

Process Intensification of Hydrogen Unit Operations Using an Electrochemical Device

U.S. Department of Energy SBIR Phase II

2011 DOE EERE Program Review

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Project ID # PD 082



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Overview

- Timeline
 - Start: August 2010
 - End: August 2012
 - 25% completed
- Budget
 - Total Project Funding
\$963,850
 - Year 1 funding: \$481k
 - Funding to-date: \$205k
- Barriers
 - 300 psig PBI pump module
 - High tolerance to reformat gases
 - Low cost
 - Efficiency
- Partners
 - PBI Performance Products, Inc.
 - Plug Power, Inc.



Teaming - Collaborators

H2Pump LLC

Responsibility

Project lead/Coordination
Requirements definition
Hardware design &
development
Test/analysis

Contacts

Glenn Eisman

PBI Performance Products, Inc.

Membrane development
Materials properties

Greg Copeland



Plug Power, Inc.

Final on-site test &
demonstration with
an integrated reformer



“Process Intensification of Hydrogen Unit Operations Using an Electrochemical Device”

Develop and demonstrate multi-functional hydrogen production technology to address the following application needs/barriers:

High efficiency (70%)

Purification (DOE targets)

100 scfh

Low cost (\$3/kg)

CO₂ tolerance

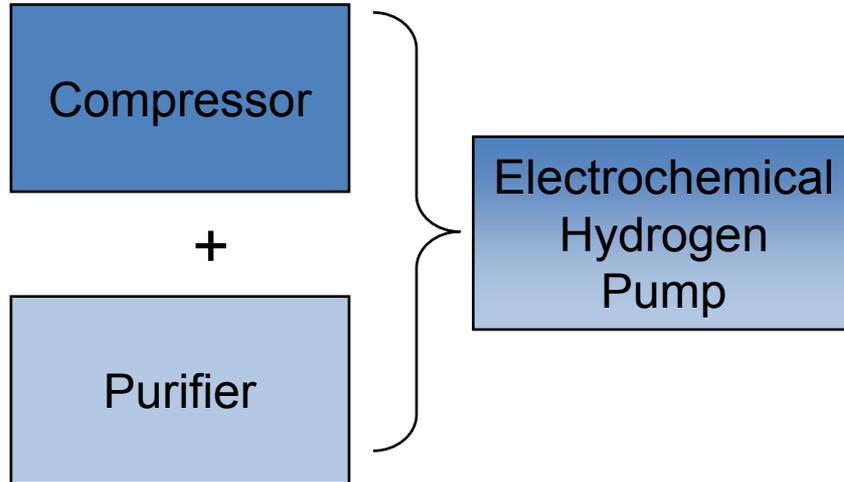
CO tolerance

Pressurization (300 psig)



Program “Asks”

“Combining multiple unit operations for the production of purified hydrogen”



H2Pump Module

Program Targets

H₂ @ 300 psi

100 scfh

CO₂ tolerance

>70% Efficiency

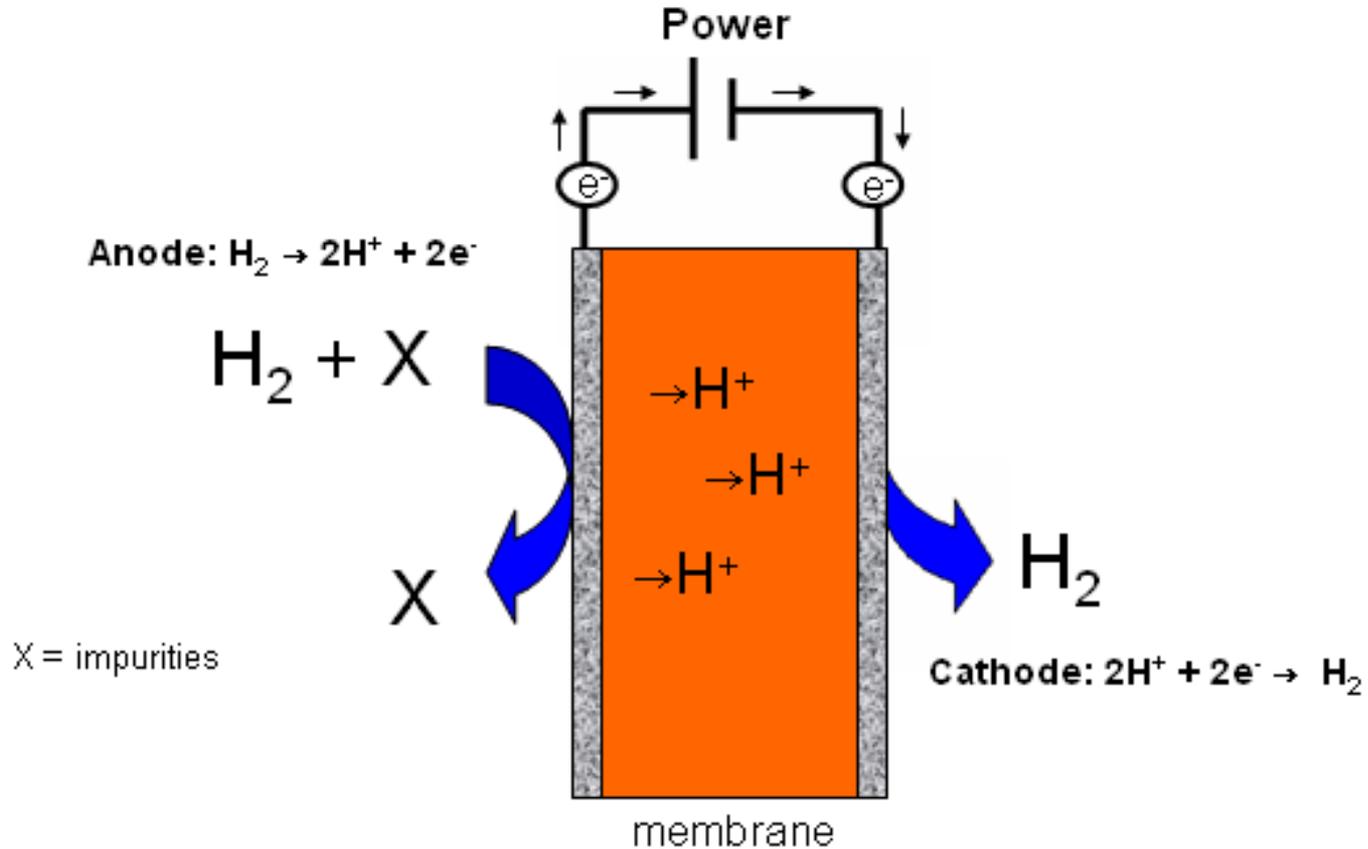
Low cost

H2Pump approach: pump module using polybenzimidazole (PBI) high temperature membrane utilized to replace multiple unit operations

Challenge: PBI membranes are structurally limited, program is based on achieving 300 psid (differential) pressure in the stack



Electrochemical Hydrogen Pump Basics



Relevance – Addressing Barriers

<u>Program Goal</u>	<u>Validation of Approach</u>
✓ Low cost (\$3/kg)	<ul style="list-style-type: none">• Field operation of 80 scfh unit demonstrated at 2.5 – 10 kW-hr/kg
✓ High efficiency (70%)	<ul style="list-style-type: none">• Electrochemical hydrogen pumping provides high efficiency solution to bulk purification and compression
✓ Purification (DOE targets)	<ul style="list-style-type: none">• Enhanced pump design integrated with specific high temperature materials can provide high purity• Further purification can be efficiently achieved with polishing steps
✓ 100 scfh	<ul style="list-style-type: none">• Easily scaled & high turn-down ratio• 450 scfh demonstrated• Field demonstration “system” scaled for 160 scfh• Next generation pump system designed for 1,600 scfh to be delivered to a customer 2Q 2011
✓ CO tolerance	<ul style="list-style-type: none">• High temperature compatible materials provide enhanced tolerance to CO
✓ Pressurization (300 psig)	<ul style="list-style-type: none">• 300 psid, 160°C operation demonstrated in multiple pumps (>200 hrs.) 3/1/2011• High temperature compatible architecture identified and currently under test



Approach - Phase I Accomplishments and Results

- **Proof of concept demonstrated**
 - 50/50 H₂/CO₂ mixture
 - 75/25 H₂/CO₂ mixture
- **Enhance the existing hardware for 300 psig operation**
 - Pressure test existing system level hardware to 300 psig. Improved reinforcement of compressive hardware and slight modifications to plate hardware cross sections
 - 300 psig 160°C pumping demonstrated for limited time on lab-scale hardware
- **Utilizing mechanically reinforced high temperature membranes**
 - Membrane materials from 4 different suppliers
 - Multiple variations on each material tested
 - 14 different high temperature membrane variations evaluated
 - Multiple mechanical reinforcements tested
 - Proper GDL selection provides significant membrane support



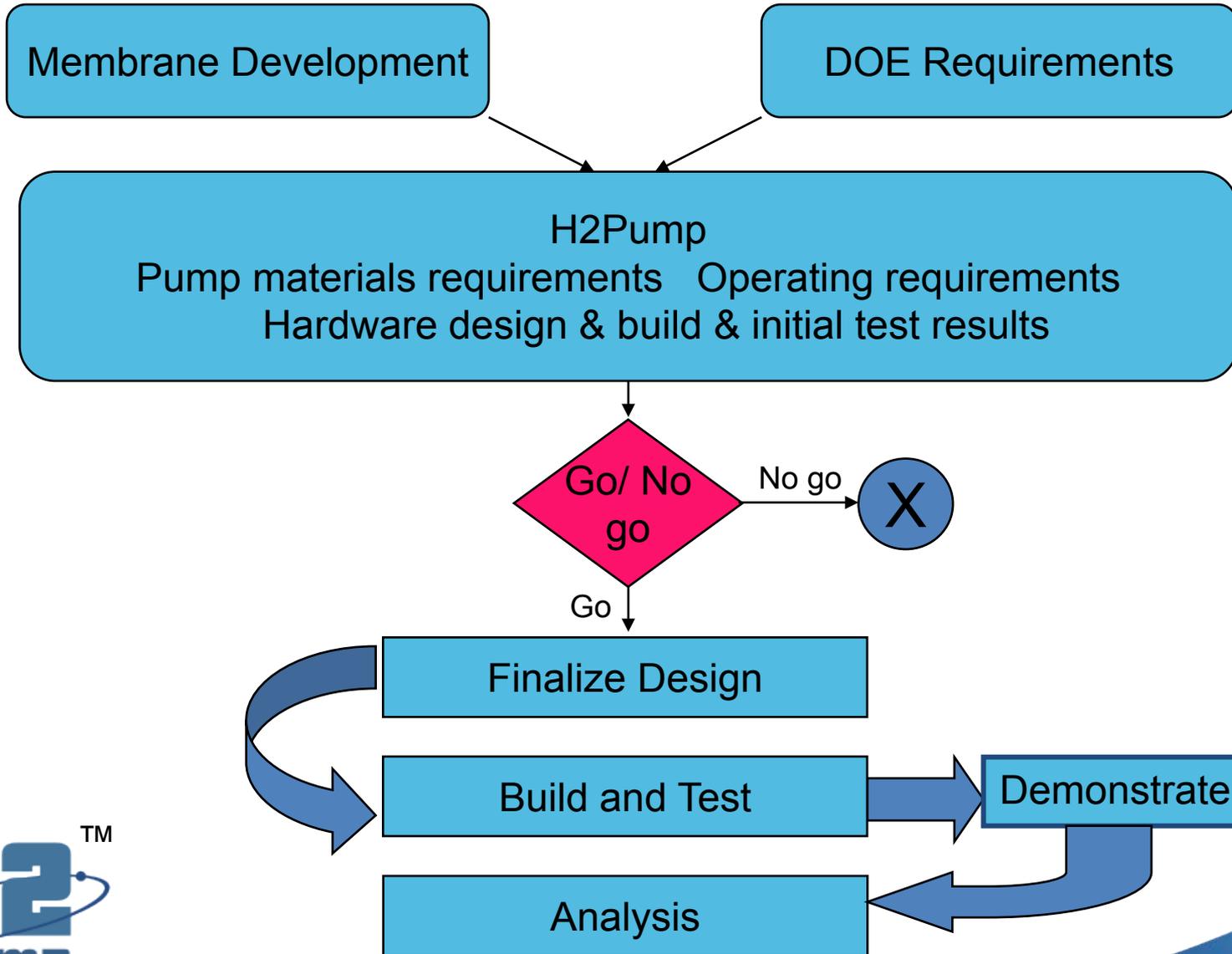
Approach – Phase II Work Plan

Barrier Identified (focus of Phase II): Lifetime performance at 300 psig

- Materials Selection and testing
 - Integrated plate / GDL structure
 - Materials selection partially completed
 - Materials testing in process
- Design for the Application
 - Membrane optimization
 - Developing improved durability and performance with program partner
 - Plate / GDL Structure
 - Identified materials
 - Working with suppliers to test samples and integrate with pumping hardware
 - Integrate electrode structure with plate / GDL
 - Pump Hardware
 - Adapt full scale pump hardware to enhanced design
 - Perform pressure, purity, efficiency, and lifetime testing with various gas mixtures
- Off-Site Integrated Testing with Reformer and Fuel Cell
 - Integrate with existing reformer and fuel cell sub-systems
 - Utilize existing test facilities at Plug Power
- Analysis
 - Performance model
 - H2A update



Approach



Schedule - Activities

Start date: August, 2010

Task	Duration (months)	Q1			Q2			Q3			Q4			Q5			Q6			Q7			Q8					
		M1	M2	M3	M4	M5	M6	M7	M8	M9	M10	M11	M12	M13	M14	M15	M16	M17	M18	M19	M20	M21	M22	M23	M24			
1 Materials Selection and Testing	6	█																										
2 Pump design and testing	16				█						█			█			█											
3 Off-site integrated fuel cell testing	6																█			█								
4 Analysis	9																█											

Phase gate go/no go decision

Q1-Q2: Materials Selection and Testing – Pump Design & Test

- PBI Performance Products engaged in polymer and membrane activities which address materials stability, mechanical strength, without significantly impacting conductivity
- H2Pump actively engaged in hardware development
 - 1) test mechanical properties of MEA candidates in an external cell
 - 2) conductivity cell developed to confirm candidate ionic transport characteristics
 - 3) cell hardware design iterations initiated
 - 4) sampling of PBI from additional sources
 - 5) pump stack design initiated



PBI Membrane Preparation/Selection

Improve material properties:

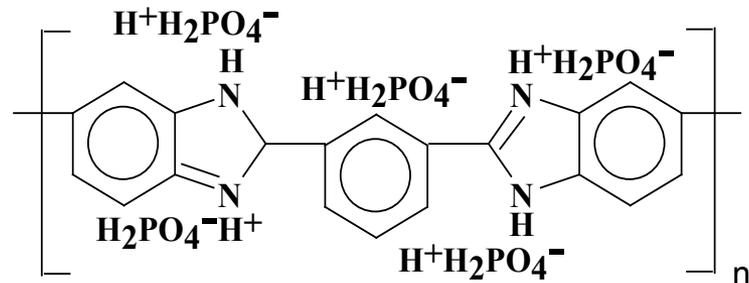
- Thermal cross-linking
- Chemical cross-linking
- Varied acid concentrations

Selection criteria / analysis

- Acid take-up
- Proton conductivity
- Physical properties
- Durability
- Cell Performance

Progress to-date

- Membrane characterization methods for early detection of material degradation
- Enhanced membrane material properties
- Implemented duplicate conductivity rigs for rapid membrane enhancement/characterization cycles



Polybenzimidazole (PBI)



Membrane Materials Stabilization



Interface between PBI membrane and electrode can affect cell performance and membrane durability

- Methods used to impact the membrane-electrode interfacial characteristics
 - Electrode coating
 - concentration, thickness and process
 - Electrode treatments
 - Acid control

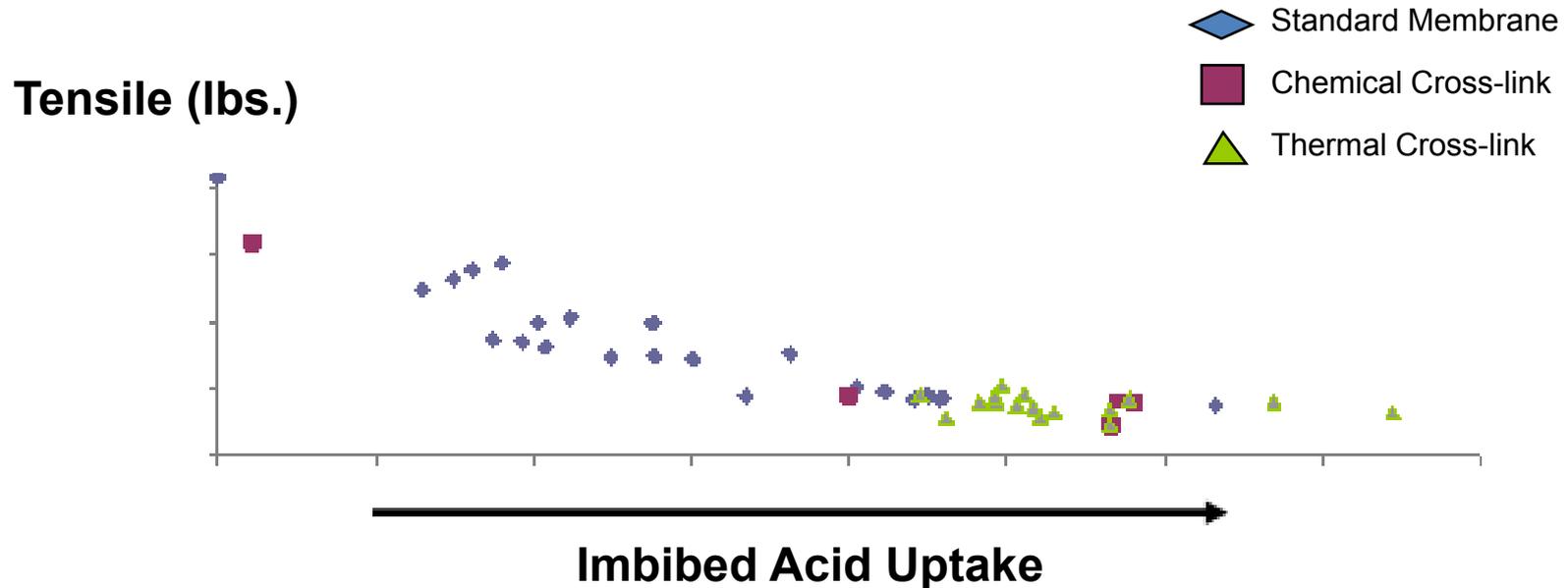
Accomplishments to-date: Membrane failure mechanism associated with early generation MEA configurations and processing method has been eliminated

Achieved repeatability in cell performance as a result of the new interface



Cross-Linking Impact on Mechanical Properties

Tensile vs. Acid Uptake



Tensile test data of various PBI membrane samples after imbibing with H_3PO_4

Cross-linking methods used to-date have not shown that there is a substantial impact on post-imbibed tensile strength at room temperature, yielding insight into polymer chain dynamics and cross-linking approach

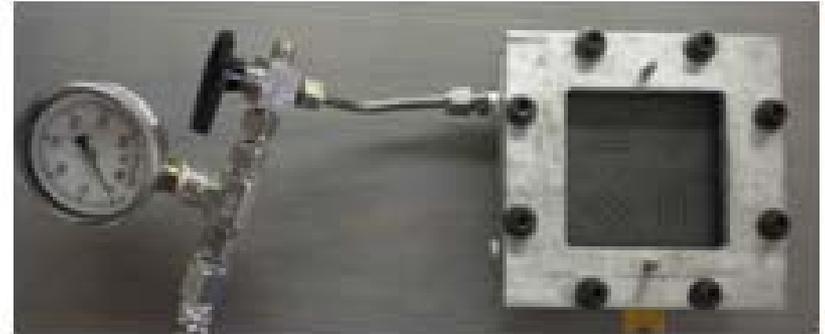
Alternative approaches to address membrane properties underway

Pump architecture should not rely on mechanical strength of membrane alone

Next Task: Implement environmental chamber on Instron to best understand membrane characteristics (test under typical operating conditions)

H2Pump Activities

- GDL and Pump support architecture identified
GDL pressure test rig (see photos)
More than 200 pressure cycles at up to 300 psi with high temperature compatible materials
Identified combined material orientation and flow channel effects on durability at pressure (drives stack design guidelines)
- Unsupported-membrane design approach
Current membrane is not capable of providing substantial pressure holding strength
Approach identified in which the membrane and cell hardware are preconfigured, thus avoiding failure due to excessive strain during application of pressure differential
- Conductivity Cell Developed
Used to identify impact of membrane changes on proton conductivity (simulated cell conditions)



Machine vs. Transverse GDL orientations can have a substantial impact on durability under high loads

Test fixture facilitates rapid material screening through cycling at high pressure differentials

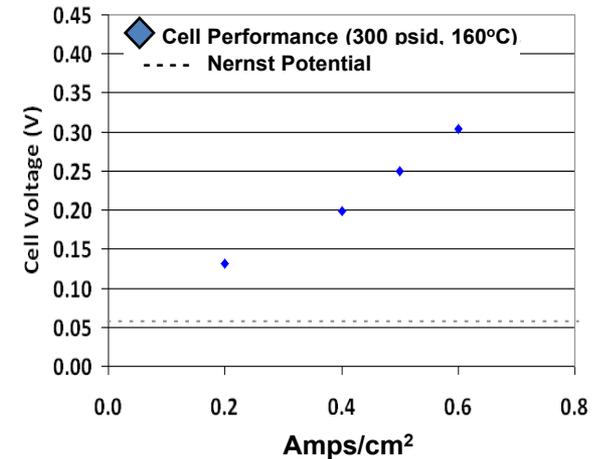
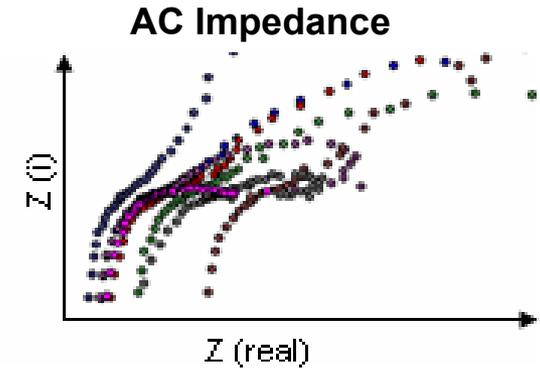
Multiple channel widths and orientations permits evaluation of machine vs. transverse direction durability



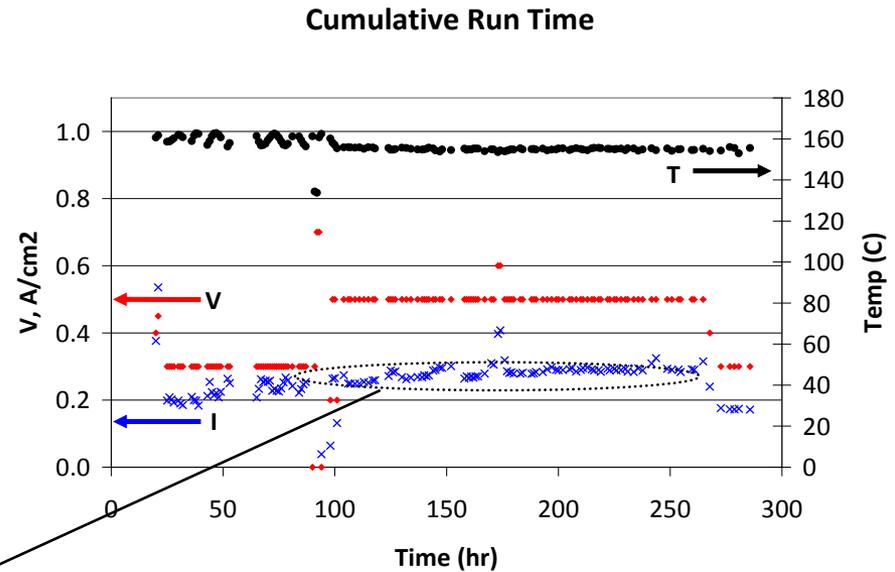
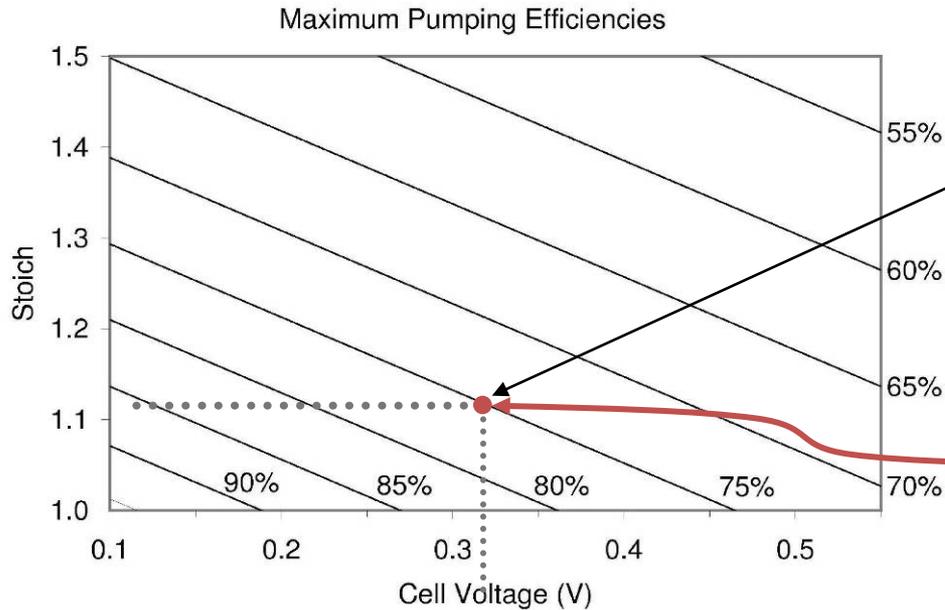
High Pressure Pump Testing

Analytical Methods

- Electrochemical Impedance Spectroscopy (EIS)
 - 8 channel stack testing capability
- Polarization curves at various pressures
 - Evaluate in conjunction with EIS
- Isolate various pressure, design, and build-related over-voltages
- Facilitate rapid design, test and evaluation cycles



Efficiency



Better than 70% efficiency (Program target met)

Future improvements anticipated

- < 1.2 stoichiometric ratio
- Reduced cell over-voltage due to improved mechanical support, reduced diffusion losses & reduced impedance



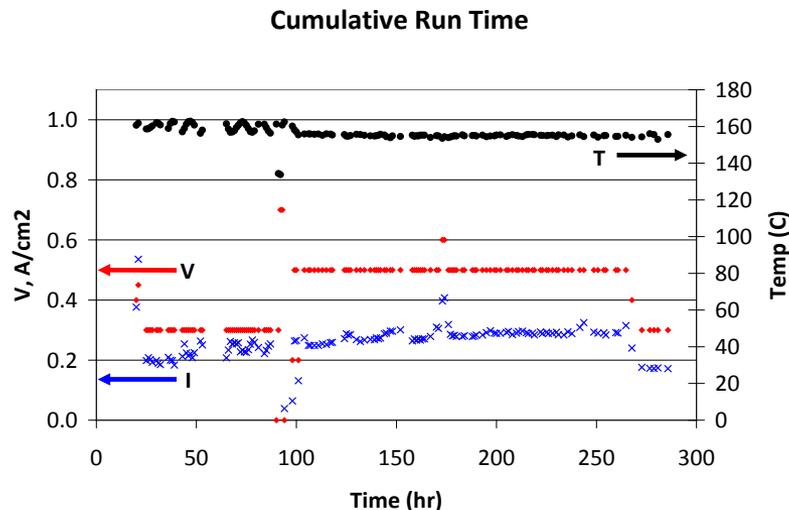
$$\varepsilon = \frac{P_{out}}{P_{in}} = \frac{P_{H2_out}}{P_{H2_in} + P_{pump}} \quad E \left(\frac{J}{mol} \right) = \frac{P}{\dot{n}} = \frac{IVNF}{I} = VNF$$

$$\varepsilon = \frac{HV_{H2}}{Stoich \cdot HV_{H2} + VNF/m}$$

Accomplishments and Progress

300 psi differential pressure achieved @ 160 °C on multiple pumps (**Program target met**)

- Design advancements
 - Implement advancements from off-line test rigs
 - GDL / support structures
 - Plate material selection
 - Cell build procedures
- PBI membrane
 - Material characterization and enhancement (H2Pump and PBI Performance Products)
 - Enhanced electrochemical interface



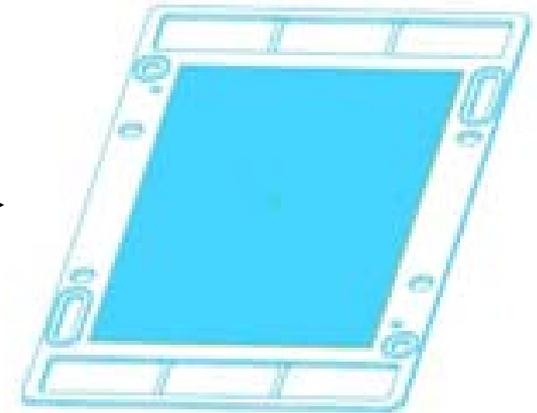
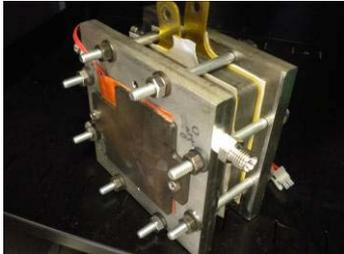
Cyclic 200 – 300 psid operation over ~ 300 hrs



Hardware Design & Development for 100 scfh Target

Large Format

- Increase active area from 50 cm² to 1000 cm²



High Pressure Design

Apply small cell and off-line test results to full size pump

Incorporate thermal management

Material properties of selected high temperature materials

Low anode pressure drop

High cathode pressure capability

50% complete to-date

Status vs. Milestones

Milestone	Schedule	Risks/Limitations	Status/Risk Mitigation	Task
Membrane Characterization	Q2	No suitable accelerated life testing available, leads to long test periods	Multiple material degradation indicator tests developed/identified (membrane, GDL, plates)	✓
Identification of Suitable 300 psi Architecture	Q4	Identification of fundamental materials or conceptual limitations Key component materials must be selected early on. Already working with suppliers to avoid delays in 2011.	Off-line test rigs developed (GDL, membrane, architecture configurations) and implemented at H2Pump and PBI Performance Products Multiple 300 psid 160°C pumps currently on test	✓
Successful operation of 300 psi pump stack	Q5	Minimal risk to scale up if design concepts can be demonstrated at lab-scale	Full-scale pump hardware design initiated. Design guidelines developed from off-line test results.	✓
Installation into and testing with modified pump system	Q6	Minimal risk: 2 demonstration systems of suitable size have already been built (1 placed in field, 1 for internal development)	Adaptation for high pressure / high temperature operation is minimal. High temperature system operation previously demonstrated at 5 kg/day scale	
Demonstration with fuel cell system	Q7	Hardware and facility availability	Commitment from partner to provide test facility, required hardware, and support	



✓ - Represents completed milestone

Project Summary

- Program is on schedule
- Close working relationship developed with PBI Performance Products
- Materials test methods developed and employed
- Multiple material options being assessed
- Membrane stability and mechanical strength enhancement efforts successfully demonstrated
- Initiated hardware and stack designs finalized
- 300 psid demonstrated in lab-scale testing > 100 hours (continuous)
- Program spending on track



About H2Pump



Dedicated solely to hydrogen recovery and recycling

Developing, manufacturing, and marketing hydrogen recycling technology for existing and emerging hydrogen-based processes and industries

Founded in October 2005

Located in Latham, New York (Albany)

Past awards: NYSERDA, Department of Energy-SBIR, and DoD STTR

Prototypes sold for both industrial and alternative energy applications

Strategic partnership signed in Nov. 2009, Oct. 2010 (InterTech Group)

Installation of first recycler in heat treating facility – Nov. 2009

12 employees



DOE SBIR Award # DE-SC0002185

Acknowledgements

DOE: Richard Farmer, DOE HQ (EERE)

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TJ Janniere**

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Jill Wager, Rhonda Staudt**

