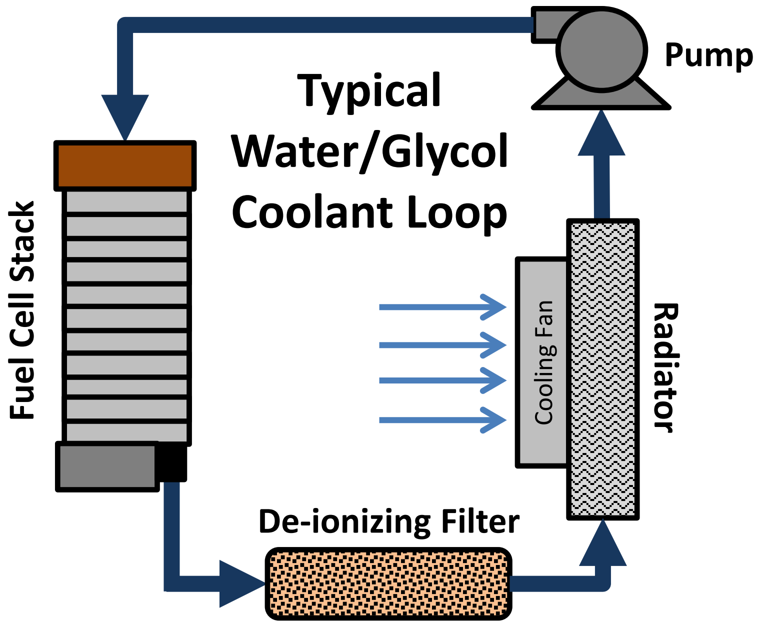


Non-ionic inhibitors circulating through pipe in Dynalene FC/LC

Inhibitor heads begin to bond to surface on pipe wall

Inhibitor fully protects wall from corrosive attack

Approach: Dynalene FC/LC Advantages in PEM Fuel Cell



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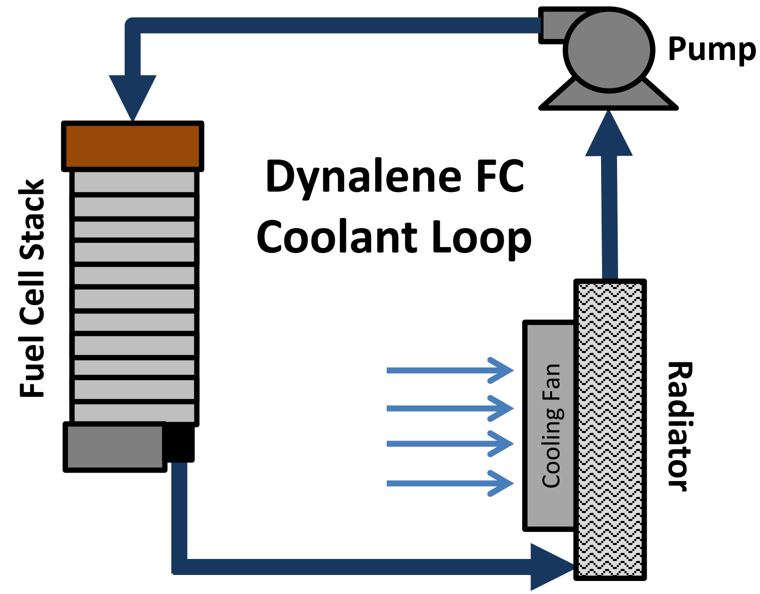


Fuel Cell Criteria	Water/Glycol Loop	Dynalene FC Loop	Dynalene LC Loop
De-ionizing Filter	Required	**Not required**	**Optional**
Pressure Drop	Higher	**Lowest**	**Lower**
Maintenance	Higher	**Lowest**	**Lower**
Clogging	Higher	**Lowest**	**Lower**
Weight	Heavier	**Lightest**	**Lighter**
Corrosion	More likely	**Less likely**	**Less likely**

Approach: Background

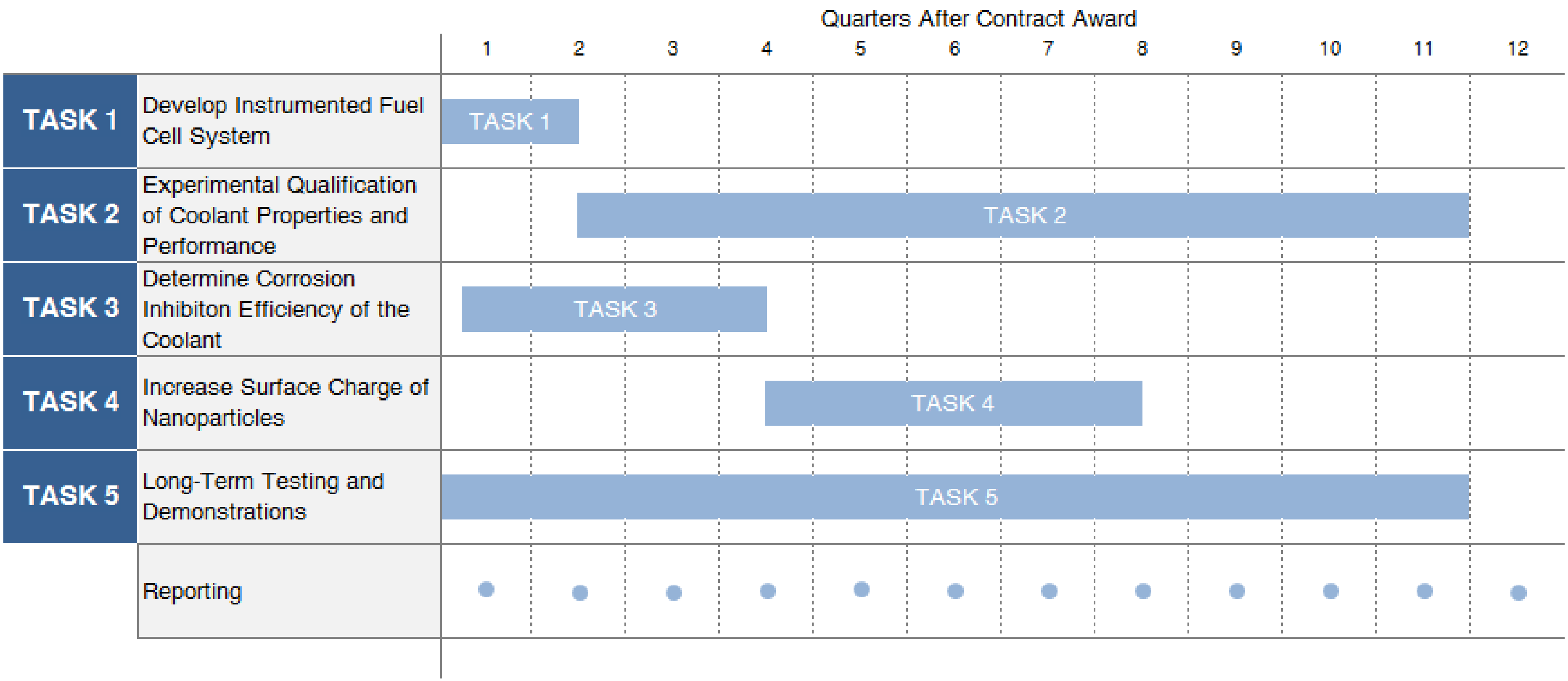
- **Fuel cell coolant development began in 2000 and was patented in 2006**
- **Expectations**
  - Low electrical conductivity over 5,000 hours of operation
  - Exceptional thermo-physical properties – similar to current automotive coolant
- **SBIR Phase I and Phase II (2004 - 2007)**
  - Provided a good recipe for a coolant fluid that demonstrated low electrical conductivity in short term tests.
- **DOE Scale-up Grant (2009 - 2011)**
  - Provided the ability to manufacture Dynalene FC in batches of 5,000 gallons.
- **SBIR Phase III (2010-2013)**
  - 5,000 hour validation of coolant in working fuel cell systems
  - Validating the efficiency of the corrosion inhibition abilities of the Dynalene FC

Approach: Phase III Key Milestones

Timeline	Objective	Status
June 2011	Design and fabricate test equipment (Dynalene, Inc., Protonex, University of Tennessee)	Completed
Sept 2013	Validate corrosion inhibitors in 5,000 hour tests (Dynalene, Inc.)	60% Completed
Sept 2012	Produce nanoparticles with at least 500 μeq/g (Dynalene, Inc.)	Completed
Sept 2013	Experimentally validate 5,000 hour coolant durability (Dynalene, Inc., Protonex, University of Tennessee)	Ongoing

Year 1 Decision Gate	Fabrication of the test equipment (Cleared)
Year 2 Decision Gate	Corrosion inhibitors validated in short term tests and nanoparticles optimized for surface charge

Approach: Tasks & Schedule



Approach: Tasks

Task 1: Development of an Experimental Test Rig to Simulate Ion Contamination in Fuel Cell System (University of Tennessee, Knoxville)

- Perform material compatibility tests to ensure best materials for test system
- Build test rig with coolant loop using FC compatible materials, automotive radiator, and voltage controlled metallic fuel cell coolant channels

Task 2: Investigation of Coolant Performance in Fuel Cell System Environment (University of Tennessee, Knoxville)

- Determine effects of voltage, temperature, time, surfactant, and nanoparticles on performance of coolant
- Investigate non-organic contaminants in tested fluids

Task 3: Determine Corrosion Inhibition Efficiency of Coolant (Dynalene)

- Material compatibility test, immersion test, electrochemical test, and electron microscopy
- Further development of non-ionic corrosion inhibitors
- 5,000 hour testing

Approach: Tasks

Task 4: Increase Surface Charge of the Nanoparticles (Dynalene)

- Target is 500 μeq/g
- Achieved greater than 800 μeq/g

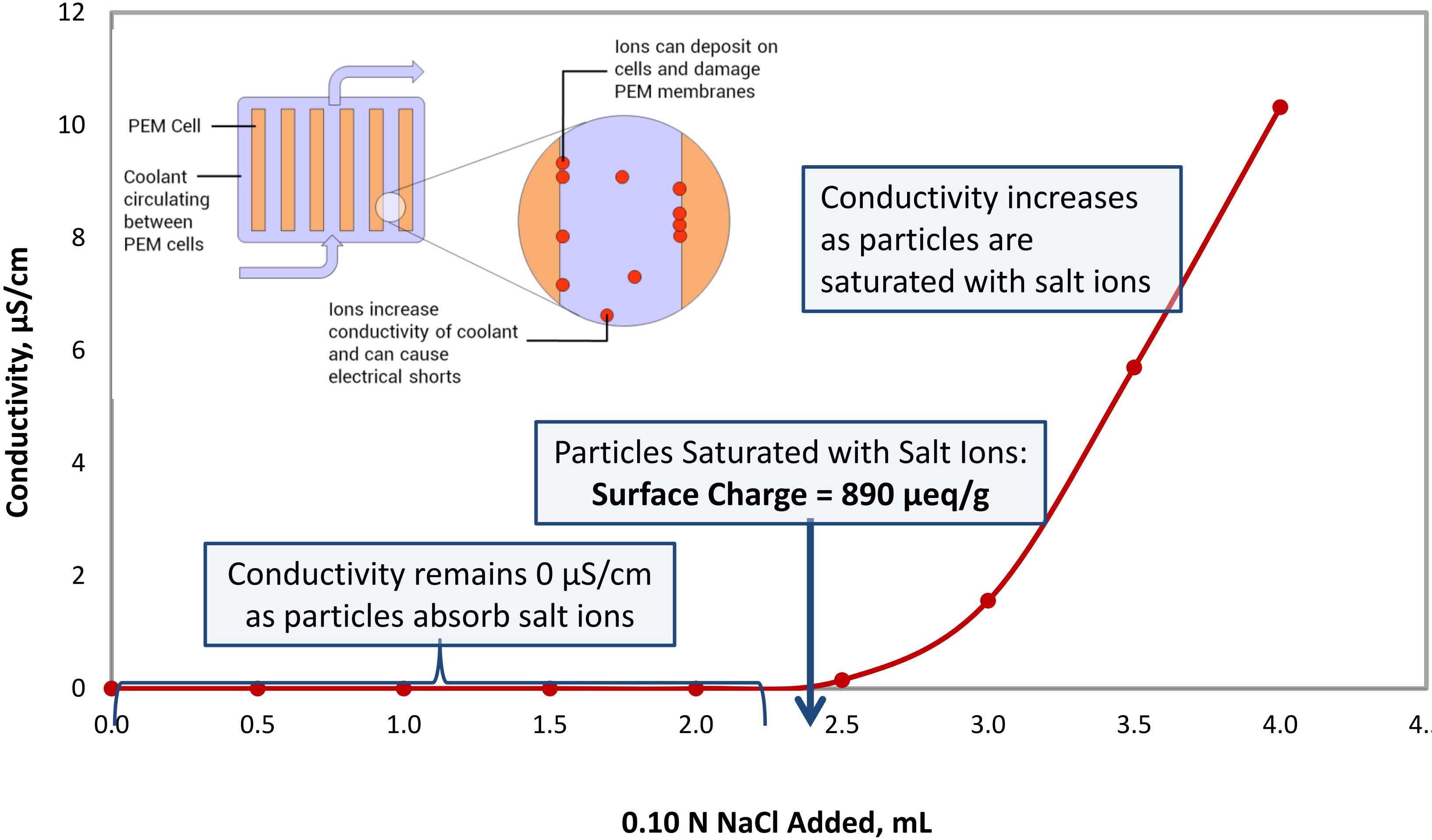
Task 5: Long-Term Testing and Demonstration (Protonex & Dynalene)

- Protonex system at the Dynalene site
- Dynalene will start 5,000 hour testing on selected coolant formulations

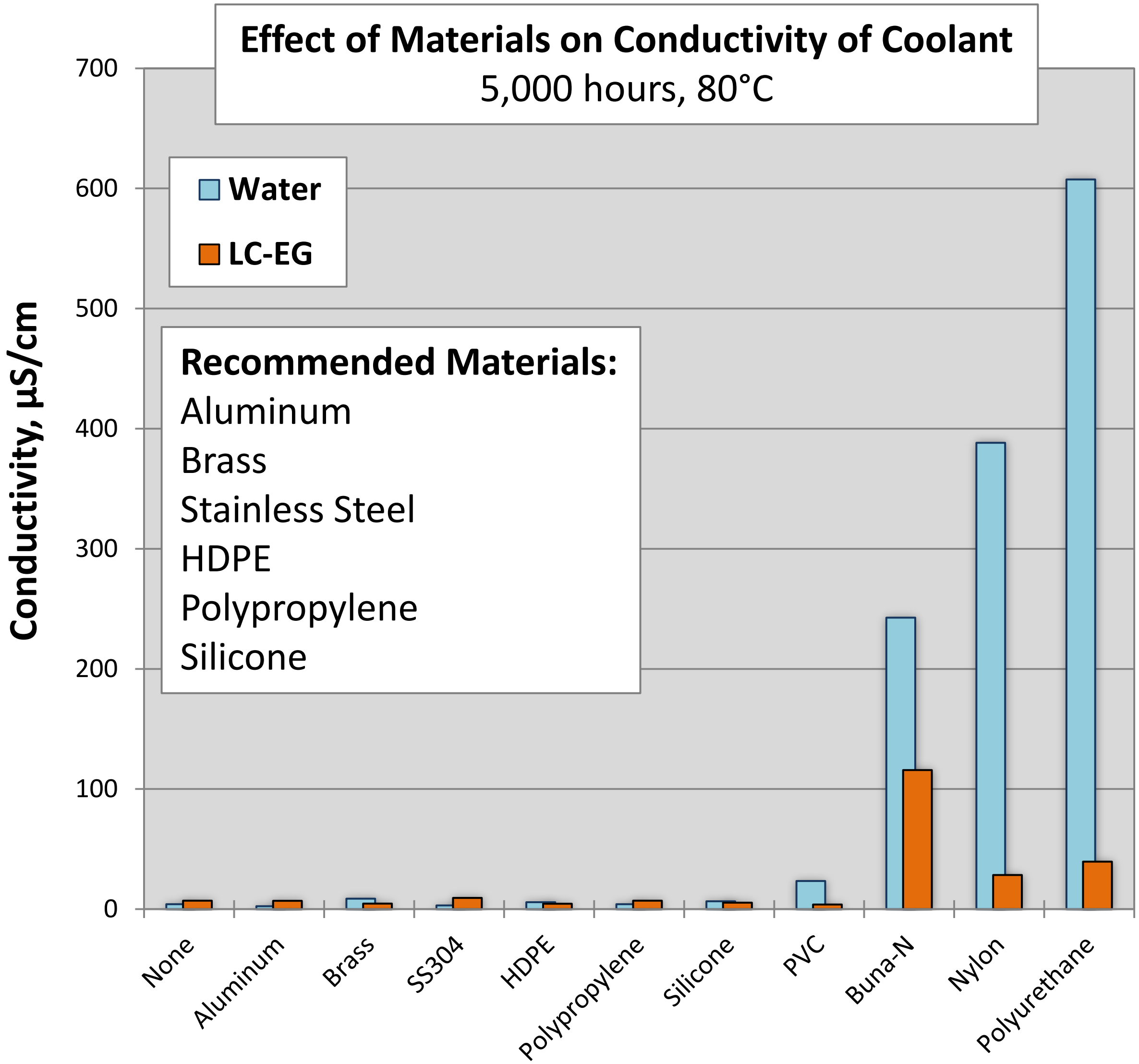
Accomplishments: Surface Charge Increase

Reformulated Particles Dispersed in BioGlycol-Water Mixture

Conductivity Measured as Salt Solution Added (0.09 wt% Particles)



Accomplishments: 5,000 Hour, 80°C Materials Testing



- Materials immersed in LC-EG & water for 5,000 hours at 80°C
- Metals performed significantly better than most polymers
- Polymers containing functionalized groups contributed many more ions than short branched hydrocarbons

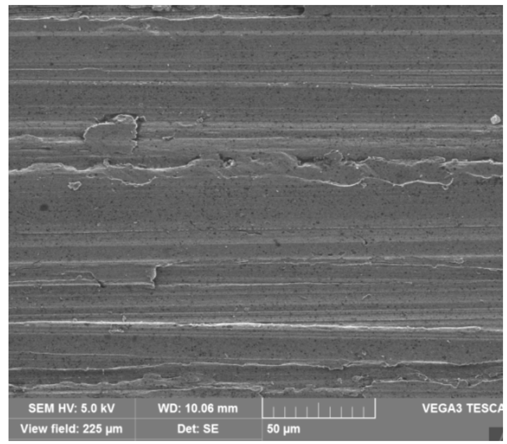
LC-EG: ethylene glycol-based coolant

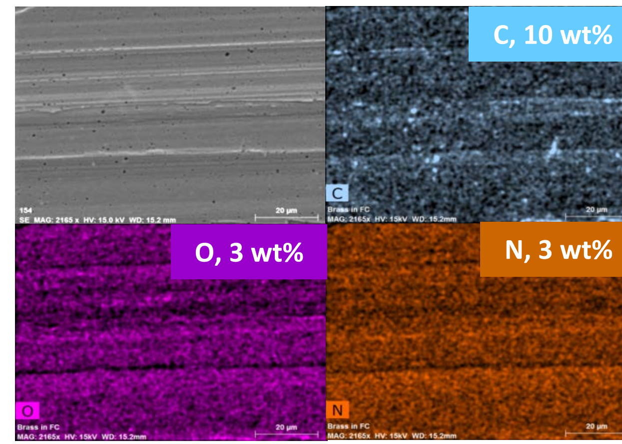
Accomplishments: SEM Analysis of Brass in FC & LC

Brass (CDA 260) Immersed in FC & LC for 2,500 hours at 80°C

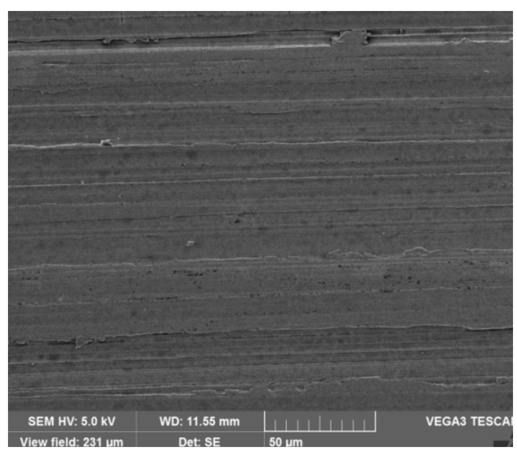
Appearance | **SEM Microscopy** | **EDS Analysis**

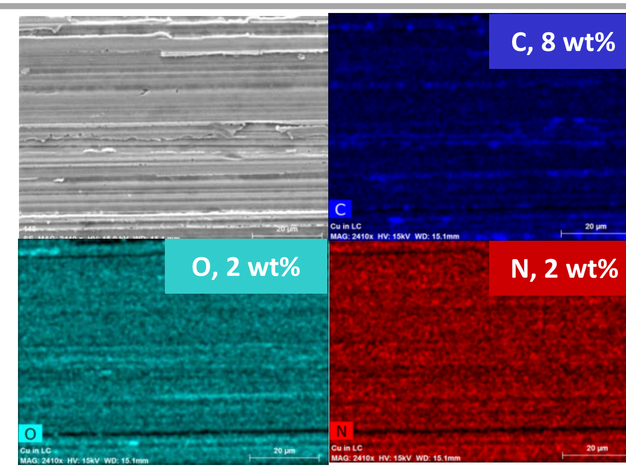
FC





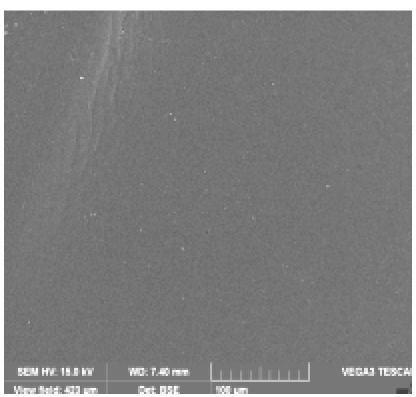
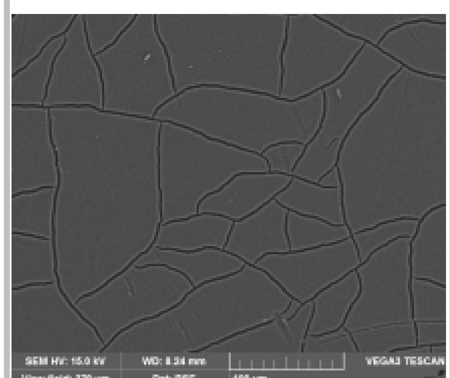
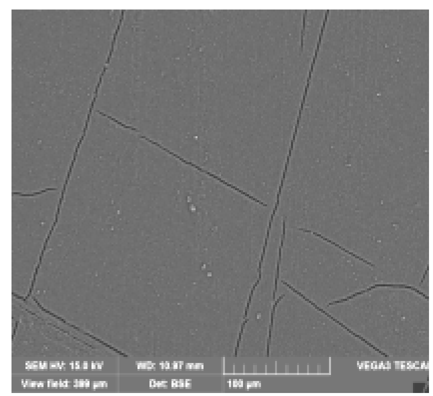
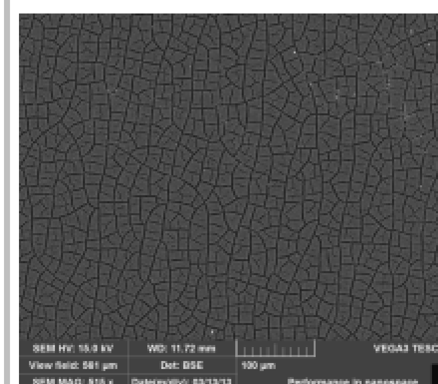
LC





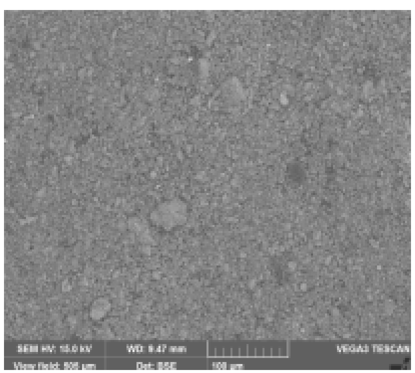
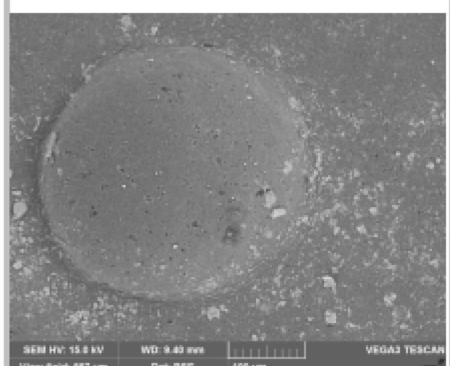
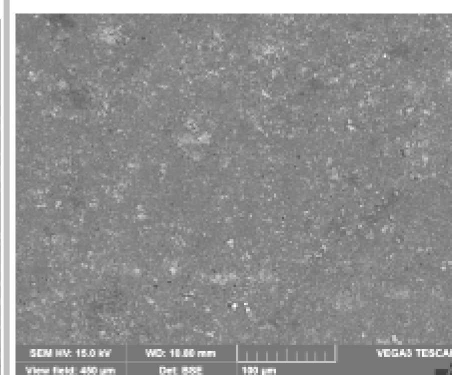
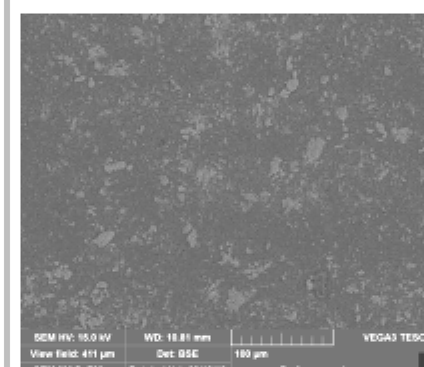
Accomplishments: SEM Analysis of Polypropylene in Coolants

Polypropylene Immersed in Coolants for 2,500 hours at 80°C

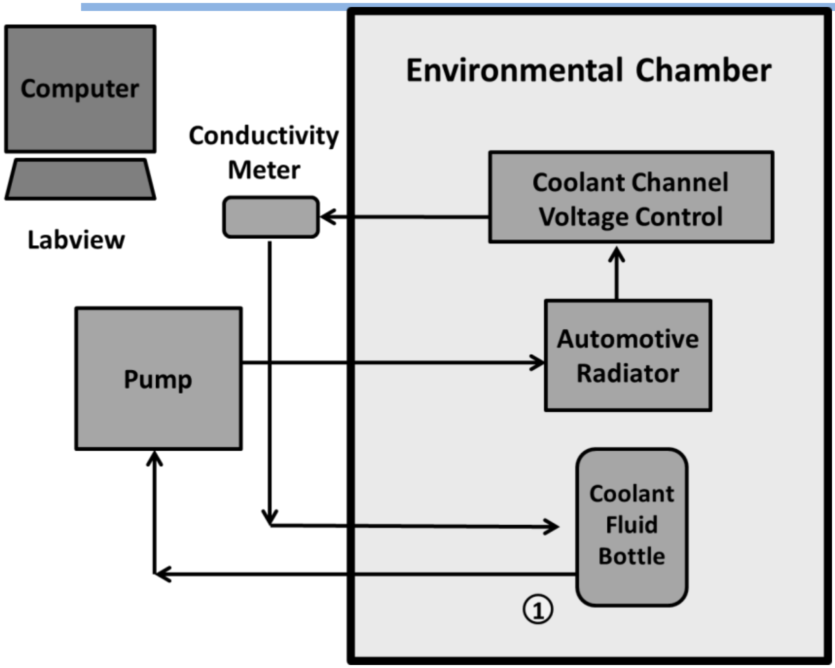
	Blank	BioGlycol-Water	FC	LC
Appearance				
Microscopy				

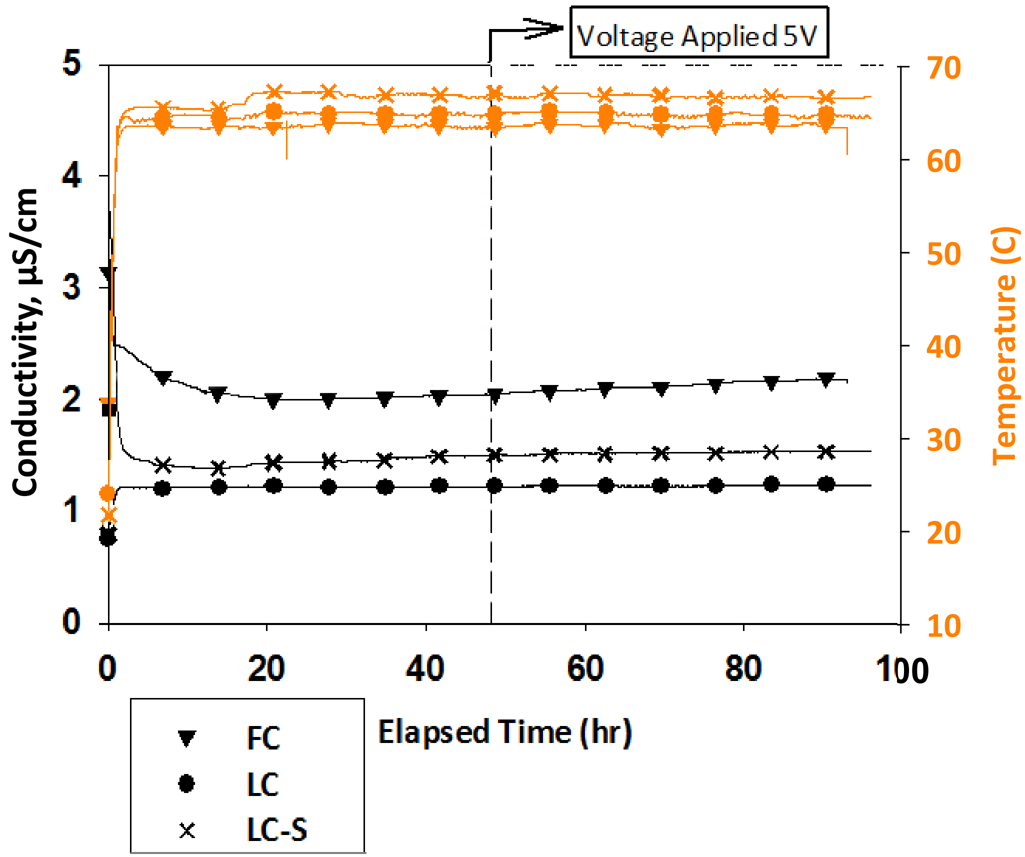
Accomplishments: SEM Analysis of Viton in Coolants

Viton Immersed in Coolants for 2,500 hours at 80°C

	Blank	BioGlycol-Water	FC	LC
Appearance				
Microscopy				

Accomplishments: Applied Voltage Test Rig

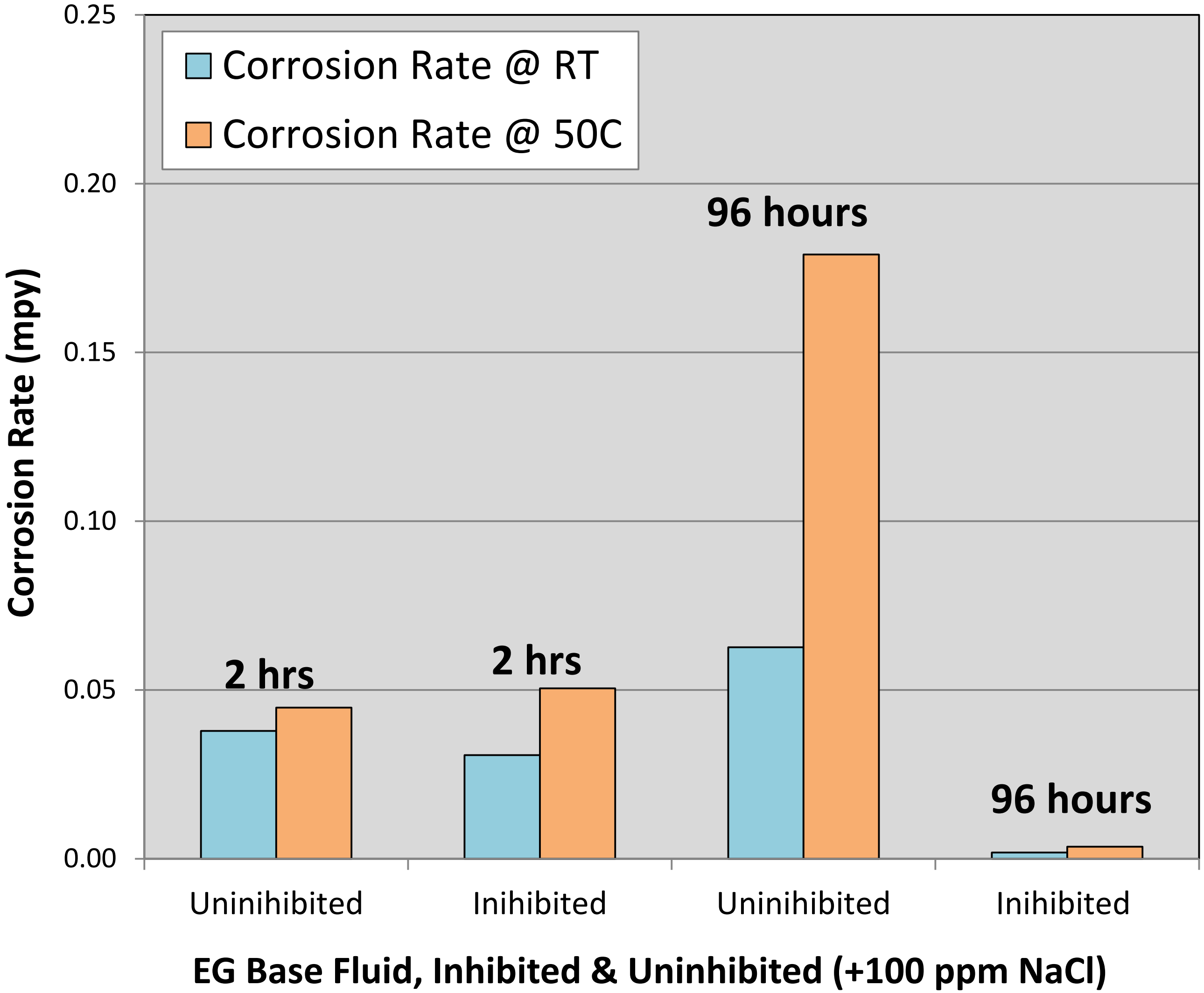




- Testing near 70°C
- Impact of 5V on conductivity growth is negligible
- Initial conductivity of Dynalene coolant with nanoparticles is higher, then gradually is reduced

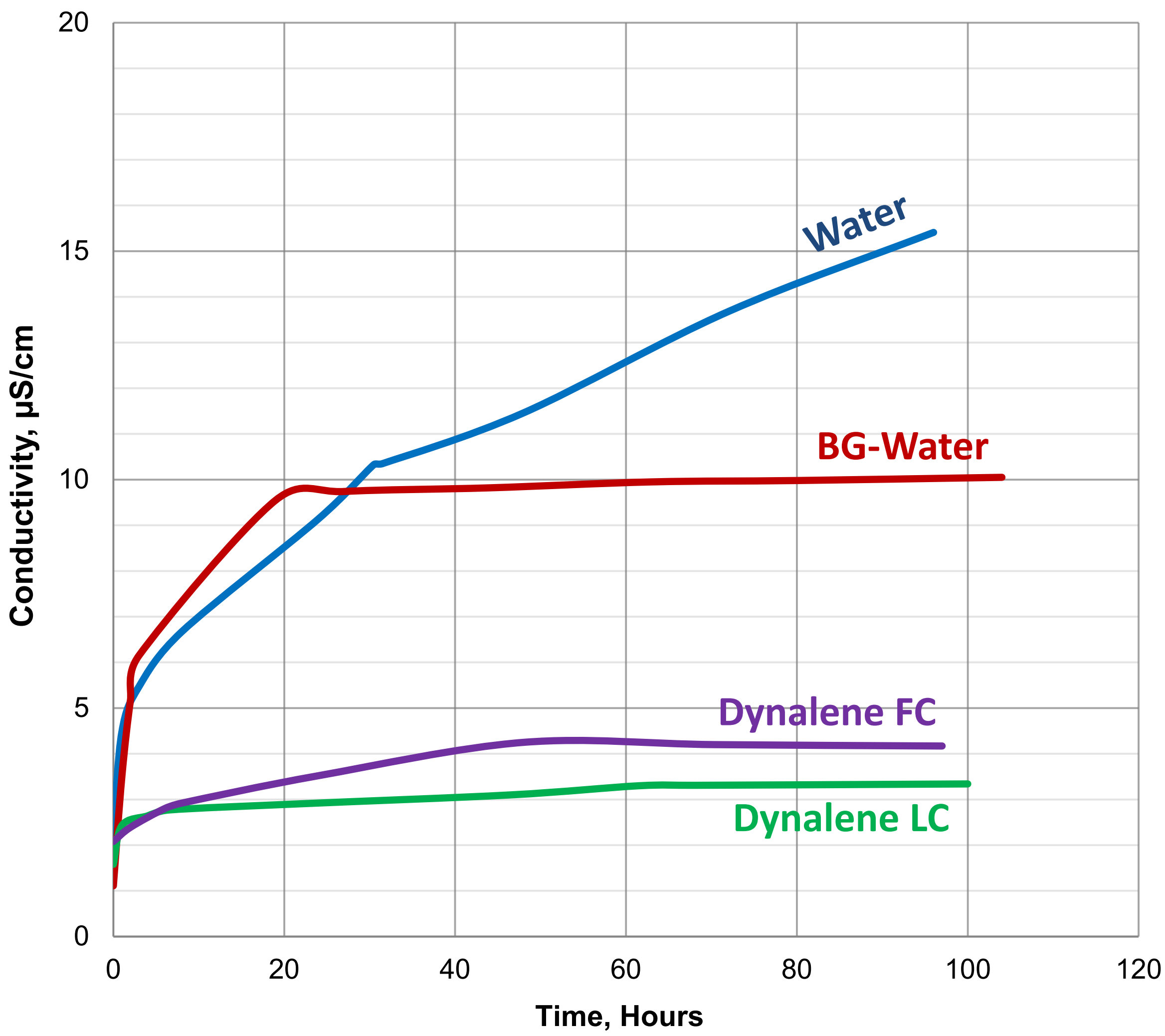
LC-S: LC with proprietary component

Accomplishments: Effect of FC/LC Inhibitor on Aluminum



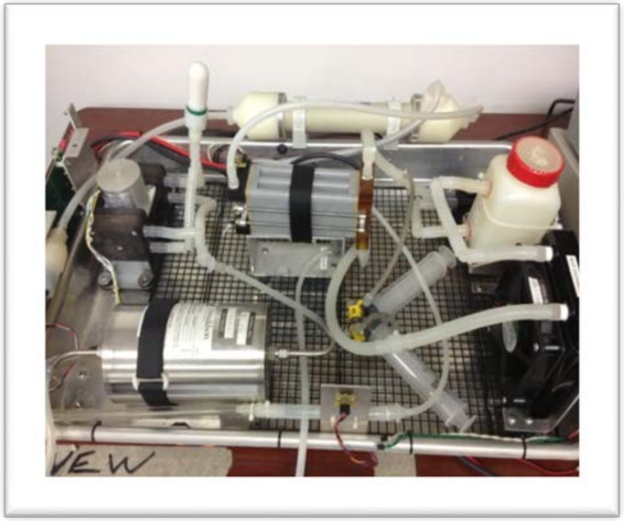
- Electrochemical results of inhibited and uninhibited aluminum at room temperature and 50°C
- FC/LC non-ionic corrosion inhibitors in EG base fluid significantly reduce corrosion rate over uninhibited EG after 96 hour immersion times
- Longer immersion times allow the inhibitors to coat the aluminum protecting the metal from corrosive attack

Accomplishments: Protonex Fuel Cell



Fuel Cell Specifications:

- Operating load of 150 W (5 Amps, 30 Volts)
- Coolant temperature of 52°C
- No loss of fuel cell performance or operating load during duration of testing



Accomplishments: Field Testing

- **10 fuel cell manufacturers testing FC and LC coolants**
- **3 manufacturers purchasing FC and LC**
- **2 fuel cell companies purchasing and testing FC and LC**
- **3 companies planning testing**

Collaborations & Acknowledgements

- University of Tennessee, Knoxville
  - Mike Manahan
  - Grace Briggs
  - Mushrif Choudhury
  - Ozgur Cekmer
  - Subhadeep Chakraborty
  - Professor Matt Mench
- Protonex Technology Corporation
  - Jeremy Hitchcock
  - Paul Osenar
- Dynalene, Inc.
  - Sreya Dutta
  - Lauren Fotiades
  - Patrick McMullen
  - Kevin Coscia
  - David Schiemer
- Lehigh University Center for Advanced Materials and Nanotechnology
- Department of Energy
  - Kathi Epping Martin
- Naval Research Laboratories
  - Karen Swider-Lyons
  - Yannick Garsany
- **All the fuel cell manufacturers who are testing and purchasing Dynalene fluids**

Future Work

- Complete validation of corrosion inhibitors in 5,000 hour tests (Dynalene)
  - **High-temperature electrochemical testing (100°C)**
- Complete compatibility and thermal degradation studies at temperature exceeding 100°C (Dynalene)
- Complete long term testing of final coolant formulation in working fuel cell system (Dynalene)
- Continuation of testing in automotive coolant rig (UTK)
  - **Testing up to 35 V**
  - **Testing of 300 hours for all coolants**
- Continuation of tested fluid species identification (UTK)
  - **New ion-chromatograph/HPLC dual channel system installed at UTK**

Summary

Accomplishments in 2012

- Coolant optimization and scale-up completed and Dynalene is capable of producing FC coolant in large quantities
- Fuel cell coolant testing skids at Dynalene and UTK completed and prepared for long term testing
- Coolant degradation tested at 120°C
- Corrosion inhibitors validated in short term testing using immersion and electrochemical methods
- Compatibility of metals and polymers completed in short term tests at 80°C and 100°C

Accomplishments in 2013

- Surface charge of reformulated nanoparticle has been increased to 890 μeq/g
- Compatibility and thermal degradation studies of polymers and metals have been performed at 80°C for 5,000 hours
- Degradation of new coolant formulations has been measured at 120°C
- Corrosion inhibitors have been validated for metals and polymers at 80°C for 2,500 hours using SEM/EDS analysis
- Coolants have been tested in working fuel cell in 100 hour tests
- Freeze/thaw tests completed and confirmed that particles remain stable in coolant

Thank You

Questions?

Technical Support Slides

- High-Temperature Testing
- SEM-EDS Pictures
- Electrochemical Results
- Coolant Properties
- Electrochemical Equations

Technical Back-Up Slides

HIGH-TEMPERATURE TESTING

SEM-EDS PICTURES

ELECTROCHEMICAL RESULTS

COOLANT PROPERTIES

ELECTROCHEMICAL EQUATIONS

Technical Slide: 120°C Testing with BioGlycol Coolants

- 65 & 80 wt% BioGlycol-based coolants exposed to 120°C for 336 hours
- Higher BioGlycol-based coolants showed less degradation at higher temperatures

Sample	Initial pH	Final pH	Initial Cond., µS/cm	Final Cond., µS/cm
65 BG-DW	5.44	3.78	1.23	14.33
65 LC	5.62	3.82	1.14	14.04
65 FC	5.03	3.76	2.13	16.4
80 BG-DW	5.57	4.34	0.38	2.07
80 LC	5.52	4.22	0.46	2.46
80 FC	5.37	4.10	0.76	4.20

65 wt% BG **65 wt% LC** **65 wt% FC**

65 wt% BioGlycol-based Coolants After Test

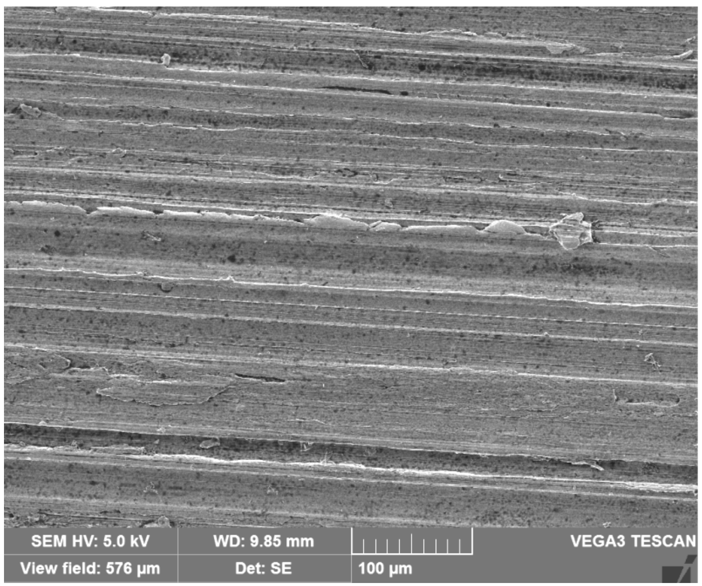
80 wt% BG **80 wt% LC** **80 wt% FC**

80 wt% BioGlycol-based Coolants After Test

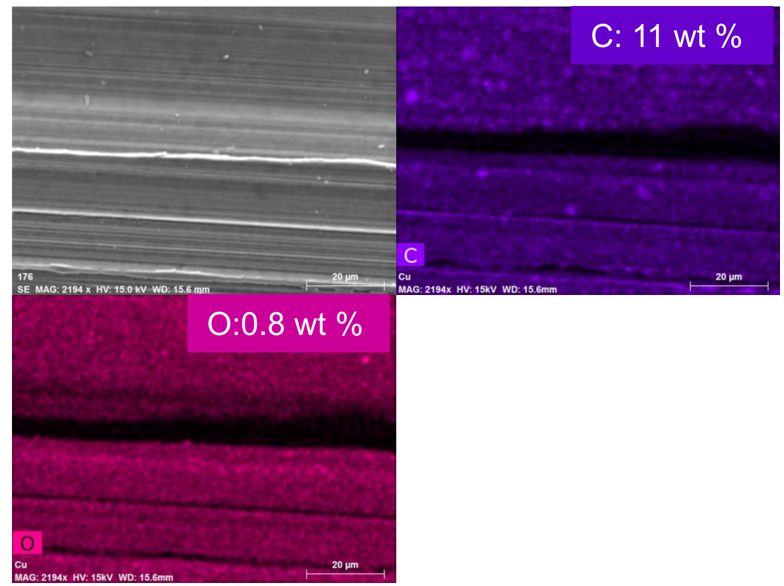
Technical Slide: Brass Blank SEM & EDS

Blank Brass Coupon, SEM/EDS

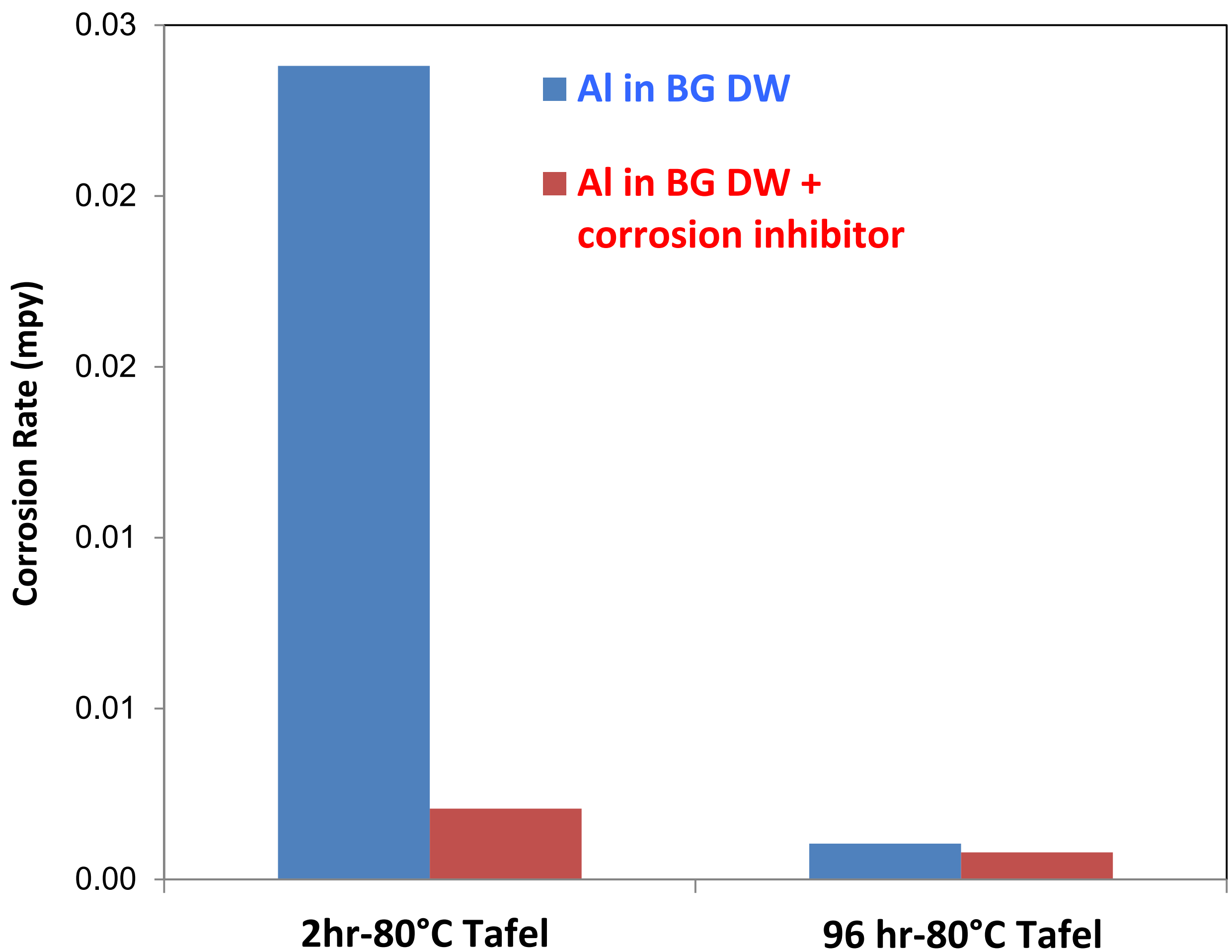
Blank Coupon SEM



Blank Coupon EDS



Technical Slide: Effect of FC/LC Inhibitor on Aluminum



- Electrochemical results of inhibited and uninhibited BioGlycol with aluminum at 80°C
- Incubation in the coolant for longer period of time protects the metal from general corrosion and pitting
- Corrosion Inhibitors form protective layer on Al on extended exposure; this imparts excellent corrosion resistance to the metal

Technical Slide: Coolant Properties

Coolant Properties

Coolant	Specific Gravity	Viscosity, cP	Thermal Cond., W/mK
Dynalene LC	1.049	31.3 @ 0°C 18.3@ 10°C	0.316
Dynalene FC	1.048	30.6 @ 0°C 17.5 @ 10°C	0.323