1. Fuel Cell Coolant Optimization and Scale-up

2. Large Scale Testing, Demonstration, and Commercialization of Fuel Cell Coolant (Phase III)

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Overview

Coolant Optimization and Scale-Up

Total project funding

- DOE share: $351K
- Cost share: $114K

Timeline

- Project start date: Sept 1st, 2009
- Project end date: Aug 31st, 2011
- 100% Complete

Barriers addressed

- System thermal management
  - Durability & Cost

Interactions/ collaborations:

- Lehigh University

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

Total project funding

- DOE share: $1.016 M
- FY11: $800K
- Planned FY12: $0K

Timeline

- Project start date: Oct 1st, 2010
- Project end date: Sept. 31st, 2013
- 35% Complete

Barriers addressed

- System thermal management
  - Durability & Cost

Interactions/ collaborations:

- Protonex
- University of Tennessee, Knoxville

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Relevance

DOE Scale-up Project and SBIR Phase III	
DOE Barriers	Dynalene Inc.
A. Durability	Understand through research and working with fuel cell manufacturers what the durability requirements are.

Coolant Durability	>5000 operating hours
Coolant Compatibility	good with typical components found in fuel cells

Relevance

DOE Scale-up Project and SBIR Phase III	
DOE Barriers	Dynalene Inc.
B. Cost	Understand what the cost of coolant should be relative to current automotive coolants.

Cost of Coolant as Production Scaled Up

Coolant Production Size	Final Coolant Cost
small scale (55 gallon batches)	$45/gallon
medium scale (1000 gallon batches)	$30/Gallon
pilot scale (5000 gallon batches)	$15 to $25 / Gallon
full scale (>100,000 gallons/year)	$10 to $15 / Gallon

Relevance

DOE Scale-up Project and SBIR Phase III	
DOE Barriers	Dynalene Inc.
C. Performance	(see table below)

Coolant Criterion	Description	Value / Characteristics
Electrical Conductivity	Electrically conductive coolants reduce the performance of a fuel cell.	< 2.0 micro Siemens/cm
Boiling Point	Similar to current automotive coolants	> 100°C
Freezing Point	Must be freeze tolerant under extreme cold conditions	< -40°C
Thermal Conductivity	Higher is better for heat transfer	> 0.4 W/m K
Viscosity	Lower is better for heat transfer and pumping	< 1.0 cP at 80°C
Specific Heat	Higher is better for heat transfer	> 3 kJ/kg K
Cost/Toxicity/Flammability	Current automotive coolant	Similar to Automotive Coolant

Approach: Coolant Characteristics

<table>
<tr><th>Characteristics</th><th>Dynalene FC (Fuel Cell Coolant)</th></tr>
<tr><td>Composition</td><td>Dynalene FC has a base fluid consisting of a glycol water mixture with a nanoparticle and non-ionic corrosion inhibitor additive package</td></tr>
<tr><td>Performance</td><td>Non-Flammable and non-toxic with physical and thermo-physical properties similar to typical automotive coolants</td></tr>
<tr><td rowspan="2">Low Electrical Conductivity</td><td>The low electrical conductivity of Dynalene FC is maintained through two mechanisms</td></tr>
<tr><td><table><tr><th>Nanoparticles</th><th>Non-Ionic Corrosion Inhibitors</th></tr><tr><td>Ion scavenging in the working coolant</td><td>Ion suppression at the wall of the coolant channel</td></tr></table></td></tr>
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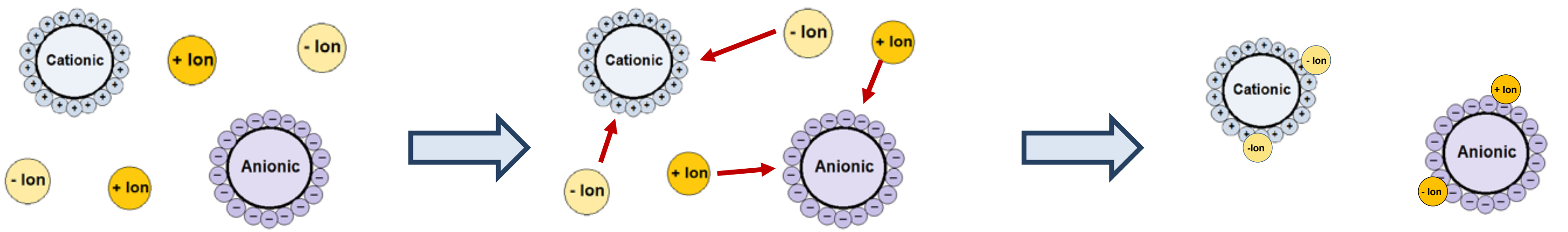
How Dynalene FC Works

The Nanoparticles

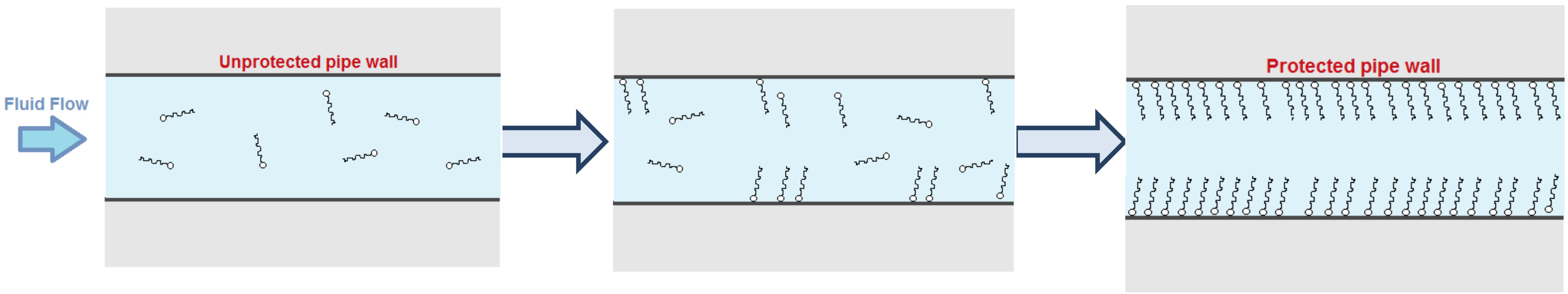


Free ions and nanoparticles in solution

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

Nanoparticles finally scavenge and neutralize free ions

The Inhibitor

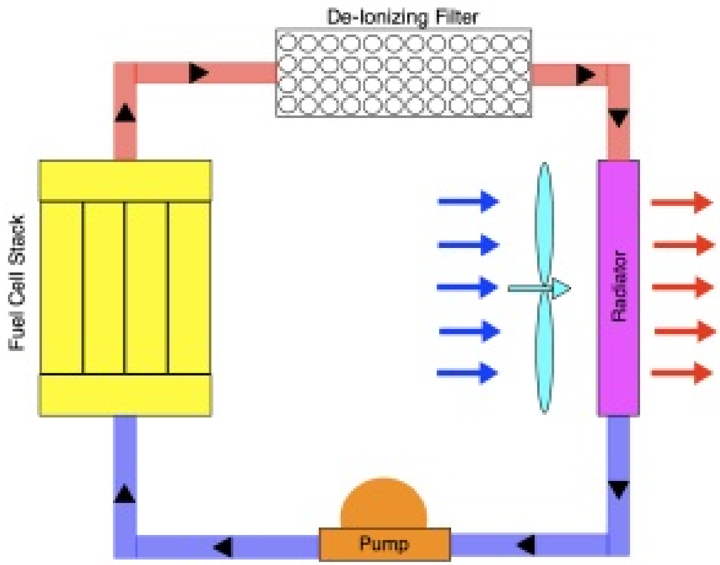


Non-ionic inhibitors circulating through pipe in Dynalene FC

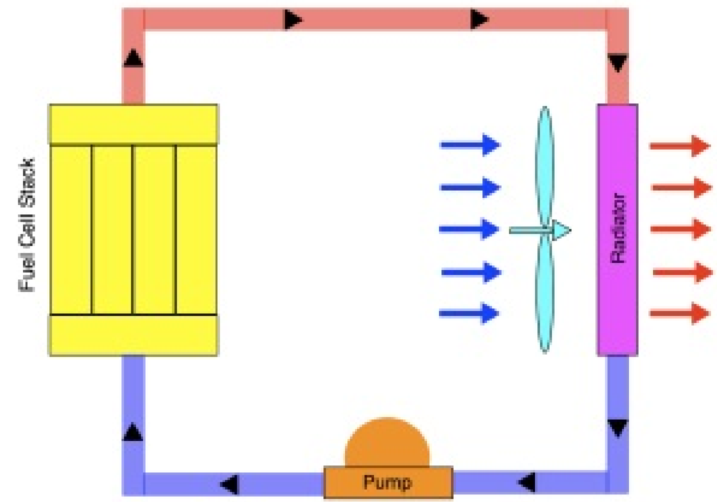
Inhibitor heads begin to bond to surface on pipe wall

Inhibitor fully protects wall from corrosive attack

Approach: Dynalene FC Advantages in a PEM Fuel Cell



vs.



Typical DI Water/Glycol Cooled System

- Needs De-ionizing Filter
- Higher Pressure Drop (Larger Pump)
- More Maintenance
- Clogging
- Heavier
- Possible Corrosion

Dynalene FC Cooled System

- No De-Ionizing Filter
- Lower Pressure Drop (Smaller Pump)
- Higher Performance
- Lower Cost
- No Clogging
- Lighter Weight
- Less Corrosive

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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

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Approach: Coolant Scale-up Tasks and Schedule

Task	Milestone	Status
1	Procure reactors	Completed
2	Optimization of 10 L Scale-up	Completed
3	Optimization of Purification in 2 L Scale	Completed
4	Nanoparticle Production in 100 L scale	Completed
5	Develop Blending Procedure for Coolant	Completed
6	Complete QA/QC Protocol for Coolant	Completed

Approach: SBIR 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.)	Ongoing
Sept 2012	Produce nanoparticles with at least 500 μeq/g (Dynalene, Inc.)	Ongoing
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

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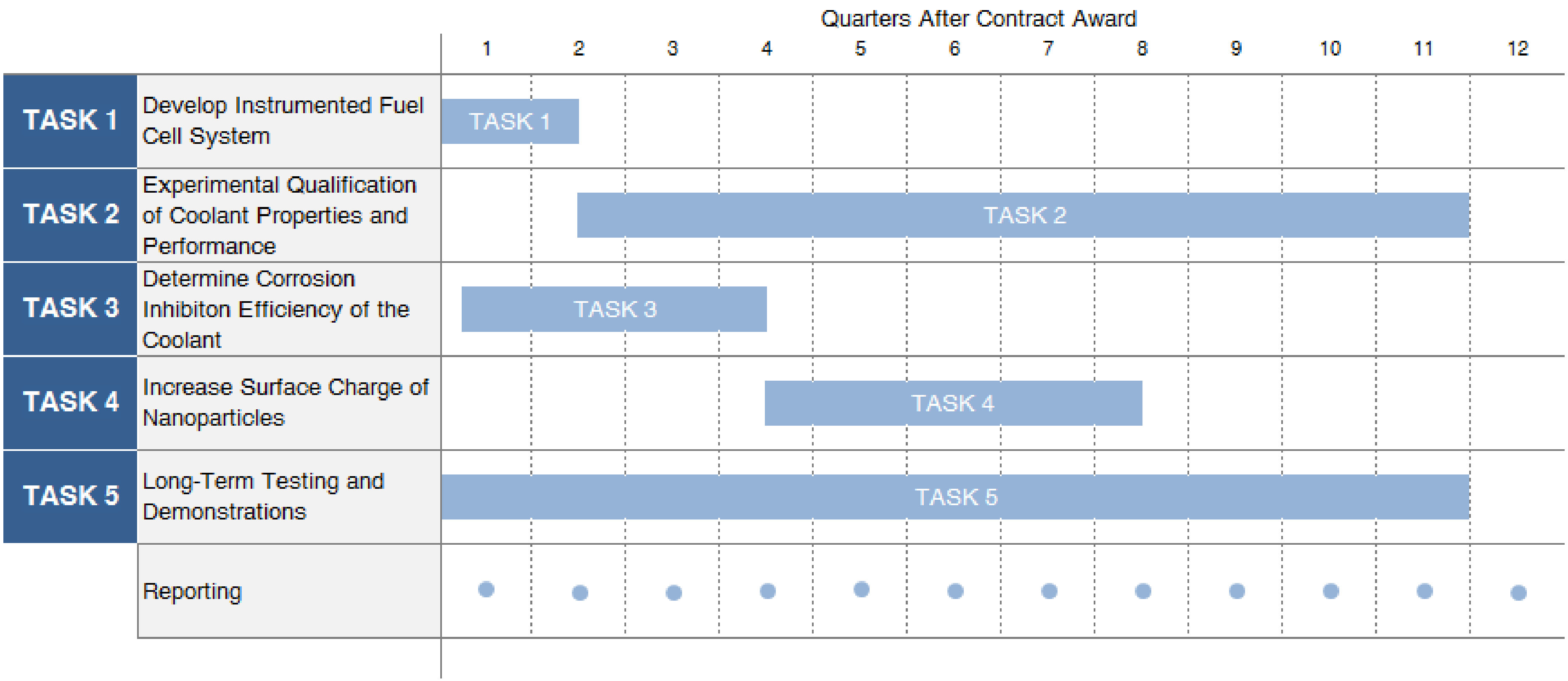
SBIR Phase III Tasks and Schedule



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Tasks and Approach

Task 1: Development of an Instrumented Fuel Cell Coolant System (University of Tennessee, Knoxville)

- Design of Flexible System (i.e. Automotive, Stationary, and Portable Specific)
- Working Temperature Range: -40°C to 120°C
- Measurement of Electrical Conductivity, pH, Temperature, Flow Rate

Task 2: Experimental Qualification of Coolant Properties and Performance (University of Tennessee, Knoxville)

- Steady Operation Durability Testing
- Non-operational Durability Testing: Zero Voltage and High Stack Voltage
- Load Cycle Impact Testing
- Advanced Coolant Accelerated Qualification Testing

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

- Material Compatibility Test, Immersion Test, and Electrochemical Test
- Further Development of Non-ionic Corrosion Inhibitors
- 5,000 Hours of Testing

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Tasks and Approach

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

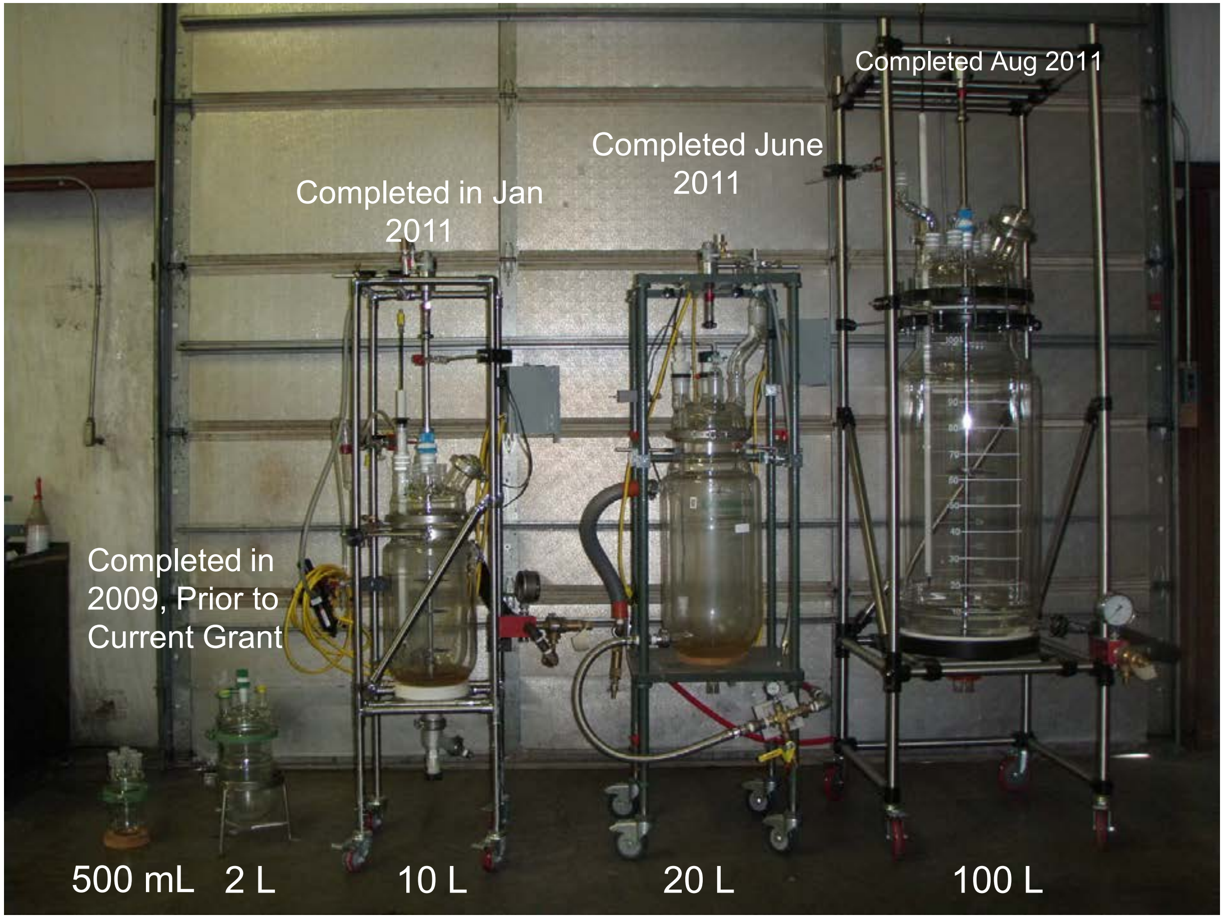
- Mainly for Anionic Particles
- Current Surface Charge is about 100 μeq/g
- Target is 500 μeq/g

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

- Two Protonex Systems: One at the Protonex Site and the other at the Dynalene Site
- Protonex will Perform 2,000 to 3,000 Hour Testing (2012-2013)
- Dynalene will Start 5,000 Hour Testing on Selected Coolant Formulations

Accomplishments: Scale-up

- Scaled up from 500 mL to 100 L Nanoparticle reactions.
- Developed QA/QC methods
- Reduced final fluid cost



Glass Reactors Used in the Scale-up of Nanoparticle Production from 500 mL to 100 L Batches

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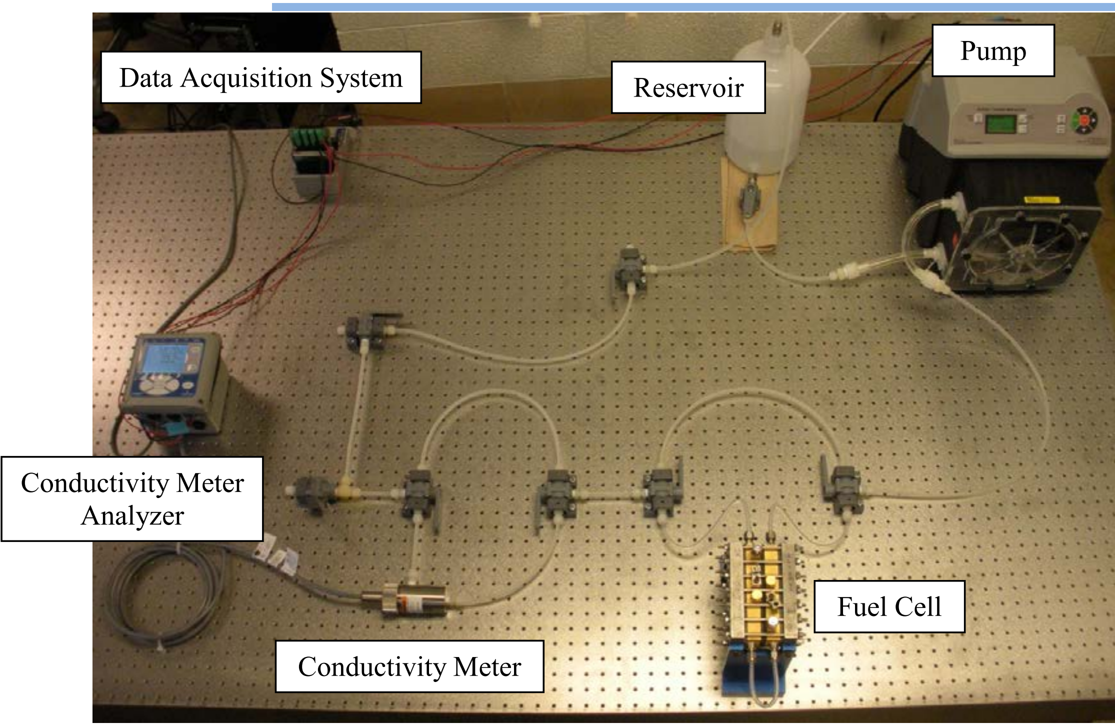
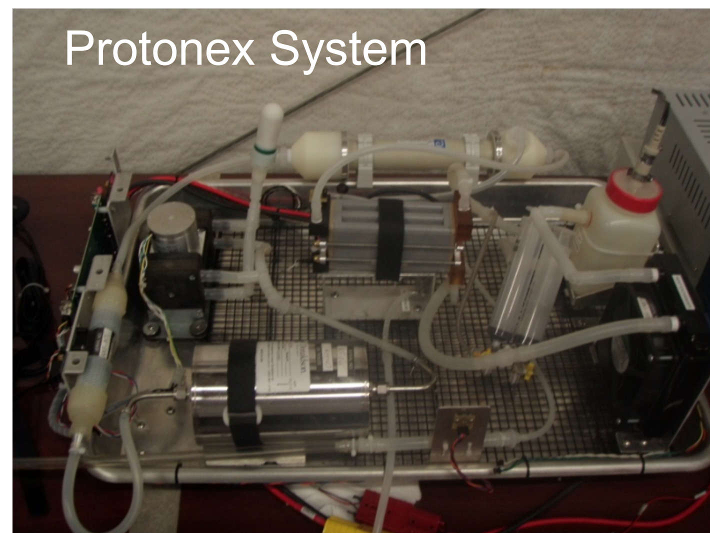
Accomplishments: Phase III



Completed design and fabrication of two separate fuel cell systems



- University of Tennessee, Knoxville (UTK) fuel cell being tested at their facility
- Protonex fuel cell being tested at Dynalene

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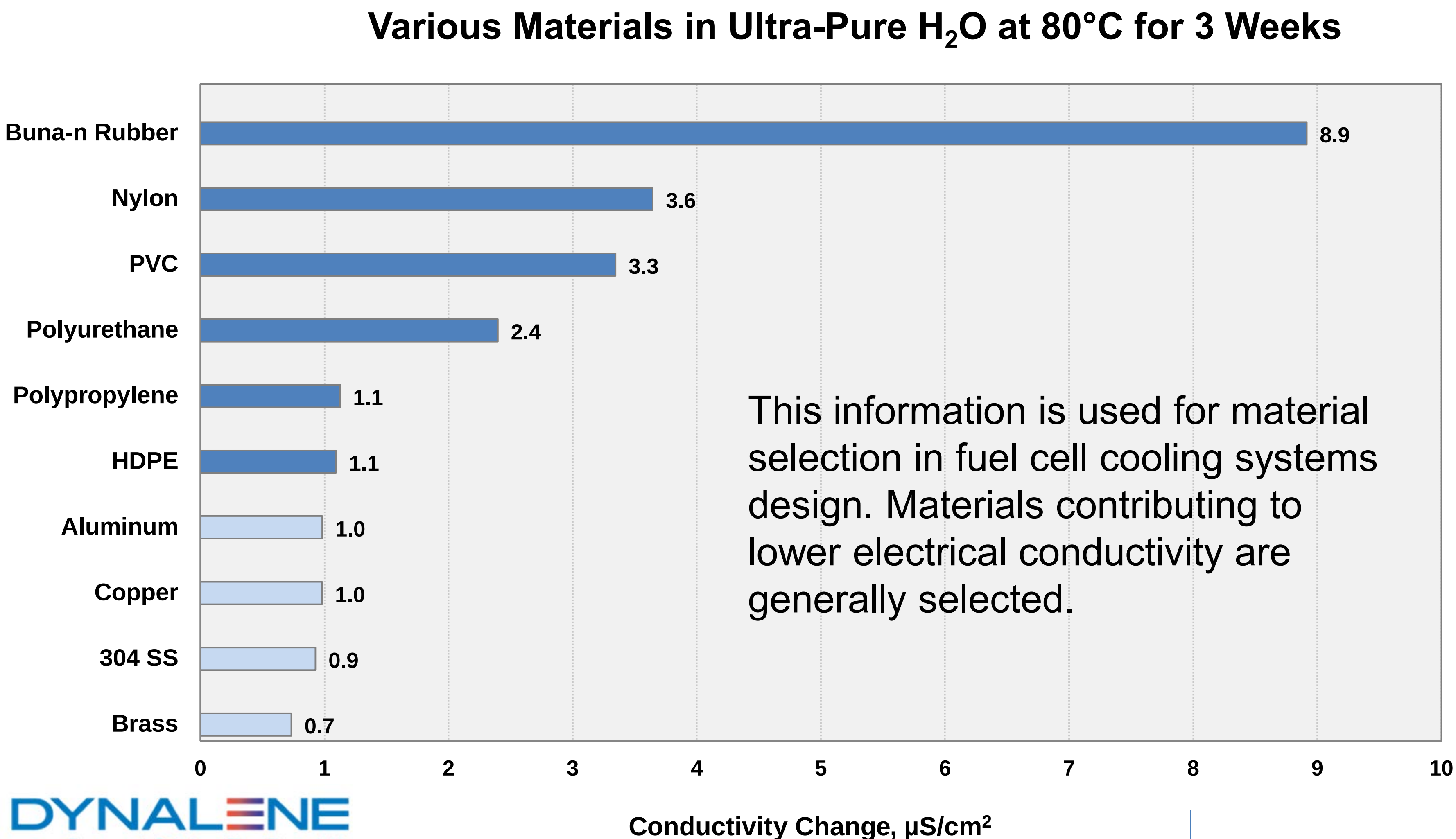
Contribution of Plastics & Metals to Electrical Conductivity



This information is used for material selection in fuel cell cooling systems design. Materials contributing to lower electrical conductivity are generally selected.

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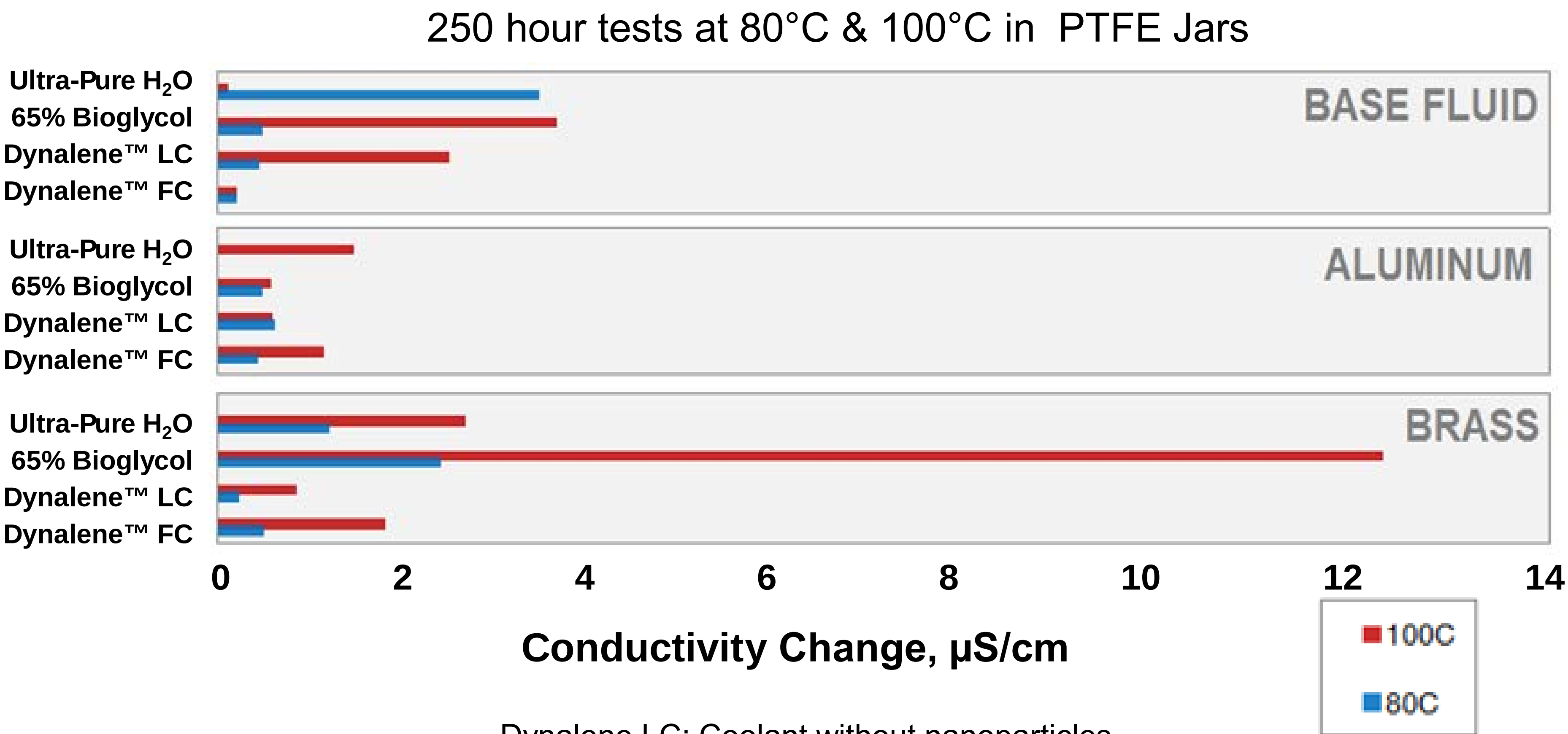
Material Testing: 80°C vs. 100°C



Dynalene LC: Coolant without nanoparticles
Dynalene FC: Coolant with nanoparticles

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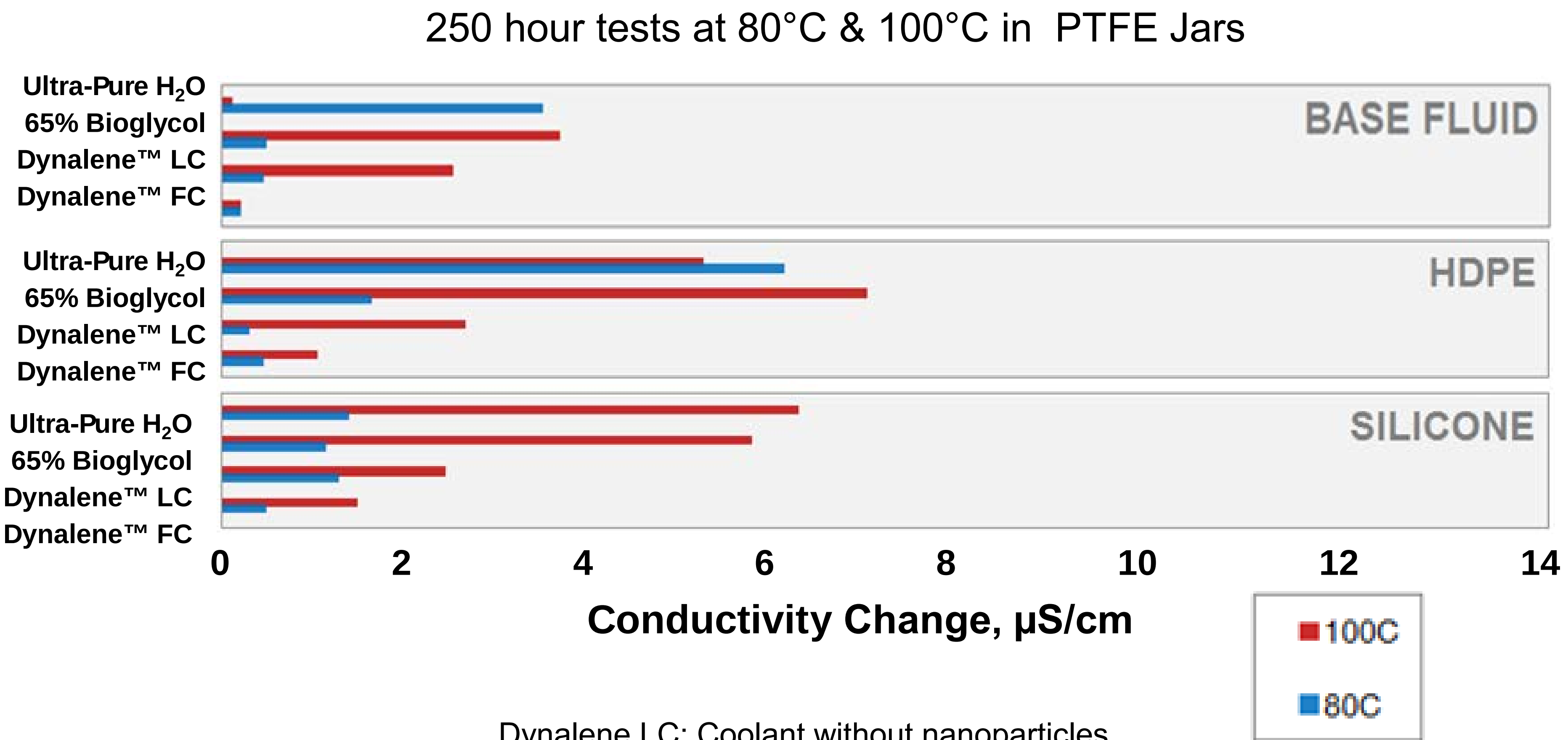
Material Testing: 80°C vs. 100°C



Dynalene LC: Coolant without nanoparticles
Dynalene FC: Coolant with nanoparticles

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Accomplishments: Degradation of Base Fluid

Testing Base Fluids at 120°C for 2 Weeks

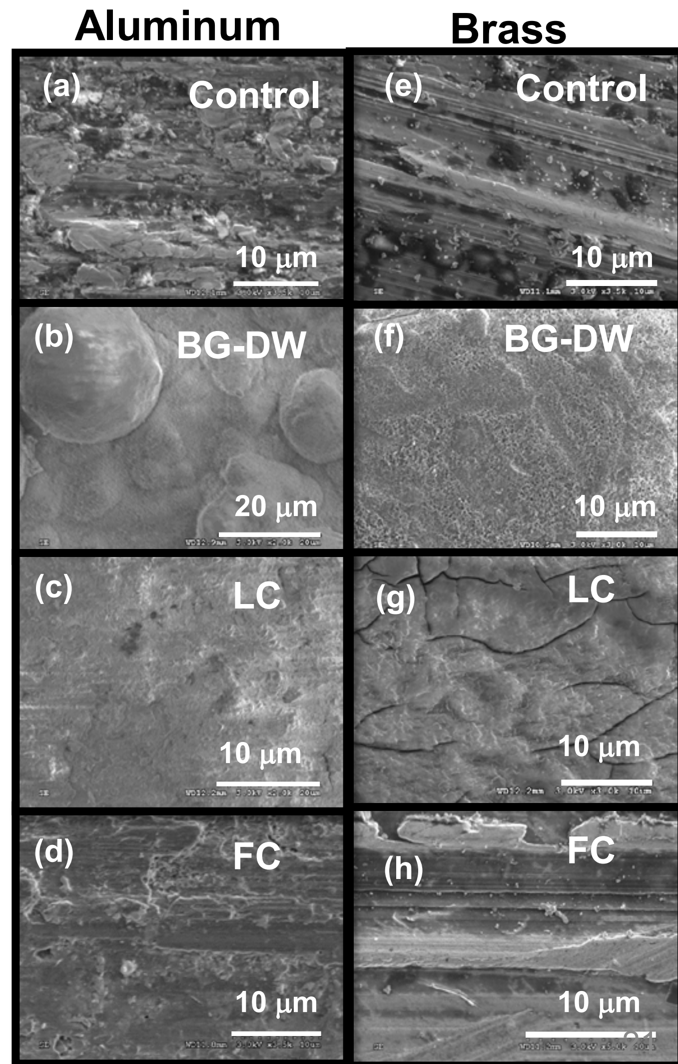
Appearance of Fluids	Initial pH	Final pH	Initial Conductivity (µS)	Final Conductivity (µS)
Ultra-Pure Distilled Water (UP DW) 	5.87	6.12	1.11	2.50
Bio Glycol (65%)-DW (35%) 	4.56	3.91	1.85	10.23
Dynalene LC 	4.59	3.87	1.85	10.69
Dynalene FC 	6.22	3.9	0.75	13.6

SEM – Aluminum & Brass

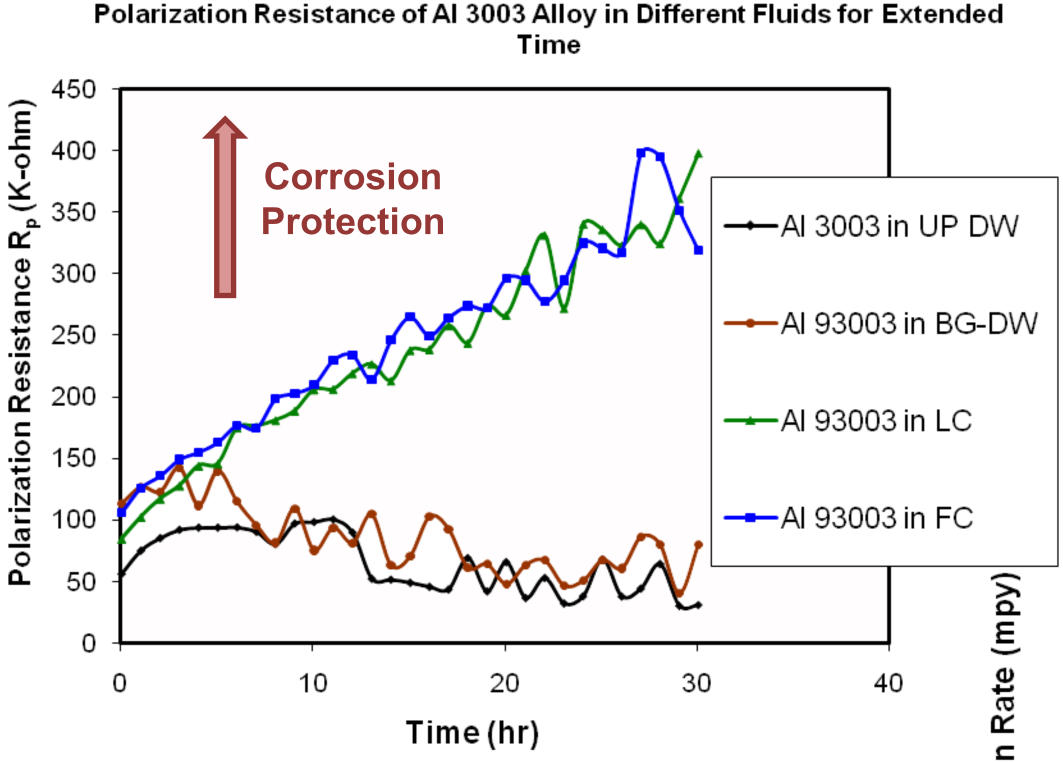
SEM images of aluminum and brass exposed to different fluids at 88°C for 2 weeks (ASTM D 1384)

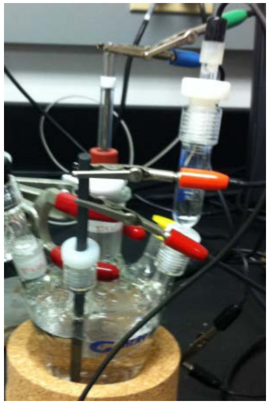
Aluminum and brass surface treated in SEM and EDS showed:

- BioGlycol (BG)-DI Water (DW): Presence of oxide rich layer on the surface
- LC: Surface layer rich in nitrogen and carbon
- FC: Clean surface with negligible oxide/organic surface layer

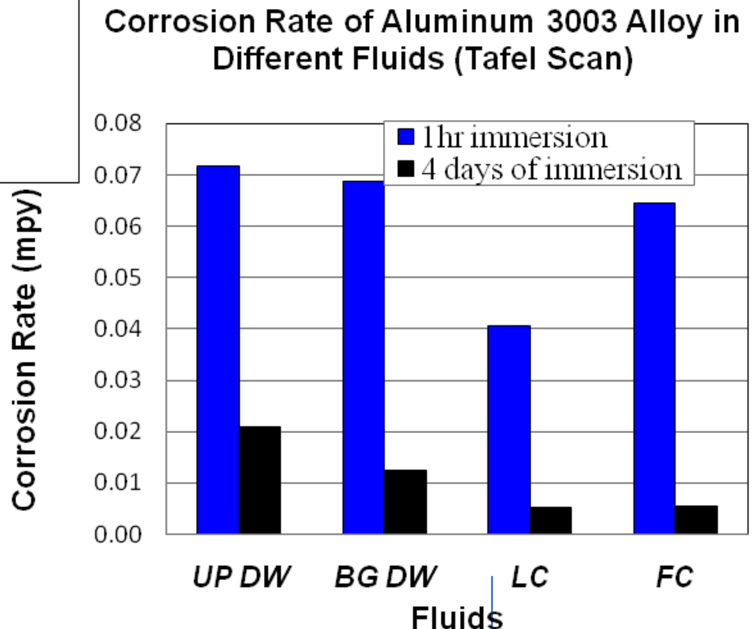


Accomplishments: Electrochemical Corrosion Study - Aluminum





Gamry 3 electrode Euro-cell



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Collaborations

- University of Tennessee, Knoxville
  - Designed and built fuel cell coolant system
  - Will test in parallel of Dynalene and send coolant samples for testing
- Protonex
  - Provided Dynalene with working fuel cell for testing
  - Perform tests on commercial fuel cell for up to 5,000 hours

Future Work

- Validate corrosion inhibitors in 5,000 hour tests
- Increase of anionic particle surface charge to match cationic at 500 μeq/g
- Perform compatibility and thermal degradation studies at temperature exceeding 100^{o}C
- Perform long term testing of final coolant formulation in three separate fuel cell systems.

Summary

- The fuel cell coolant optimization and scale-up has been completed and Dynalene is capable of producing Dynalene FC coolant in large quantities.
- The fuel cell coolant testing skids at Dynalene and the University of Tennessee, Knoxville have been completed and are ready for long term testing.
- The corrosion inhibitors were validated in short term testing using immersion and electrochemical methods.

Thank You

Questions?

Technical Support Slides

- SEM-EDS Pictures
- Coolant Properties
- Electrochemical Equations

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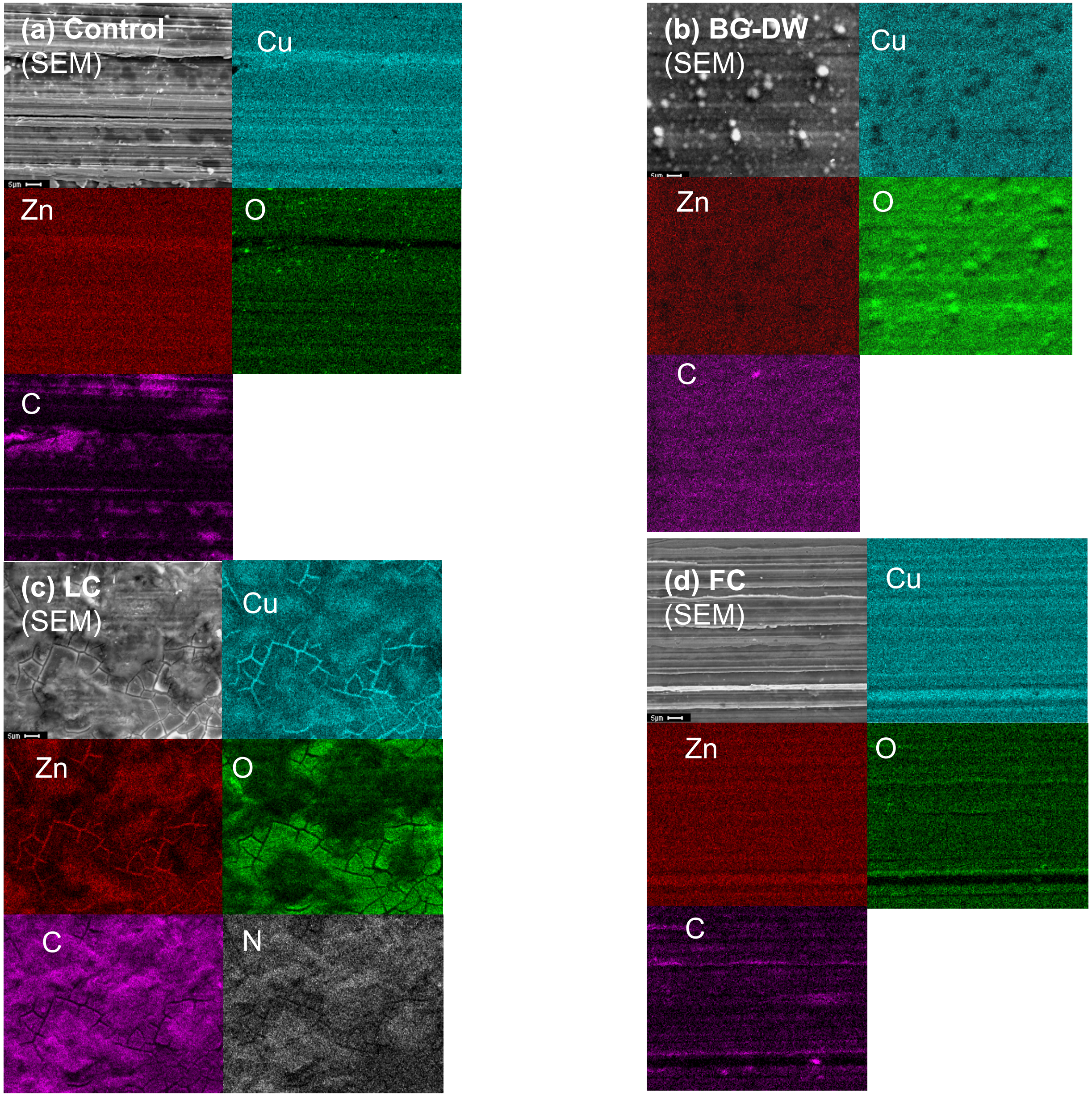
Technical Back-up Slide for EDS



Elemental EDS maps on Brass coupons after ASTM D 1384: (a) As-received and treated in (b) BG-DW, (c) Dynalene LC and (d) Dynalene FC

Technical Back-up Slide

Coolant Properties

Coolant	Specific Gravity	Viscosity (cP)	Thermal Conductivity (W/m-K)
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

Electrochemical Equations

Rp and Corrosion Rate (CR)

From Stern-Geary Equation:

$$R_P = \frac{\beta_a \times \beta_c}{2.3 I_{Corr} (\beta_a + \beta_c)}$$

where,

$\beta_a, \beta_c = \text{Tafel constants}$

$I_{Corr} = \text{Corrosion current}$

Also,

$$CR = \frac{I_{Corr} \times K \times EW}{d \times A}$$

where,

$\text{K} = \text{constant}$

$\text{EW} = \text{equivalent weight}$

$\text{d} = \text{density}$

$\text{A} = \text{sample area}$

$$\therefore CR \propto \frac{1}{R_P}$$