# V.H.3 Developing Improved Materials to Support the Hydrogen Economy\*

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\*Congressionally directed project

## **Objectives**

- DOE application approval.
- Manage ongoing projects.
- Receive, review and approve round two projects.
- Release round 3 request for proposal (RFP).
- Receive, review and approve round three projects.
- Collect and review monthly project reports for go/no-go results.
- Initiate Phase I and Phase II projects as appropriate.
- Prepare quarterly reports from individual project reports.

## **Technical Barriers and Technical Targets**

The Edison Materials Technology Center (EMTEC) will solicit and fund hydrogen infrastructure related projects that have a near term potential for commercialization. The subject technology must be related to the U.S. Department of Energy goals as outlined in the multi-year plan titled, "Hydrogen, Fuel Cells and Infrastructure Technologies Program Multi-Year Research, Development and Demonstration Plan." Preference will be given to cross-cutting materials development projects that lead to the establishment of manufacturing capability and job creation. Specific barriers for the projects include:

- Capital Costs
- Operation and Maintenance (O&M)

- On-Board Hydrogen Storage
- System Cost
- Durability/Operability
- Hydrogen Embrittlement of Pipelines
- Control and Safety
- Device Configuration Designs
- System Design and Evaluation
- Grid Electricity Emissions
- System Efficiency
- Electricity Costs
- Liability Issues
- Variation in Standard Practice of Safety Assessments for Components and Energy Systems
- System Weight and Volume
- High-pressure Conformability
- Materials of Construction
- System Life-Cycle Assessments
- Lack of Understanding of Hydrogen Physisorption and Chemisorption
- Reproducibility of Performance
- Charging/Discharging Rates
- Hydrogen capacity within the storage medium (doped and undoped alanates)
- Facility of hydrogen desorption and adsorption in optimized systems
- Renewable Integration
- Electricity Costs
- High-Purity Water Availability
- Fuel Cell Manufacturing and Process Costs
- Fuel Cell / Stack Durability
- Fuel Cell Electrode Performance
- Feedstock Issues
- Carbon Dioxide Emissions
- Impurities
- High Cost and Low Energy Efficiency of Hydrogen Liquefaction
- Fuel Processor Manufacturing
- Thermal, Air and Water Management

## Approach

The Edison Materials Technology Center (EMTEC) has used the U.S. Department of Energy goals as outlined in the multi-year plan titled, "Hydrogen, Fuel Cells and Infrastructure Technologies Program Multi-Year Research, Development and Demonstration Plan" to find and fund projects with near-term commercialization potential. An RFP process aligned with this plan requires performance-based objectives with go/no-go technology based milestones. EMTEC manages this project for the DOE using the protocols that include an RFP solicitation process, white papers, and proposals with peer technology and commercialization review (including DOE), EMTEC project negotiation and definition, and DOE cost share approval. Our RFP approach specifies proposals/ projects for hydrogen production, hydrogen storage or hydrogen infrastructure processing which may include sensor, separator, compression, maintenance, or delivery technologies. EMTEC is especially alert for projects in the appropriate subject area that have cross-cutting materials technology with near term manufacturing opportunities. To date EMTEC has selected projects which have been continuing development projects preparatory to commercialization. EMTEC's overriding objective is technology commercialization.

## Accomplishments

- EMTEC released RFP3 (white paper): July 8, 2005.
- DOE Renewal Application Approved: July 13, 2005.
- Round 3 white paper due date: August 12, 2005.
- Invitation for full Round 3 proposals: September 9, 2005.
- Full Round 3 proposal due date: October 10, 2005.
- Round 3 proposal review completed: December 30, 2005.
- Round 3 down-select and site visit announcement: January 3, 2006.
- EMTEC hosted the 1<sup>st</sup> Symposium on Manufacturing of Membrane Electrode Assemblies (MEAs) for Hydrogen Applications, August 9-11, 2005 at the DoubleTree in downtown Dayton, Ohio. This symposium drew 133 participants from 10 countries. EMTEC continued the planning and organizing efforts for the 2<sup>nd</sup> MEA Manufacturing Symposium to be held August 22-24 at the Dayton Marriott.
- EMTEC hosted a highly successful half-day Fuel Cell Manufacturing Short Course on March 2, 2006 at the Engineers Club of Dayton, OH. This course was presented by fuel cell experts, Dr. Jack Brouwer of the National Fuel Cell Research Center, University of California, Irvine and Dr. Tom Zawodzinski of Case Advanced Power Institute, Case Western Reserve University.
- EMTEC attended and presented at the DOE Hydrogen Program Review in Crystal City, May 16-19, 2006.

- EMTEC continued the negotiations for Round 2 and 3 proposals, including limited Phase II proposals, culminating in a total award of 23 active projects to date. Kick-off meetings were held and reporting and invoicing procedures were set and announced to each new active project team. A project execution summary is included in Figure 1. Individual progress reports for the projects funded through this period are included in this report.
- Through June 30, 2006, the EMTEC advisory team has reviewed over 114 project proposals and accomplished over 42 site visits.

## **Future Directions**

- Assess options for release of Round 4 RFP.
- Manage ongoing projects.
- Initiate Phase I and Phase II projects as appropriate.
- Collect and review monthly project reports for go/no-go results.

Hydrogen Program Performance							iedule	st - Ohiochiuo	Potential	st. Position	namn
EFC #		Project Title			Project Funding	Match Ratio	On Sch	On Cot	Comm	Reinve	Prinr/O
H1-01	Nanocatalyst Developme for Hydrogen Generation	Faraday	\$ 360,287	2.89	þ	α	r	Þ	Ē		
H1-09- 2A	Novel Spiral Stackable Reactor (SSR) for Low-Cost Hydrogen Production (II)			Catacel Corp	\$ 528,737	2.17	•	q		þ	Ē
H1-11	Commercialization of EM Solid State Welding for High Pressure Hydrogen Storage (I)			IAP Research Inc.	\$ 324,769	3.25	0	ά	k	þ	ſ
H1-15	Low Cost MEMS Hydrog	drogen Sensor for Transportation Safety (I)		Makel Engineering	\$ 260,727	1.99	0	d.	Ť	Ē	Ē
H1-16- 2A	Development of Improved Materials for Integrated Photovoltaic- Electrolysis Hydrogen Generation Systems (I-II)			MWOE	\$ 674,875	2.08	0	ά	t	Þ	ŕ
H2-20- 2A	Regenerative Solid Oxide Fuel Cell (II)			UDRI	\$ 390,000	1.95	0	ά	k	Ł	Ē
H2-15	High Temperature Electrochemical Cells for Hydrogen Production and Regenerative Fuel Cell Systems (I)			NexTech	\$ 215,000	2.15	0	þ	k	Þ	Ē
H2-21	Novel Ceramic Hydrogen Sensors for Fuel Cell Applications (I)			NexTech	\$ 215,073	2.15	С	ά	x	t	Ē
H2-12	High Strength, Low Cost Microballoons for Hydrogen Storage (I)			Powdermet	\$ 218,267	2.23	C	þ	×	¢	2
H2-3	Hydrogen Storage Using Porous Silicon (I)			Dephi	\$ 216,742	2.46	C	þ.	k	F	Ē
H2-08	Electrochemical Coal Gasification with Novel Electrodes (I)			Ohio Univ.	\$ 70,070	2.00	0	o	þ	þ	C
H2-22	Fabrication of Metal-Carbon Nanostructure Composites (I)			Univ. of Toledo	\$ 73,334	1.55	0	q	c	þ	C
H2-18	Novel Materials for Reversible Hydrogen Storage (I)			Hy-Energy	\$ 232,588	2.09	0	q	c	þ	С
H2-11	Development of Complex Metal Hydrides for Hydrogen Storage Applications (I)			GFS Chemicals	\$ 197,092	2.18	0	ø	c	þ	2
H2-16	Novel Intermediate-Temperature Reversible SOFC for a Renewable Energy System that can Co-produce Power and Hydrogen (I)			NFCRC	\$ 224,085	2.24	þ	ø	¢	þ	С
H2-23	Reel to Reel Processing for Continuous Thermal Pressing of the Catalyst Film onto a Membrane for High Volume, Low Cost Commercialization of Hydrogen Generating MEAs (I)			PET	\$ 201,897	2.21	0	q	k	þ	С
H3-14	Electrochemical Conversion of Biomass to Hydrogen (I)		тмі	\$ 162,000	1.62	o	ø	¢	þ	С	
H2-2	Improved Liquifaction Process (I)		Praxair	\$ 200,804	2.01	0	q	c	þ	С	
H3-19	Methanol Reformed Hydrogen For Portable PEM Fuel Cell Systems (I)		)	Protonex	\$ 264,573	2.65	þ	þ	¢	þ	С
H3-3	Development of High Pressure Electrolyzers for Backup Power Systems (I)		ns (I)	Proton Energy	\$ 207,526	2.10	þ	þ	¢	þ	C
H3-7	Scalable Steam Methane Reformer System for Distributed Hydrogen Production (I)			Catacel Corp	\$ 209,998	2.10	þ	þ	k	þ	С
H3-13	Low-Cost Manufacturing of Multi-Fuel Reactors for an Innovative High- Efficiency Planar Reformer (I)		Delphi	\$ 219,084	2.19	þ	q	¢	þ	С	
H3-5	Innovative and Cost-Effective Micro-Process for Roll-to-Roll Solution Casting of Multi-Layer PEMS (I)		Chemsultants	\$ 203,657	2.11	0	þ	¢	þ	С	
I - Phase I II - Phase II III - Phase III			0000	LEGEND							

FIGURE 1. Project Execution Summary

## **Results: Individual Projects**

**Faraday Technology, Inc.** (EFC-H1-1); Nanocatalyst Development Employing Electrically Mediated Processing for Hydrogen Generation

The objective is to develop a low-cost, mass fabrication technology for catalyzation of membrane electrode assemblies (MEAs) for polymer electrolyte membrane (PEM) electrolyzers and regenerative fuel cells. In Phase I, we are:

- 1. Developing an optimum catalyst for improved electrolyzer performance.
- 2. Demonstrating electrodeposition of electrolyzerspecific catalysts onto the electrode support.
- 3. Demonstrating scale-up of the catalyzation process.

## Accomplishments

- Developed non-carbon gas diffusion layer (GDL) support structures for catalyst.
- Determined optimum alloy catalyst for a regenerative fuel cell/electrolyzer: Pt<sub>4</sub>Ru<sub>4</sub>Ir alloy.
- Design and assembly of an alpha-scale electrocatalyzation fixture; beta-scale fixture in design stage.
- Have demonstrated capability to electrodeposit catalyst for PEM fuel cell applications; require extension of this capability to alloy catalysts.

## Special Recognitions & Awards/Patents Issued

**1.** One U.S. Patent (# 6,080,504) previously awarded. No additional patents have been applied for, but are anticipated.

## FY 2006 Publications/Presentations

**1.** Poster presentation at Fuel Cell Seminar 2005, November 2005, Palm Springs, CA.

**2.** Will be presenting at the 2<sup>nd</sup> Annual Symposium on MEA Manufacturing for Hydrogen Applications.

**Catacel Corp.** (EFC-H1-09-2A): Novel Stackable Structural Reactor (SSR<sup>TM</sup>) for Low-cost Hydrogen Production

The Phase II project plan for SSR will finalize the development of the product, and prepare for commercialization:

- Validate long-term catalyst durability under a range of operating conditions.
- Validate mechanical durability under expected operating conditions.
- Examine key cost drivers, be ready with alternatives.

- Prepare for manufacturing and quality control needs.
- Gain extensive understanding of customer needs, and their cost/benefit tradeoffs.
- Demonstrate that SSR is superior in a real environment.

## Accomplishments

- Two catalyst formulations have shown good conversion and stability when tested up to 150 hours in simulated field conditions.
- Materials have been exposed to simulated conditions for 1,000 hours without damage. After being exposed to severe oxidation for 1,000 hours, full-size parts and can easily be removed from customer tubes without damage to the tube.
- Details and methods of construction have been demonstrated that will allow SSR to be cost-competitive with current catalyst systems
- Most of the assembly and coating methods have been demonstrated at bench scale.
- Final negotiations are underway with a customer to put in a small plant to showcase the technology. This plant will be brought on-line 2Q 2007.

## Special Recognitions & Awards/Patents Issued

- U.S. Patent filed 7/27/05, "Reactor Having Improved Heat Transfer."
- Trademark application filed 9/12/05 for "SSR".

## FY 2006 Publications/Presentations

• Poster, "Novel Catalyst Development for Steam Reforming of Natural Gas", The Ohio Fuel Cell Symposium, May 23-24, 2006.

## IAP Research, Inc. (EFC-H1-11-1A):

Commercialization of Electromagnetic (EM) Solid State Welding for High Pressure Hydrogen Storage

- Establish the feasibility of using electromagnetic solid state welding for pressure sealing applications.
- Determine applicability to DOE Hydrogen Multi-Year plan for compressed hydrogen storage.
- Establish near-term commercialization potential in heating, ventilation and air conditioning, chemical processing and power industries.

## Accomplishments

- Developed modeling methodology for EM solid state welding.
- Established solid state welding parameters for key pressure sealing applications.

• Successfully validated through experiments EM solid state welding of pipe coupling application relevant to hydrogen transmission.

## FY 2006 Publications/Presentations

**1.** Presentation to EMTEC Board of Governors - Hydrogen Program Review, February 8, 2006.

**Makel Engineering, Inc.** (EFC-H1-15-2A): Low Cost MEMS Hydrogen Sensor for Transportation Safety

The objective is the development of a low-cost, high performance hydrogen safety sensor for hydrogen powered vehicles. The proposed system meets a need for a low-cost sensor for on-vehicle safety, pipeline/ fueling station monitoring for hydrogen distribution, and has the potential for use in closed-loop fuel cell control loops. In order to meet this emerging market need, Makel Engineering, Inc. (MEI) is adapting our hydrogen sensing technology and integrating recently developed hydrogen-sensitive nanomaterials into a highly manufacturable system platform. MEI will work with our partners to produce a second generation prototype system that will be tested on hydrogen powered vehicles.

## Accomplishments

Phase I of this project was focused on the adaptation of MEI's hydrogen microsensor technology, originally developed for NASA safety applications, for the automotive environment. The Phase I effort resulted in a prototype sensor system with components that are rated for commercial vehicle conditions, the most challenging being operating in temperatures from -40°C to 125°C. The bill of materials for the initial prototype suggests that the system's manufacturing cost would be a few hundred dollars.

In addition to adapting MEI's current state-of-theart hydrogen detection technology, our team pushed the development of an Argonne National Laboratory (ANL) sensor technology based on hydrogen nanoclusters. This technology has the potential to offer very high sensitivity detection while significantly lowering the persensor cost and simplifying the manufacturing process. Demonstration sensors utilizing this technology were produced in Phase I.

**Midwest Optoelectronics, LLC** (MWOE) (EFC-H1-16-2A): Development of Improved Materials for Integrated Photovaltaic-Electrolysis Hydrogen Generation Systems

• Develop and produce integrated photovoltaicelectrolysis (IPE) hydrogen generation systems based on the high-efficiency, multiple-junction amorphous silicon (a-Si) photoelectrode.

- Achieve a solar-to-hydrogen conversion efficiency of 8%, a durability of 20,000 hours and a hydrogen production cost less than \$10/kg, when optimized.
- Achieve a small-scale production of 8 ft<sup>2</sup> IPE hydrogen generation panels with a throughput of 20 panels per week at the end of the project.

## Accomplishments

- Successfully designed and fabricated full scale 4 ft x 2 ft IPE panels. These are the first large-area integrated photovoltaic electrolysis panel for solar hydrogen generation. Each of these panels produce hydrogen at a rate of 100–120 cc/min under sunlight.
- Completed two mini-production runs with six panels in each set of production.
- Established an outdoor solar testing site for testing IPE panels under outdoor conditions.
- Identified failure mechanisms such as the degradation of the conductive coating, the leakage of the electrolyte and aging of the membrane in electrolyte. Redesigned the device and solved the degradation problems after extensive testing and development.
- A new conductive coating material, which provides much higher device performance and stability, has been identified and used. Electrolysis components have been redesigned to solve the leaking mechanism. Many new membrane materials have been explored and tested and a membrane with much stronger resistance to caustic solution, good ion conductivity and excellent gas separation property has been used to replace the old membrane.
- Panels has gone through extreme cold winter conditions and summer weather conditions. So far, most of the panels have been under outdoor testing for more than half a year.
- MWOE has had meetings with the Toledo Port Authority which manages Toledo Express Airport. The project being discussed and planned is for MWOE to install solar hydrogen generation panels in a prominent spot at the Toledo Express Airport so that the passengers will be exposed to this important project.
- In the process of building a high efficiency, large area a-Si deposition machine in collaboration with the University of Toledo (UT) with the grant support from the Ohio Department of Development. This system will allow MWOE and UT to make largearea a-Si photoelectrodes with improved efficiency.
- MWOE and the University of Toledo have reached and signed an exclusive, worldwide license agreement.

## Special Recognitions & Awards/Patents Issued

**1.** MWOE's chairman, Dr. Xunming Deng received 2006 Outstanding Researcher Award from Chinese Association of Science and Business, New York, NY.

**2.** MWOE's academic partner, the University of Toledo, filed one patent application in the U.S., Japan and Germany, and filed another patent application in the U.S. The inventors for both patent applications are Dr. Xunming Deng and Dr. Liwei Xu. MWOE owns exclusive and worldwide rights to these technologies.

**3.** MWOE and the University of Toledo filed a patent application for the Integrated Photovoltaic Electrolysis process. Inventors are Mahabala Adiga, Xunming Deng, Aarohi Vijh and Liwei Xu. MWOE owns exclusive and worldwide rights to these technologies.

## FY 2006 Publications/Presentations

- Presentation, "Solar Generation of Hydrogen

   a New Industry", University of Science and Technology of China, Hefei, China, July 3, 2005.
- Presentation, "Photoelectrochemical Production of Hydrogen from Water Using SunlightInstitute of Semiconductors", Chinese Academy of Sciences, Beijing, China, July 4, 2005.
- Poster presentation at the DOE hydrogen review meeting, May, 2006.
- Presentation, "Sputter Deposition of Fe<sub>2</sub>O<sub>3</sub> Films for Photoelectrochemical Hydrogen Production", 208th Meeting of the Electrochemical Society, Los Angeles, CA, October 16-21, 2005.

## **University of Dayton Research Institute**

(EFC-H2-20-2A): Regenerative Solid Oxide Fuel Cell (RSOFC)

- Select anode, cathode, and electrolyte materials.
- Improve microstructure using pore formers.
- Test the novel materials using solid oxide electrolysis cells (SOECs).
- Establish the commercial viability of those materials for SOFC/SOEC electrolyzer systems.

## Accomplishments

- Fabricated anode-supported RSOFC cells with a thin electrolyte ( $\sim 10\mu m$ ).
- Optimized anode microstructure.
- Demonstrated RSOFC cell testing for  $H_2$  and electricity generation.
- Achieved the energy conversion efficiency of the power output more than 85% at 50 mA/cm<sup>2</sup>.

**NexTech Materials** (EFC-H2-21): Novel Ceramic Hydrogen Sensors for Fuel Cell Applications

- Develop ceramic based sensor formulation that is sensitive to low concentrations of hydrogen in air (1,000 ppm 1%) for use as a safety sensor.
- Demonstrate that sensor does not give false alarms in the presence of interference gases (CO, CH<sub>4</sub>, VOCs, etc.).
- Demonstrate response time of less than 30 seconds in the presence of 1% hydrogen.
- Test proposed novel platform for improved hydrogen sensor.
- Fabricate a prototype based on the best performing sensor system and platform.

## Accomplishments

- Identified sensor formulation with high sensitivity to hydrogen.
- Quantitative responses to 250 ppm hydrogen to 1.0% hydrogen.
- Demonstrated high level of interference resistance to both carbon monoxide and methane.
- Demonstrated 20 second response time to 1.0% hydrogen with 48 second recovery time.
- Demonstrated improvements using novel sensor platform.
  - Doubled sensitivity to 1% hydrogen.
  - Faster response times (20 seconds versus 2.3 minutes).
  - Four-fold decrease in power requirement.
  - Improved baseline stability and sensitivity degradation.
  - Lower production cost.
- Built and tested prototypes based on identified sensor formulations and novel platform design.
- Demonstrated improved performance over other commercial ceramic-based hydrogen sensor.

## Special Recognitions & Awards/Patents Issued

**1.** A draft patent application has been prepared.

## FY 2006 Publications/Presentations

**1.** Presentation at EMTEC Hydrogen Review Meeting (Dayton, OH, February 8, 2006).

**2.** Poster presented at 2005 Fuel Cell Seminar (Palm Springs, CA).

**1.** Presentation at 6<sup>th</sup> DoD Logistic Fuel Processing Conference (Panama City, FL, May 2006).

**Powdermet Inc.** (EFC-H2-15): High Strength, Low Cost Microballoons for Hydrogen Storage

- Investigate the idea of using high strength to weight chemical vapor deposition (CVD) coating on a lightweight scaffold microballoon to create high strength hydrogen storage vessels.
- Calculate the theoretical storage limits of coated microballoon for hydrogen storage technology.
- Increase crush and burst strength of commercial microballoons by adding a light weight high strength coating.
- Change the hydrogen permeability of microballoons by using a CVD coating approach.
- Validation of four key components of microballoon storage including manufacturing, strength, filling technology and long-term storage.
- Investigate new materials or previously unused materials for hydrogen microballoons.

## Accomplishments

- Proved theoretical storage >15% by weight hydrogen in a high strength coated microballoon concept.
- Calculated specific microballoon sizes as well as coating thicknesses and strengths and materials needed for >15% hydrogen storage by weight.
- Filled coated microballoon with hydrogen at 2,500 psi internal pressure.
- Long term (months have been verified) hydrogen storage at pressure >2,000 psi in CVD coated microballoons.
- Creation of optimally sized carbon microballoons for use as light weight scaffolding in hydrogen storage concept.
- Engineered coatings to control hydrogen permeability in microballoons.
- Proof of concept for creation of stronger microballoons by adding a high strength CVD coating.

## FY 2006 Publications/Presentations

**1.** American Ceramic Society Cocoa Beach Conference on Advanced Ceramics & Composites, January 22-27, 2006 Presentation "High Strength, Low Cost Microballoons for Hydrogen Storage."

## **Delphi Electronics & Safety and Packer**

**Engineering, Inc.** (EFC-H2-3): Hydrogen Storage using Porous Silicon

• Demonstrate hydrogen storage capacity for porous silicon (predicted at 6-7%).

- Evaluate system application requirements for storage system using porous silicon.
- Provide rapid technical feasibility for this novel concept in storage.

## Accomplishments

- In just seven months took a completely new concept and demonstrated best-in-class results for hydrogen storage.
- Conducted a theoretical analysis of the maximum storage rate for porous silicon and validated it against lab tests and electron microscope photos of the material (6.1%).
- Developed a system model, Ishikawa diagrams, and thermodynamic relationships.
- Assessment of silicon availability and supply, targeting a 100x reduction in cost.

## Special Recognitions & Awards/Patents Issued

**1.** Three inventions from Phase I are of being filed as provisional patent applications.

**2.** The base invention for hydrogen storage using porous silicon has been pending since 2003 – no response has been received from the USPTO.

## **Publications/Presentations**

**1.** An abstract on the technical feasibility work has been submitted to the Society of Automotive Engineers for consideration as a publication for their Proceedings for the 2007 World Congress.

## **EERL Lab of Chemical and Biomolecular Department, Ohio University** (EFC-H2-08): Electrochemical Coal Gasification with Novel Electrodes

The main objective of this project is to evaluate the feasibility of electrolyzing coal slurries to produce hydrogen at intermediate temperature. The followings are the specific tasks for this objective:

- Redesign the coal electrolytic cell to work at high temperatures.
- Set up a bench-scale continuous system in order to work at high temperatures.
- Evaluate the performance of the new electrolytic cell to provide a scale-up design for alternative production of hydrogen.

## Accomplishments

• Preliminary results showed that increasing temperature from 60°C to 80°C increases the experimental time form 4.9 hours to 6.3 hours; the utilization of coal is enhanced.

- New system for operation at 140°C to 160°C has been set up.
- New coal electrolyte cell has been designed and manufactured; evaluation information about the new set-up and cell at higher temperature will be derived.

**The University of Toledo** (EFC-H2-22): Fabrication of Metal-Carbon Nanostructure Composites

- To fabricate self-standing carbon nanostructures on a catalyst perform.
- To incorporate metal species 'into' and 'onto' the carbon nanostructures via electrophoresis.
- To design a compact device for hydrogen with enhanced storage capacity.

## Accomplishments

- The processing parameters (pre-conditioning of the alloy wires, selection of carburizing gas and, carburization temperature-time, etc.) for the creation of carbon nanotubes have been optimized.
- The process of microscale coating on the alloy surface has been optimized.
- Characterization has been carried out.

**Hy-Energy LLC.** (EFC-H2-11): Novel Materials for Reversible Hydrogen Storage

Hy-Energy is developing lightweight reversible hydrogen storage materials based on novel complex hydride – amide mixtures. Materials are being prepared and tested for viability as a practical means of storing hydrogen for use with a variety of fuel cell applications. The hydrogen uptake and release properties being tested are:

- Reversible hydrogen storage capacity.
- Rates of hydrogen uptake and release.
- Thermodynamic stability (reversible pressure vs. temperature).
- Cycle-life properties (capacity and sorption rates as a function of cycling).

## Accomplishments

- An initial set of samples were prepared and characterized.
- The second sample showed reversibility (reversible capacity 3 wt%).
- Pressure composition temperature isotherm (PCT) measurements showed three plateaus, thus, multiple phase transition. This is a good indication that the reversible capacity can be significantly improved.
- The largest plateau was at 70 bar (250°C) demonstrating that this phase transition is unstable

and should be able to provide hydrogen at practical pressures (1-5 bar) and much lower temperatures.

## Special Recognitions & Awards/Patents Issued

1. Hydrogen storage materials patent submitted.

## FY 2006 Publications/Presentations

**1.** Some results presented at the MRS Spring Meeting (April 17-21, San Francisco, CA) and at DOE Hydrogen Program, 2006 Annual Merit Review, (May 16-19, 2006, Arlington, VA).

**GFS Chemicals, Inc.** (EFC-H2-11-1A): Development of Complex Metal Hydrides for Hydrogen Storage Applications

- Manufacture pure Li<sub>3</sub>AlH<sub>6</sub> plus material doped with 1) aluminum rich compounds of titanium and carbon, 2) volatile carbonyl compounds of Fe and Ni.
- Execute qualifying sample testing and characterization at University of Dayton Research Institute (UDRI), Dayton.
- Develop hydrogen absorption-desorption data on qualified samples at Hy-Energy LLC.
- Draw conclusions re use of doped Li<sub>3</sub>AlH<sub>6</sub> substrates in hydrogen technologies.

## Accomplishments

- Successful manufacture of pure Li<sub>3</sub>AlH<sub>6</sub> on a scale of dozens of grams.
- Preparation of samples of Li<sub>3</sub>AlH<sub>6</sub> mechanically doped with Al<sub>4</sub>C<sub>3</sub>, TiAl, TiAl<sub>3</sub>.
- Onset of testing of pure Li<sub>3</sub>AlH<sub>6</sub> (UDRI) plus upgrade of UDRI test regimen to accommodate sensitivity of substrates to oxygen and moisture.

## FY 2006 Publications/Presentation

**1.** Presentation at the EMTEC Hydrogen Program Review Meeting Feb. 8, 2006 (Dayton).

**UC-Irvine, National Fuel Cell Research Center** (EFC-H2-16): Novel Intermediate-Temperature Reversible SOFC for a Renewable Energy System that can Co-Produce Power and Hydrogen

- Evaluate novel reversible solid oxide fuel cell (SOFC) materials sets produced with wet-chemicalroute synthesis through preliminary manufacturing (dry pressed button cells) and performance tests.
- Select the best performing materials sets for manufacturability, performance and stability features.

- Manufacture planar anode-supported reversible SOFC button cells using the selected materials set by tape-casting, screen-printing, and co-firing processes.
- Accomplish electrochemical and stability tests of planar anode-supported reversible SOFC button cells.
- Investigate market and commercialization potential of reversible SOFC technology.

## Accomplishments

- Fabricated pure phase LSGMC electrolyte with higher conductivity (0.192 S/cm at 750°C) than the literature (0.171 S/cm at 750°C) [1].
- Synthesized SYT nano powders by a unique sol-gel process.
- Manufactured highly active SSC composite cathode (cathodic current density reaches 1 A/cm<sup>2</sup> at an overpotential of 48 mV at 800°C in air).

#### Special Recognitions & Awards/Patents Issued

**1.** First-place winner of student micrograph competition of the Carl Zeiss Center of Excellence. Graduate student: Thomas Pine, with micrograph "*Thermal Facets on the Free Surface of Annealed Yttrium-Doped Strontium Titanate (SYT) Ceramic Pellet.*"

**2.** Second-place winner of student micrograph competition of the Carl Zeiss Center of Excellence. Graduate student: Grace Qin, with micrograph *"Crystal Tree of Perovskites."* 

#### FY 2006 Publications/Presentations

**1.** Two presentations entitled "*Electrocatalytic Properties of SOFC Cathode/LSGMC Interfaces*" and "*Sol-Gel Synthesis and Electrochemical Characterization of SYT Powders for Reversible SOFCs*" at the 30th International Conference & Exposition of The American Ceramic Society, January 22-27, 2006, Cocoa Beach, Florida.

#### References

1. T. Ishiharaa, T. Shibayamaa, S. Ishikawaa, K. Hosoib, H. Nishiguchia, Y. Takitaa, *J. Eur. Ceram. Soc.*, **24** (2004) 1329-1335.

**Precision Energy and Technology (PET)** (EFC-H2-23): Reel to Reel Processing for Continuous Thermal Pressing of the Catalyst Film onto a Membrane for the High Volume, Low Cost Commercialization of Hydrogen Generating Membrane Electrolyte

- Manufacture a system to continuously thermal press a catalyst film to a membrane via "reel to reel" process.
- Build the system into a Western Technologies Associates "reel to reel" processing machine for full production:

- Costs Reel to Reel appears to reduce total manufacturing time by 80%.
- Durability PET's review of raw materials has noted potential failure in carbon cloth processing and potential issues with bonding.
- Energy Efficiency PET's efforts in reel to reel continuous processing focuses on the bonding issues on a small rolled area versus the plate bonding currently used. This small continuous area approach may provide greater control and less stress to the material during bonding, thus improving stability of the process.

## Accomplishments

- Bonding process utilizing a matrix of materials.
  - Ionic membranes Nafion<sup>®</sup>, GEFC
  - Electrodes Ballard, E-Tek, GrafTech, Toray
- Laminator process equipment 75% complete.
- Materials characteristics 90% complete.

#### FY 2006 Publications/Presentations

- Electrochemical Society has invited PET to present in Feb 2007 on Manufacturing Membrane Electrode Assemblies (MEAs).
- PET will be presenting at the MEA symposium on Manufacturing of MEAs.

**Technology Management, Inc.** (EFC-H3-14): Electrochemical Conversion of Biomass to Hydrogen

The objective is to demonstrate a biomass to hydrogen system suitable for scale-up to a distributed network of hydrogen generation stations. Phase 1 will focus on demonstrating the feasibility of integrating biomass-derived syngas with the fuel cell/electrolyzer system. Technology Management, Inc. (TMI) of Cleveland, Ohio, believes that a unique opportunity exists to demonstrate hydrogen production from a biomass-fueled system using regenerative fuel cell technology at high efficiency, with low emissions.

#### Accomplishments

- Cells and small stacks with sealing exceeding target values.
- Production and capture of hydrogen via electrolysis of water.
- Characterization of sub-kilowatt stack operating on synthetic biogas.

**Praxair, Inc.** (EFC-H2-2): Improved Hydrogen Liquefaction Process

• Reduce the cost for hydrogen liquefaction.

- Reduce the electrical power consumption for hydrogen liquefaction.
- Increase the liquid hydrogen production rate for existing plants.
- Reduce hydrogen distribution costs.

This project will attempt to improve the electrical efficiency of hydrogen liquefaction. Our goal is to reduce the power consumption by about 20%. If this can be accomplished, assuming that the current efficiency is 25%, the final efficiency would be over 30%, which will exceed the 2010 target. Improving efficiency will reduce cost as long as the added capital cost is low enough. Therefore, maintaining low capital cost is another important goal of the project. Any improvement made in this project will apply to both small-scale (existing) and large-scale (future) plants.

## Accomplishments

- Experimental system designed and assembled.
- Preliminary candidate materials identified.

**Protonex Technology Corporation** (EFC-H3-19): Methanol Reformed Hydrogen For Portable PEM Fuel Cell Systems

Design, develop and demonstrate an advanced fuel cell system in the power range of approximately 250 Watts based on a methanol-reformed hydrogen fueling solution.

## Accomplishments

- A design concept for an integrated 250 Watt methanol reformer/fuel cell system was completed. The balance-of-plant components (BOP) for the system were selected.
- A full reformer prototype was developed. Several reforming reactors were constructed and tested successfully. The units produced reformate streams of over 70% hydrogen with carbon monoxide levels in the range of 20-200 parts per million (ppm).
- A reformer/fuel cell breadboard was constructed. A fuel pump on the breadboard pumped methanol/ water mix to a reformer that generated a hydrogenrich stream. The stream was sent to a fuel cell stack that produced up to 100 Watts electrical output. The exhaust stream from the stack was recycled to a combustor to provide the heat required for the reforming reactions. A control system on the breadboard maintained operating temperatures in the reformer and monitored and recorded data.

## Proton Energy Systems, Inc. (EFC-H3-3):

Development of High Pressure Electrolyzers for Backup Power Systems

Evaluate and test candidate electrolyzer cell stack materials that will optimize 2,000 psi hydrogen generation for cost, performance, and manufacturability. Specifically:

- Identify and acquire several membrane types for evaluation.
- Evaluate physical properties (e.g. conductivity, permeability, etc.) of 2-5 candidate membranes.
- Integrate one or two of the best membrane candidates into a single cell test for evaluation at up to 2,000 psi.

## Accomplishments

Project awarded on April 1, 2006 and currently in the materials procurement stage. No significant accomplishments to report at this time.

**Catacel Corp.** (EFC-H3-07-1A): Scalable Steam Methane Reformer System for Distributed Hydrogen Production

The overall objective of this project is to demonstrate the technical and commercial feasibility of a scalable steam methane reformer system for the distributed production of hydrogen:

- Document one or more specific configurations of a 20 kg/day (10 kw fuel value) hydrogen generation system based on the Catacel heat exchanger platform.
- Demonstrate that lab-scale versions of key components will perform as expected.
- Demonstrate that the system is scalable to commercial sizes.
- Show that the cost of hydrogen produced from such a system will meet or exceed U.S. Department of Energy targets of \$2.50/gge.

## Accomplishments

Since the project began only in early April, there have only been limited accomplishments to date, as follows:

- Considerable work has been performed on construction of the heat exchanger platform. We anticipate having a unit available to test in mid-July.
- Water-gas-shift catalysts have been prepared by NexTech, have been coated on foils, and are scheduled for testing at the University of Toledo in July.

• Preliminary analysis has been performed from a system perspective. This analysis showed the rough heat and size balance between components, and revealed the operating points to be used for the testing phases of the project.

**Chemsultants, International, Inc.** (EFC-H3-5-1A): An Innovative and Cost Effective micro-process for rollto-roll solution casting of Multi-layer Proton Exchange Membranes with superior Performance, Transport and Mechanical properties in High Temperature/Low RH Operating Environments.

Prove the feasibility of:

- A new manufacturing process for roll to roll production of multilayer proton exchange membranes.
- Multi-layer membranes comprised of interspersed, discrete layers of hydrophilic zirconium particles and recast Nafion<sup>®</sup> polymer.
- Solution casting polymer in a layered structure via a novel, advanced process to manufacture thin caliper (12-20 um) membranes.

## Accomplishments

- A project outline meeting was held with Case Western Reserve personnel and Chemsultants Inc. on May 31, 2006. The scope of work to be performed was discussed, including the synthesis of the zirconium/phosphonate nano-particles.
- Synthesis of the zirconium phosphate sulfophenylphosphonate particles was initiated. The process followed those found in published articles from the University of Perugia, Italy. Preparation, synthesis and purification of the particles spanned

about two weeks time frame. The particles will be used in further work being conducted with re-cast Nafion<sup>®</sup> solution.

- A visit was made to Graftech International (Advanced Energy Technology Inc.) to investigate the use of the PEM-LITE<sup>®</sup> System in evaluating ion transport phenomenon through fuel cell membranes and MEA components. The system has been successfully used in experiments that characterized the gas diffusion and catalyst activity of fuel cell components and compared results to actual fuel cell stack data. The instrument is of interest for our work with the nano-particles and multi-layered membranes.
- Literature searches were conducted on the following subjects: hydrophilic particles, nano-particles in membranes, multi-layered membranes and casting procedures and ion transport through PEMs.

## **EMTEC Conclusions**

EMTEC is energetically pursuing the near term commercialization of the hydrogen infrastructure technology that in many cases has been developed with previous DOE support. The EMTEC programmatic collaborative approach is well suited to accelerate technology to market. EMTEC has selected high quality projects in the appropriate subject area that have cross-cutting materials technology with near term manufacturing opportunities.

## FY 2006 Publications/Presentations

**1.** EMTEC presented an overview of the hydrogen program at the DOE Program Review in Washington, D.C., May 23 through May 26, 2005.