



2006 DOE Hydrogen, Fuel Cells & Infrastructure Technologies Program Review

Development of Polybenzimidazole-based High Temperature Membrane and Electrode Assemblies for Stationary Applications

May 16, 2006
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FC-2

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OVERVIEW

Timeline

- ❖ Project start date Aug 2003
- ❖ Scheduled end date July 2006
- ❖ Request to extend to FY07 due to funding cuts in 06

Budget

- ❖ Total project funding \$7.29 M
 - DOE share \$ 5.84 M
 - Plug share \$1.46 M
- ❖ Funding received in FY04 \$1.50 M
- ❖ Funding received in FY05 \$2.05 M
- ❖ Funding for FY06 \$0.94M
- ❖ Funding requested for FY07 \$0.89

Barriers

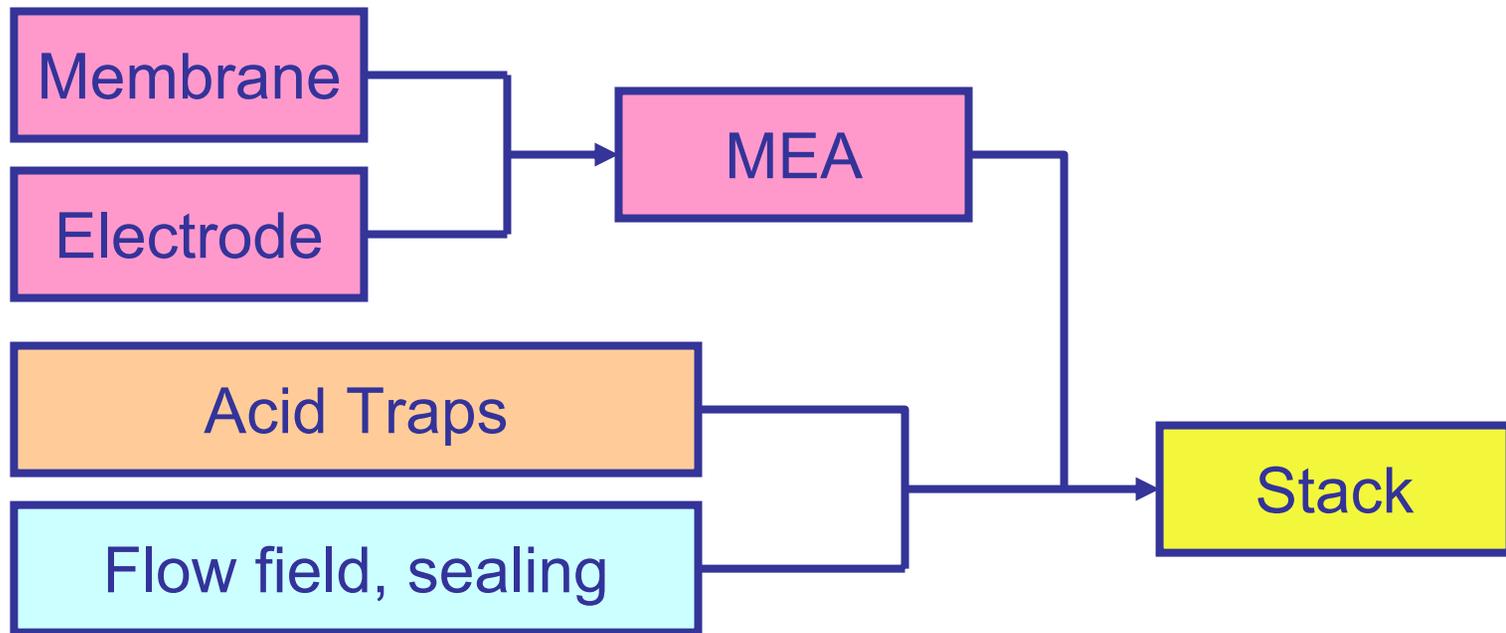
- ❖ O. Stack material and manufacturing cost
- ❖ P. Durability

Subcontractors

- ❖ Rensselaer Polytechnic Institute (RPI)
 - Polymer Science Laboratory
 - Fuel Cell Center
- ❖ PEMEAS
- ❖ Albany Nano Tech
- ❖ Entegris
- ❖ University of South Carolina

OBJECTIVES

- ❖ To identify and demonstrate an MEA based on a high-temperature polybenzimidazole (PBI) membrane that can achieve the performance, durability and cost targets required for stationary fuel cell applications



APPROACH

- ❖ Membrane (Task 1-4) *70% complete*
 - ✓ Formulate and characterize polymers
 - ✓ Improve membrane mechanical stability
 - Scale up process and fabricate full size MEAs
- ❖ MEA (Task 5-8) *60% complete*
 - ✓ Conduct 50cm² screening tests at RPI
 - ✓ Conduct parametric tests to fully characterize MEA performance
 - Assemble and test a full size short stack
- ❖ Stack (Task 9-12) *50% complete*
 - ✓ Characterize acid absorbing materials
 - ✓ Optimize flow fields and sealing
 - ✓ Develop novel electrodes using nanotechnology
 - Cost assessment

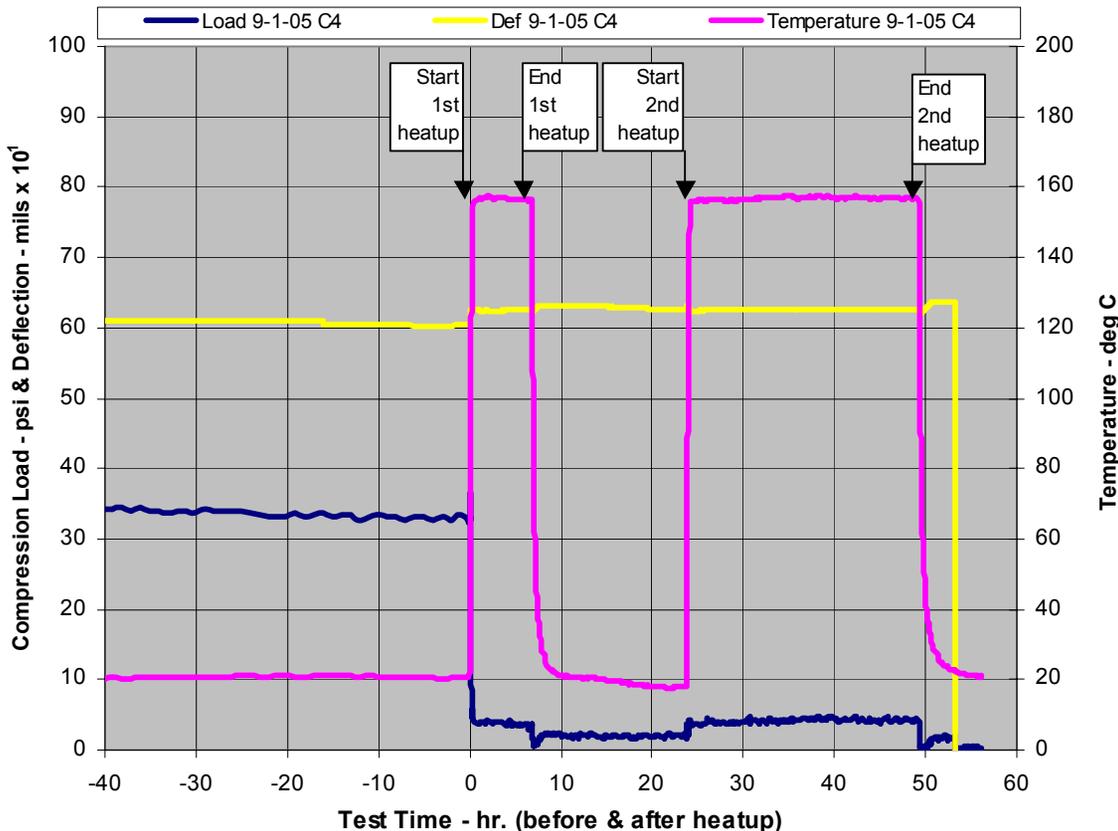
- ✓ Progress made

TECHNICAL ACCOMPLISHMENTS

MEMBRANE (TASK 2)

- ❖ MEA mechanical behavior studied at room and operating temperature
- ❖ Stress relaxation of small coupons (non- operating)

MEA Load Loss with Temperature of RPI Unfilled Membrane
After 600 hr. of Stress Relaxation



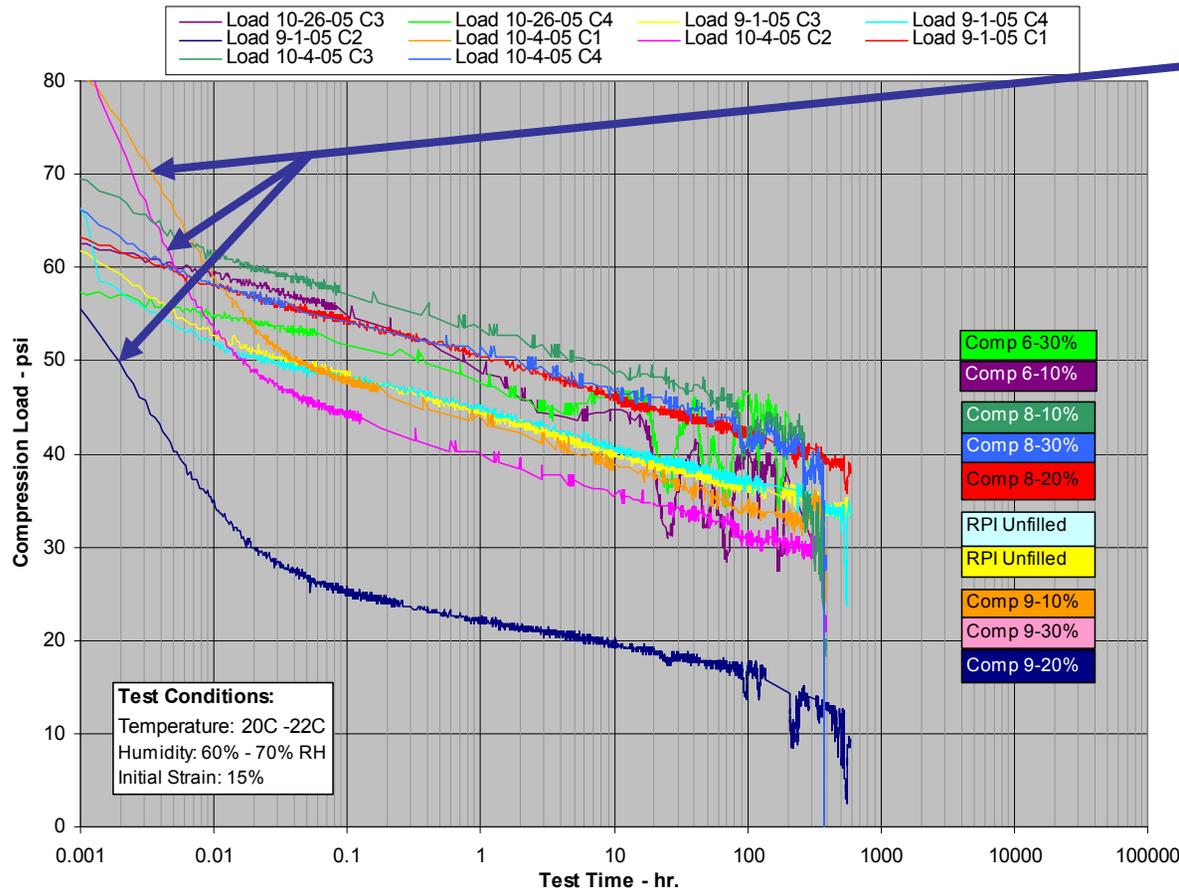
- MEA stress relaxed to 30 psi in 600 hours, and further reduced below 5 psi during heat up to operating temperature
- MEA behavior at room temperature is important for stack assembly.
- MEA behavior during heat up is important for stack design

TECHNICAL ACCOMPLISHMENTS

MEMBRANE (TASK 2)

- ❖ RPI prepared 4 filled membranes using the best formulation (Type 2) from Task 1
- ❖ MEA stress relaxation characterized at room temperature

Stress Relaxation Filled & Unfilled Test MEAs



Composition 9 relaxed faster in initial minutes, but exhibited the same long term behavior

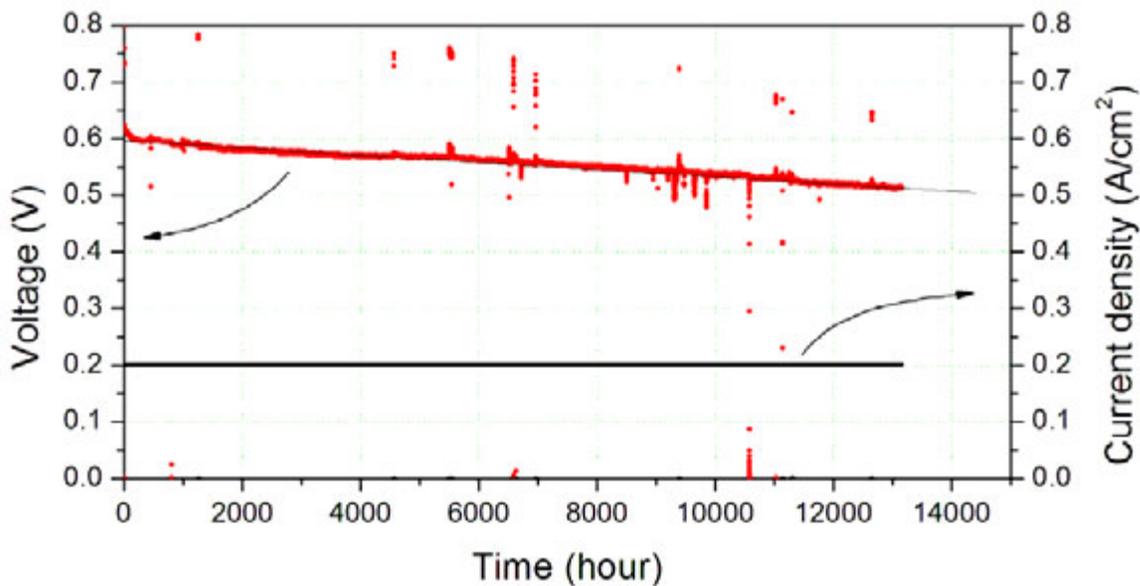
- All membranes filled and unfilled membranes stress relax to very low loads
- Low load enables bonding concept for stack



TECHNICAL ACCOMPLISHMENTS

50 CM² CHARACTERIZATION (TASK 5)

- ❖ Long term durability @ 120°C- Type 2 unfilled membrane
- ❖ H₂/ Air, No humidification



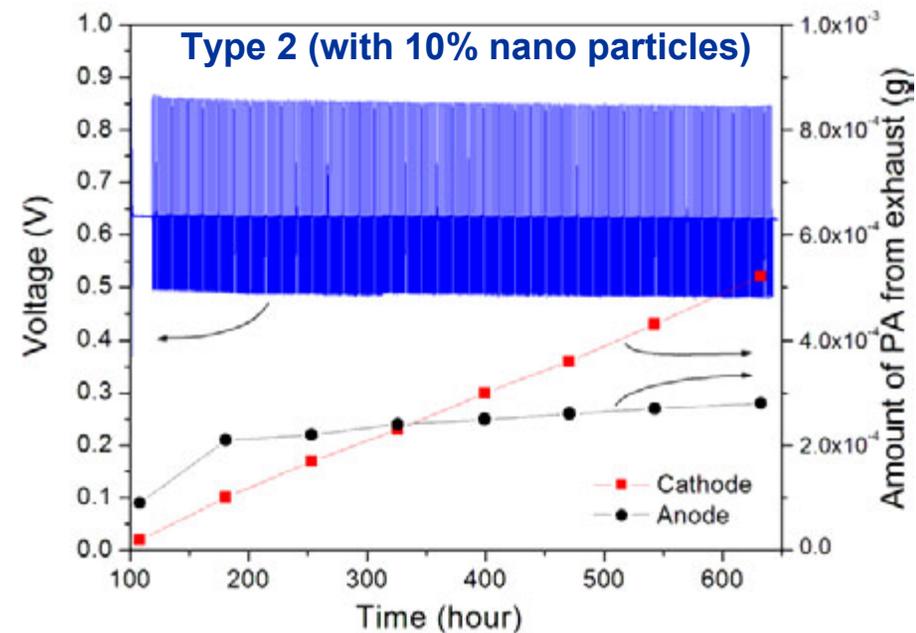
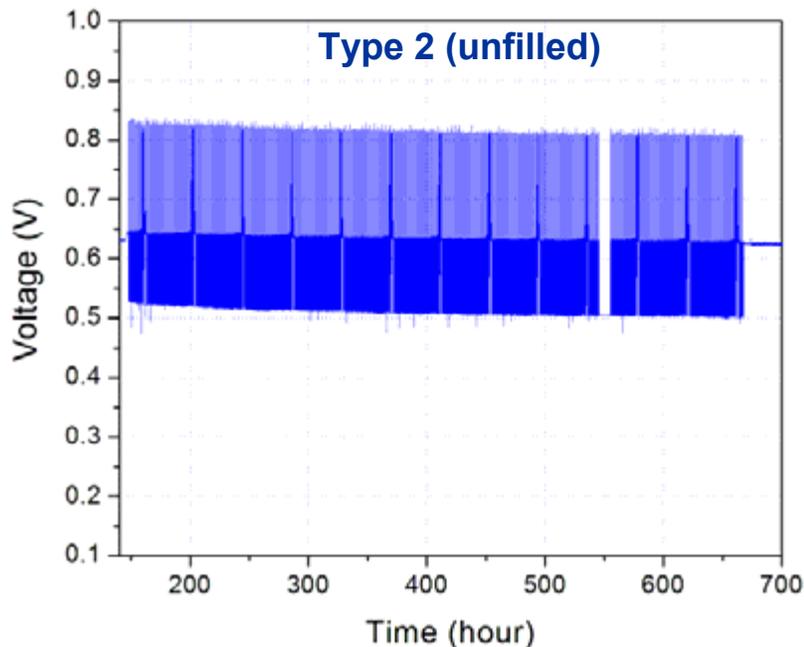
- Steady State Operation
- Degrade rate 6.3 μ V/hour projecting a 32,000 hour stack life
- 13,000 hour complete



TECHNICAL ACCOMPLISHMENTS

50 CM² CHARACTERIZATION (TASK 5)

- ❖ Load cycle testing unfilled and filled comparison- encouraging results
- ❖ 160°C 2 minutes at OCV, 30 minutes at 0.2 A/cm², 30 minutes at 0.6 A/cm²
- ❖ Unfilled projected stack life 6,000 hours vs. filled at 14,600 hours



Both cells:

1 mg/cm², Pt/ C cathode & anode
H₂/ air
No humidification

Degrade rate:

33 μV/ hour @ OCV
27 μV/ hour @ 0.2 A/cm²
40 μV/ hour @ 0.6 A/cm²

Degrade rate:

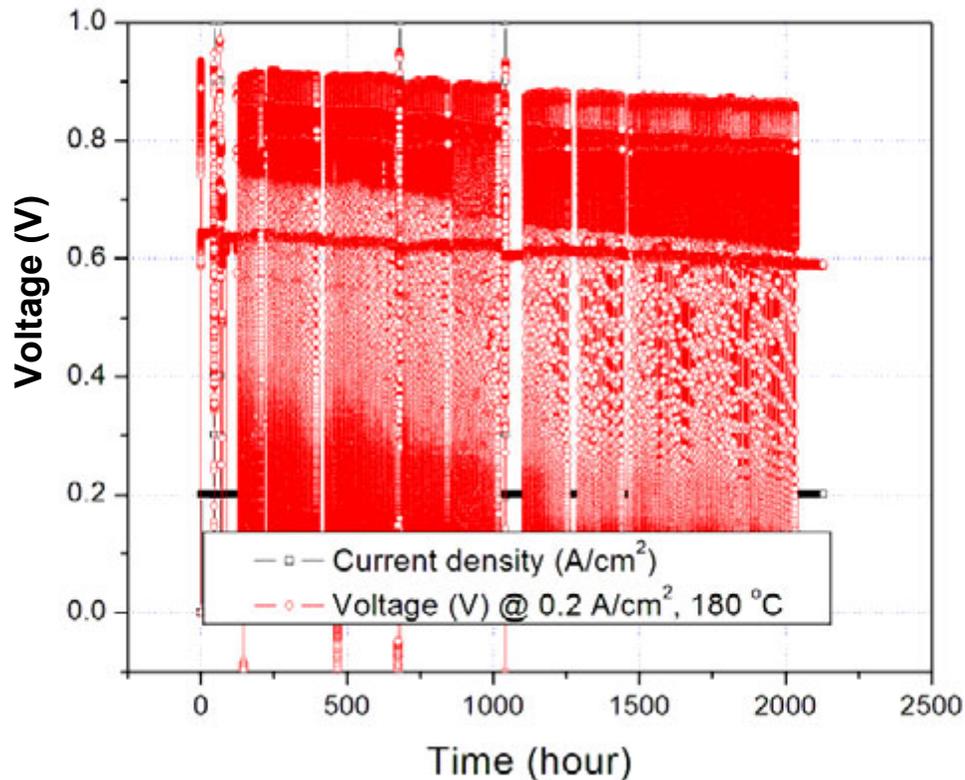
20 μV/ hour @ OCV
12 μV/ hour @ 0.2 A/cm²
19 μV/ hour @ 0.6 A/cm²

Acid loss:

18.2 ng/cm²hr @ cathode
9.8 ng/cm²hr @ anode
28 ng/cm²hr @ total

TECHNICAL ACCOMPLISHMENTS 50 CM² CHARACTERIZATION (TASK 5)

- ❖ Start-up Shut-down testing of a Type 2 membrane
- ❖ 180°C, H₂/air, No humidification

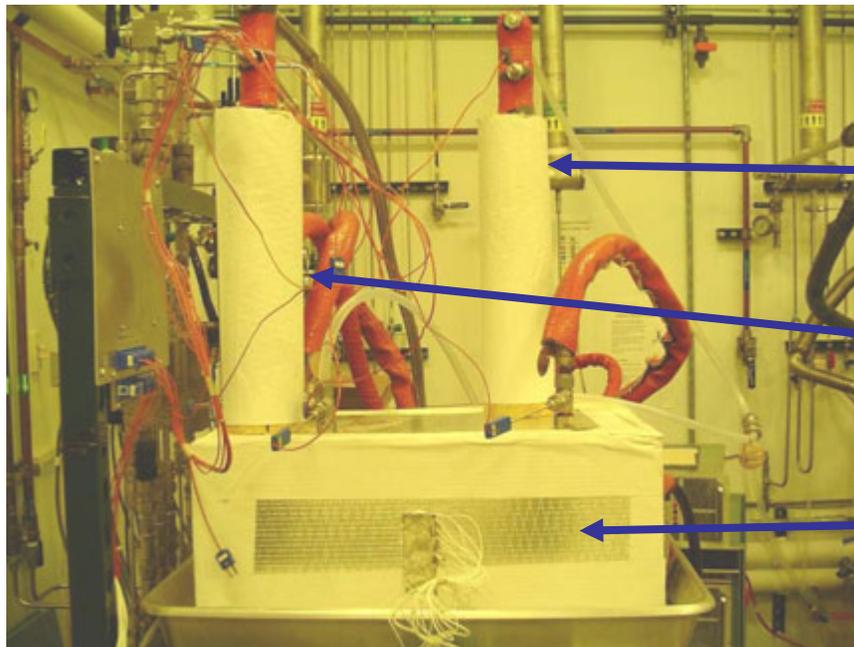


- Over 110 cycles completed
- Degradation rate @ 0.2 A/cm²
~ 0.13mV per cycle
- > 1000 hours total time

TECHNICAL ACCOMPLISHMENTS ACID MANAGEMENT (TASK 9)

- ❖ Acid loss mechanisms defined, loss rates characterized and controllable
- ❖ 2,000 hours of acid trap testing completed on full size Type 2 MEAs
 - 6 non-detects passed the cathode trap
 - 3 non-detects passed the anode trap

Phos acid is trapped preventing contamination of the rest of the system



Cathode Acid Trap

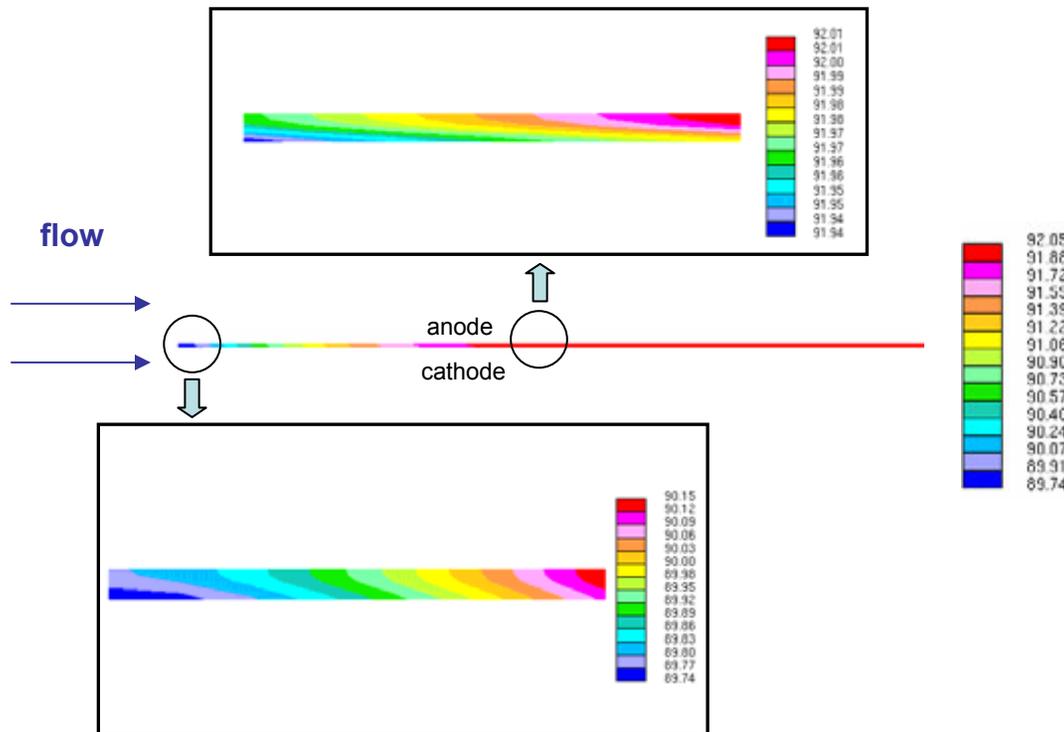
Anode Acid Trap

16 cells 440 cm² module

TECHNICAL ACCOMPLISHMENTS

ACID MANAGEMENT (TASK 9)

- ❖ Predict changes in σ of H_3PO_4 as function of time, temperature & current density
- ❖ Applied equations and parameters used in spreadsheet model to 2D CFD model

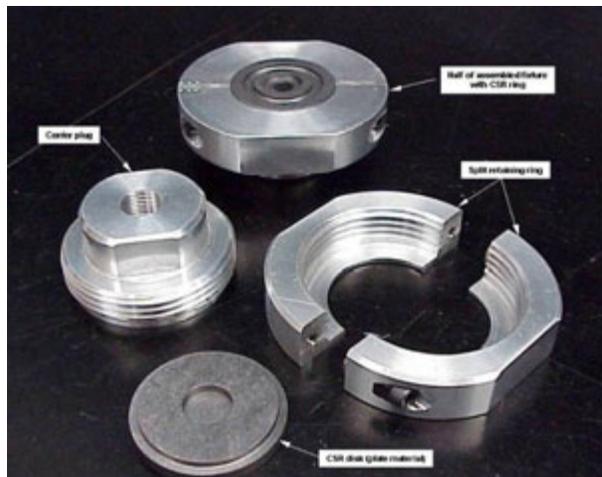


- Model shows change in acid concentration over the length of a channel (89-92%).
- Sensitivity study of known variables when FY07 funding is available.

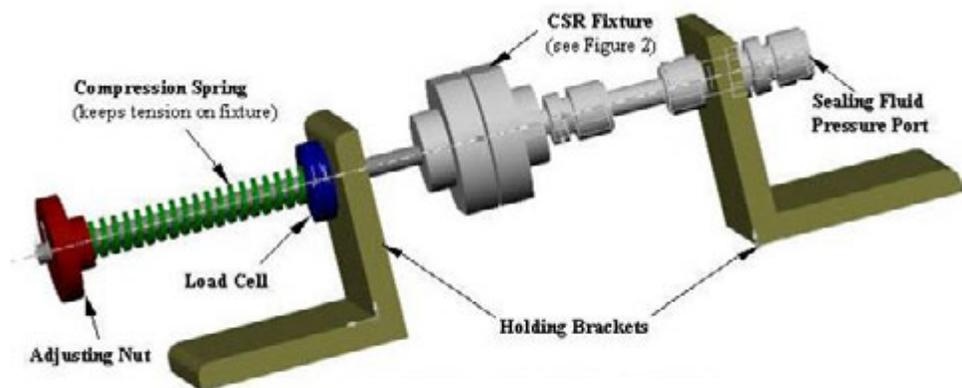
2D CFD model Ave. H_3PO_4 weight (%)

TECHNICAL ACCOMPLISHMENTS STACK DESIGN (TASK 10)

- ❖ Brainstormed plate bonding and sealing concepts
 - Evaluated conductive and non-conductive materials
 - 11 bonded samples exposed to phosphoric acid and coolant at 180°C - 6 failed
 - Lap shear tests and sealing pressure tests performed - 2 survived
 - Large scale bond line optimization in progress with MEAs
- ❖ 2 patents filed regarding aspects of the bond and seal concept
- ❖ Big cost saving potential by eliminating gaskets, assembly time, end hardware



CSR holding fixture

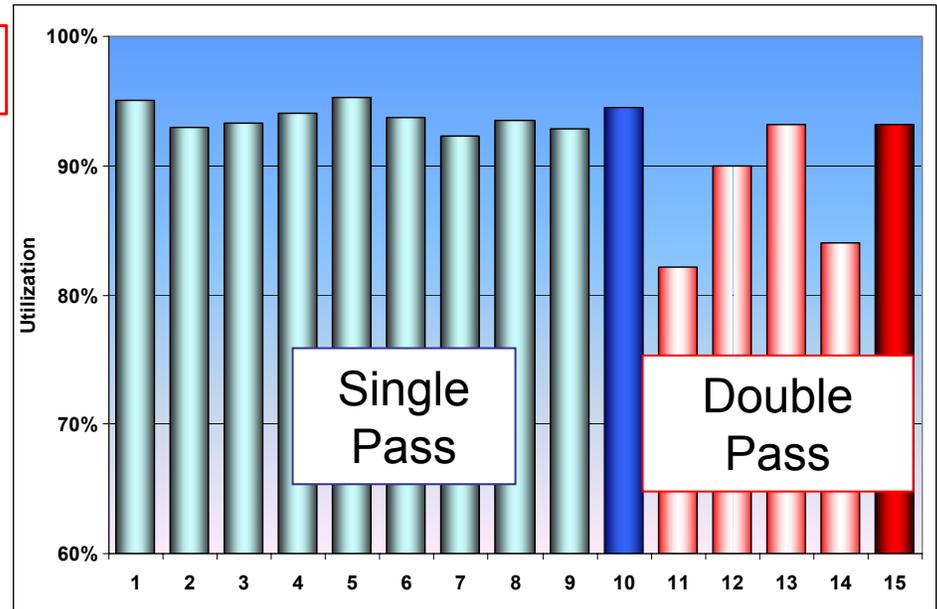
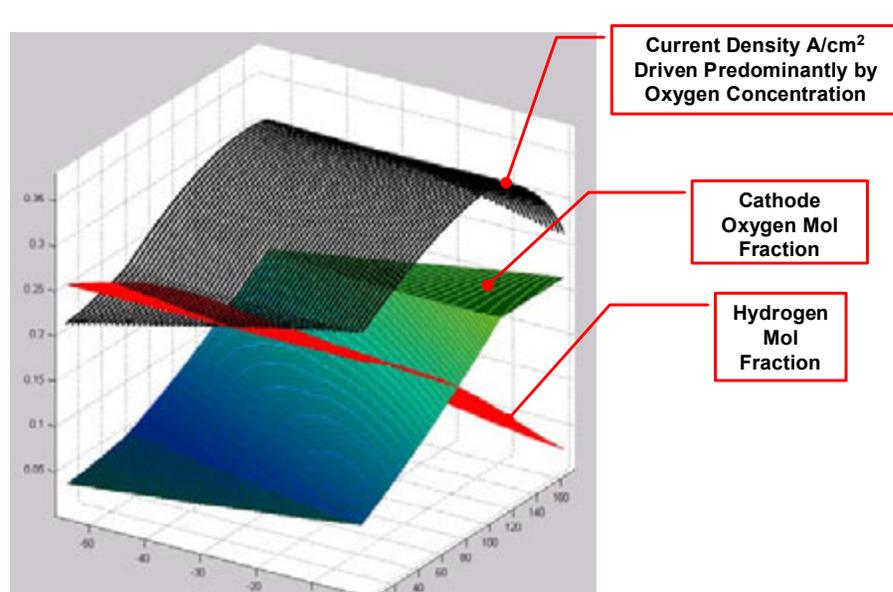


Sealing Pressure Tester

TECHNICAL ACCOMPLISHMENTS

PBI SPECIFIC FLOW FIELD DESIGN (TASK 10)

- ❖ Modeled and tested 50 cm² test cells to obtain optimal performance
- ❖ Modeled and tested full size flow field geometry in single and double-pass



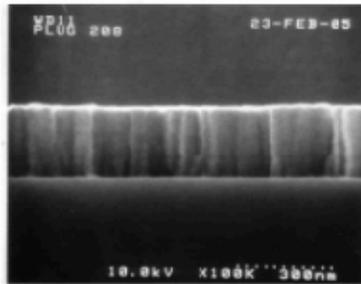
- Single pass, single cells showed little sensitivity to channel geometry achieving greater than 90% utilization
- Double pass configuration showed significant variability in single cell, but offers benefits with multiple cells



TECHNICAL ACCOMPLISHMENTS ELECTRODE (TASK 11)

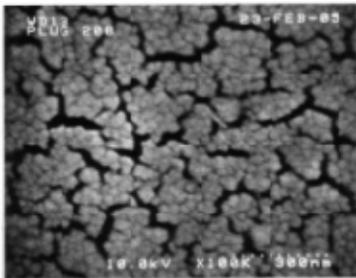
- ❖ Sputter deposition and spray treatments created with extremely low Pt loaded electrodes
- ❖ Stacked structures created with 2-7 $\mu\text{g}/\text{cm}^2$ Pt were tested in half-cell rig
- ❖ Activities were discontinued at ANT due to near term viability
- ❖ Activities were discontinued at RPI's Fuel Cell Center due to funding cuts

Cross-sectional View

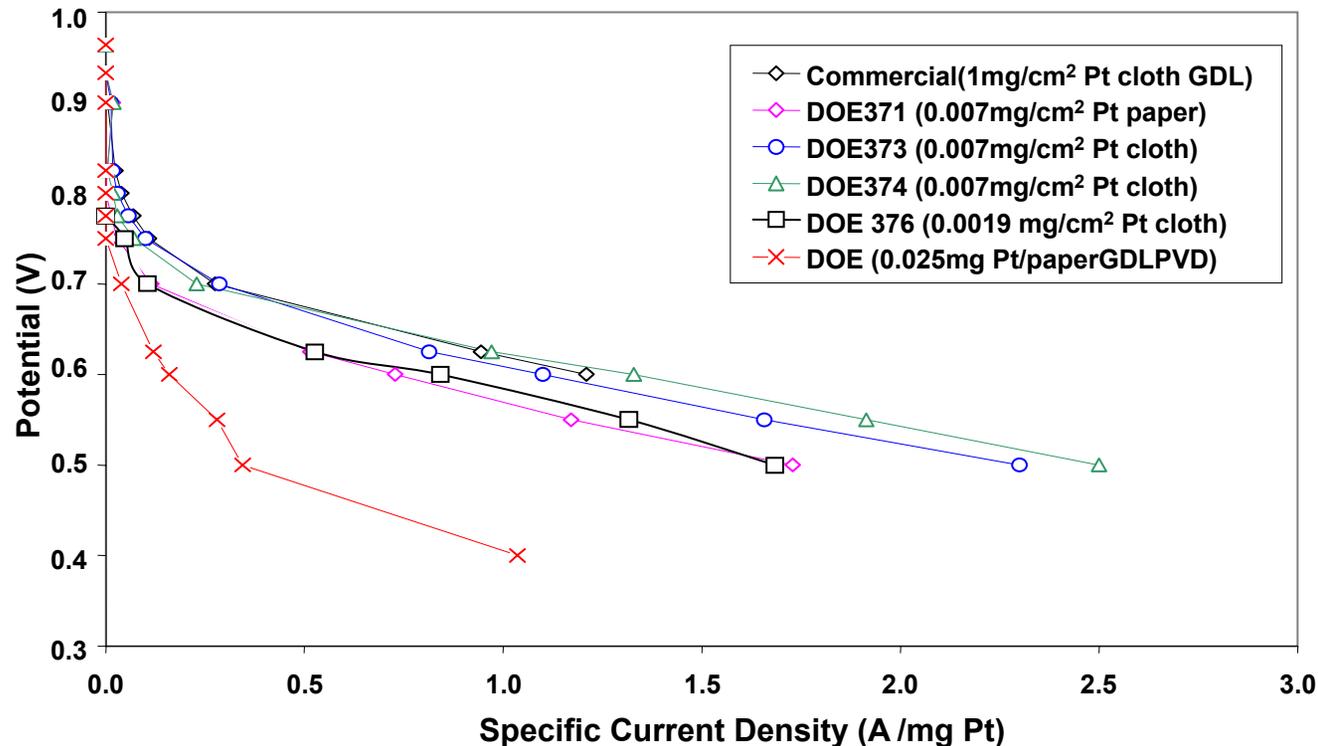


80 minutes Pt

Top View



Performance Comparison of Commercial and ANT Electrodes



TECHNICAL TARGETS- STATIONARY

Characteristic	Units	Calendar Year			
		2004 Status	2005 Status	2006 Outlook	2010 Target
Membrane conductivity, operating temperature	Ohm-cm ²	0.10	0.10	0.10	0.10
Oxygen crossover	mA/cm ²	5	5	5	2
Hydrogen crossover	mA/cm ²	5	5	5	2
Cost	\$/ kW		50	50	5
Operating temperature	°C	160	160	160	170
Durability	Hours	5,000	>15,000	>15,000	40,000
Survivability	°C	-20	-30	-30	-40

- **Conductivity:** Meets DOE target at 160°C
- **Cross over:** Need to confirm with final membrane
- **Cost:** PEMEAS and Entegris cost estimates delayed due to funding cuts on FY 06.
- **Temperature:** Routinely run at 180°C
- **Durability:** 20,000 hours demonstrated by PEMEAS in 50cm² testing, 13,000 hours demonstrated by RPI at 120°C
- **Survivability:** Data available from PEMEAS

RESPONSES TO REVIEWERS' COMMENTS - 2005

- ❖ “ With one year into the program, fundamental problems still exist: acid evaporation, low power density, and membrane mechanical properties.”
 - Acid retention is not the issue, acid management is the issue- our solution was presented at the 2005 review. A PAFC consultant helped Plug Power and PEMEAS understand acid loss, and in reviewing the literature, they dealt with it just as we need to.
 - Agree that mechanical stability is an issue and is clearly our focus.
 - We believe we have a stack design that will accommodate the SR of the membrane, and plan to demonstrate this.
 - Increase in power density is not likely due to the fundamental limitations of this PAFC type fuel cell
- ❖ “ Another reviewer commented that the acid management is well addressed.” “Acid loss appears to be manageable.”
- ❖ “ There is substantial effort remaining to be completed and no indication of a comprehensive cost analysis.” “ Plug Power should do a ballpark screening on cost earlier in the program.”
 - A cost estimate from PEMEAS was planned to occur in the later part of the program but the 2006 funding cuts have moved this activity to FY07, pending further funding. Costing can not be performed until MEA design is finalized but commercial costs have been assessed.
 - Entegris has provided a cost estimate for production quantities of bonded cells that significantly reduce cost.

FUTURE WORK

Remainder of 2006:

- ❖ Continue operational characterization of Type 2 membrane.
- ❖ Identify and confirm possible mechanism for improved degrade of filled membranes
- ❖ Build full size prototypes and demonstrate stack sealing concept with Entegris
- ❖ Complete long term acid trap testing

2007:

- ❖ Re-start work at USC on acid model
- ❖ Scale-up membrane & procure MEAS from PEMEAS utilizing their latest electrode
- ❖ Test a full size module with improved membrane, flow field and sealing concept
- ❖ PEMEAS and Entegris will deliver price estimate for MEA and stack
- ❖ Demonstrate 1,000 hours life with low degradation rate and project 40,000 hours life

PUBLICATIONS AND PRESENTATIONS

Publications:

High Temperature Polybenzimidazoles for Fuel Cell Membranes via a Sol-gel Process.

Xiao, L.; Zhang, H.; Scanlon, E.; Ramanathan, L.S.; Choe, E.W.; Rogers, D.; Apple, T.; Benicewicz, B.C. *Chem. Mater.*, 2005, accepted, in revision.

Advanced MEA Testing Systems for PEMFC. Gordon Research Conference on Fuel Cells, Bryant University, Smithfield, RI, 7/19/05. D. Giuliano, L. Xiao, B.C. Benicewicz.

Presentations:

Synthesis and Evaluation of Poly(2,5-benzoxazole) as a Proton Exchange Membrane Fuel Cell Candidate. ACS Meeting, Chemical Education Division, San Diego, CA, 3/15/05.

M. Kienzler, E.W. Choe, E. Scanlon, B.C. Benicewicz.

Advanced MEA Testing Systems for PEMFC. Gordon Research Conference on Fuel Cells, Bryant University, Smithfield, RI, 7/19/05. D. Giuliano, L. Xiao, B.C. Benicewicz.

Study of Steady-State and Dynamic Long Term Stability of PBI-Based Membranes.

Gordon Research Conference on Fuel Cells, Bryant University, Smithfield, RI, 7/19/05. S. Yu, B.C. Benicewicz.

Study of Steady-State and Dynamic Long Term Stability of PBI-Based Membranes.

Gordon Research Conference on Fuel Cells, Bryant University, Smithfield, RI, 7/19/05. S. Yu, B.C. Benicewicz.

High Temperature Technology Development, 2005 Fuel Cell Seminar, Palm Springs, CA, 11/17/05, R. Staudt

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