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Low Cost, Durable Seals For PEM Fuel Cells

Jason Parsons
UTC Power Corporation

June 8, 2010

Henkel
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Freudenberg
NOK

Virginia Tech
VIRGINIA POLYTECHNIC INSTITUTE AND STATE UNIVERSITY

FC053

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Overview

Timeline

- Start: Apr 2007
- End: Sept 2010
- 90% Complete

Barriers

- A: Fuel cell seal durability
- Fuel cell seal cost is also being evaluated

Budget

- Total Project Funding
  - DOE: $1,980K
  - Contractor: $1,320K
- Funding Received in FY09
  - $430K
- Funding for FY10
  - $100K

Partners

UTC Power A United Technologies Company **(Project Lead)**

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Freudenberg NOK

Virginia Tech Virginia Polytechnic Institute and State University

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Collaborators

Collaborator	Project Role	Team Leads
UTC Power A United Technologies Company	•Material specification •Seal interactions •Modeling •Seal design •Stack design	•Jason Parsons
Henkel A Brand like a Friend	•Materials development •Development support and consulting	•Dr. Matthew Burdzy
Freudenberg® NOK	•Seal concept evaluation •Rapid prototyping •Process development	•Mark Belchuk
Virginia Tech 1872 VIRGINIA POLYTECHNIC INSTITUTE AND STATE UNIVERSITY	•Material characterization •Accelerated testing	•Dr. David Dillard

Relevance to DOE Targets

The Goal: Develop a low cost, non-silicone, durable seal material that is broadly applicable to the PEM fuel cell industry and sealing techniques amenable to high volume manufacture of PEM cell stacks.

DOE Targets/Goals/Objectives	Project Goals
Durability ***Barrier A*** Transportation: 5,000 hr Stationary: 40,000 hr	Durability - 4000 hr bulk material testing at up to 90 C - Up to 4000 hr air aging and CSR at up to 120 °C - 4000 hr accelerated out-of-cell testing - 2000 hr in-cell verification testing
Low Cost ***Barrier B*** - Barrier not explicitly identified for seals - DTI study suggests target of $3.91/kW @ 500k stacks/year - Fuel Cell Tech Team suggests $2.00/kW @ 500k stacks/year	Low Cost - Evaluate seal material and production method against suggested targets

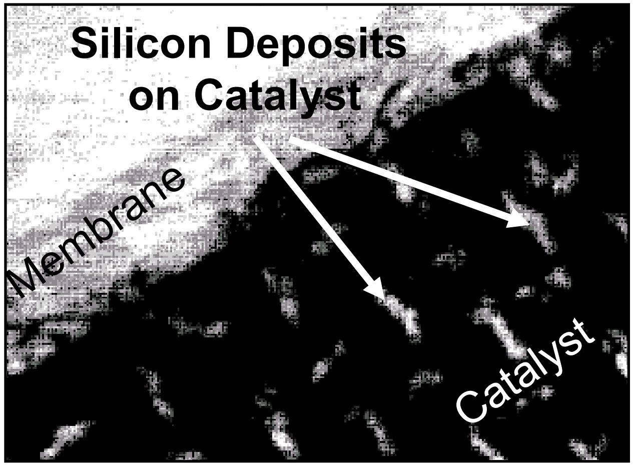
Background

Material selection concept

Material Category	Stress Relaxation	Chemical Stability	Processibility	Low Cost
LIM Silicone	–	–	+	+
Fluoropolymers	O	+	O	–
Existing Hydrocarbons	+	+	O	O
LIM Hydrocarbon	+	+	+	+

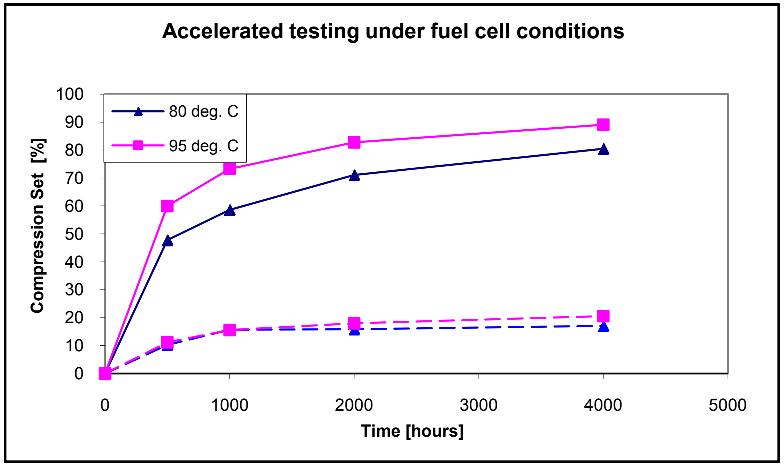
+ Excellent   O Fair   – Poor   *LIM = Liquid Injection Moldable

Silicones are know to breakdown and chemically contaminate the fuel cell



Source: M. Schulze, et. al., Journal of Power Sources 127 (2004) 222-229

Hydrocarbon elastomers can retain load better than silicones in PEM environments



Source: UTCFC-DOE Topic 1, Contract #DE-FC36-04GO14053 , Merit Review, PEM Cell Stack Activities, 2005

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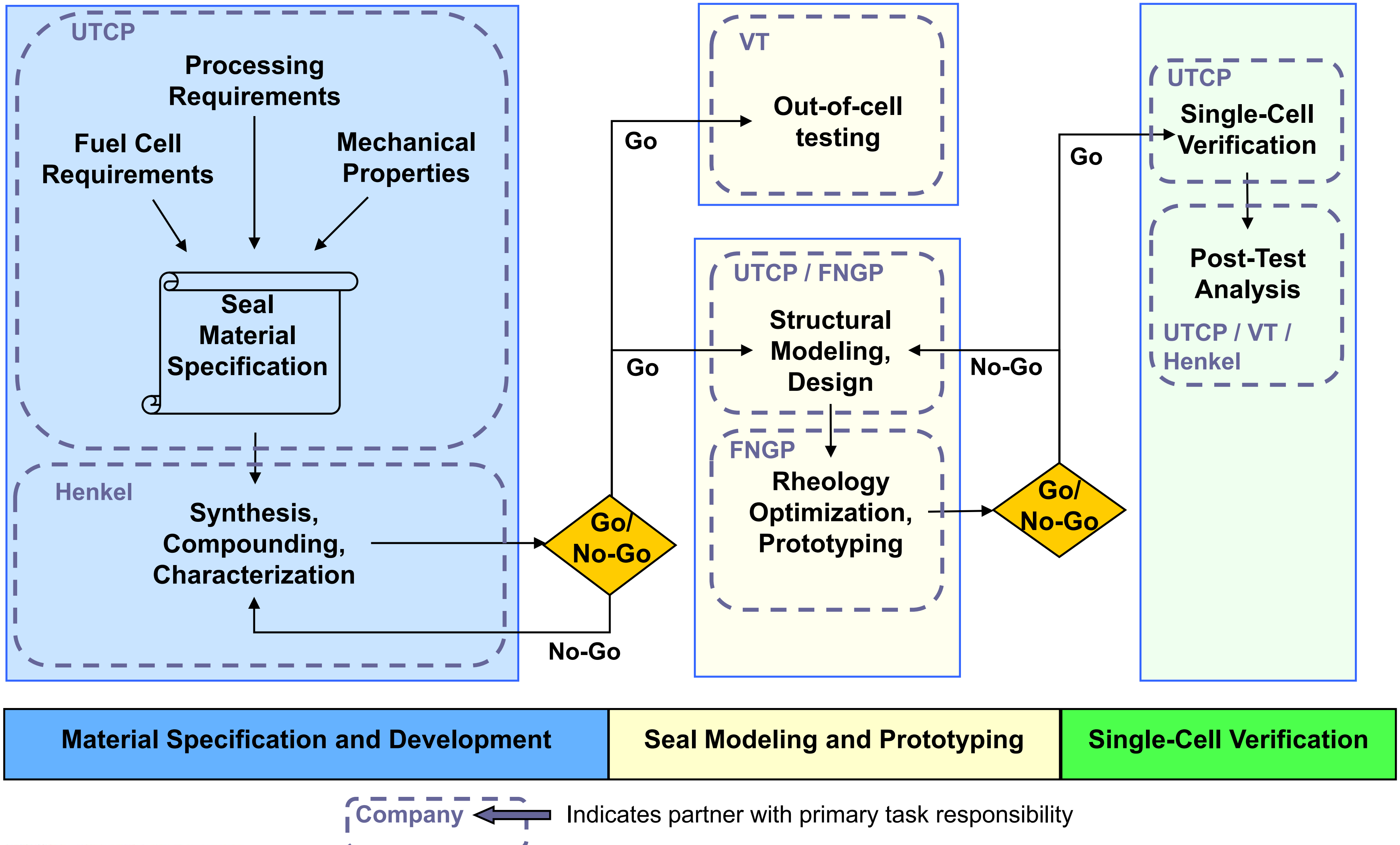
Approach



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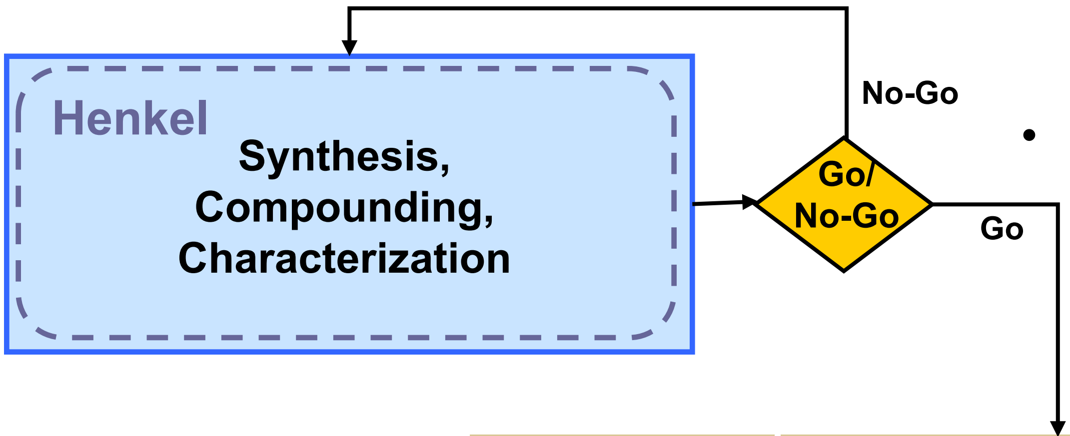
FY09 / FY10 Milestones

Milestones or Go/No-Go Decision	Progress Notes	% Comp
Milestone: Material characterization report for the leading candidate material	Most required data has been collected	90
Go/No-Go: Full-size prototype design selection	Primary going forward design has been selected. Tool fabrication is underway.	100
Milestone: 4000 hr bulk material testing	Testing continues, but greater than 4000 hours has been achieved for all planned testing.	100
Milestone: 4000 hr accelerated out-of-cell testing	Greater than 4000 hours has been achieved on planned CSR testing.	100
Milestone: 2000 hr In-Cell verification testing	FCS2 on PEN film cell configuration testing achieved 1700 hours. Testing was terminated due to a test stand issue.	70
Milestone: Complete single-cell post-test analysis	Not started	0

Accomplishments and Progress

Summary of Materials

- 100's of experiments
- Most promising candidates released for additional evaluation
- Scale up and commercialization efforts are underway*



Iteration	Description
FCS0	One-part material meeting all minimum material requirements
FCS1	Two-part material with improved curing and mechanical characteristics
FCS2	One-part material based on FCS1; purpose: to eliminate the potential for shot-to-shot mixing variability in the production of SMORS
FCS3	One-part material with improved tensile strength, tear strength and elongation (not yet released)

*Please contact Dr. Matthew Burdzy at Henkel Corporation for any sample requests

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Accomplishments and Progress

Material properties vs. specification

- All material candidates meet or exceed all minimum project goals
  - Exception: FCS3 viscosity is higher than planned for in the material specification
    - Initial molding trials indicate that the higher viscosity should not be an issue

- In terms of key initial properties, all four also meet or exceed most of the ultimate project goals

Henkel LIM Hydrocarbon Elastomer Property Table for DOE						
Properties	Project Requirements		FCS0	FCS1	FCS2	FCS3
	Minimum	Ultimate				
Process Properties						
LIM processable	Yes	Yes	Yes	Yes	Yes	Yes
Viscosity @ room temperature (cPs)	<= 700,000	<= 600,000	~ 500,000	~ 543,000	~ 543,000	~ 1,000,000
Mold temperature (°C)	< 135	<= 110	120 to 130	120 to 130	120 to 130	120 to 130
Mold time (second)	<= 400	<= 60	60 to 120*	60 to 120*	60 to 120*	60 to 120*
Mechanical Propreties						
Hardness (Shore A)	15 to 68	30 to 55	31	30	30	49
100% Modulus (Mpa)	0.25 to 3.5	1 to 2.5	0.75	0.68	0.69	1.91
Tensile strength (Mpa)	>= 0.5	>= 0.8	1.3	1.3	1.1	4.86
Elongation (%)	> 125	> 150	163	171	160	222
Tear strength Die C (kN/m)	>= 2.7	>= 5.0	3.7	3.7	3.9	16
Environmental Requirements						
Temperature resistance (°C)	-40 to 85	-40 to 90	-40 to 90	-40 to 90	-40 to 120	-40 to 120
Notes						
*cure schedule: 120 second in the mold @ 120°C and then 1 hour post cure @ 130°C						

Green: Meets minimum & ultimate goal
Lt Green: Meets minumum goal
Yellow: Does not meet project goal, but may be acceptable
Red: Does not meet project goal

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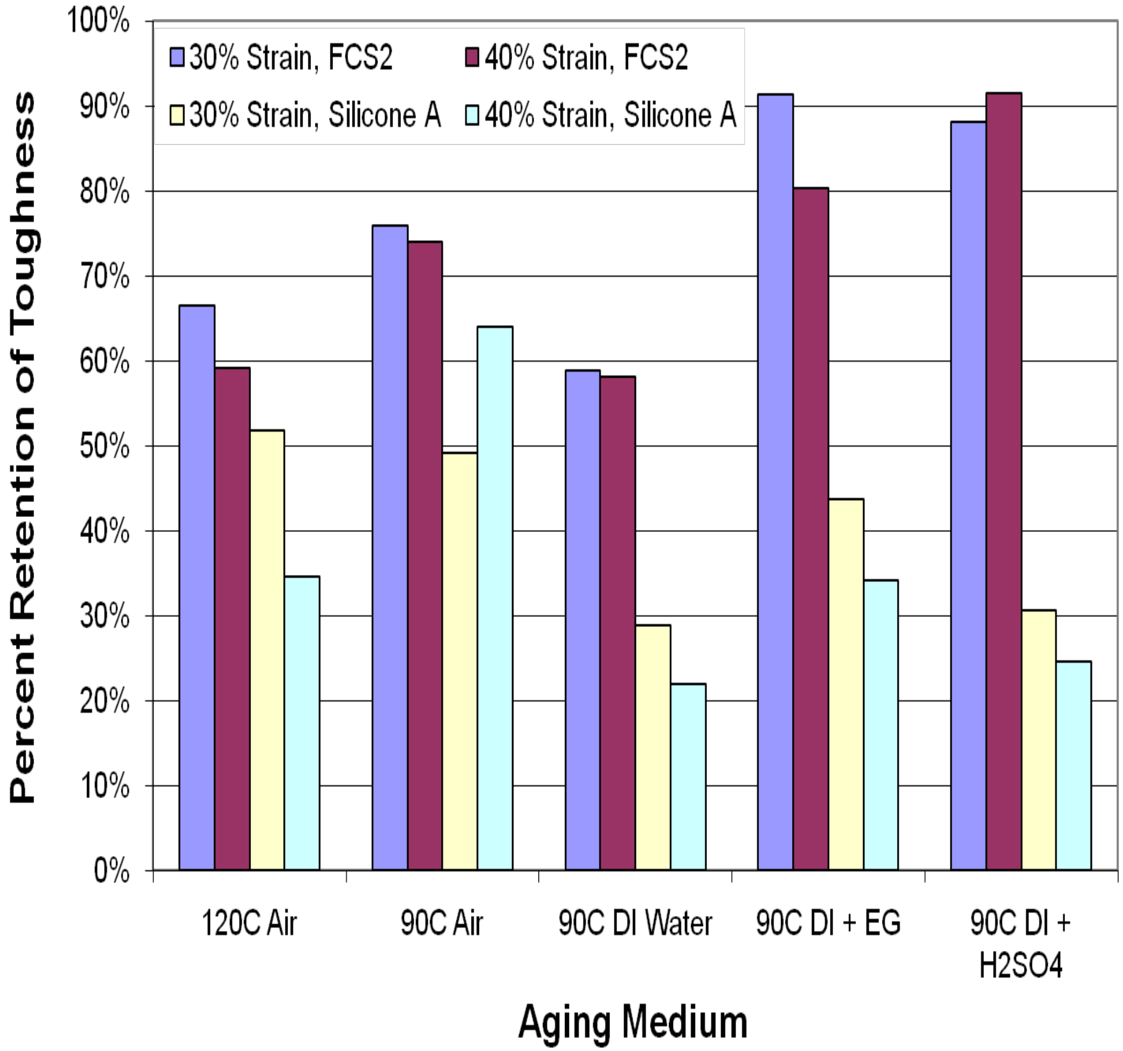
Bulk Material Testing

Accomplishments and Progress

Comparison with a Fuel Cell Grade Silicone

- 8 weeks (~ 1300 hours)
- Specimens are aged while subjected to bending
  - Results in maximum tensile strain of either 30 or 40%
- Toughness accounts for combined changes in tensile strength and elongation
- Two key results:
  - Strain level has less effect on FCS2 than on Silicone A
  - FCS2 exhibits better retention of toughness



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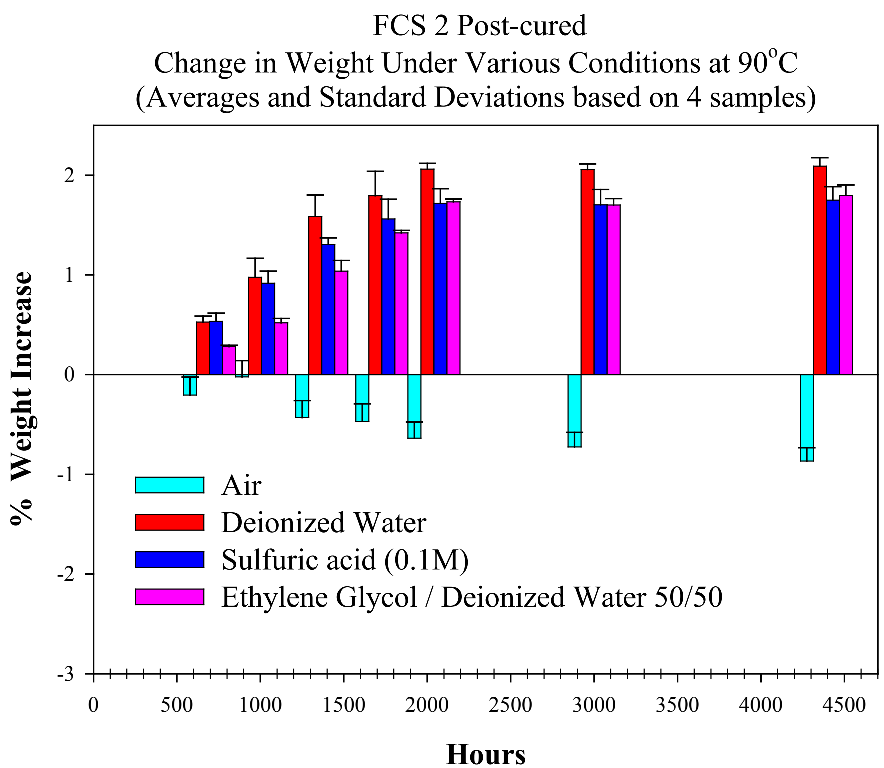
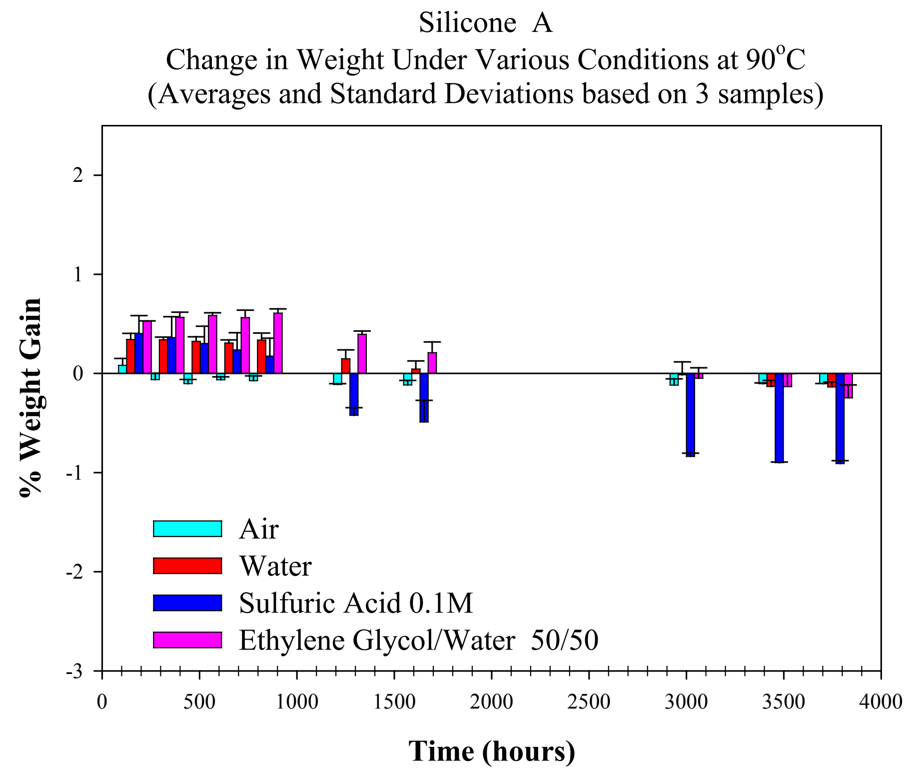
Accomplishments and Progress

Comparison with FC Grade Silicone @ 90 C

- Measuring mass uptake with time
- All changes for both materials are small
- FCS2 stabilizes after 2000 hours
- Silicone A is stable in air, but may not be stable in the aqueous environments





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Accomplishments and Progress

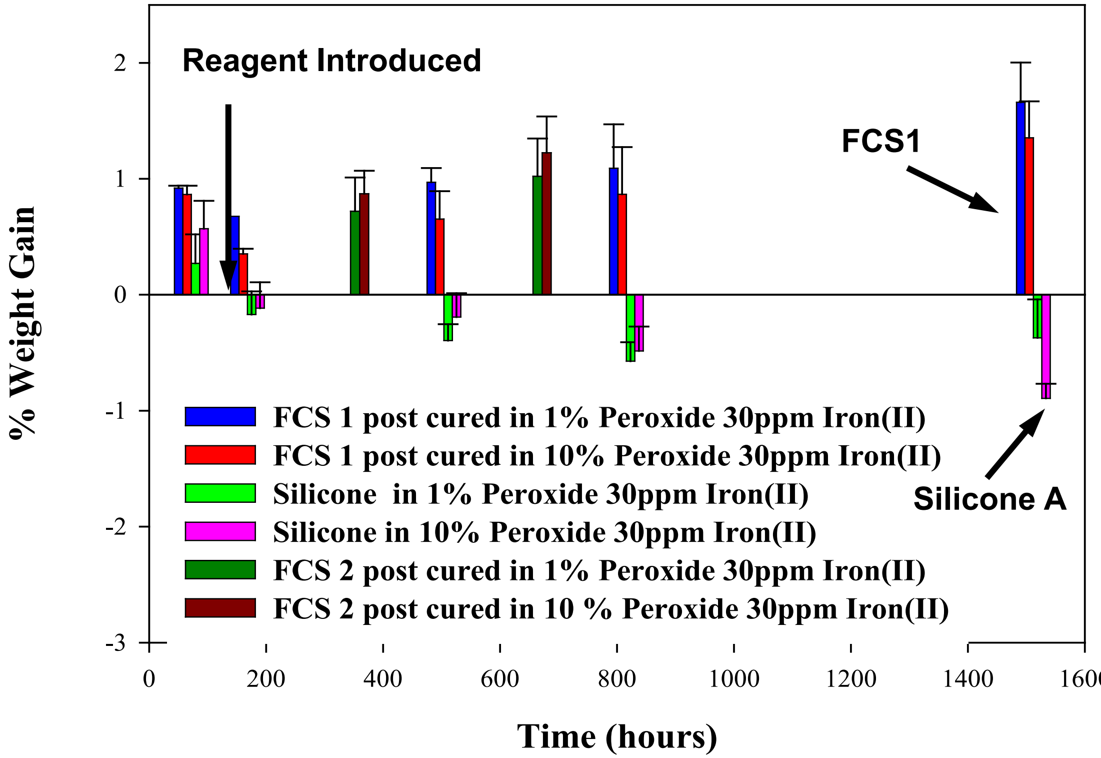
Testing at VT – Degradation using Fenton's Reagent

Experimental Setup:

- Water bath set at 90 ˚C,
- Solution of 1% and 10% peroxide,
- 30 ppm per mass of solution of Iron II sulfate heptahydrate,
- Each sample is immersed in the peroxide solution
- The peroxide solution is replaced every week.

Key Results:

- Shortly after reagent introduction, Silicone A transitions from weight gain to weight loss
- Weight gain for FCS1 in peroxide solution is similar to that seen in other immersion media after 1500 hours
- Silicone A is affected by the 10% solution



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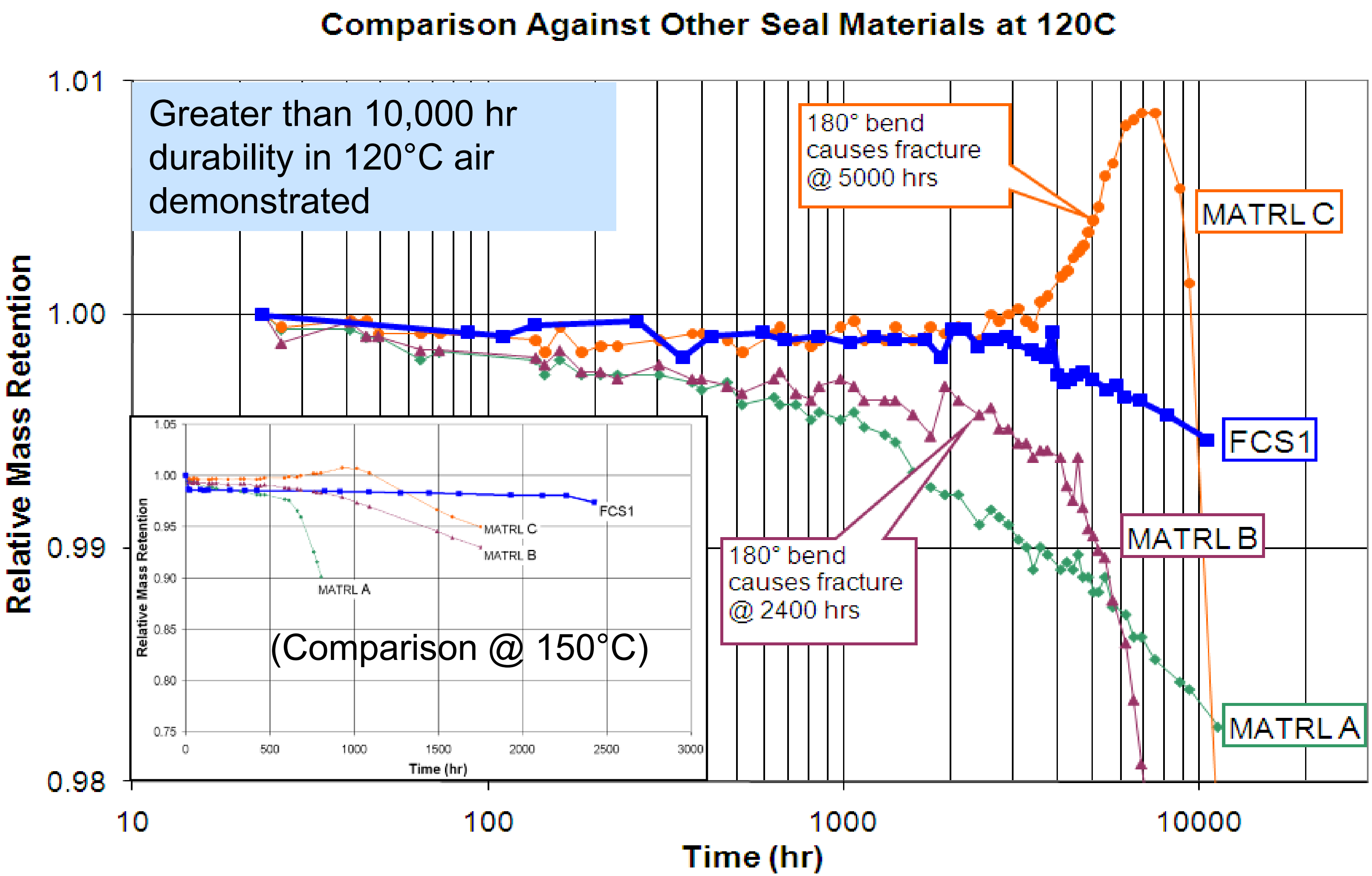
Accomplishments and Progress

Thermal Stability in Air @ 120 C

- FCS1 compared with three EPDM-based fuel cell seal materials (A, B, & C)



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Compression Stress Relaxation (CSR) Testing

Accomplishments and Progress

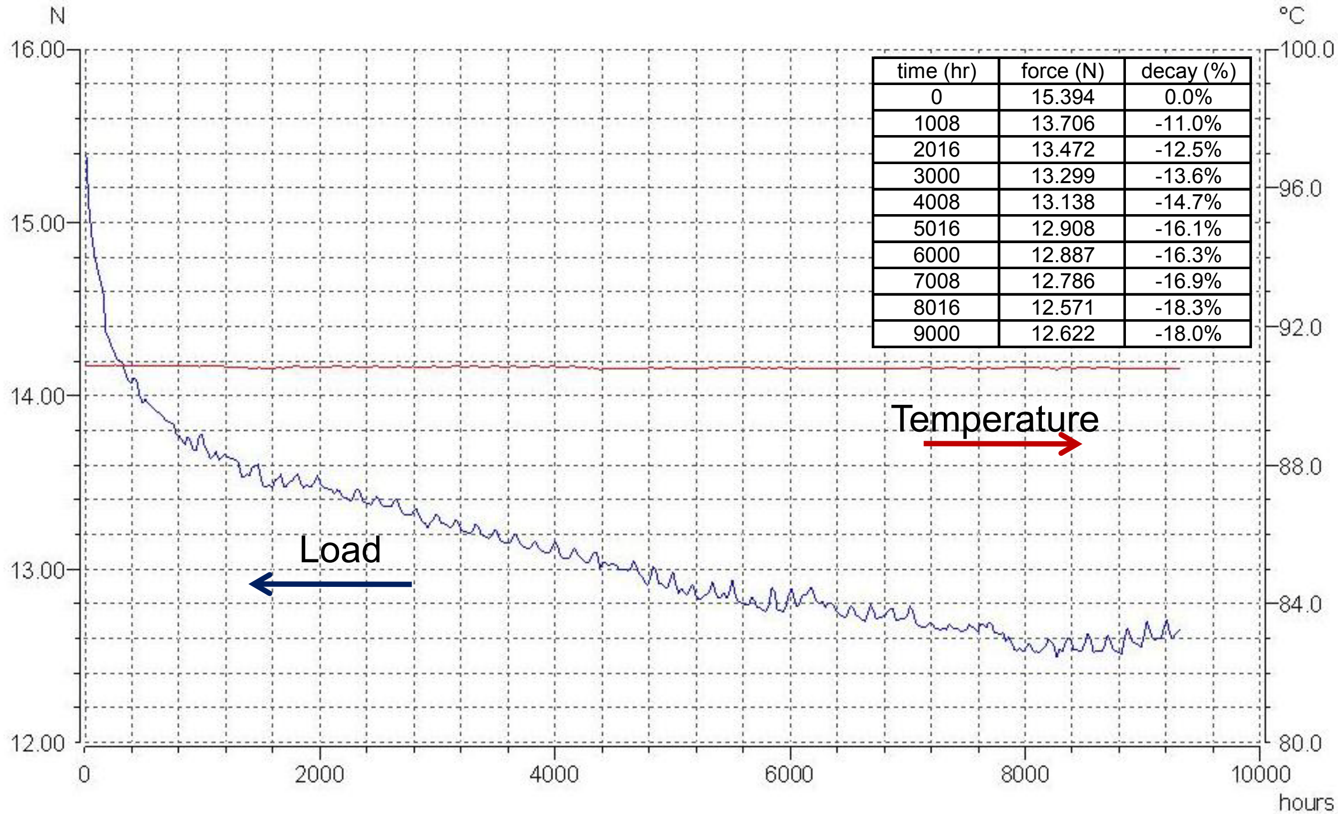
Subscale seal testing

- **Sub-scale Molded O-ring Seals (SMORS)**
  - Seal liquid injection molded over substrate by FNGP
  - Produced on same equipment used to make full-size prototypes
  - Used for compression testing
  - Single bead facilitates detection of leaks
  - Cross-section mimics full-size part
  - Used to verify results obtained in bulk material tests

Accomplishments and Progress

CSR Testing on FCS2 SMORS @ 90 C in Air

1. Continuous CSR @ 90 ˚C in air
2. 19.4% compression
3. 15.4 N initial loading force
4. Automotive operating lifetime (< 20% decay after 9000 hrs)



time (hr)	force (N)	decay (%)
0	15.394	0.0%
1008	13.706	-11.0%
2016	13.472	-12.5%
3000	13.299	-13.6%
4008	13.138	-14.7%
5016	12.908	-16.1%
6000	12.887	-16.3%
7008	12.786	-16.9%
8016	12.571	-18.3%
9000	12.622	-18.0%

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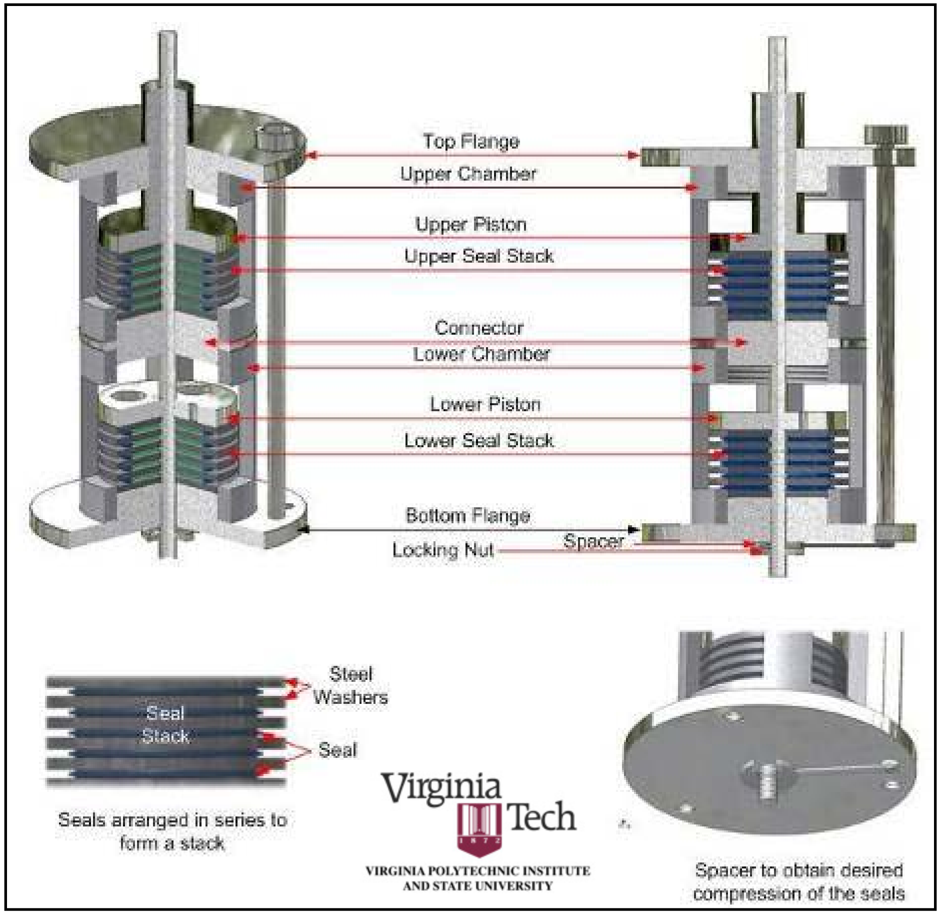
CSR Fixture: Original & Modified Design



Micrometer to precisely set and hold compression

Environmentally sealed load cell for continuous monitoring of seal load

Lower sample chamber and seal stack retained

Original Design

- Advantage: Tracking of both compressive (momentary) properties and load relaxation over time in identical environments and one rig
- Disadvantage: Intermittent measurements can introduce noise when working with low durometer seals

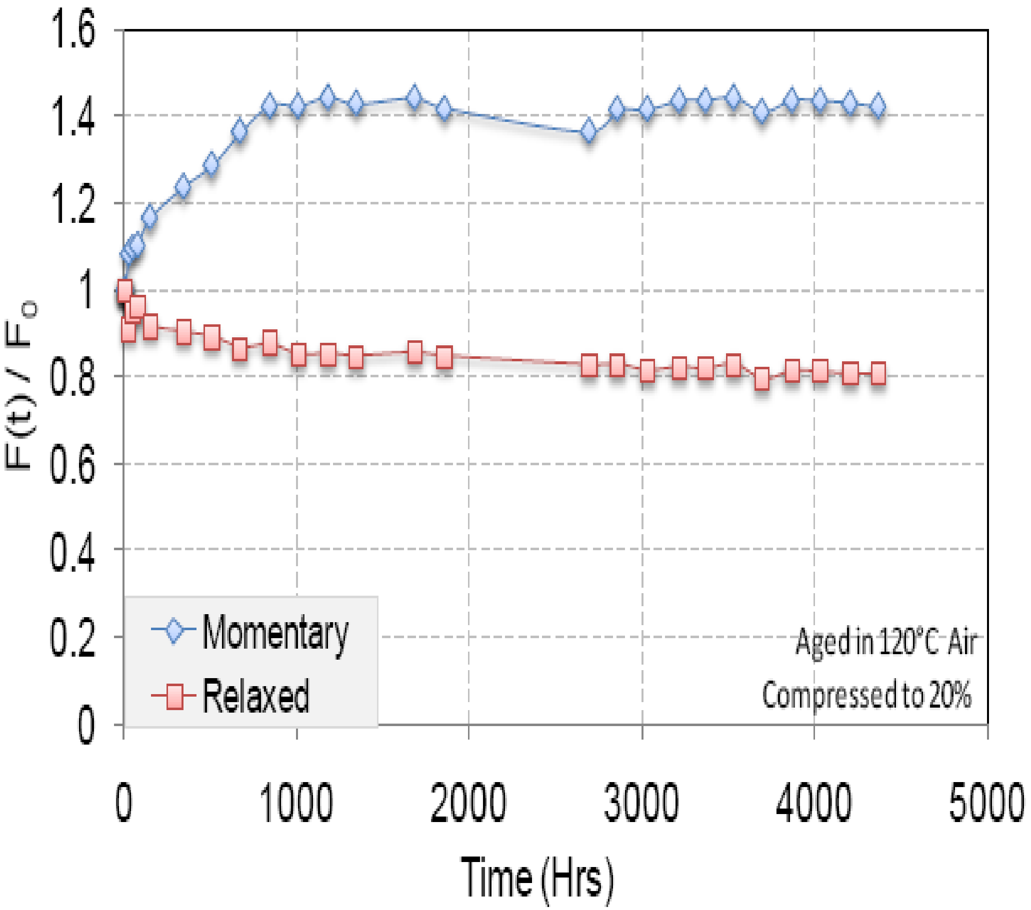
Modified Design

- Advantage: Continuous load relaxation measurement should reduce noise in the data
- Disadvantages:
- Samples used for tracking changes in momentary properties aged separately
- Introduces other potential sources of noise

Accomplishments and Progress

CSR Testing on FCS2 – Intermittent Measurements

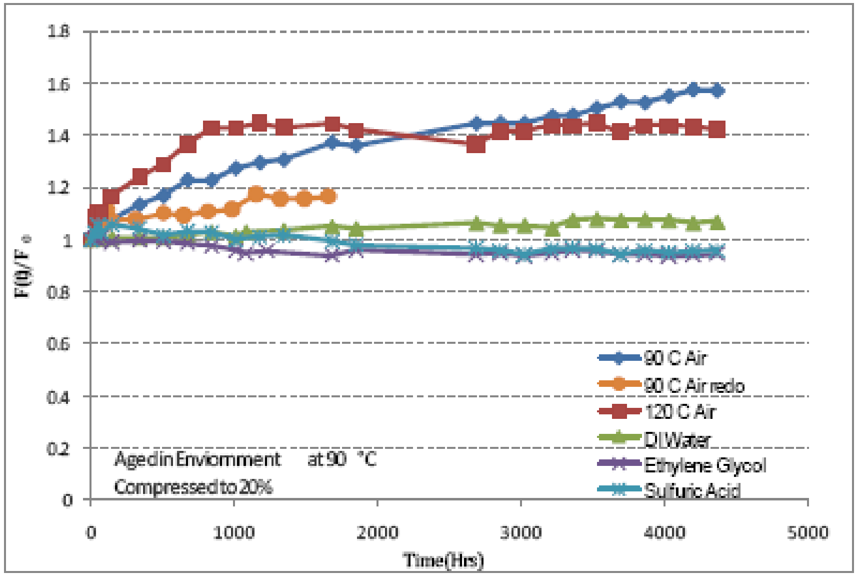
- Momentary data tracks compressive modulus at 20% compression
- Relaxed data tracks load relaxation under a constant 20% compression
- Restart of a previous test
  - Restarted due to excessive noise in the early intermittent data
  - Result of previous test: 30% load loss after 6000 hours
- Result of current test:
  - 20% load loss after more than 4000 hours
  - Rate of load loss: 1.2% per 1000 hours
- Significance of Result
  - Less than 30% load decay expected after 10,000 hours



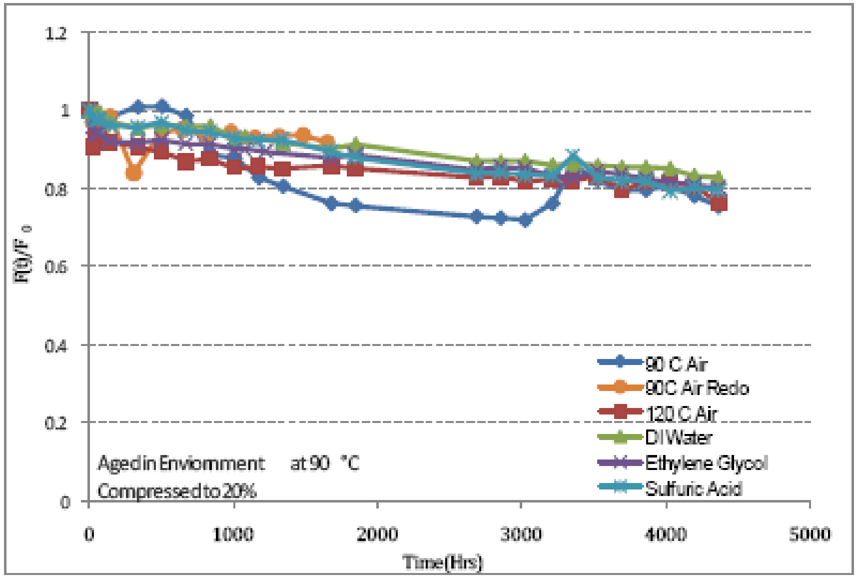
Accomplishments and Progress

CSR Testing on FCS2 – Intermittent Measurements

- Little effect seen for aging in fluid environments
  - Max load loss after more than 4000 hours is < 25%
- The effect of air exposure manifests in the momentary data as an increasing modulus
  - Possible causes being investigated
  - Correlation to load relaxation results appears to be weak
- Maximum rate of load loss: < 6% per 1000 hours in 0.1 M H_2SO_4 (pH=1)



Momentary



Relaxed

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Full-size Component Development and Test

Accomplishments and Progress

Full-Size proto-type development

- Staged Approach to Component Development
- Seals over molded onto plastic subgasket
  - Benefit: Industry alignment
  - 1700 hours of in-cell testing completed
- Integrated Molded Seal (IMS) MEA
  - Using hot-runner (low-volume) tooling
    - Parts are being produced in a variety of MEA configurations
    - Purpose: Determine the effects of altering parameters such as GDL type and configuration
  - Using cold-runner (high volume) tooling
    - Tool design is underway
  - IMS Benefit: Addresses cost by combining
    - Unitization of the MEA
    - Molding of the seal
    - Placement of the sealing features
  - Primary IMS Challenges
    - Control of flash into the active area
    - Rapid part removal from the mold

Full Size Prototype Made Using Hot-Runner LIM* tool

*LIM = Liquid Injection Molding

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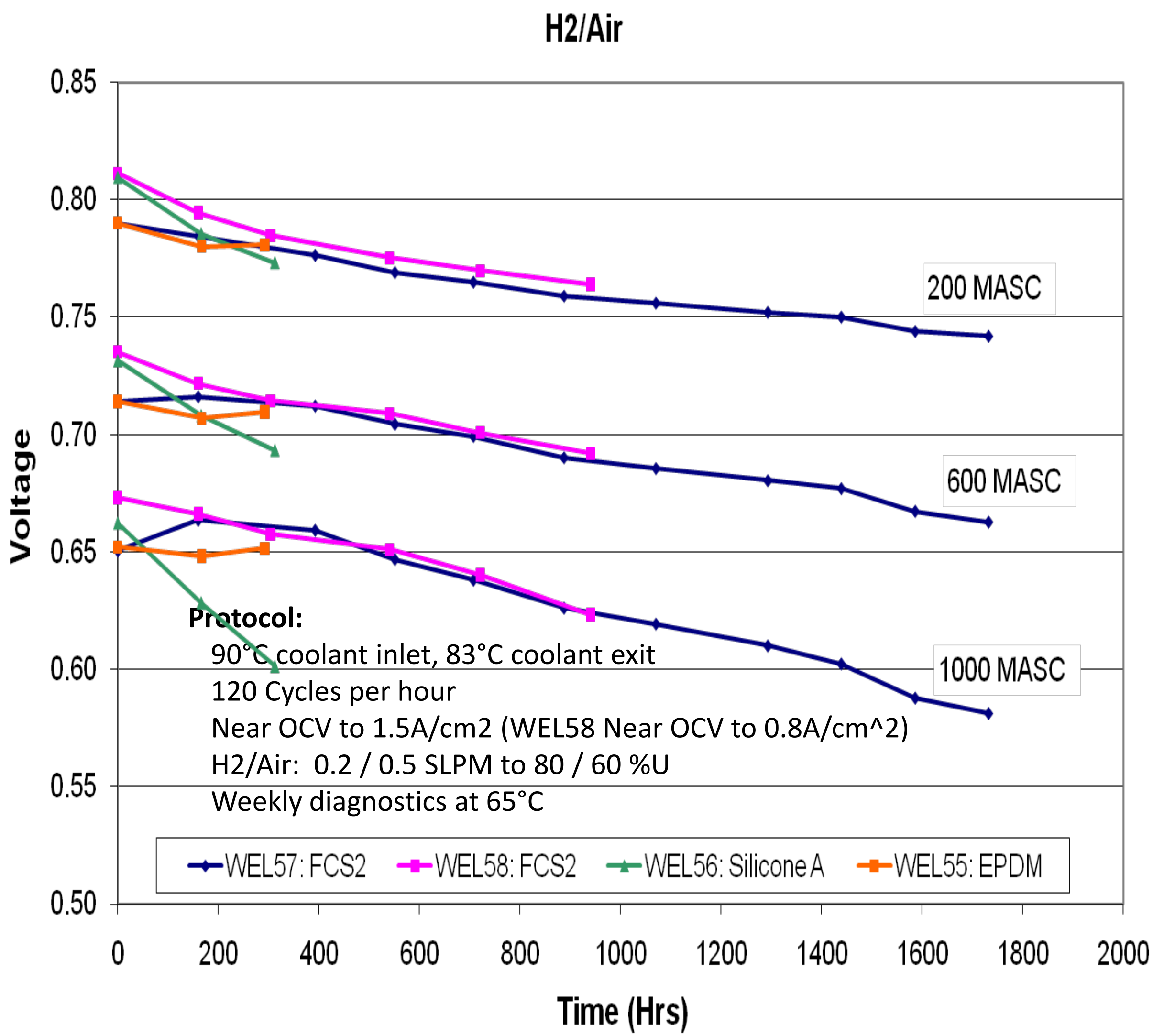
Accomplishments and Progress

Single Cell Testing: FCS2 over molded on PEN

- Over 1700 hours accumulated before WEL 57 removed due to a test stand issue
- Accelerated cyclic test designed to stress all cell components
- At BOL, FCS2 cells have similar performance to baseline EPDM and silicone cells
- A review of the weekly diagnostics indicates performance decay is not attributable to seals
- Additional testing is planned



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Future Work

- Out-of-Cell testing
  - Continue benchmarking against other materials
- Full-size prototype development
  - Additional over-molding of seals on subgaskets for additional full-size in-cell evaluation
  - Perform additional molding trials with hot-runner tooling
    - Incorporate any lessons learned in the final prototype design
  - Complete cold-runner tool fabrication for final prototype design
- In-cell testing
  - Complete 2000 hr verification testing using full-size seals
  - Complete post-test analysis for in-cell testing

Summary

- <u>Project Goal</u>
  - Develop a durable and low-cost PEM fuel cell seal material for broad PEM fuel cell industry use
- <u>Materials selection and development</u>
  - Material properties for available candidates meet most ultimate project goals
  - FCS3 expected to meet all key project goals
  - Scale-up and commercialization plans are underway
- <u>Out-of-cell testing</u>
  - Bulk material testing
    - Over 10,000 hours 120°C air durability demonstrated
    - Stability in FC relevant fluid environments at 90°C demonstrated
  - CSR Testing
    - Over 6,000 hours durability at 120°C in air demonstrated
    - Over 4,000 hours durability at 90°C in FC relevant fluid environments demonstrated
- <u>In-cell testing</u>
  - Over 1,700 hours of in-cell verification testing completed
    - No evidence of cell contamination by the seals present in cell diagnostics
  - Additional testing planned

Acknowledgements

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  - Dr. Robert Moore
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