

Process Intensification of Hydrogen Unit Operations Using an Electrochemical Device

U.S. Department of Energy SBIR Phase II

2012 DOE EERE Program Review

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Project ID # PD 082



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Overview

- Timeline
 - Start: August 2010
 - End: August 2012
 - 80% completed
- Budget
 - Total project funding
 - DOE share: \$963,860
 - Cost share: \$0
 - Funding in FY 11: \$456,140
 - Planned funding for FY 12: \$507,720
- Barriers
 - A: Reformer Capital Costs
 - D: Feedstock Issues
 - E: Greenhouse Gas Emissions
- Partners
 - PBI Performance Products, Inc.



Teaming - Collaborators

H2Pump LLC

Responsibility

Contacts

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

Glenn Eisman

PBI Performance
Products, Inc.

Membrane development &
materials properties

Greg Copeland



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)

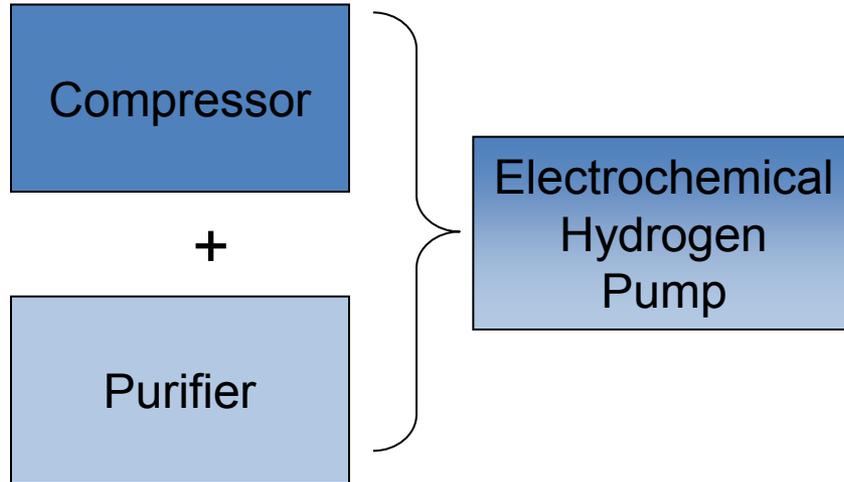
CO₂ tolerance

Pressurization (300 psig)



Program “Asks”

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



Program Targets

H₂ @ 300 psi

100 scfh

CO₂ tolerance

>70% Efficiency

Low cost

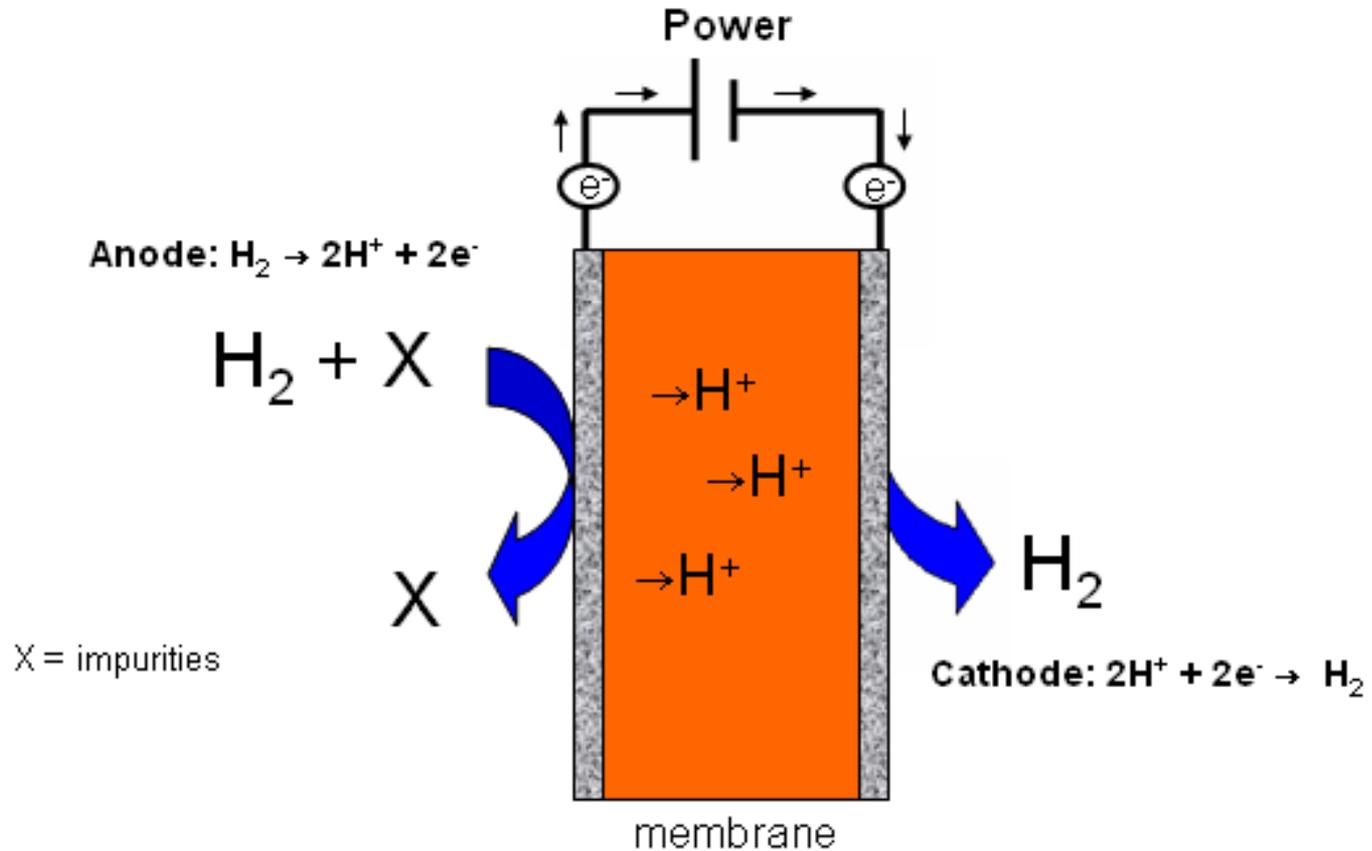
H2Pump Module

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

Challenge: PBI membranes are structurally limited, the 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 the Approach</u>
✓ Low cost (\$3/kg)	• Field operation of 80 scfh unit demonstrated at 2.5 – 10 kW-hr/kg
✓ High efficiency (70%)	• Electrochemical hydrogen pumping provides high efficiency solution to bulk purification and compression
✓ Purification (DOE targets)	• Enhanced pump design integrated with specific high temperature materials can provide high purity • Further purification can be efficiently achieved with polishing steps
✓ 100 scfh	• Easily scaled & high turn-down ratio • Field demonstration systems scaled for 1600 scfh
✓ CO ₂ /CO tolerance	• High temperature compatible materials provide enhanced tolerance to CO/CO ₂
□ <i>Pressurization (300 psig) demonstrated at scale using PBI membranes</i>	• <i>300 psig generated at 160°C operation - demonstrated in multiple pumps in the lab (50 cm²) >1700 hrs.</i> • <i>Developed and tested high temperature cell architecture</i> • <i>Cell hardware scale up completed and validated using low temperature membranes</i>



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



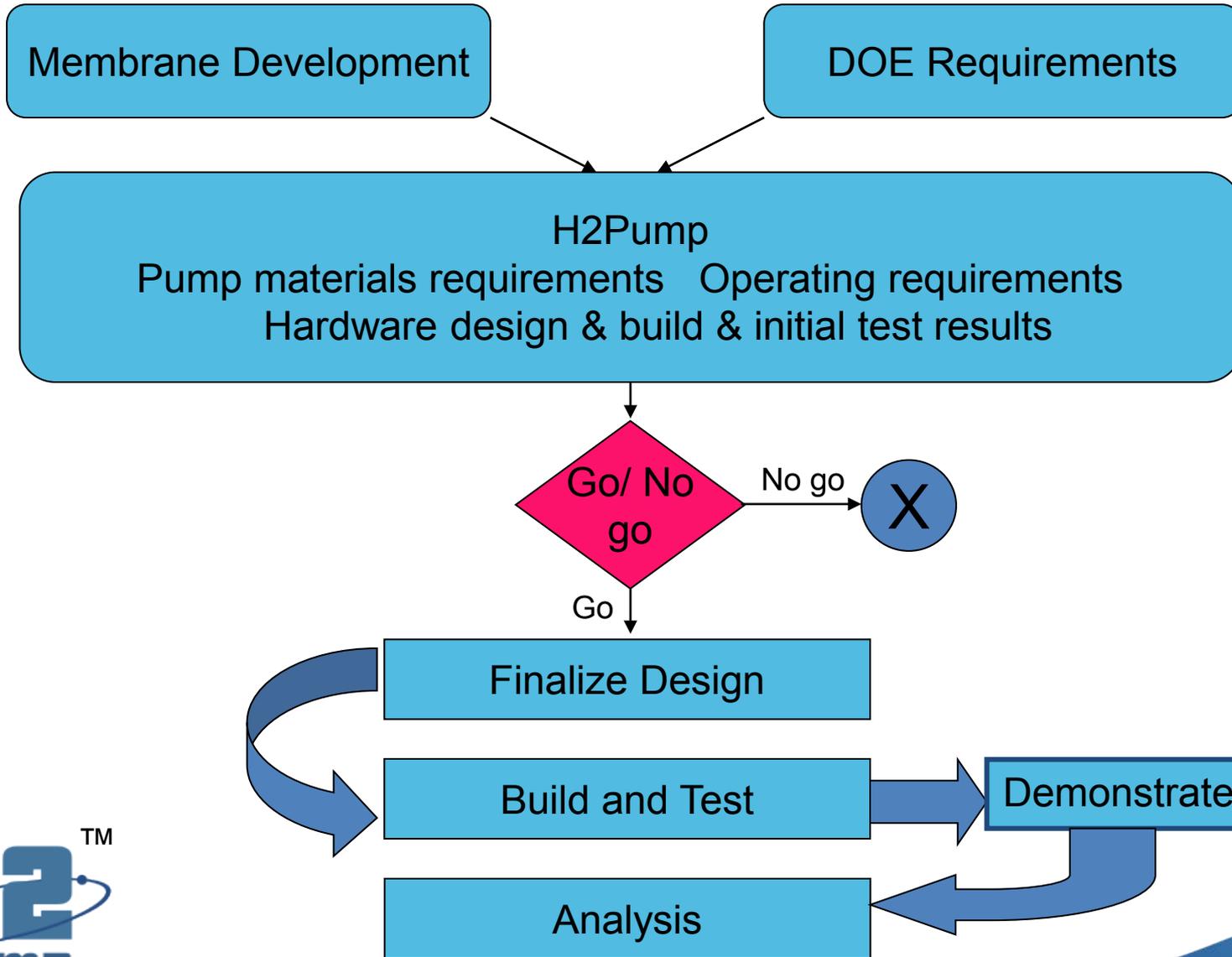
Approach – Phase II Work Plan

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
- Demonstration: Integrated reformer test and evaluation
- Analysis
 - Performance model
 - H2A update



Approach



Schedule - Activities

Start date: August, 2011

		Q1			Q2			Q3			Q4			Q5			Q6			Q7			Q8		
TASK	Duration (months)	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	█																							
2	Pump design and test							█						█	█	█	█	█	█	█	█	█	█	█	█
3	Integrated pump – reformer test																								
4	Analysis																								

Stage Gate - Go/No Go Decision

Current Program Timing

- Successfully passed go/no go check point in August 2011
- Demonstration now to take place at H2Pump’s facility
- Program on track (schedule and cost)



PBI Membrane Preparation/Selection

Program Objectives:

Improve material properties:

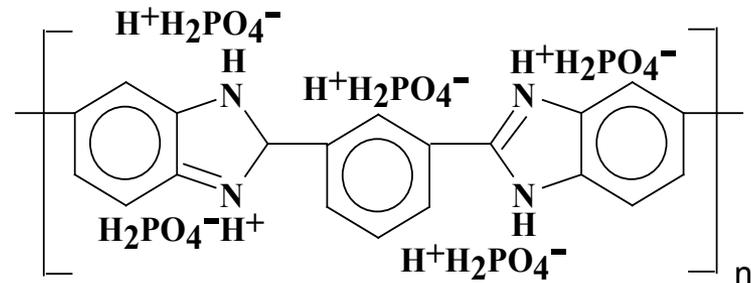
- Thermal cross-linking
- Chemical cross-linking
- Varied acid concentrations

Selection criteria / analysis

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

3/2011-3/2012 (Q3 – Q6) Activities:

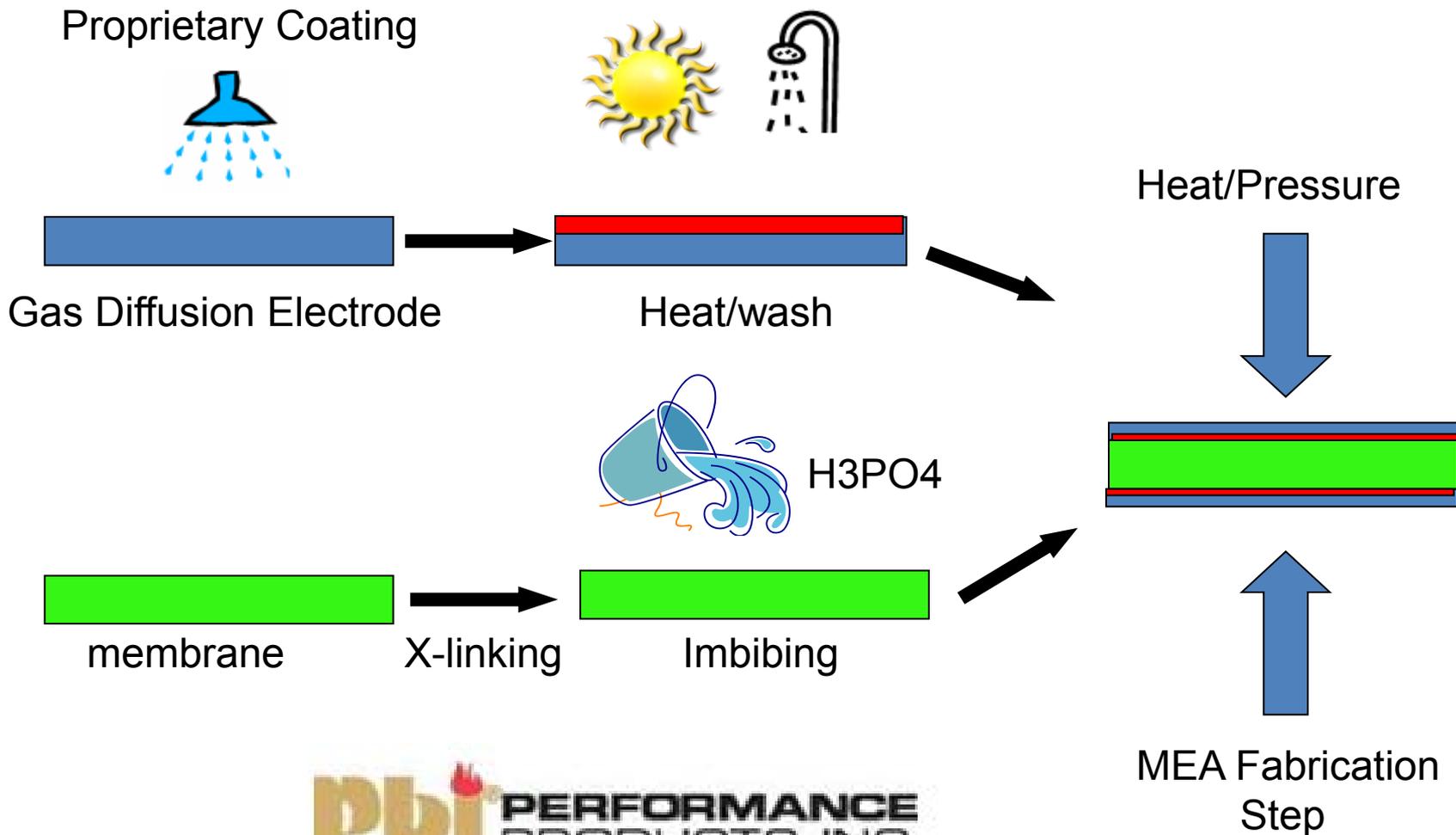
- Supplied 50 cm² membranes and electrodes to assess reproducibility
- Refined proprietary membrane stabilization process
- Developed Standard Operating Procedure (SOP) for 50 cm² MEA's
- Initiated large scale (1000 cm²) materials handling procedures



Polybenzimidazole (PBI)



PBI Materials Processing Procedures



H2Pump Activities

Task 1 Materials selection and testing

Task 2 Pump engineering and testing

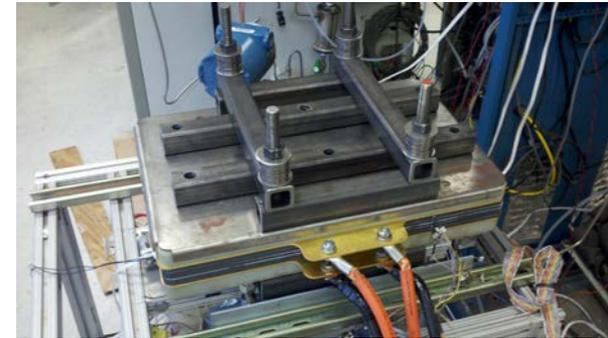
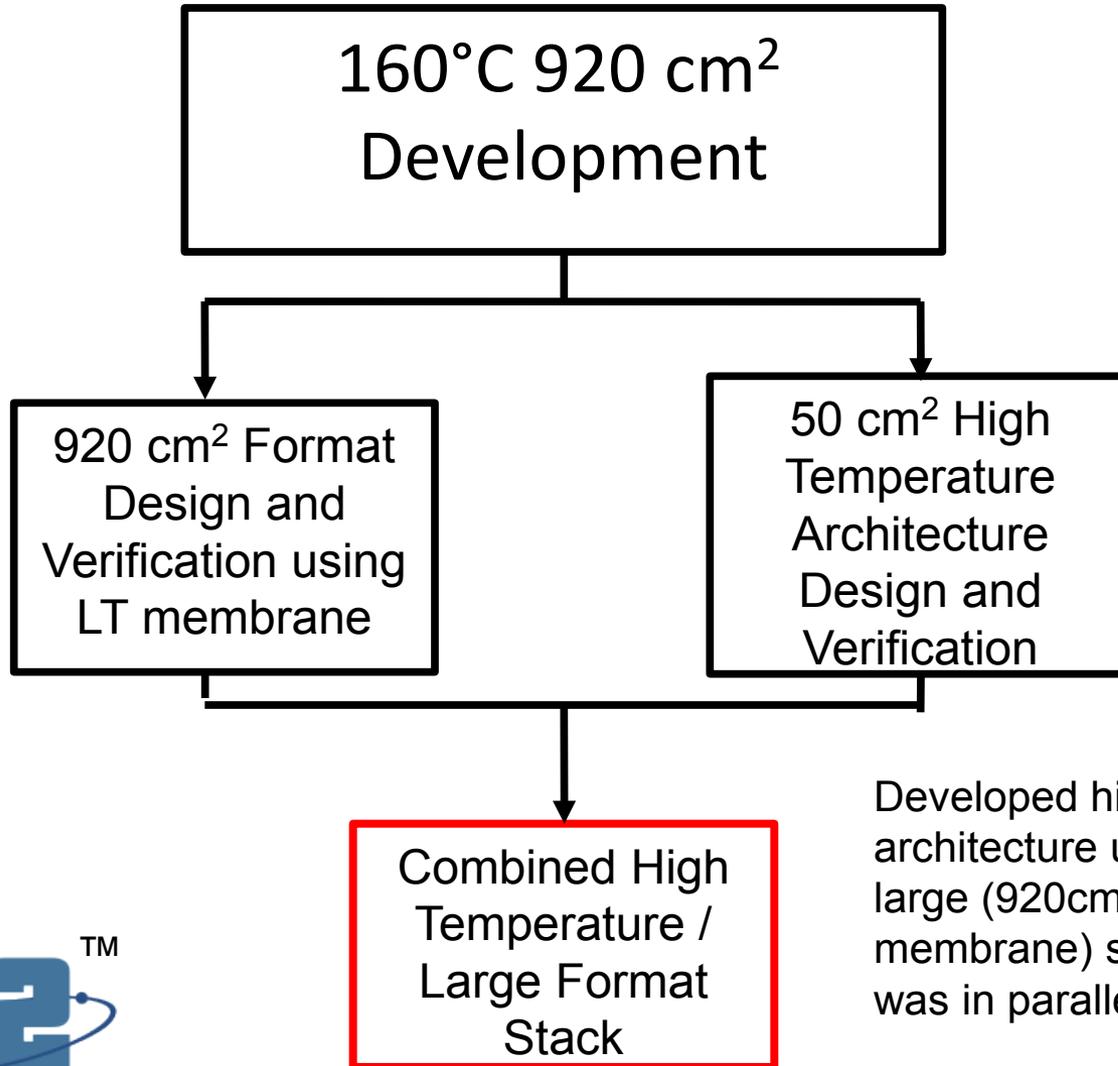
Task 3 Demonstration

3/2011-3/2012 (Q3 – Q6) Activities:

- Performance testing
- MEA sealing and support method development
- Large format cell/stack engineering and scale up
- Demonstration preparations



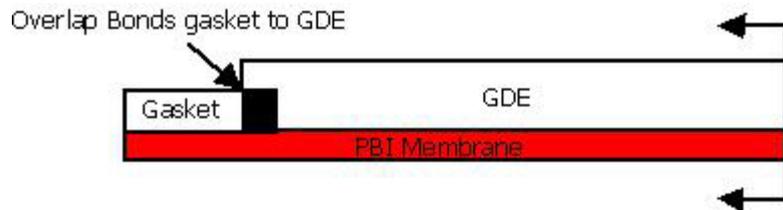
Development Effort



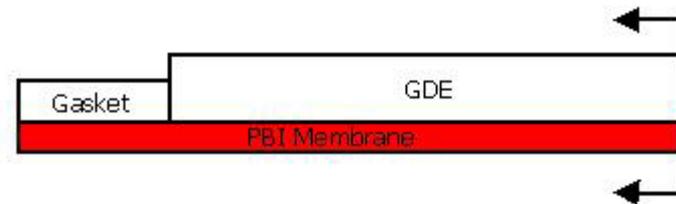
Developed high temperature architecture using 50cm² MEA and large (920cm²) hardware (LT membrane) so hardware development was in parallel, minimizing time

PBI MEA Architecture Evolution

- 1st Generation
 - Gasket overlap configuration
 - Hand cast gaskets
 - Non-optimized flow field
 - High degradation
- 2nd Generation
 - Gasket overlap configuration
 - Commercially available sheet gasket
 - Optimized flow field
- 3rd Generation
 - Line to line gasket configuration
 - Commercially available sheet gasket
 - Optimized flow field
 - Higher risk

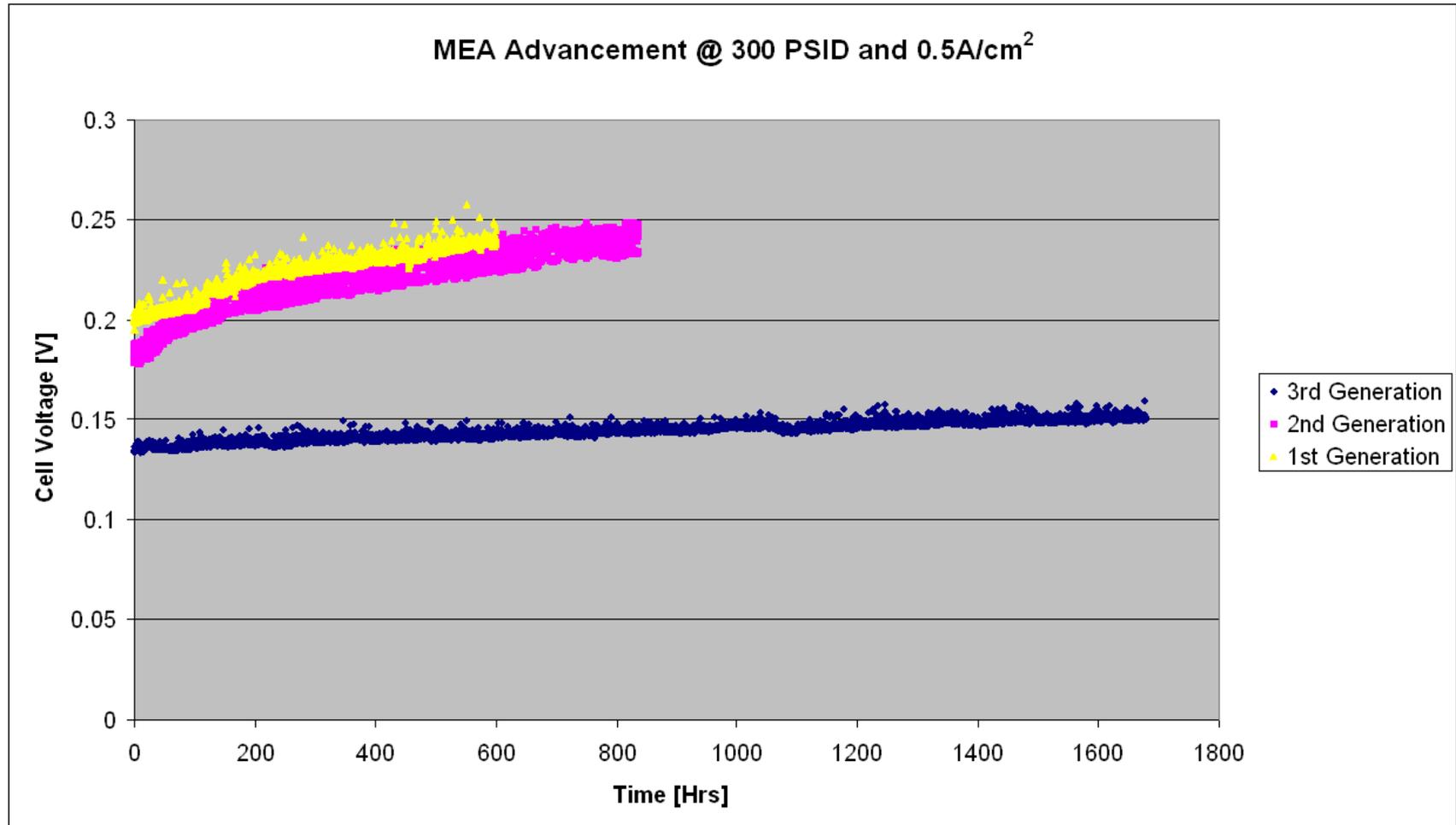


TM Gasket overlap configuration



Line to line gasket configuration

PBI MEA-Hardware Architecture Advancements



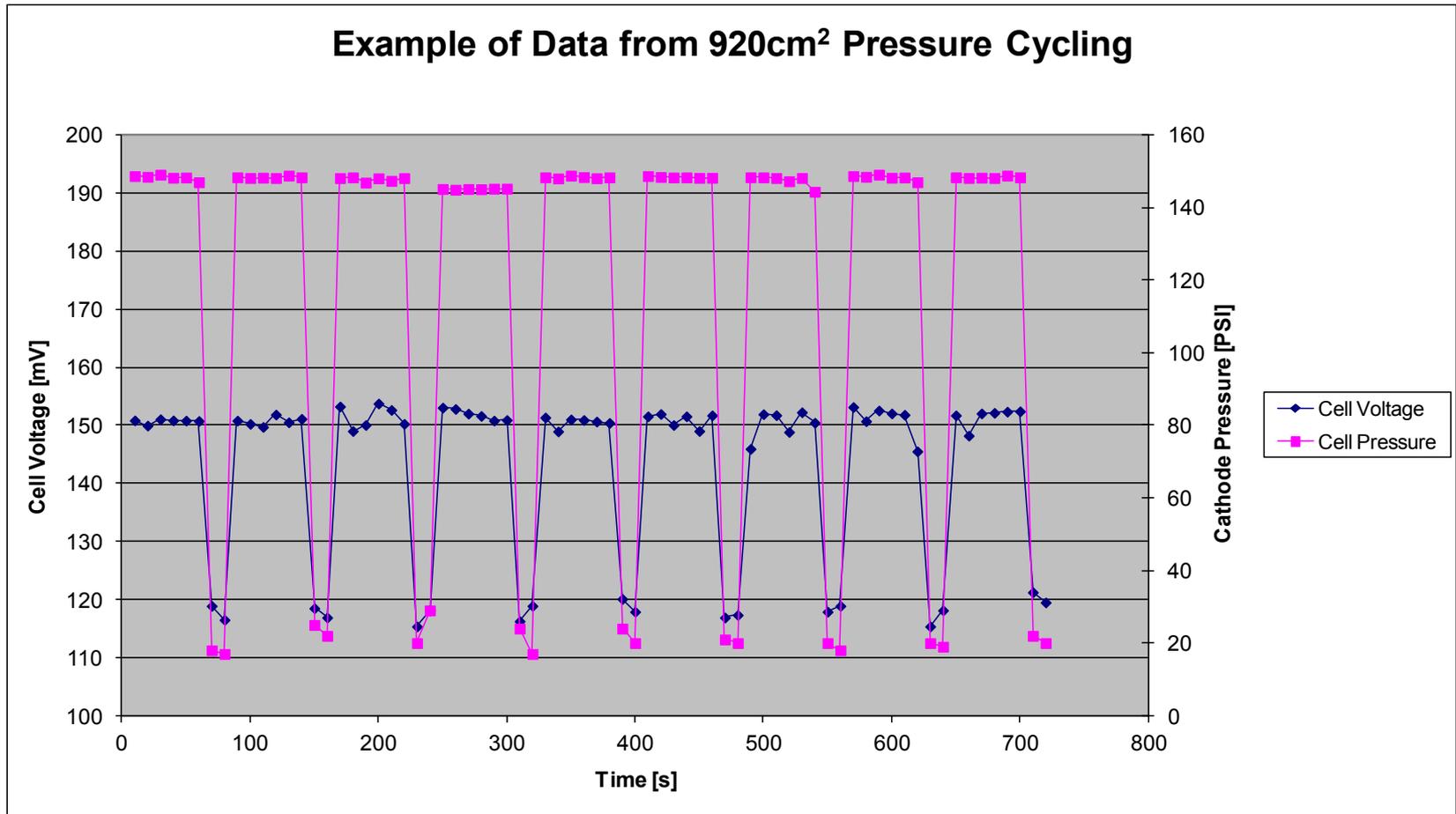
Performance & Degradation Improvement Verified on 50cm² MEA.
Improvements based on MEA & cell hardware design modifications

Cell Development Summary

- Completed HT MEA architecture and seal development on 50 cm² format
- Continuing to perform tests to identify significant operating parameters
- Confirmed large format design via LT membrane testing
 - Completed 2350 pressure cycles without failure or degradation
- Adapting HT architecture to large cell active area in progress



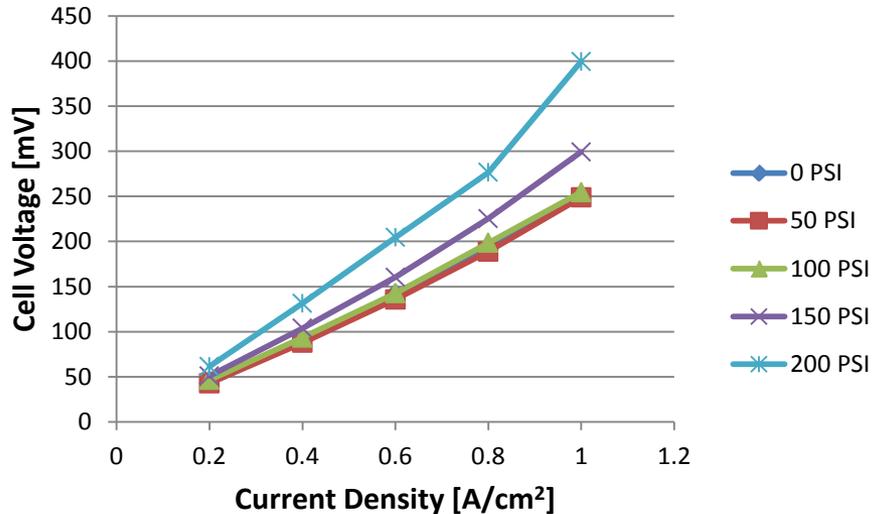
920 cm² Pressure Cycling Test Utilizing LT Membrane to Verify Stack Hardware Viability



2350 pressure cycles achieved without evidence of performance loss or hardware failure

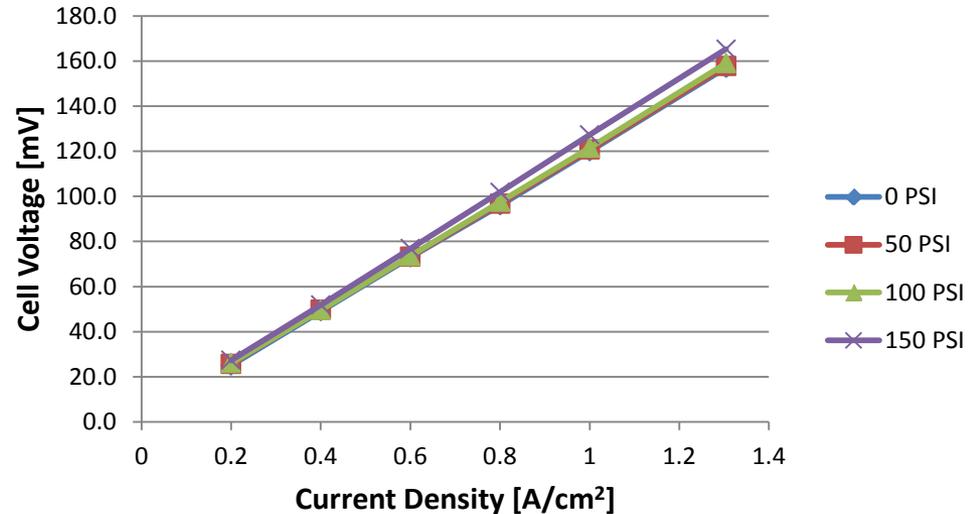
New 920cm2 Stack Hardware Design Improves Performance at High Pressures

**Nernst Corrected Polarization Curve
Rev. 1 – 5 mil membrane**



Previous stack hardware

**Nernst Corrected Polarization Curve
Current Rev. - 2 mil membrane**



Improved Stack hardware

Previous hardware had pressure induced lift off causing loss of performance™ at high pressures

Large format design demonstrated at 250 psi on low temp membranes

Confirmed hardware support design flexibility



Stack Development Summary

Final materials selection

- Commercially available cloth GDE
- Commercially available sheet gasket material

Stack Sizing

- 10 Cell, 0.75 A/cm^2 (690 A) \longrightarrow 100 SCFH product flow

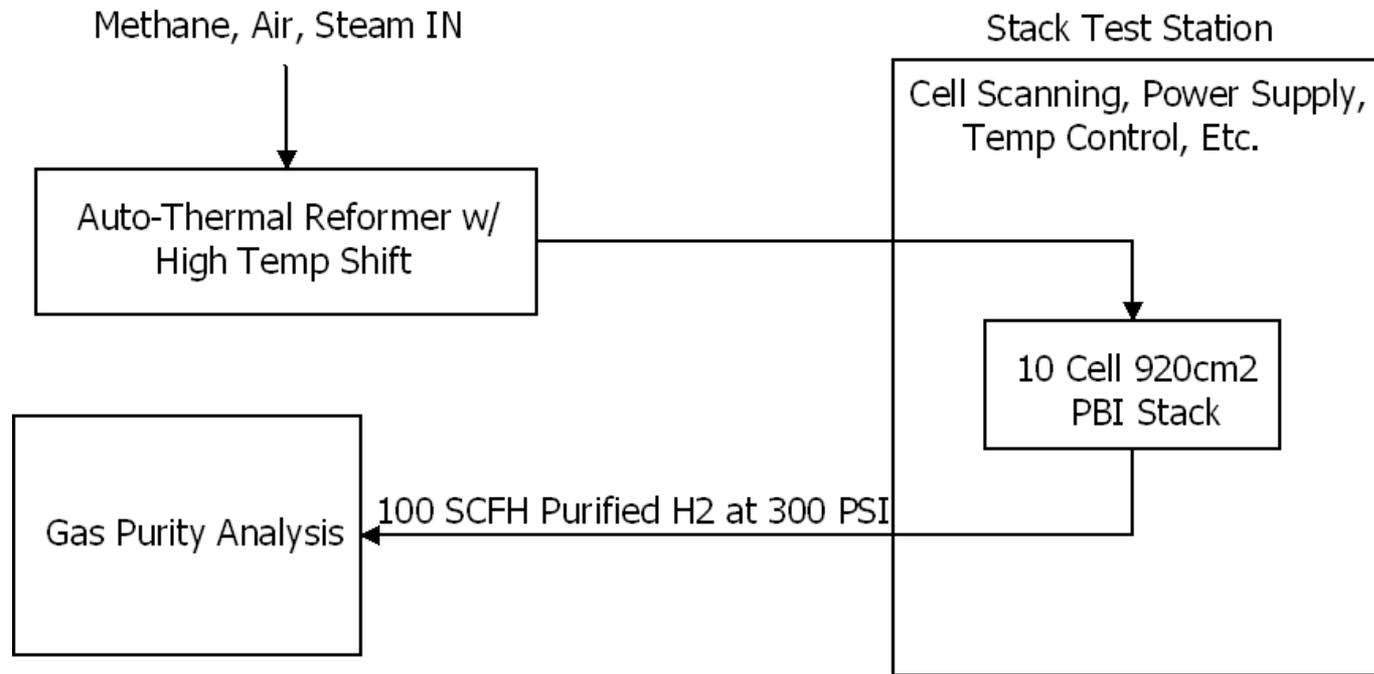
Thermal Management

- Cartridge heaters in end plates to reach operating temperature
- $1.25 \text{ kW}_{\text{th}}$ generated internal to stack at 100 SCFH pumping rate
- Down-selected coolant
- Coolant flow rates based on operating model



Demonstration P&ID

- Build and test HT 920 cm² stack with PBI
 - Stack Station
 - Reformer
 - Gas analysis



Stack Station/Facilities

- Acquired high temperature stack test station
 - Features include cell scanning, temperature control, alarm shutdowns, data acquisition and logging
- Test station-pump integration activities initiated
- Facilities
 - Permitted for hydrogen
 - Existing ventilation system rated for 1600 SCFH
 - Safety system and policies in place



Reformer

- Acquired reformer from Plug Power
- Reformer-Pump integration design completed
 - Flow rates for methane, air and water determined
 - Sub-system P&ID and electrical schematic completed
 - Integrated into safety shutdown circuits for stack test station and lab



ATR with HTS

Status vs. Milestones

Milestone	Schedule	Risks/Limitations	Status/Risk Mitigation
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 3000 hrs. operation at 100 psig Multiple 300 psid tests conducted (50 cm ²)
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 Design guidelines developed from off-line test results
Installation 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 Preliminary testing of large cell at high pressures (PBI)
Demonstration with fuel cell system	Q7	Hardware and facility availability	Program plan changed to test on site @ H2Pump, acquired reformer technology from Plug Power

Future Activities

Next period will focus on:

Stack development

- Order stack hardware and supporting fixtures and dies
- MEA fabrication
 - Large format MEA pressing procedure development

Stack station

- Installation & integration with lab utilities and safety systems
- Modification/Debug

Reformer set-up and integration with the pump module

Gas purity and H₂A analyses



Project Summary

- Program is on schedule and budget
- Multiple 50 cm² PBI cells operated >1000 hrs. (each) @ 300 psig (differential)
- Materials processing finalized @ PBI Performance Products
- 920 cm² hardware, and GDL materials selected, designed, built, and tested
- Large scale membrane and electrode fabrication process developed
- Stack design demonstrated to over 2300 pressure cycles
- Acquired necessary hardware for the final demonstration (reformer)
- P&ID developed for integrated reformer, pump demonstration



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 with the InterTech Group (Charleston, SC)

Installation of HRS-100™ recyclers in 2011, 2012

13 employees



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